BioNorm II

Pre-normative research on solid biofuels for improved European standards

Specific Targeted Research or Innovation Project

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Contents

1 Introduction

BIONORM II:
PRE-NORMATIVE RESEARCH ON SOLID BIOFUELS FOR IMPROVED EUROPEAN STANDARDS

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To develop the market for solid biofuels within the EU standards are urgently needed. Based on a mandate given by the EC, CEN TC 335 "Solid Biofuels" currently develops such standards based on the available knowledge. In the past several Technical Specifications (TS) (or pre-standards) have been developed, which are currently upgraded to European Standards (EN). Other TS's are on its way. Applications in industry however have shown that additional information has to be integrated and/or considerable gaps in knowledge still exist. This makes it difficult to develop the still needed Technical Specifications and/or upgrade them to European Standards. Against that background the goal of the BioNorm II project was it to support the ongoing standardisation efforts especially for the development of improved solid biofuel specifications. For that purpose BioNorm II was subdivided in four Work Packages, which concentrate on sampling, test methods, quality measures and biofuel specifications. The results of this pre-normative work is transferred directly into the ongoing standardisation process to allow a fast and reliable development of improved European Standards and acceptable Technical Specifications. Thus, the following report can present only a selection of the most important project results within the last 3 years of scientific work.

Key words: solid biofuels, standards, sampling, test procedures, quality measures, biofuel specification

1 BACKGROUND

The development of European Standards (EN) is seen as a major driver to expand the market for solid biofuels. This market expansion is needed to fulfil the aims defined within the European Commissions' (EC) White Paper on Renewable Energy, the Directive on "Green" Electricity from Renewable Energy (EU-Directive COM(2000) 279 final), and the new proposal for the Renewable Energy Directive as well as various political goals on the national level.

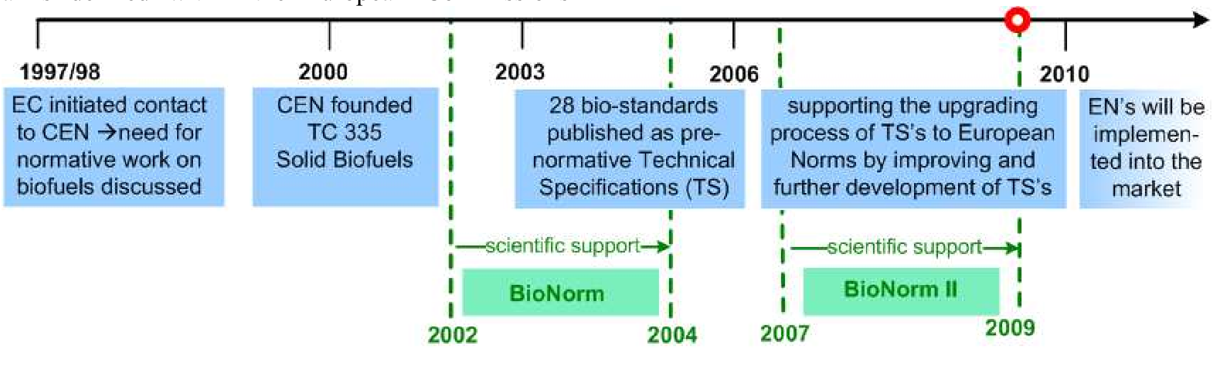

Figure 1: Timescale of the project history and its focus on the development of European standards; the red point on the scale marks the present situation

Against this background the aim of the BioNorm II project was initiated to carry out pre-normative research in the field of solid biofuels in close collaboration with the work of CEN TC 335 "Solid Biofuels" (Figure 1). Due to the wide range of standards to be developed by CEN TC 335 this project focuses on such aspects urgently needed by industry to increase the markets for solid biofuels where significant pre-normative R&D need is given. This applies in particular for an acceptable and transparent definition of key properties of solid biofuels traded on the market, taking into account the given needs from the production and provision chain as well as the end user. Furthermore, reference test methods and rapid test methods are necessary to prove if the defined key properties are met.

Additionally, rules for sampling and sample planning as well as quality measures especially adapted to the needs of solid biofuels are urgently needed.

The BioNorm II project is the successor of the successfully completed previous BioNorm project. The BioNorm II project consortium is a team of 25 experienced partners from 11 European countries (many partners are also active members of CEN TC 335) and the German Biomass Research Centre (DBFZ) is the coordinator of this project that runs 3 years (2007 – 2009). Within the project framework four work packages (WP I – IV) exist in which fuel characteristics of solid biofuels and methods to test and verify them are developed:

WP I: Sampling, sample reduction and planning
WP II: Chemical and physical testing procedures
WP III: Quality measures
WP IV: Specifications of biofuels

2 PROJECTS' RELATION TO THE EUROPEAN STANDARDISATION PROCESS

The project is in continuous contact with the CEN TC 335 "Solid Biofuels" team to ensure a close connection and interaction between the standardisation bodies and the scientific work in this area (Figure 2). Based on this the BioNorm II project aims in the field of standardisation of solid biofuels are:

- to validate existing Technical Specifications (TS) taking experiences from their practical implementation into account, particularly by improving the existing CEN/TS 14961 "Solid biofuels – Fuel Specification and Classes" in cooperation with WG 2 of CEN/TC 335 by
    - including specifications given by the combustion unit and
    - including rules for conformity of the products with their specified requirements,
- further improvement of existing reference tests, particular in terms of precision,
- to develop new reference methods as well as rapid on site test methods,
- working out sampling and sample reduction methods for selected biomasses and the development of sampling plans, and
- to develop adapted quality measures in accordance with the ISO 9000 / 9001.

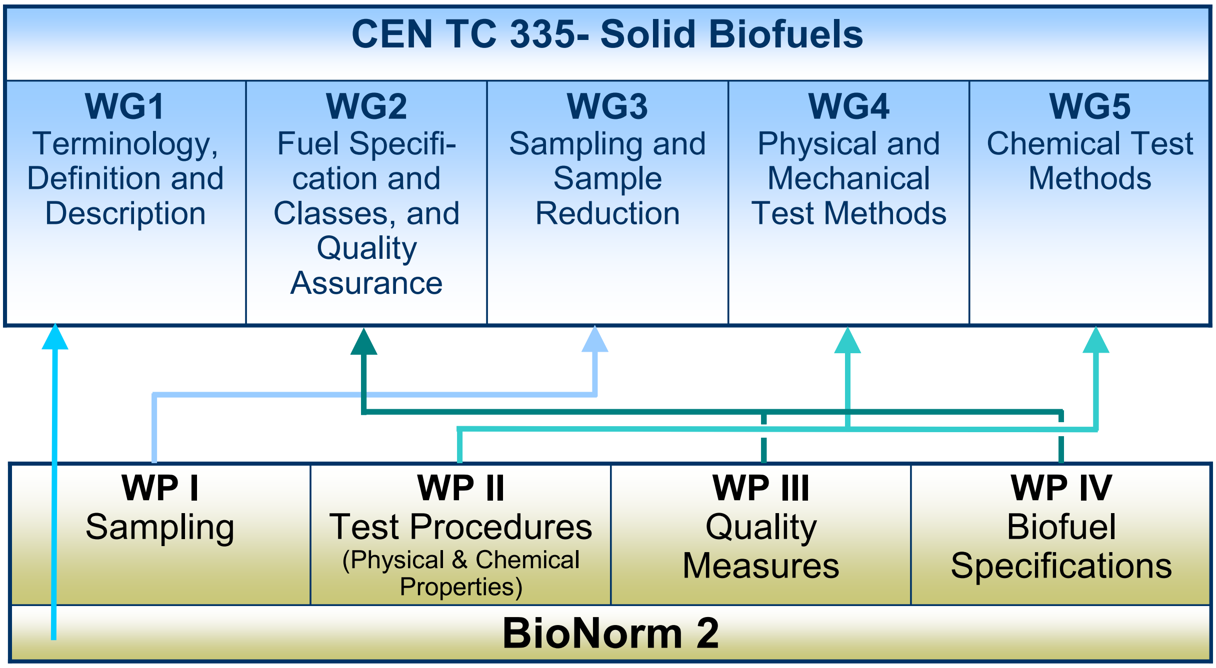

Figure 2: Main structure and interactions between BioNorm II working packages and CEN/TC 335 working groups

3 WORK PERFORMED

Within the following explanations the content of each Work Package is shortly presented.

3.1 Sampling, sample reduction and planning (WP I)

With the background knowledge, the experiences and the approved methodology of the first BioNorm project WP I extended the work on increments and sample reduction by a second selection of materials and test methods. In the focus of the assessment came solid biofuels from South European countries (e.g. agricultural residues from the production and processing of olives and grapes) as well as general common wood fuels like pellets, wood chips or bark.

Sampling and sample reduction experiments have been carried out for pellets and olive residues at different plants in Italy and for wood chips, bark and wood chips from stem wood at a plant in Sweden. Samples have been taken from static as well as from moving streams. For reduction purpose different methods depending on the kind of biofuel like riffle box, coning and quartering and the long pile method have been used. Each exercise was performed according to a defined procedure with regard to sample sizes and sampling times. The analysis of the respective biofuels for relevant parameters was performed according to CEN Technical Specifications.

Sample planning experiments have been carried out with grape and olive residues as well as with 6 mm pellets in Italy. In Sweden sample planning experiments were carried out for bark.

3.2 Chemical and physical testing procedures (WP II)

Biofuel properties and its combustion features are determined by several critical parameters, which can be spilt in physical and chemical properties. In the first BioNorm project a number of different test methods was developed and elaborated as CEN/TS ("Technical Specification") within the last years.

One part of the WP II team was engaged in determining further physical fuel properties (like the impurities and bridging properties) applying self-constructed test devices and methods. During the second project period three test principles for the determination of impurities were tested, guidelines for three individual round robins exercises were developed and test material for the round robin tests prepared.. With regard to the determination of bridging properties the testing principle was pre-tested and results on influencing factors were determined in order to be able to set the guidelines for the round robin test. A second bridging testing apparatus was provided for tests, so that both apparatuses are to be used in the round robin trials.

The second part of WP II verified existing test methods for the determination of chemical fuel properties by testing their practicability and accuracy. The aim was the development of new or improved test methods in the framework of the European standardisation process for solid biofuels. To guarantee regular, cheap and consistent biofuel quality tests online test methods and rapid tests for laboratories are necessary. In the second project period the largest round robin test with 40 European laboratories was performed, where two different samples were tested fivefold according to relevant CEN Technical Specifications. Furthermore a XRF method for rapid laboratory testing was developed as well as rapid test methods for the determination of chlorine, sodium and potassium, which is currently tested for cheap outdoor use. Additionally the reference test methods for Cl and S were improved and online test methods for industrial biofuel production reviewed.

3.3 Quality measures (WP III)

Within WP III the work was focused on the development a Quality Management System in order to guarantee a defined biofuel quality. Based on the results of the former BioNorm project where a Quality Assurance and Quality Control System was developed WP III now concentrates on Quality Planning, Quality Improvement and Quality Policy.

Thus the work started with activities on quality planning and quality improvement. In order to get an overview about end user requirements, interviews and investigations were performed. This gave a detailed overview about user requirements along the whole biofuel supply chain. Furthermore Critical Control Points and quality influencing factors in the different supply chains of wood pellets, wood chips, hog fuel, straw and olive residues were identified. With the help of this information a detailed overview for wood pellets, wood chips, hog fuel, straw and olive residues was prepared. In this so called "CCP table" Critical Control Points, quality influencing factors and quality issues (sampling, testing, documentation) with regard to biofuel parameters according to CEN/TS 14961 were analysed and evaluated. This gives information about the relevance of Critical Control Points in the biomass supply chains, appropriate sampling and testing methods as well as quality improvement potential. Additionally a summary about logistic aspects (transport, handling, storage) within the five biomass supply chains was written. Finally the WP III drafted a guideline for the enlargement of the quality assurance standard prEN 15234 (part 2 ff). Some of the prepared biofuel supply chains were directly converted into the first standard draft (part 2 pellets, part 4 wood chips).

3.4 Specifications of biofuels (WP IV)

In WP IV the work is concentrated on the support of the European standardisation Committee CEN TC 335 by defining fuel specifications and classes of the most traded solid biofuels. Thus supply chain related fuel properties as well as combustion and fuel handling related fuel properties have to be analysed and determined for each specific biofuel (e.g. wood chips, pellets, briquettes). This pre-normative work is continuation of the results of the first BioNorm project, which resulted in "Solid biofuels – Guide for a Quality Assurance System", CEN Technical Report (CEN/TR 15569) and a draft to the CEN/prEN 14961. This standard will be transferred in the first full European Norm (EN) for solid biofuels at the beginning of 2010.

Therefore the collection of feedback from the practice, scientific institutions and other BioNorm II partners to the draft standard, its evaluation and harmonisation have been the main tasks of WP IV. As prEN 14961-1 also includes wood waste one aim was to clarify by the help of a detailed study, which fractions of wood waste can be defined as solid biofuel. The encouraged work finished in a new draft of the prEN 14961 with the integration of more detailed specifications and for further solid biofuels. Furthermore, some modifications of existing thresholds were needed but also the extension of the prEN 14961 to multi-part standard, in which concrete fuel quality requirements for different solid biofuel product used in households and industry can be seen as a result of the feedback analysis of companies and several associations and in collaboration with the CEN/TC 335 WG 2. Also combustion tests with small-scale boilers (< 50 kW) and by using the most traded solid biomass fuels (pellets, briquettes, wood logs, wood chips and olive cakes) have been carried out in order to identify and assess the properties, which are defined (or needed) by the combustion appliances and other end-user devices. The team also benefits the upgrading of CEN/TC 335 standards to ISO standards.

4 CONCLUSIONS

The two BioNorm projects were one of the most important measures of the EC to promote the development of standards within the field of solid biofuels. Without this support for the determination of the scientific basis it would have not been possible to develop in such a relative short time period reliable and acceptable standards covering the whole range of aspects needed to build the foundation for the fast and successful development of the market for solid biofuels in Europe. And the market development during the last couple of years shows very clearly that well prepared standards can indeed help to develop the markets in a sustainable way.

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- DONG Energy Generation A/S (DONG), DK
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- Swedish University of Agricultural Sciences (SLU)
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- Università Politecnica delle Marche (UNIVPM), ITL

2 Working Package I
- Sampling -

Coordinated by: Robert Samuelson, SLU

Involved partners:

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2.1 EVALUATION OF METHODS FOR SAMPLING AND SAMPLE REDUCTION OF SOLID BIOFUELS FROM NORTHERN EUROPE

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Designed sampling experiments were carried out on three different biofuel materials. The aim of the project was to assess the bias that might be introduced when samples are taken from static heaps (for example containers or vehicles) instead of from moving streams, and to define the number of increments needed to provide a representative bulk sample. The results showed that bias between the sampling methods was obtained only for a few parameters, in particular parameters where different particle sizes can segregate i.e. particle size distribution of wood chips and mechanical durability in pellets. Individual variances for sub-lots, increments and sub-sample tests were estimated using Analysis of Variance (ANOVA). These variances were then used to determine the minimum number of increments needed to obtain a specified, required precision. To verify that the results were valid also for sampling over a longer period of time, the variation in the materials was studied during 10-12 weeks. In all cases the results in the two examinations were consistent. In addition, the precision of three sample reduction methods were evaluated. The results showed that no one method of sample reduction gave a significant smaller between sub-sample standard deviation than the others for all test methods and materials.

Keywords: BioNorm, solid biofuels, sampling, increment number, bias

1 INTRODUCTION

In the recently-completed BioNorm project, statistically designed sampling experiments were carried out in Sweden with three kinds of wood fuel – sawdust, wood chips from forestry residues consisting of side branches and tops discarded in the tree felling operation and wood pellets in which the moisture content, particle size distribution and ash content were determined. In Denmark similar experiments were carried out with straw bales in which moisture, ash and chloride content were analysed [1].

Therefore, Working Package I of the EU-project BioNorm II extended this already completed work by applying broadly the same methodology to a second selection of materials and test methods, i.e. other materials that are of general interest across the EU; different kinds of wood chips, pellets with enhanced ash content and bark from conifer.

Chemical and physical analyses of biofuels are always afflicted with measuring uncertainty also named measuring error. This measuring error may be divided into two types, namely systematic error or bias, and experimental error. Bias affect the accuracy (how close the average of the analytical result is to the “true value”), and is often caused by a single factor like erroneous calibration, inappropriate analytical method, incomplete dissolution of the sample, etc. This error gives rise to an analytical result that is always too low or too large compared to the “true result” and should always be eliminated as far as possible. Experimental error on the other hand is due to several factors where each factor has small impact on the result and may affect the result in both positive and negative direction. Examples of such factors are variations in humidity, ambient temperature, pipette volumes, etc. This type of error does not influence the accuracy of the analytical result as the bias does. Instead the experimental error affects the precision, which is the distribution of the analytical result around the “true value”. A low precision means that the results from repeated analyses have a large spread around the “true value” and vice versa. Experimental errors cannot be eliminated, but can be handled by suitable statistical methods.

The objectives of the experiments in this paper were to assess the relative bias of the two studied sampling methods for solid biofuels, heap sampling and conveyor sampling, and to derive recommendations on how to decide the numbers of sampling increments needed to obtain an accepted sampling precision. In addition, the behaviour of three different sample reduction methods was evaluated.

2 EXPERIMENTAL

2.1 Experimental design

2.1.1 Bias testing and variance estimation

The experimental design used for the evaluation of sampling methods is a nested (hierarchical) design described in [2]. Three different biofuel materials were studied in the sampling experiments, namely bark, wood chips and 8mm pellets from stem wood including bark. A detailed description of the materials is shown in table 1.

Table 1: Detailed description of biofuel materials

Sample	**Sample origin**
Bark	Bark from Scots pine with a nominal top size of 100mm
Wood chips	Wood chips from stem wood including bark with a nominal top size of 16mm. Conifer: Deciduous 95:5.
Pellets	8mm pellets produced from stem wood including bark (see wood chips above)

Five lorry-loads (about 40 ton each) of bark were sampled from a heap tipped on an asphalted plane and from a stopped conveyor, respectively. The sampling from the stopped conveyor was carried out by loading the bark material onto the conveyor and sampling the material along the conveyor belt until the appropriate sample volume was reached. The reason to this was that the feed of material on the conveyor was too irregular to obtain a well defined volume when using the stopped conveyor method described in [3]. From the sub-lots, four increments of three different increment sizes (20L, 10L and 5L) were collected by both methods.

Five sub-lots of wood chips were sampled both from a falling stream at the end of a moving conveyor right after the chipper, where the stem wood logs were chipped successively by conveyor feeding, and from the formed heap. The amount of material in the sub-lots was about 10 ton. From each sub-lot, four increments of three different increment sizes each (10L, 4L and 2L) were sampled by both methods.

The 8mm pellets were sampled both from a falling stream at the end of a moving conveyor in the storage area and from the formed heap. The amount of material in the sub-lots was about 10 ton. From each sub-lot, four increments of the increment sizes 8L, 4L and 2,5L were collected by both methods.

The sampling experiments were carried out during a period of one week in all cases.

For all materials two sub-samples of each increment were tested for moisture content [4] and ash content [5]. In addition, particle size distribution [6], gross calorific value [7] and mechanical durability [8] were analyzed in wood chips, bark and pellets, respectively. The sieve apertures used in the analyses of particle size distribution were 16mm, 8mm, 5mm, 3mm and 2mm.

2.1.2 Long time variation

To verify that the results from the sampling experiments in section 2.1.1 were valid also for sampling over a longer period of time, the variation in the materials was studied during 10-12 weeks. One to two sub-lots a week were sampled for all materials, obtaining a total number of sub-lots of 19, 11 and 10 for bark, wood chips and pellets, respectively. Four increments per sub-lot were aggregated to a laboratory sample that was divided into two sub-samples by using coning & quartering for bark and riffle box for the other materials. The sub-samples were then analyzed for the same parameters as in section 2.1.1.For the investigations described in 2.1.1 and 2.1.2 the sampling experiments with bark were carried out at the power plant of Skellefteå Kraft AB in Hedensbyn Skellefteå, while the other two materials were sampled at Skellefteå Kraft AB pellet plant in Storuman.

2.1.3 Assessment of sample reduction methods

For the assessment of sample reduction methods, 10kg, 15kg and 30kg of wood chips, pellets and bark respectively, were homogenized and divided into 16 sub-samples using the coning & quartering, long pile and riffle box methods. Each sub-sample was then analyzed for the analytical parameters mentioned above.

2.2 Statistical analyses

2.2.1 Evaluation of bias

No method of sampling biofuels is considered to be a reference method, so the data can be used only to assess the bias of one method relative to another.

The bias is evaluated by using a paired comparison design, where the difference between the mean values of the two sampling methods for each sub-lot and increment size is calculated. The mean value of these differences is then tested by the Student´s t-test ($\alpha = 0.05$) for the hypothesis that the difference is not significant different from zero. If the mean of the differences is significant different from zero there is a bias between the sampling methods, otherwise not. The bias testing was also performed for the three increment sizes separately. To ensure that the significant test is valid, the difference values where tested for normality with $\alpha = 0.05$. In addition, a graph was constructed where the average of the conveyor sampling results for each sub-lot was plotted against the corresponding heap sampling results. If there is no bias between the sampling methods, a relatively even distribution of the points on both sides of the diagonal line in the graph should be seen for each sample size.

2.2.2 Evaluation of sampling variances

Basic information on the variability between sub-lots, increments and sub-sample tests due to sampling was provided by Analysis of Variances (ANOVA) of the analytical data.

From the individual analytical results of the sub-samples A and B, y_{bst}, the average of each increment, $\hat{y}_{bs}$, was calculated. The averages of the increments were then used to calculate the average of each sub-lot, $\hat{y}_b$, and finally the grand average, $\hat{y}$, was calculated from the results of all sub-samples A and B.

The values for $\hat{y}$, $\hat{y}_b$, $\hat{y}_{bs}$ and y_{bst} were then used together with the number of sub-lots N, the number of increments I and the number of sub-sample tests T for the calculation of Sum of Squares (SoS) for the sub-lots, the increments and the sub-sample tests according to table 2.

Table 2 Formulas for calculation of Sum of Squares (SoS).

	SoS	DoF
Sub-lots	$IT\sum(\hat{y}_b-\hat{y})^2$	N-1
Samples	$T\sum\sum(\hat{y}_{bs}-\hat{y}_b)^2$	N(I-1)
Test	$\sum\sum\sum(y_{bst}-\hat{y}_{bs})^2$	NI(T-1)

The SoS were then divided by the Degrees of Freedom (DoF) to give the Mean Square (MS) of the sub-lots, the increments and the sub-sample tests, respectively.

From the MS-values the individual variances σ^2_N, σ^2_I and σ^2_T for the sub-lots, the increments and the sub-sample tests were estimated from the formulas (1) to (3).

$$MS_N = IT\,\sigma^2_N + T\,\sigma^2_I + \sigma^2_T \qquad (1)$$

$$MS_I = T\,\sigma^2_I + \sigma^2_T \qquad (2)$$

$$MS_T = \sigma^2_T \qquad (3)$$

Significance tests of variances

Significance tests of differences in variances between increment sizes were performed on Mean Square-values since these have well defined Degrees of Freedom. The differences in MS between the increment sizes were tested for their significance by using the F-distribution with α = 0.05. If no significant difference is found between increment sizes within a sampling method, an average of the variance may be calculated and the differences between the methods are then tested for their significance as before. If there is a significant difference in variances between increment sizes an average value cannot be calculated and the significance test of the difference between sampling methods has to be performed for the individual increment sizes.

2.2.3 Repeatability

For the analytical methods where a general analysis sample is used for the determination, a repeatability standard deviation was calculated. This was accomplished by repeating the analysis 5-6 times for one single general analysis sample. The result was used to assess the significance of the repeatability of the analytical method compared to the overall analytical variability.

2.2.4 Evaluation of long time variation

A suitable statistical method for the evaluation of long term variation is the Shewhart control chart. In this method the values from the analyses of a fuel parameter are collected over a long period of time. From these values the average **ŷ** and the standard deviation **s** are calculated. A chart is then constructed where the individual values are plotted in time order and where the average **ŷ** together with ±**1s** and ±**2s** are shown in the chart as control lines.

The feature with the Shewhart control chart is that you easily get a measure of the long term variation in the data. In addition, it indicates how much variation about the average ŷ that can be expected to occur by chance and if you have any outliers or trends in the data.

In addition, a comparison of the corresponding total standard deviations in the experiments described in sections 2.1.1 and 2.1.2 was performed by using a significance test of variances (F-distribution). If no significant differences between the variances are found, the results may be considered as representative for the material under study and can, for example, be used for the calculation of the number of sub-lots, increments or sub-sample tests necessary to obtain a certain level of precision in the analytical result.

2.2.5 Assessment of sample reduction methods

The evaluation of the precision for the various sample reduction methods was performed by calculating the variance and the coefficient of variance of the individual results of the analytical parameters for each material and reduction method. The differences in the variances were then tested for its significance by using the F-distribution (α = 0.05).

3 RESULTS

3.1 Evaluation of bias

In table 3 the analytical results together with the standard deviation, the coefficient of variance (CV) and the number of sub-lots tested (n) for the studied parameters in bark, wood chips and 8mm pellets are shown. The data for each individual parameter was represented by the average of all sub-lots analyzed in the two experiments described in 2.1.1 and 2.1.2.

For the ash content repeatability standard deviations of 0,0057%, 0,012% and 0,013% for bark, wood chips and pellets, respectively were obtained. For gross calorific value in bark the repeatability standard deviation was 0,017 MJ/kg.

Table 3: Analytical results, standard deviations, coefficients of variation and number of sub-lots for a) bark, b) wood chips and c) 8mm pellets

a)

Parameter	**Average**	**Std.dev.**	**CV**	**n**
Moisture (%)	57,3	2,57	4,49	24
Ash (%)	2,1	0,50	23,6	24
Gross Calorific Value (MJ/kg)	20,7	0,21	1,03	24

b)

Parameter	Average	Stddev	CV	n
Moisture (%)	45,1	3,72	8,24	21
Ash (%)	0,62	0,19	30,7	21
Particle size <16>8mm (%)	14,5	3,3	22,8	21
Particle size <8>5mm (%)	41,5	3,2	7,7	21
Particle size <5>3mm (%)	31,0	3,2	10,4	21
Particle size <3>2mm (%)	4,9	1,1	23,1	21
Particle size <2mm (%)	7,4	2,1	28,8	21

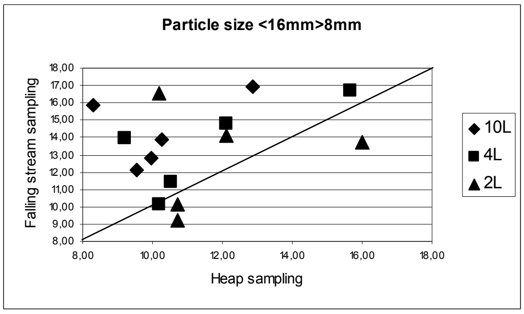
c)

Parameter	Average	Stddev	CV	n
Moisture (%)	8,4	0,62	7,30	25
Ash (%)	0,62	0,048	7,78	25
Mechanical durability (%)	97,6	0,15	0,15	25

No bias between the sampling methods was found for the moisture content and ash content in all materials.

For the particle size distribution in wood chips, on the other hand, a pronounced bias is found which is significant for the 10L increment size (see figure 1). From the graphs one can see that the largest particle sizes are overrepresented in the conveyor sampling while the smallest particle sizes are overrepresented in the heap sampling.

a)



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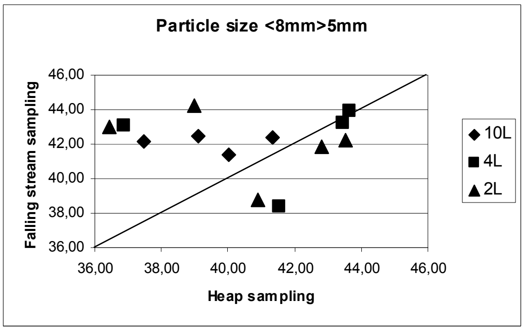
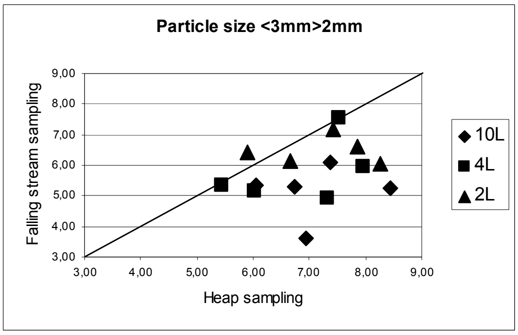


c)



d)

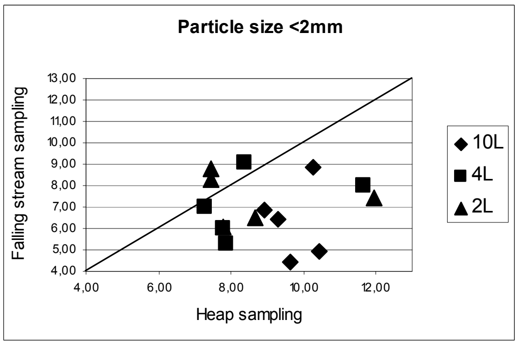


Figure 1: Test of bias between sampling methods for particle size distribution in wood chips by plotting conveyor sampling vs. heap sampling. a) <16>8mm, b) <8>5mm, c) <3>2mm and d) <2mm. The unit is %.

A significant bias between the sampling methods for the mechanical durability was also observed for all increment sizes for the 8mm pellet material. The t-value was 4,94 compared to a t_{crit} of 2,14. The result is shown in figure 2.

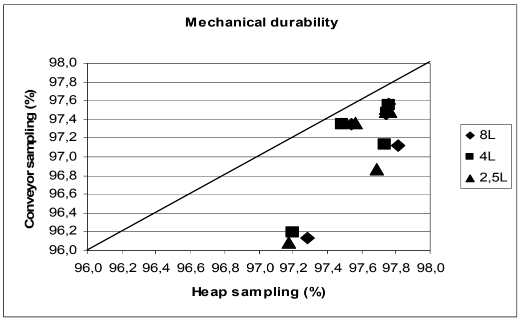


Figure 2: Test of bias between sampling methods for mechanical durability in 8mm pellets by plotting conveyor sampling vs. heap sampling. The unit is %.

3.2 Evaluation of variances

For all materials easy interpretable results for most analytical parameters have been obtained with small differences in variance both between increment sizes and sampling methods. The only differences of importance were the significantly higher precision of the 10L increment for all parameters in the heap sampling of wood chips, and the significantly higher precision of heap sampling for mechanical durability in 8mm pellets.

For some materials and parameters significant differences in sub-sample test variances were found both between increments and sampling methods. However, these variances were negligible compared to the increment and sub-lot variances and had therefore no impact on the final results.

In table 4 the individual, variances for moisture and ash content for the various biofuel materials are summarized according to equations (1) to (3).

Table 4: Individual variances for sub-lots (σ2N), increments (σ2I) and sub-sample tests (σ2T) for moistiure and ash content.

	Moisture (%)^2			**Ash (%)^2**		
Biofuel	σ^2_N	σ^2_I	σ^2_T	σ^2_N	σ^2_I	σ^2_T
Bark	3,12	4,89	0,68	0,38	0,026	0,019
Wood chips	1,98	10,5	0,058	0,027	0,023	0,00040
Pellets (8mm)	0,80	0,16	0,0032	0,0050	0,00035	0,00033

The individual variances obtained for gross calorific value in bark were σ^2_N=0,077, σ^2_I=0,0045 and σ^2_T=0,0042 (MJ/kg)^2, while the corresponding values for the mechanical durability in pellets were σ^2_N=0,19, σ^2_I=0,019 and σ^2_T=0,0061 (%)^2.

3.2 Evaluation of long time variances

In table 6, a comparison of the short time and long time variations of bark, wood chips and 8mm pellets are shown. The only significant difference in variation between the two investigations was found for the fraction <3>2mm in the particle size distribution in wood chips.

Table 6: Comparison of the overall standard deviation over short and long time for a) bark, b) wood chips and c) 8mm pellets. The units are %, except for gross calorific value where the unit is MJ/kg.

a)

Parameter	**Overall std.dev. Task I.1 short time**	**Overall std.dev. Task I.2 long time**	**F-value overall variance**	**F_{crit}**
Moisture	2,21	2,31	1,09	2,9
Ash	0,63	0,42	2,25	5,8
Calorific value	0,26	0,18	2,09	5,8

b)

Parameter	**Overall std.dev. short time**	**Overall std.dev. long time**	**F-value overall variance**	**F_{crit}**
Moisture	2,53	3,33	1,73	5,9
Ash	0,16	0,15	1,14	3,1
Particle size <16>8mm	3,03	4,21	1,93	5,9
Particle size <8>5mm	2,02	3,43	2,88	5,9
Particle size <5>3mm	3,22	3,77	1,37	5,9
Particle size <3>2mm	0,45	1,32	8,60	5,9
Particle size <2mm	1,16	2,49	4,61	5,9

c)

Parameter	**Overall std.dev. short time**	**Overall std.dev. long time**	**F-value overall variation**	**F_{crit}**
Moisture	0,71	1,26	3,15	6,0
Ash	0,068	0,049	1,93	3,6
Mechanical durability	0,24	0,26	1,17	6,0

In figure 3 an example of a Shewhart control chart is shown for moisture in bark. For all materials and parameters no outliers or trends in data were found.

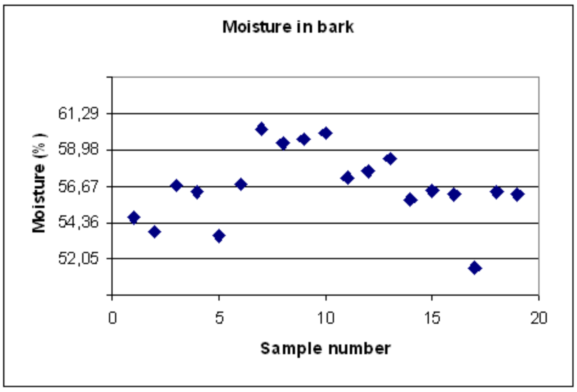


Figure 3: Shewhart control chart for moisture in bark.

3.3 Assessment of sample reduction methods

Table 5 presents the coefficient of variance, CV_r, for the sample reduction of the materials under study when using the coning & quarteriang (C&Q), the long pile (LP) and the riffle box (RB) methods.

Table 5: Evaluation of the sample reduction methods coning & quartering (C&Q), long pile (LP) and riffle box (RB) for bark, wood chips and pellets determined as coefficient of variances (CV_r). a) moisture and ash content b) gross calorific value, GCV (bark) and mechanical durability (pellets).

a)

	CV_r, moisture (%)			CV_r, ash (%)		
Sample	**C&Q**	**LP**	**RB**	**C&Q**	**LP**	**RB**
Bark	0,57	0,59	-	6,3	4,8	-
Wood chips	0,30	0,34	0,23	7,8*	2,6	2,4
Pellets	0,56	0,46	0,86	5,2	6,8	4,4

* a different laboratory sample was used in this case

b)

	CV_r, GCV (MJ/kg)		CV_r, mechanical durability (%)		
Sample	**C&Q**	**LP**	**C&Q**	**LP**	**RB**
Bark	0,45	0,37	-	-	-
Pellets	-	-	0,13	0,10	0,13

For the particle size distribution the CV_r varied from about 8% for the fraction <16mm>8mm to about 2% for the fraction <8mm>5mm. No significant difference between the reduction methods was found for the materials and parameters.

4 DISCUSSION

4.1 Bias

The bias found for particle size distribution and mechanical durability for wood chips and 8mm pellets, respectively, is probably caused by segregation of different particle sizes. In the pellet heap the fine particles will stay in the middle, while the larger pellets will flow along the sides where the sampling is accomplished. This gives an overrepresentation of small particles in the conveyor sample. These small particles will then be destroyed in the analysis of mechanical durability and removed in the sieving step, with a lower durability as a consequence.

For particle size distribution in wood chips, on the other hand, an overrepresentation of the large particles was found in the conveyor sample. This cannot be explained by segregation of the particles in the heap, since the small particles tend to stay in the middle of the heap. Instead it might be some sort of discrimination of particle sizes in the falling stream.

4.2 Variances

The significant higher precision for the 10L increment of the heap sampling of wood chips is probably caused by chance since all parameters were affected and since no difference was found for the conveyor sampling.

The significant lower precision for mechanical durability in the conveyor sampling of 8mm pellets is probably connected to the bias discussed in section 4.1.

4.3 Calculation of number of increments

The minimum number of increments to be taken from a lot or a sub-lot of a biofuel material to obtain a specified precision of the analytical parameter of interest depends on the heterogeneity of that parameter in the material in combination with the design of the sampling method. Some energy companies are analyzing every single sub-lot, i.e. lorry load, and using the result of each individual sub-lot for the payment of the biofuel. Other companies are using the average of a number of sub-lots, i.e. all lorry loads during one month, as the bases for the biofuel price. Still other companies are dealing with much larger lots, i.e. shipments of 30 000 ton, which are usually divided into sub-lots of about 2 500 ton from which the sample increments are collected.

Below two sampling designs are described in detail together with the corresponding formula for the calculation of minimum increment number.

4.3.1 Analysis of one sub-lot (10-50 ton)

The sub-lot, i.e. a lorry load, is sampled by a specified number of increments. These increments are aggregated to give a laboratory sample which is sent to the analytical laboratory. This laboratory sample is then divided into a number of sub-samples, which are analyzed for the specified parameter.

The uncertainty of this analysis may be calculated by the following equation

$$e = t\sqrt{\frac{\sigma_I^2}{I} + \frac{\sigma_T^2}{T}} \qquad (4)$$

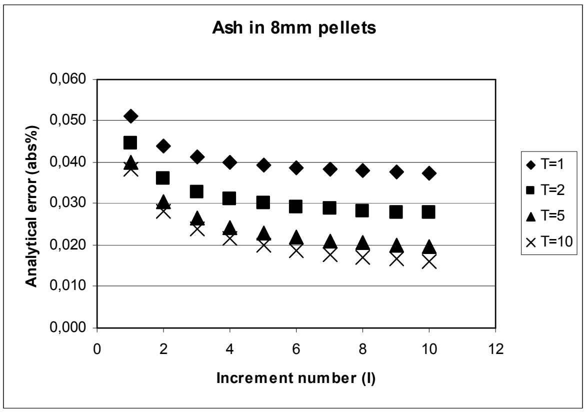
where e is the uncertainty of the analysis; σ^2_I and σ^2_T are the increment and the sub-sample test variances, respectively; I is the number of increments, T is the number of sub-sample tests and t is the Student´s value for α = 0.05 which is approximately 2.

By rearranging equation (4) the minimum number of increments needed (*I*) to obtain a specified precision (*e*) for a specified parameter is determined according to the following formula

$$I=\frac{4\sigma_I^2}{e^2-\frac{4\sigma_T^2}{T}} \qquad (5)$$

The required precision, e, and the number of sub-samples, T, may be decided by the producer and the customer in agreement. The values for σ2I and σ2T may be determined by the company for the material under study or may be determined from earlier experience.
In addition, by constructing a graph where the precision, e is plotted versus the number of increments, I, for certain values of sub-sample tests, T, the minimum number of increments for a certain required precision may also be determined.
To demonstrate the effect of the relation between σ2I and σ2T on the sampling error, three different situations are described in figure 5.

a)



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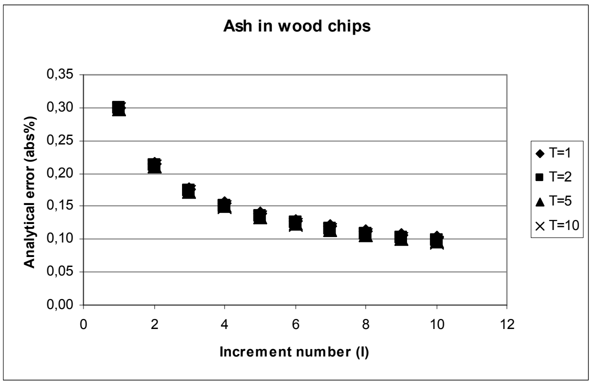

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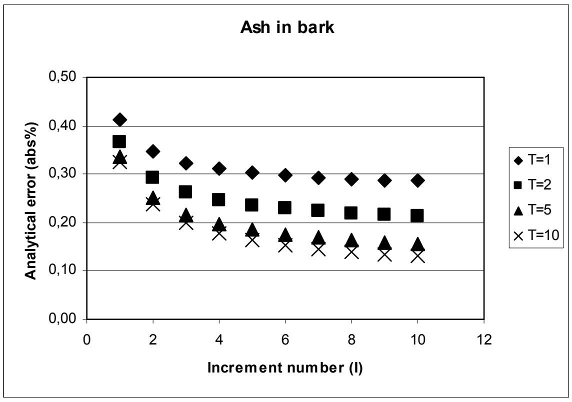


Figure 5: The effect of the increment numbers on the precision for various numbers of sub-sample tests. a) ash in 8mm pellets, b) ash in wood chips c) ash in bark,.

As can be seen from figure 5 the relation between the increment and the sub-sample variance is of great importance how the increment number influences the precision. For ash in 8mm pellets and bark, where σ^2_I and σ^2_T are of the same order, both the increment number and the number of sub-sample tests determine the analytical error
On the other hand, for wood chips where σ^2_T is much smaller than σ^2_I the increment number alone determine the sampling error without influence of the number of sub-sample tests.

4.3.2 Analysis of several sub-lots (10-50 ton each)
A number of sub-lots are sampled by a specified number of increments per sub-lot. The increments from each sub-lot are aggregated to give a laboratory sample which is sent to the analytical laboratory. Each laboratory sample is then divided into a number of sub-samples, which are analyzed for the specified parameter. The final result is obtained by averaging the results from each sub-lot which is then an estimation of the studied parameter in the original lot.

The uncertainty of this analysis may be calculated by the following equation

$$e=t\sqrt{\frac{\sigma_N^2}{N}+\frac{\sigma_I^2}{NI}+\frac{\sigma_T^2}{NT}} \qquad (6)$$

where e is the uncertainty of the analysed parameter in the lot; σ^2_N, σ^2_I and σ^2_T are the sub-lot, the increment and the sub-sample variances, respectively; N is the number of sub-lots averaged, I is the number of increments, T is the number of sub-sample tests and t is the Student´s value for α = 0.05 which is approximately 2.

The minimum number of increments needed (*I*) to obtain a specified precision (*e*) for a specified parameter is determined according to the following formula

$$I=\frac{4\sigma_I^2}{Ne-4\sigma_N^2-\dfrac{4\sigma_T^2}{T}} \qquad (7)$$

The required precision, *e,* the number of sub-lots, *N*, and the number of sub-samples, *T*, may be decided by the producer and the customer in agreement. The values for σ^2_N, σ^2_I and σ^2_T may be determined by the company for the material under study or may be assumed from earlier experience.

However, the equations (6) and (7) are valid only under the circumstances where the number of sub-lots sampled and analyzed is negligible compared to the total sub-lots in the lot. If instead all sub-lots in the lot are sampled and tested, the contribution from the sub-lot variance in the final result will diminish and the equations (6) and (7) will change as follows:

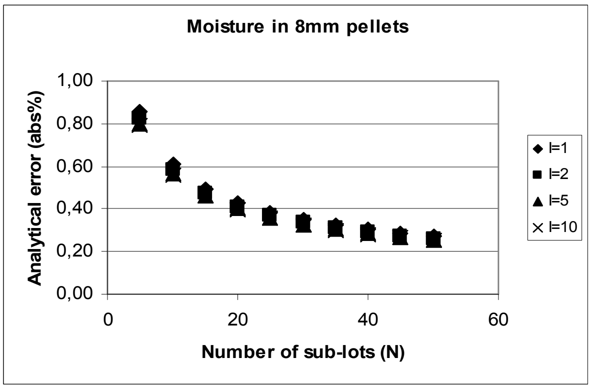
$$e=t\sqrt{\frac{\sigma_I^2}{NI}+\frac{\sigma_T^2}{NT}} \qquad (8)$$

$$I=\frac{4\sigma_I^2}{Ne^2-\dfrac{4\sigma_T^2}{T}} \qquad (9)$$

In a similar way as in section 4.3.1, the minimum number of increments for a certain required precision may also be determined by constructing a graph where the precision, *e*, is plotted versus the number of sub-lots, *N*, for certain numbers of increments, I.

To demonstrate the effect of the relation between σ^2_N and σ^2_I on the sampling error, three different situations are described in figure 6 according to equation (6) where a limited number of sub-lots are analyzed.

a)



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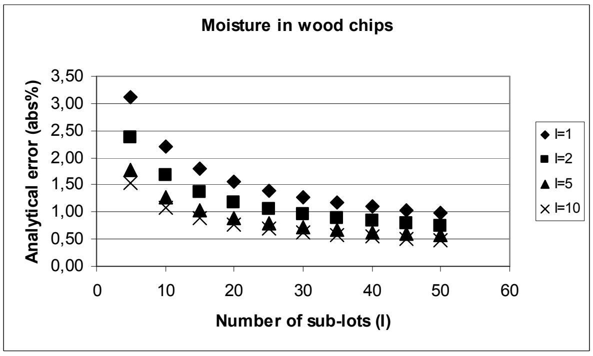

c)

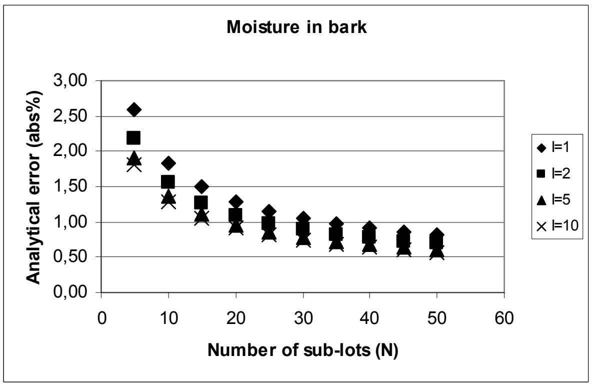

Figure 6 The effect of number of sub-lots on the precision for various increment numbers when a limited number of sub-lots are sampled and tested. The number of sub-sample tests = 1. a) moisture in 8mm pellets, b) moisture in wood chips c) moisture in bark,.

Even in this case the relation between the individual variances determines the influence of the increment number on the sampling error. As can be seen from table 4 for moisture content in pellets, the sub-lot variance is much larger than both the increment variance and the sub-sample variance. This means that the increment number and the number of sub-sample tests are of little importance for the uncertainty of the overall analytical result.

For the moisture content in wood chips on the other hand, where the increment variance is much larger than the sub-lot variance the increment number has a large impact on the final precision. A similar effect is obtained for bark, but less pronounced.

The corresponding graphs for the sample scheme where all sub-lots in the lot are tested are shown in figure 7. Here we see a considerable change in behaviour for moisture in pellets, where the increment number has a much larger impact on the precision due to the elimination of the effect from the sub-lot variance. For wood chips and bark a less pronounced effect is found.

However, for all cases the minimum number of increments can be decreased by increasing the number of sub-lots averaged.

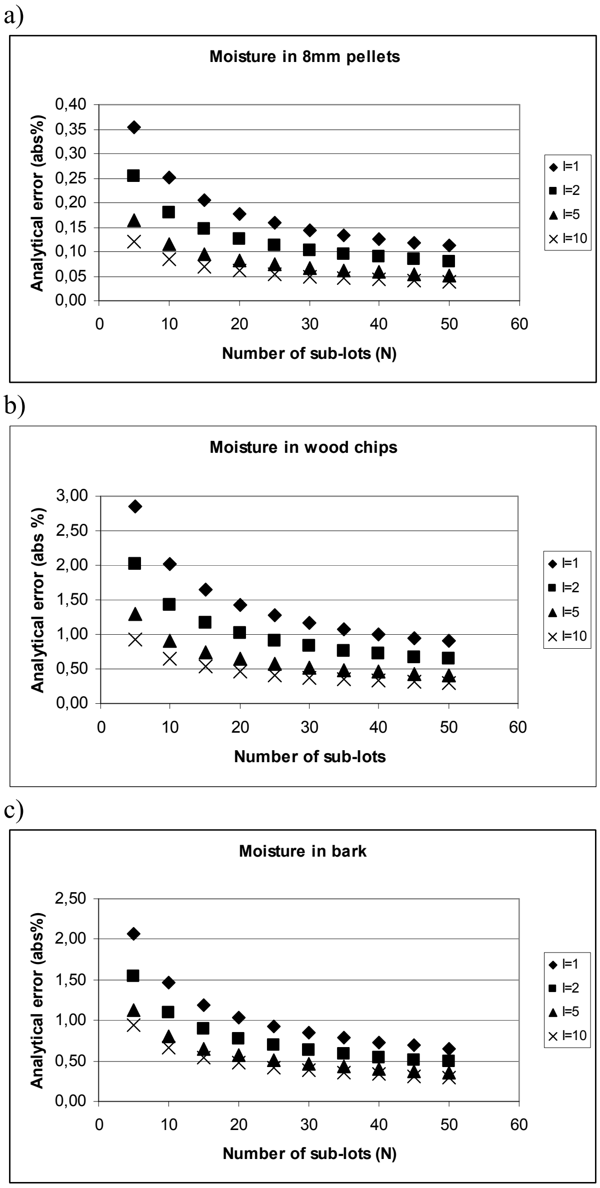

Figure 7: The effect of number of sub-lots on the precision for various increment numbers when all sub-lots in a lot are sampled and tested. The number of sub-sample tests = 1, a) moisture in 8mm pellets, b) moisture in wood chips c) moisture in bark,.

4.4 Long time variances

Good agreement between the overall standard deviations for all parameters in the two examinations of bark and 8mm pellets was obtained.

Also for wood chips a good agreement between the two tasks was found even if a significant difference in overall standard deviation was acquired for the particle sizes <3>2mm. The reason for this is due to the inexplicably small variance obtained in the first task for this fraction. However, this would anyway indicate that the obtained values of variation may be considered as reliable and representative for the studied materials.

4.5 Assessment of sample reduction methods

No one method of sample reduction gave a significant smaller between sub-sample standard deviation than the others for all three of the test methods. Thus for the determination of the studied parameters, the choice of sample reduction method in most cases is decided from a practical point of view, i.e. time consumption, number and size of sub-samples, nominal top size, etc.

5 CONCLUSIONS

The following conclusions can be drawn from this work:

- Bias between the sampling methods was obtained only for particle size distribution in wood chips and mechanical durability in 8mm pellets.
- All materials showed small differences in variance both between increment sizes and sampling methods.
- The individual sub-lot, increment and sub-sample test variances estimated by the ANOVA method can be used to evaluate the minimum increment number needed to obtain a specified required precision.
- Depending on the sampling scheme different formulas for the calculation of the minimum increment number will be used.
- Long time studies verified the individual variance values σ^2_N, σ^2_I and σ^2_T, which would indicate that the obtained values of variation may be considered as reliable and representative for the studied materials.
- Similar precisions were obtained for the sample reduction methods coning & quartering, long pile and riffle box when dividing bark, wood chips and 8mm pellets.

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7 ACKNOWLEDGEMENTS

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2.2 EVALUATION OF METHODS FOR SAMPLING AND SAMPLE REDUCTION OF SOLID BIOFUELS FROM SOUTHERN EUROPE

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Three Mediterranean materials were investigated for sampling properties: olive residues, grape residues and 6mm pellet. Designed sampling experiments were carried out on the three different biofuel materials. The aim of the project was to assess the bias that might be introduced when samples are taken from static heaps (for example containers or vehicles) instead of from moving streams, and to define the number and size of increments needed to provide a representative bulk sample. The results showed that bias between the sampling methods was obtained only for a few parameters. Individual variances for sub-lots, increments and sub-sample tests were estimated using Analysis of Variance (ANOVA). These variances were then used to determine the minimum number of increments needed to obtain a specified, required precision. To verify that the results were valid also for sampling over a longer period of time, the variation in the materials was studied during some weeks. The results in the two examinations were consistent only for grape residues, while for the other materials explications are done. In addition, the best reduction method was found for each material, after comparison between different methods: long pile, coning & quartering and riffle box.

Keywords: BioNorm, solid biofuels, mass reduction, sampling, increment number, bias

1 INTRODUCTION

In the recently-completed BioNorm project, statistically designed sampling experiments were carried out in Sweden with three kinds of wood fuel – sawdust, wood chips from forestry residues consisting of side branches and tops discarded in the tree felling operation and wood pellets in which the moisture content, particle size distribution and ash content were determined. In Denmark similar experiments were carried out with straw bales in which moisture, ash and chloride content were analysed [1].

Therefore, Work Package I of the EU-project BioNorm II extended the already completed work by applying broadly the same methodology to a second selection of materials and test methods, including solid biofuels that are specially relevant for Southern Europe: agricultural residues from the production and processing of olives and grapes as well as pellet.

Chemical and physical analyses of biofuels are always afflicted with measuring uncertainty also named measuring error. This measuring error may be divided into two types, namely systematic error or bias, and experimental error. Bias affect the accuracy (how close the average of the analytical result is to the “true value”), and is often caused by a single factor like erroneous calibration, inappropriate analytical method, incomplete dissolution of the sample, etc. This error gives rise to an analytical result that is always too low or too large compared to the “true result” and should always be eliminated as far as possible. Experimental error on the other hand is due to several factors where each factor has small impact on the result and may affect the result in both positive and negative direction. Examples of such factors are variations in humidity, ambient temperature, pipette volumes, etc. This type of error does not influence the accuracy of the analytical result as the bias does. Instead the experimental error affects the precision, which is the distribution of the analytical results around the “true value”. A low precision means that the results from repeated analyses have a large spread around the “true value” and vice versa. Experimental errors cannot be eliminated, but can be handled by suitable statistical methods.

The objectives of the experiments in this paper were to assess the relative bias of the two studied sampling methods for solid biofuels, heap sampling and conveyor sampling, and to derive recommendations on how to decide the sizes and numbers of sampling increments needed to obtain an accepted sampling precision. In addition, the behavior of three different sample reduction methods was evaluated.

2 EXPERIMENTAL

2.1 Experimental design

2.1.1 Bias testing and variance estimation

The experimental design used in this investigation is a nested (hierarchical) design described in [2].

Three different biofuel materials were studied in the sampling experiments, namely olive residue, grape residue and 6mm pellets from whole trees of beech.

Five lorry loads of olive and grape residues were both sampled from a heap and from a falling stream at the end of a moving conveyor, respectively [3]. From each sub-lot, four increments of three different increment sizes (10L, 5L and 2L) were taken by both methods. Two sub-samples of each increment were then tested for moisture [4], ash [5], nitrogen [6], and seven major elements (Al, Ca, Mg, Na, P, Si and K) [7].

The 6mm pellets were sampled either from heap and from moving conveyor. Five heaps were formed by collecting, for each heap, pellets derived from 10 pellets sacks. The sampling from moving conveyor means that each sub-lot was constituted by four pellet sacks randomly collected from the ending part of the production line. For the heap method, from each sub-lot four increments of three different increment sizes (8L, 4L and 2,5L) were taken. For the conveyor method, from each pellet sack of a sub-lot, the three different increment sizes (8L, 4L and 2,5L) were taken. Two sub-samples of each increment were tested for moisture [4], ash [5], and mechanical durability [8].

2.1.2 Long time variation

To verify that the results from the sampling experiments in section 2.1.1 were valid also for sampling over a longer period of time, the variation in the materials was studied during some weeks. The sampling over time was different for the three materials, depending on their availability, especially for seasonal biomasses. Four sub-lots a week during 10 weeks were sampled for pellet, obtaining a total number of sub-lots of 40. Four sub-lots a week during 5 weeks were sampled for grape residues, obtaining a total number of sub-lots of 20. The reason of this shorter time is that the grape residues production could not cover all the 10 weeks as for pellet. Since the olive residues derive from abroad, just one shipment could be investigated, taking samples from 10 lorries deriving from the shipment. For each material, four increments per sub-lot were aggregated to a laboratory sample that was divided into two sub-samples by using coning & quartering for grape residues and riffle box for the other materials. The sub-samples were then analyzed for the same parameters as in section 2.1.1.

For the investigations described in 2.1.1 and 2.1.2 the sampling experiments with olive and grape residues were carried out at C&T power plant in Termoli, while the sampling of pellet was performed in two different pellet production plants. In particular, for the investigation described in 2.1.1 samples were taken at Energy Pellet production plant in Riese Pio X where whole trees of beech trees were used; for the investigation described in 2.1.2 the sampling was performed at G.L.M. Ecowood production plant in Pergola, where different kind of forest materials were used (oak, Turkey oak, chestnut wood, beech, pine, fir, etc).

2.1.3 Assessment of sample reduction methods

For the assessment of sample reduction methods, 15kg of olive residues, grape residues and pellet were homogenized and divided into 16 sub-samples using the coning & quartering, long pile and riffle box methods [9]. Each sub-sample was then analyzed. In particular pellet sub-samples were tested for ash content [5] and durability [8]; the other materials were tested for moisture [4], ash [5], Nitrogen [6] and Potassium content [7].

2.2 Statistical analyses

2.2.1 Evaluation of bias

No method of sampling biofuels is considered to be a reference method, so the data can be used only to assess the bias of one method relative to another.

The bias is evaluated by using a paired comparison design, where the differences between the mean values of the two sampling methods for each sub-lot and increment size is calculated. The mean value of these differences is then tested by the Student´s t-test ($\alpha = 0.05$) for the hypothesis that the difference is not significantly different from zero. If the mean of the differences is significantly different from zero there is a bias between the sampling methods, otherwise not. The bias testing was also performed for the three increment sizes separately. To ensure that the significant test is valid, the difference values where previously tested for normality with $\alpha = 0.05$. In addition, a graph was constructed where the average of the conveyor sampling results for each sub-lot was plotted against the corresponding heap sampling results. If there is no bias between the sampling methods, a relatively even distribution of the points on both sides of the diagonal line in the graph should be seen for each sample size.

2.2.2 Evaluation of variances

Analysis of variance (ANOVA)

Basic information on the variability between sub-lots, increments and sub-sample tests due to sampling was provided by Analysis of Variances (ANOVA).

From the individual analytical results of the sub-samples A and B, y_{bst}, the average of each increment, $\hat{y}_{bs}$, was calculated. The averages of the increments were then used to calculate the average of each sub-lot, $\hat{y}_b$, and finally the grand average, $\hat{y}$, was calculated from the results of all sub-samples A and B.

The values for $\hat{y}$, $\hat{y}_b$, $\hat{y}_{bs}$ and y_{bst} were then used together with the number of sub-lots N, the number of increments I and the number of sub-sample tests T for the calculation of Sum of Squares (SoS) for the sub-lots, the increments and the sub-sample tests according to Table 1

Table 1: Formulas for calculation of Sum of Squares (SoS).

	SoS	DoF
Sub-lots	$IT\sum(\hat{y}_b-\hat{y})^2$	N-1
Samples	$T\sum\sum(\hat{y}_{bs}-\hat{y}_b)^2$	N(I-1)
Test	$\sum\sum\sum(y_{bst}-\hat{y}_{bs})^2$	NI(T-1)

These SoS were then divided by the Degrees of Freedom (DoF) to give the Mean Square (MS) of the sub-lots, the increments and the sub-sample tests, respectively.

From the MS-values the individual variances σ^2_N, σ^2_I, σ^2_T and the corresponding standard deviations s_N, s_I, s_T for the sub-lots, the increments and the sub-sample tests were estimated from the formulas (1) to (3).

$$MS_N = IT\,\sigma^2_N + T\,\sigma^2_I + \sigma^2_T \qquad (1)$$

$$MS_I = T\,\sigma^2_I + \sigma^2_T \qquad (2)$$

$$MS_T = \sigma^2_T \qquad (3)$$

Significance tests of variances

Significance tests of differences in variances between increment sizes were performed on Mean Square-values since these have well defined Degrees of Freedom. The differences in MS between the increment sizes were tested for their significance by using the F-distribution with $\alpha = 0.05$. If no significant difference is found between increment sizes within a sampling method an average of the variance may be calculated and the differences between the methods are then tested for their significance as before. If there is a significant difference in variances between increment sizes an average value cannot be calculated and the significance test of the difference between sampling methods has to be performed for the individual increment sizes.

2.2.3 Repeatability

For the analytical methods where a general analysis sample is used for the determination, a repeatability standard deviation was calculated. This was accomplished by repeating the analysis 5-6 times for one single general analysis sample. The result was used to assess the significance of the repeatability of the analytical method compared to the overall analytical variability.

2.2.4 Evaluation of long time variation

A suitable statistical method for the evaluation of long term variation is the Shewhart control chart. In this method the values from the analyses of a fuel parameter are collected over a long period of time. From these values the average $\hat{\mathbf{y}}$ and the standard deviation **s** are calculated. A chart is then constructed where the individual values (considered as mean values of sub-samples) are plotted in time order and where the average $\hat{\mathbf{y}}$ together with **±1s** and **±2s** are shown in the chart as control lines.

The feature with the Shewhart control chart is that you easily get a measure of the long term variation in the data. In addition, it indicates how much variation about the average $\hat{y}$ that can be expected to occur by chance and if you have any outliers or trends in the data.

In addition, a comparison of the corresponding total standard deviations in the experiments described in sections 2.1.1 and 2.1.2 was performed by using a significance test of variances (F-distribution). If no significant differences between the variances are found, the results may be considered as representative for the material under study and can, for example, be used for the calculation of the number of sub-lots, increments or sub-sample tests necessary to obtain a certain level of precision in the analytical result.

2.2.5 Assessment of sample reduction methods

The evaluation of the precision for the various sample reduction methods was performed by calculating the variance and the coefficient of variance of the individual results of the analytical parameters for each material and reduction method. The differences in the variances were then tested for its significance by using the F-distribution ($\alpha = 0.05$).

3 RESULTS

3.1 Evaluation of bias

No bias between the sampling methods was found for olive residues except for the 2L-increments of Aluminium and Magnesium where the heap sampling showed higher values than the conveyor sampling (see Figure 1). The t-values were 3.3 and 3.7 for Al and Mg, respectively compared to a t_{crit} of 2,78.

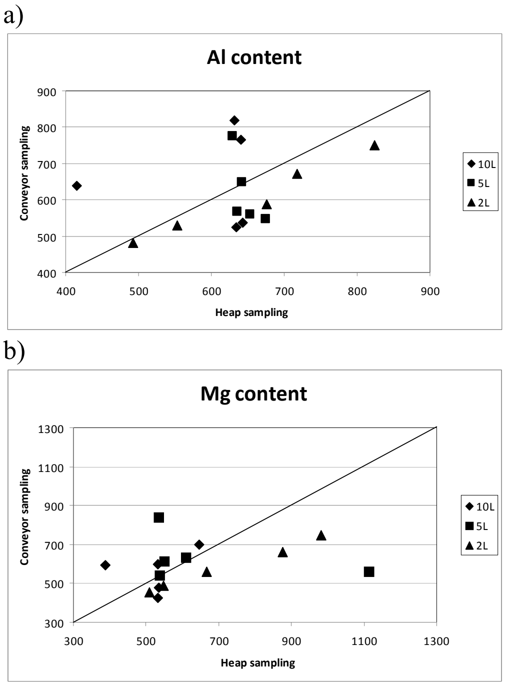


Figure 1: Test of bias between sampling methods for Aluminium (a) and Magnesium (b) in olive residues by plotting conveyor sampling vs. heap sampling. The unit is mg/kg DM.

For grape residues a bias between the sampling methods was found for the 2L-increment of Potassium, where the conveyor sampling gave higher Potassium content than the heap sampling (see Figure 2). The t-value was 5.78 compared to a tcrit of 2,78. In all other cases no difference between the sampling methods was found.

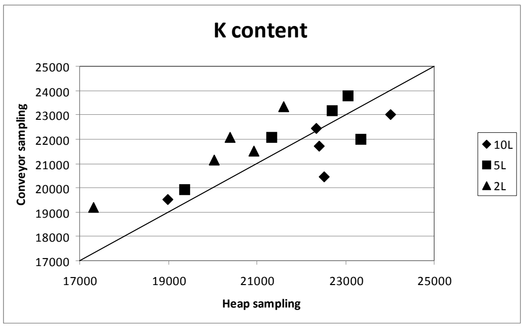


Figure 2: Test of bias between sampling methods for Potassium in grape residue by plotting conveyor sampling vs. heap sampling. The unit is mg/kg DM.

3.2 Evaluation of variances

For the pellet good results for ash content were obtained, without any significance in variances. Concerning the durability parameter high variance with the 8L-increment, probably due to the repeated riffling to reduce the size.

For the olive and grape residue a much more complicated situation was observed with differences in variance between both increment sizes and sampling methods that in many cases were contradictory. This was true especially for the major metallic elements. In general however it seems that the variability is higher with the 2L-increment.

In Table 2 the individual "pure" variance for moisture and ash content for the various biofuel materials are summarized according to equations (1) to (3).

Table 2 Individual variances for sub-lots (σ2N), increments (σ2I) and sub-sample tests (σ2T).

	Moisture (%)			Ash (%)		
Biofuel	σ^2_N	σ^2_I	σ^2_T	σ^2_N	σ^2_I	σ^2_T
Pellets 6(mm)	0,008	0,016	0,014	0,0004	0,0004	0,0071
Olive residue	0,120	0,105	0,028	0,832	0,658	0,527
Grape residue	6,06	0,746	1,88	0,198	0,519	0,202

3.3 Evaluation of long time variances

In Table 3, a comparison of the short time and long time variations of olive and grape residues and 6mm pellets are shown. The only significant difference in variation between the two investigations was found for many chemical elements in olive residues and for all parameters in 6mm pellet.

Table 3: Comparison of the overall standard deviation over short and long time for a) olive residues, b) grape residues and c) 6mm pellets. The units are %, except for the chemical elements values where the unit is mg/kg.

a)

Parameter	Overall std.dev. Task I.1 short time	Overall std.dev. Task I.2 long time	F-value overall variance	F_{crit}
Moisture	0,50	0,29	2,97	3,6
Ash	0,39	0,56	2,06	6,0
Aluminium	18	185	106	6,0
Calcium	344	1468	18,2	6,0
Magnesium	250	121	4,27	3,6
Sodium	43	40	1,16	3,6
Phosphorus	30	158	27,7	6,0
Silicon	264	1203	20,8	6,0
Potassium	526	1068	4,12	6,0
Nitrogen	0,091	0,088	1,07	3,6

b)

Parameter	Overall std.dev. Task I.1 short time	Overall std.dev. Task I.2 long time	F-value overall variance	F_{crit}
Moisture	2,64	3,53	1,34	3,5
Ash	0,30	0,41	1,37	3,5
Aluminium	54	24,7	2,19	3,5
Calcium	1542	685	2,25	3,5
Magnesium	100	118	1,18	3,5
Sodium	95	97,8	1,03	3,5
Phosphorus	203	242	1,19	3,5
Silicon	146	507	3,47	3,5
Potassium	1643	1931	1,18	3,5
Nitrogen	0,056	0,066	1,18	3,5

c)

Parameter	Overall std.dev. Task I.1 short time	Overall std.dev. Task I.2 long time	F-value overall variance	F_{crit}
Moisture	0,087	0,59	62,9	5,7
Ash	0,040	0,29	36,0	5,7
Durability	0,030	1,01	729	5,7

In Figure 3 an example of a Shewhart control chart is shown for moisture in grape residues.

For olive residue no outliers were detected but a suspicious trend for the first samples for all metallic elements was found. For grape residue a suspicious outliers was detected for Nitrogen according to Dixon´s outlier test, but no trend in the data was found. Finally for 6mm pellets no outliers or trends were detected except for ash content where a suspected outlier was found according to Dixon´s outlier test.

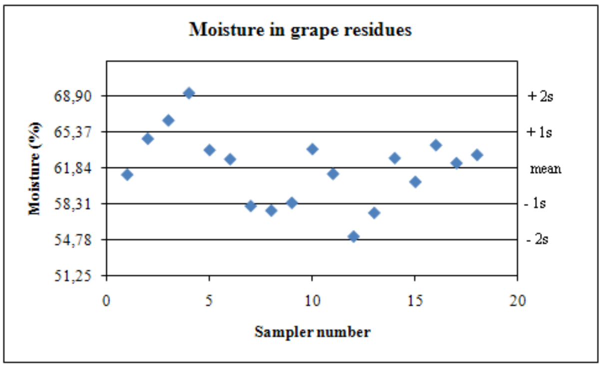


Figure 3: Shewhart control chart for moisture in grape residues.

3.4 Assessment of sample reduction methods

Table 5 presents the coefficient of variance, CV_r, for the sample reduction of the materials under study when using the coning & quartering (C&Q), the long pile (LP) and the riffle box (RB) methods.

Table 5: Evaluation of the sample reduction methods coning & quartering (C&Q), long pile (LP) and riffle box (RB) for olive and grape residues and pellet determined as coefficient of variances (CV_r). a) ash and Nitrogen content b) moisture and Potassium content c) durability.

a)

	CV_r, Ash (%)			**CV_r, Nitrogen (%)**		
Sample	**C&Q**	**LP**	**RB**	**C&Q**	**LP**	**RB**
Olive res.	34,1	37,7	22,7	39,7	38,3	40,4
Grape res.	8,1	9,1	10,0	13,3	5,4	6,9
Pellet	12,6	21,5	28,7	-	-	-

b)

	CV_r, Moisture (%)			**CV_r, Potassium (mg/kg)**		
Sample	**C&Q**	**LP**	**RB**	**C&Q**	**LP**	**RB**
Olive res.	0,79	0,30	1,55	17	16	28
Grape res.	2,3	1,2	1,1	17,1	6,8	14,6

c)

	CV_r, Durability (%)		
Sample	**C&Q**	**LP**	**RB**
Pellet	0,3	0,2	0,2

Some significant difference between the reduction methods was found for the materials and parameters, but not so heavy to prevent choosing the most practical ones.

4 DISCUSSION

4.1 Bias

No explanation has been found for the bias between heap and conveyor method when sampling Aluminium and Magnesium in olive residue, and Potassium in grape residue, even if a small increment size is more sensitive to inherent heterogeneity of the biofuel material.

4.2 Variances

An explanation to the odd behavior of grape and olive residues might be the high degree heterogeneity of these materials.

4.3 Calculation of number of increments

The minimum number of increments to be taken from a lot or a sub-lot of a biofuel material to obtain a specified precision of the analytical parameter of interest depends on the heterogeneity of that parameter in the material in combination with the design of the sampling method. Some energy companies are analysing every single sub-lot, i.e. lorry load, and uses the result for the payment of each individual sub-lot of the biofuel. Other companies are using the average of a number of sub-lots, i.e. all lorry loads during one month, as the bases for the analyses of the original lot. Still other companies are dealing with much larger lots, i.e. shipments of 30 000 tonnes, which are usually divided into sub-lots of about 2 500 tonnes from which the sample increments are collected. Below two sampling designs are described in detail together with the corresponding formula for the calculation of minimum increment number.

4.3.1 Analysis of one sub-lot (10-50 tonnes)

The sub-lot, i.e. a lorry load, is sampled by a specified number of increments. These increments are aggregated to give a laboratory sample which is sent to the analytical laboratory. This laboratory sample is then divided into a number of sub-samples, which are then analysed for the specified parameter.

The uncertainty of this analysis may be calculated by the following equation

$$e = t^* \sqrt{(\sigma^2_I/I + \sigma^2_T/T)} \qquad (4)$$

where e is the uncertainty of the analysis; σ^2_I and σ^2_T are the increment and the sub-sample variances, respectively; I is the number of increments, T is the number of sub-sample tests and t is the Student´s value for $\alpha = 0.05$ which is approximately 2.

By rearranging equation (4) the minimum number of increments needed (I) to obtain a specified precision (e) for a specified parameter is determined by the increment variance (σ^2_I) and the sub-sample variance (σ^2_T) of that parameter together with the number of sub-samples analysed (T) according to the following formula

$$I=\frac{4\sigma_I^2}{e^2-\frac{4\sigma_T^2}{T}}\text{, where} \qquad (5)$$

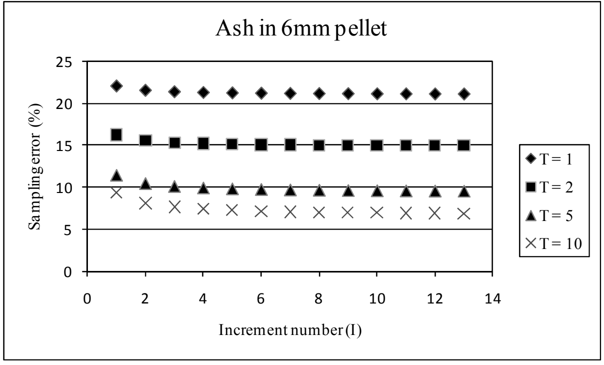
I = minimum number of increments needed for the required precision
e = required precision (twice the standard deviation) for the specified analytical parameter
σ^2_I= increment variance for the specified parameter
σ^2_T = sub-sample variance for the specified parameter
T = number of sub-sample tests for the specified parameter

The required precision, *e,* and the number of sub-samples, *T*, may be decided by the producer and the customer in agreement. The values for σ^2_I and σ^2_T may be determined by the company for the material under study or may be determined from earlier experience.

In addition, by constructing a graph where the precision, *e*, is plotted versus the number of increments, *I*, for certain values of sub-sample tests, the minimum number of increments for a certain required precision may also be determined.

To demonstrate the effect of the relation between σ^2_I and σ^2_T on the sampling error, three different situations are described in figure 4.

a)



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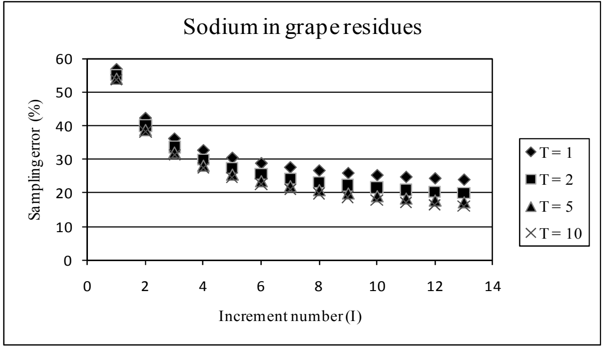

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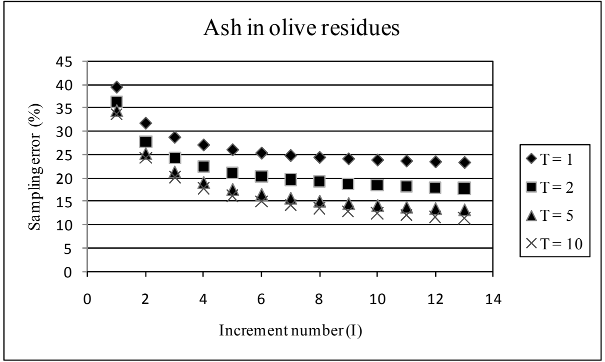

Figure 4: The effect of the relation between σ^2_I and σ^2_T on the number of increments needed to obtain a required precision. a) ash in 6mm pellets, b) Sodium in grape residues c) ash in olive residues.

As can be seen from Figure 4, the relation between the increment and the sub-sample variance is of great importance how the increment number influence the sampling error. For ash in 6mm pellets, where $\sigma^2_T >> \sigma^2_I$, the number of increments have almost no effect on the sampling error.

On the other hand, for Sodium content in grape residues where $\sigma^2_T << \sigma^2_I$ the increment number alone determine the sampling error without influence of the number of sub-sample tests.

For ash in olive residues, where σ^2_T and σ^2_I are of the same order, both the increment number and the number of sub-sample tests determine the sampling error.

4.3.2 Analysis of several sub-lots (10-50 tonnes each)

A number of sub-lots, i.e. all lorry loads during one month, are sampled by a specified number of increments per sub-lot. The increments from each sub-lot are aggregated to give a laboratory sample which is sent to the analytical laboratory. Each laboratory sample is then divided into a number of sub-samples, which are analysed for the specified parameter. The final result is obtained by averaging the results from each sub-lot which is then an estimation of the studied parameter in the original lot.

The uncertainty of this analysis may be calculated by the following equation

$$e = t^*\sqrt{(\sigma^2_N/N + \sigma^2_I/NI + \sigma^2_T/NT)} \qquad (6)$$

where e is the uncertainty of the analysis; σ^2_N, σ^2_I and σ^2_T are the sub-lot, the increment and the sub-sample variances, respectively; N is the number of sub-lots averaged, I is the number of increments, T is the number of sub-sample tests and t is the Student´s value for α = 0.05 which is approximately 2.

The minimum number of increments needed (*I*) to obtain a specified precision (*e*) for a specified parameter is determined by the sub-lot variance (σ^2_N), the increment variance (σ^2_I) and the sub-sample variance (σ^2_T) of that parameter, respectively, together with the number of sub-lots averaged (*N*) and the number of sub-samples analysed (*T*) according to the following formula

$$I=\frac{4\sigma_I^2}{Ne-4\sigma_N^2-\frac{4\sigma_T^2}{T}}$$, where

I= minimum number of increments per sub-lot needed for the required precision
N = number of sub-lots averaged
e = required precision (twice the standard deviation) for the specified analytical parameter
σ^2_N = sub-lot variance for the specified parameter
σ^2_I = increment variance for the specified parameter
σ^2_T = sub-sample variance for the specified parameter
T = number of sub-sample tests for the specified parameter

The required precision, e, the number of sub-lots, N, and the number of sub-samples, T, may be decided by the producer and the customer in agreement. The values for σ^2_N, σ^2_I and σ^2_T may be determined by the company for the material under study or may be determined from earlier experience.

In a similar way as in section 4.3.1, the minimum number of increments for a certain required precision may be determined by constructing a graph where the precision, e, is plotted versus the number of increments, I, for certain numbers of sub-sample tests.

Even in this case the relation between the individual variances determine the influence of the increment number on the sampling error. As can be seen from Table 2 for moisture content in grape residues, that the sub-lot variance is much larger than both the increment variance and the sub-sample variance. This means that the increment number and the number of sub-sample tests are of little importance for the uncertainty of the overall analytical result.

For the ash content in 6mm pellets the number of sub-sample tests are the most important factors for the sampling error.

4.4 Long time variances

Good agreement between the overall standard deviations for all parameters in the two examinations of grape residues was obtained.

For olive residues a worse agreement between the two tasks was found, where the F-test indicated that there is a significant difference for half of the parameters. The reason for this is that the two shipments derive from two different countries.

For 6mm pellet is shown that there is a large, significant difference in variation between the two investigations for all three parameters. The reason for this disagreement is that the sampling of pellet in the two examinations was performed in two different power plants using different woody materials.

4.5 Assessment of sample reduction methods

No one method of sample reduction gave a significant smaller between sub-sample standard deviation than the others for all three of the test methods. Thus for the determination of the studied parameters, the choice of sample reduction method in most cases is decided from a practical point of view, i.e. time consumption, number and size of sub-samples, nominal top size, etc.

5 CONCLUSIONS

The following conclusions can be drawn from this work:

- Bias between the sampling methods was obtained only for a few chemical parameters that are Potassium in grape residues and Aluminium and Magnesium in olive residues, and in particular with the smallest increment size.

- Olive and grape residues showed differences in variance between both increment sizes and sampling methods that in many cases were contradictory. This was true especially for the major metallic elements and in general the variability is associated to small increments.
This discrepancy was probably caused by a high heterogeneity of the materials.

- The individual sub-lot, increment and sub-sample variances estimated by the ANOVA method was used to evaluate the minimum increment number needed to obtain a specified required precision.

- Long time studies verified the individual variance values σ^2_N, σ^2_I and σ^2_T for grape residues, which would indicate that the obtained values of variation may be considered as reliable and representative for the studied material. A different situation is found for olive residues and 6mm pellet, where the different origin of material has an important role in the evaluation of different variances.

- Different precisions were obtained for the sample reduction methods coning & quartering, long pile and riffle box when dividing olive residues, grape residues and 6mm pellets. But the differences are such that the choice is decided from a practical point of view.

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3 Working Package II.1
- Test methods for physical properties -

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3.1 METHODS FOR DETERMINING IMPURITIES IN SOLID PARTICULATE BIOFUELS

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Two methods for determining impurities in solid biofuels were developed and tested (visual identification for impurities with a nominal top size above 2 mm and wet sedimentation for impurities with a nominal top size below 2 mm). The procedures aim at describing two size classes of added impurities depending on the nominal particle size of the impurities. One size class consists of impurities with a nominal top size (NTS) equal to or above 2 mm and the other size class consists of impurities with a nominal top size below 2 mm. The methods were tested on various types of solid biofuel samples (sawdust, bark and wheat straw pellets, chips of debarked Norway spruce, forest wood chips and logging residues) added with various concentrations of sand and soil with a nominal top size below 2 mm and impurities larger than 2 mm consisting of metal, stones and plastic. The method for impurities with a nominal top size above 2 mm and the wet sedimentation of impurities with a nominal top size below two mm was found suitable and by combining the two methods the complete range of all particle sizes of impurities can be detected in solid biofuels such as wood chips and hog fuel.

Keywords: Impurities, Solid biofuel, Quality characterisation

1 INTRODUCTION AND OBJECTIVES

Impurities as e.g. sand, soil, metals, plastics and similar which have been introduced in solid biofuels during the production and/or storage, is a well-known quality-reducing factor which increases the share of ash and potentially lowers the ash melting point. This can lead to fouling and slagging at over heaters and boiler surfaces. The impurities influence in various degrees the combustion behaviour depending on the fuel type, combustion technology and end user skills. First class wood pellets intended for sophisticated pellet boilers at the domestic market should not contain any impurities, whereas an increasing degree of mineral impurities will always be present in wood chips, hog fuel and particularly in solid biofuel from logging residues and clear-cuttings.

Mineral impurities in the raw material for pellet production could for instance be caused by inappropriate storage conditions of the raw material e.g. by storing the raw material of sawdust, shavings, wood chips etc. near dirt roads where fine mineral matter of soil and sand can be carried by wind into the raw biomass. Mineral impurities in the produced pellets can also be related to the production of pellets. A certain amount of fine impurities of mineral origin is difficult to avoid in wood chips. Impurities can be caused by inappropriate storage of raw wood near gravel roads, dragging of entire trees across the forest floor, producing chips from forest residues and roots, etc. Metal fragments, plastic parts and similar should under no circumstances be included in any solid biofuels. Such evidence is for example given when the level of ash content increases from approximately 0.2 % in the raw material to more than 0.5 % in the produced pellets, as reported by OHMAN ET AL [1]. The authors conclude that the increased ash content is a result of contamination with inorganic material during the drying of the sawdust, where the drying was performed by direct use of exhaust fumes from burning bark fuel with very high ash content.

Compared to fine mineral impurities like sand and soil, metal fragments, plastic parts, larger stones and similar can relatively easier be identified. The total amount of ash-forming elements, including sand and soil, can be determined by a simple measurement of the ash content of the solid biofuel. However, as there can be a considerable natural variation in the ash content in solid biofuels, the ash content will not necessarily on its own be sufficiently determining whether the ash originates from mineral contamination or just from indigenous ash-generating components in the fuel. The indigenous ash-forming components in biomass vary considerably between different types of biomass, but also within the same type of biomass [2].

The objective of the study is to conduct a comprehensive evaluation and identification of the best appropriate methods for the determination of impurities in solid biofuels in a round robin test with five European laboratories.

2 MATERIAL AND METHODS

The procedures aim at describing two size classes of added impurities depending on the nominal particle size of the impurities [3]. One size class consists of impurities with a nominal top size (NTS) equal to or above 2 mm and the other size class consists of impurities with a nominal top size below 2 mm. The first type is identified by the visual inspection and the latter by wet sedimentation. Separating the impurities using a nominal top size of 2 mm follows the procedure described for compost [4].

Sample preparation

Samples were prepared from various raw materials (see specific raw materials for each method below) with a batch of debarked Norway spruce as the only raw material applied for both tested methods. All sample size reduction was performed according to the principles described in the "Theory of Sampling" [5] using riffle boxes with minimum slot openings of 3 times the applied fuel or strip mixing with minimum 20 increments according to CEN/TS 14780 [6].

2.1 Sieving and visual inspection

Test principle

A sample is fractionated on a set of sieves with aperture size of 40 mm, 16 mm, 8 mm and 2 mm respectively, separating the sample into the four fractions > 40 mm, 16 - 40 mm, 8 – 16 mm and 2 – 8 mm and a bottom fraction < 2 mm. Non-biofuel particles in the four largest fractions are sorted out by hand and their mass is determined with 0.1 g accuracy.

The amount of impurities that can be identified visually in each of the four largest size fractions is calculated as follows, using the fraction 8 – 16 mm as example:

$I_{8\text{-}16\,\mathrm{mm}}$:
$$I_{8-16\,\mathrm{mm}} = \frac{w_{8-16\,\mathrm{mm}}}{m} \cdot 100\ \%$$

The amount of impurities is calculated either as the mass in each fraction or the total mass of impurities in the sample.

Test equipment

A set of sieves consisting of four sieves with 40 mm, 16 mm, 8 mm and 2 mm aperture size was applied for the analyses. The three larger sieves are mounted with round hole sieves and the 2 mm sieve is mounted with metal wire mesh. The four applied sieves have a diameter of 400 mm giving an effective sieve area of each sieve of 1200 cm^2. The geometry of the apertures, the thickness of the sieves, the hole distances and the opening of the holes are in accordance with the demands in ISO 3310 Part 1 and 2 [7, 8].

Biofuel test samples

Three 9 litres fuel samples were prepared from three types of solid biofuel: debarked Norway spruce, wood chips and logging residues. As the wood chips were collected from natural wood chips storages any impurities originating from the production process were avoided by sorting out the biomass particles for the test samples by hand. Subsequently, each sample was added a selection of impurities with a NTS > 2 mm consisting of stones, plastic and metal. The samples were conditioned to moisture equilibrium with the ambient temperature. The drying was conducted by convection drying on shelves in the laboratory at the Danish Technological Institute. The mass of the biofuel sample was determined with 0.1 gram accuracy and the number and mass of each impurity were determined with 0.01 gram accuracy.

The biofuel samples including the impurities were packed in plastic buckets with tight lids to secure the transhipping between the partners in the round robin.

2.2 Wet sedimentation

Test principle

A test portion of fuel chips is washed in deionised water. The detached impurities and fine particles with a NTS < 2 mm are separated through a 2 mm mesh wire sieve. The ash content of the biofuel sample and the corresponding impurity sample was determined according to CEN/TS 14775[9].

The amount of impurities is calculated as the relative difference between the ash content in the two fractions.

Test equipment

A set of two containers are required for flushing off the impurities from the fuel chips. The inner container (sample container) consists of a metal bucket with Ø 200 mm and height 272 mm. The container is equipped with a mesh wire bottom with an aperture size of 2 mm. The outer container (flushing container) consists of a metal bucket with Ø 250 mm and height 340 mm.

The impurities contained in the water of the flushing container are separated from the water fraction through an ash free filter paper. The water is evacuated using a vacuum pump connected to a

Buchner funnel with a diameter of 240 mm and a Buchner flask with a volume of 10 liters.

Biofuel test samples

6 types of biofuel samples consisting of chipped debarked Norway spruce, forest wood chips and logging residues were prepared for the round robin (Table 1). The debarked Norway spruce was added a soil slurry containing mineral particles in a concentration equivalent to 1 and 2 times the natural ash content of the debarked Norway spruce. After adding the slurry the samples were left to dry so the mineral matter attach to the surface of the wood chips particles imitating a natural occurring contamination e.g. originating from handling and dragging the wood through the forest floor.

Table 1: Samples for wet disintegration of impurities from wood chips and hog fuel.

Sample type	Impurity	Conc.
Debarked N.s.	Natural	-
Debarked N.s.	Soil slurry	×1
Debarked N.s.	Soil slurry	×2
Forest wood chips	Natural	-
Forest wood chips	Natural	-
Logging residues	Natural	-

3 METHODOLOGY

3.1 Sieving and visual inspection

Upon receipt of the test samples the material was left in the buckets with the lids off in order to equilibrate to the moisture level in the laboratory. After equilibration, but previous to the sieving and visual inspection, the mass of each sample was determined with an accuracy of 0.1 gram and then recorded. The sieving was performed manually or mechanically by means of 4 sieves with Ø400 mm with an aperture size of 45 mm, 16 mm (round holes) and 2 mm (wire mesh) respectively starting with the sieve that has the largest aperture size. The fraction below 2 mm was collected in a bottom tray. Each sample was analyzed three times.

By means of a mechanical sieving procedure the set of sieves was placed in a sieving apparatus and the sample was placed on the upper sieve. During the sieving operation the particles pass through the four sieves according to the particle size and the aperture size. The material that was retained on each sieve was quantitatively transferred to a clean, flat surface where the impurities were identified visually and collected manually by means of tweezers. The impurities of each fraction size were sorted according to origin i.e. stone, metal or plastic and the amount of particles in each group was recorded. Finally, the total weight of each type of material in each fraction was determined with an accuracy of 0.1 gram in accordance with a manual sieving procedure the sample was placed on the sieve with 45 mm aperture size which was placed on a bottom tray. The sieve was moved horizontally until no more particles were observed passing through the sieve into the bottom tray. The impurities were collected and recorded according to the above description concerning mechanical sieving.

3.2 Wet sedimentation

The inner container is placed in the outer container and places the sample in the inner container. The washing is conducted using 7 l of deionised water at ambient temperature in the flushing container. The impurities is detached by stirring/pressing the particles through the water column with a spoon for 60 seconds and after which the sample container are lifted the full way up and down through the water column 5 times to rinse down the impurities to the outer container. The washing procedure is repeated 3 times (60 seconds stirring and 5 times lifting) to completely detaching the impurities.

After the washing the inner container is lifted out and the water to remove the water from the inner container. Finally the fuel sample is removed by hands from the inner container and placed it in a drying container and the particles from the inner side of the inner container are flushed into the 'fuel sample' part. To secure that all particles are collected the procedure is finalized by gently tiping the inner container and rinsing any particles of the outer side and the bottom of the sample container into the flushing (impurity part) container (Figure 1).

Figure 1: Gently tip the inner container and rinse any particles of the bottom of the inner container into the outer container.

The drying container with the wet sample is dried in a drying oven for 24 hours at 105 ± 2°C. This part is now representing the solid *biomass* fraction of the sample. The impurities contained in the water fraction are separated from the water through a Buchner funnel and Buchner flask using vacuum. The impurities are retained using a ash free filterpaper and upon the separation the ash free filter paper with the impurities is dried 24 hours at 105 ± 2°C. This part is now representing the solid *impurity* fraction of the sample.

The ash content of the two fractions is determined according to CEN/TS 14775 [9] and based on these two results the relative impurity level can be calculated

4 RESULTS AND DISCUSSION

4.1 Sieving and visual inspection

Data evaluation

The amount of impurities that can be identified visually when studying each fraction size is calculated in the following way:

Impurities > 45 mm, $I_{>45mm}$:

$$I_{>45mm} = \frac{w_{>45mm}}{m} \cdot 100 \quad \%$$

Impurities 16 - 45 mm, $I_{16-45mm}$:

$$I_{16-45mm} = \frac{w_{16-45mm}}{m} \cdot 100 \quad \%$$

Impurities 8 - 16 mm, $I_{8-16\,mm}$:

$$I_{8-16mm} = \frac{w_{8-16mm}}{m} \cdot 100 \quad \%$$

Impurities 2 - 8 mm, $I_{2-8\,mm}$:

$$I_{2-8mm} = \frac{w_{2-8mm}}{m} \cdot 100 \quad \%$$

where:

m is the total weight of the sieved sample , in g

$w_{>45mm}$ is the total weight of non-biofuel material in the sample with a size of > 45 mm, in g

$w_{16-45mm}$ is the total weight of non-biofuel material in the sample with a size of 16 – 45 mm, in g

w_{8-16mm} is the total weight of non-biofuel material in the sample with a size of 8 – 16 mm, in g

w_{2-8mm} is the total weight of non-biofuel material in the sample with a size of 2 – 8 mm, in g

Prior to calculation of the repeatability and reproducibility a Cochran test for outliers was performed.

Repeatability

The repeatability conditions are the conditions where independent analyses are obtained using the same method, equipment and operator in the same laboratory.

The repeatability limit or repeatability (r) is the maximum value from which, with a probability of 95 %, the absolute value of the difference between two results obtained by using the repeatability conditions can be seen.

For each type of sample the repeatability is calculated using an analysis of variance with one classification criteria (here the laboratory). With the results of the ANOVA the component of variance between method and component of variance of residuals is calculated. These components are used for calculation of repeatability.

The standard deviation of repeatability (s_r^2) is equal to the square root of the error mean square:

$$s_r = s_{error} = \sqrt{MS_{error}}$$

Reproducibility

The reproducibility conditions are the conditions where independent analyses are obtained using the same method but in different laboratories and with different operators and different equipment.

The reproducibility limit or reproducibility R is the maximum value from which, with a probability of 95 %, the absolute value of the difference between two results obtained by using the reproducibility conditions can be seen.

The results of the ANOVA (see above) are also used for the calculation of the reproducibility.

The standard deviation of reproducibility is

$$s_R = \sqrt{MS_{lab} + MS_{error}}$$

Results

The visual inspection of solid impurities with an NTS > 2 mm gave consistent results between the five participating laboratories. The total mass of impurities that was added to the samples is indicated with a black line in Figure 2 – 4. Except for the forest wood chip sample that was analyzed at BLT, all laboratories reported masses of the identified impurities which were less than 1 % different from the mass of the original added impurities. A separation of the result into size classes also confirm that the method delivers consistent results because the variation of the standard deviation in the fractions is relatively moderate.

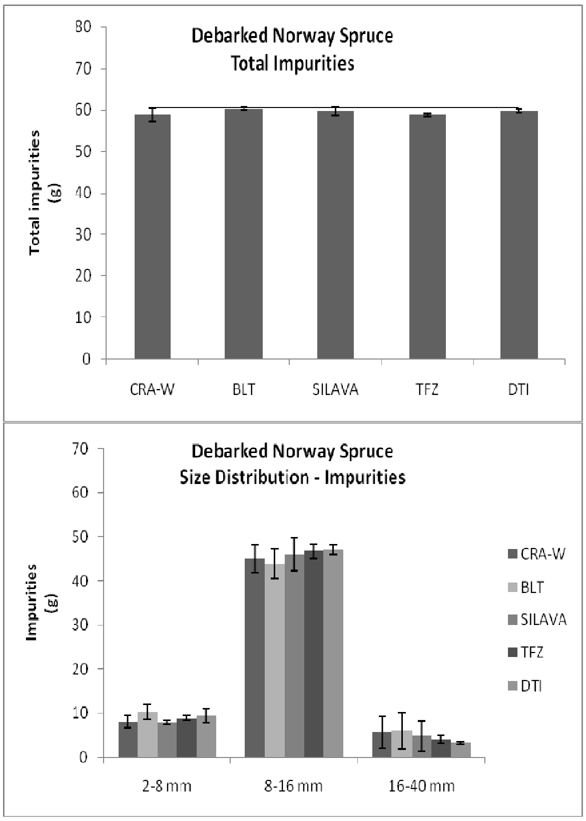

Figure 2: Total mass (g) impurities with an NTS > 2mm (g) and mass (g) of the impurities according to the size class for logging residues for Norway spruce.

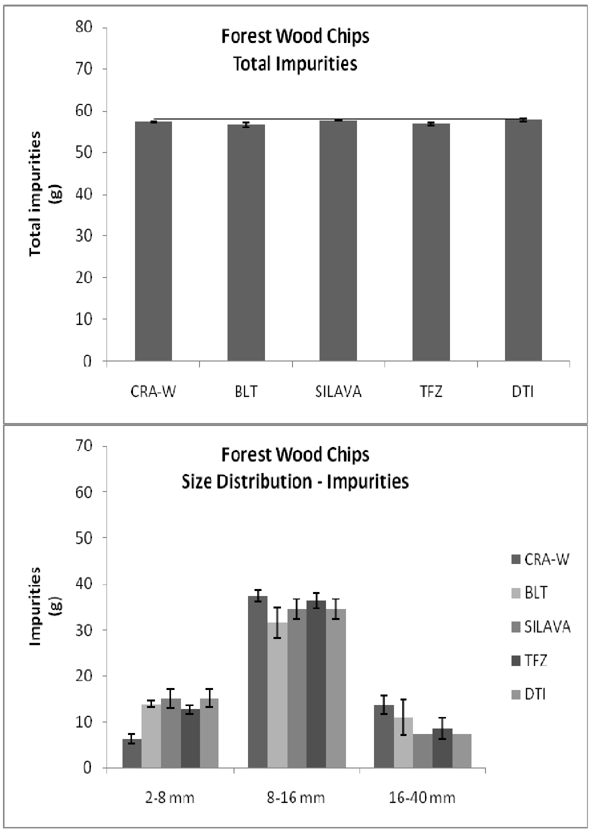

Figure 3: Total mass (g) impurities with an NTS > 2mm (g) and mass (g) of the impurities according to the size class for wood chips.

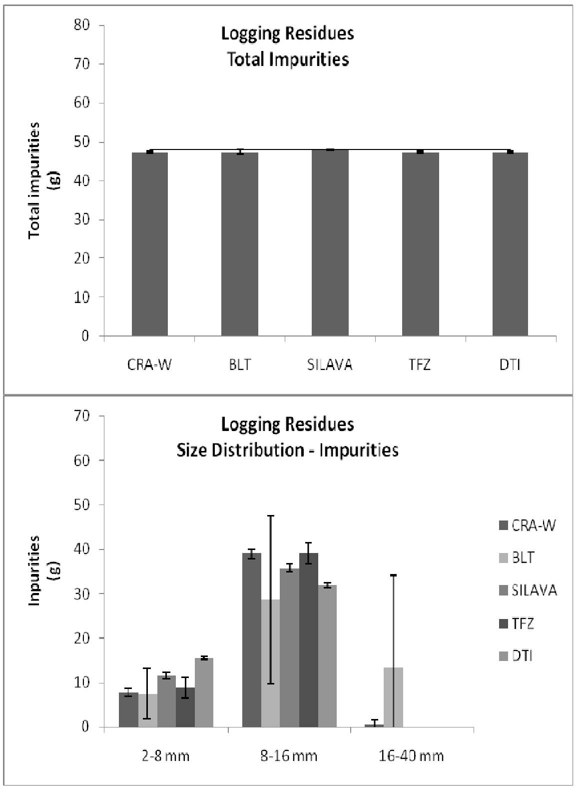

Figure 4: Total mass (g) impurities with an NTS > 2 mm (g) and mass (g) of the impurities according to the size class for logging residues.

Table 2: Repeatability and reproducibility of the tested fuels

Fuel type	**N**	**S_r (g)**	**S_r %**
Debarked Norway spruce	15	0.871	0.933
Forest wood chips	15	0.322	0.335
Logging residues	14	0.322	0.335

N Number of values
Sr Estimate of repeatability
sR Estimate of reproducibility

The repeatability was in all tests found to be lower than the reproducibility and in all cases the figures was very low, which also is indicated in the Figure 2 – 4. The highest repeatability and reproducibility was found for debarked Norway spruce, which is somewhat surprising as this material in theory should be easier to sort out impurities from than the more heterogeneous forest wood chips and logging residues (Table 2). However, as the results are very similar from all laboratories a small particle not found will have a quite high influence on the result.

The Cochran test for outliers rejected one results from the logging residues as outlier and therefore only 14 individual data is use for the calculation of repeatability and reproducibility. Due to the relatively few replications (14 and 15 for each fuel type) the calculation is not absolutely robust from a statistical point of view.

4.2 Wet sedimentation

Data evaluation

In the present, the data evaluation is based on descriptive statistics, i.e. histograms.

The intensity of impurity contamination can be calculated either as:

- the difference between the ash content in the biofuel part and the impurity part (content on the ash free filter paper) or
- the relative difference between the ash content in the biofuel part and the impurity part (content on the ash free filter paper) and the corresponding figure calculated based on the difference between the ash content in the biofuel part and the impurity part (content on the ash free filter paper) using a relevant debarked wood sample (e.g. Norway spruce) as reference material.

Results

The results from the disintegration of mineral impurities from solid biofuel in water are presented below. The six samples can be divided into two groups, each of three fuels: a) one group consisting of original fuel with the impurities they may contain from the production and b) a group consisting of debarked, chipped Norway spruce with added mineral impurities in known concentrations. The first group contained two types of forest wood chips, where Type 1 is relatively more dirty compared to Type 2, and a logging residue. The ash content in Type 1 forest chips varied moderately among laboratories but in all tests the difference between the solid biomass fraction and the mineral part is highly significant (Figure 5). It is not possible to identify whether the variation obtained in one laboratory is due to the analysis or a result of the sample reduction. For the Type 2 forest wood chips the results are consistent and as with the Type 1 the difference between the solid biomass fraction and the mineral part is highly significant (Figure 6). The logging residues contained lower amount of impurities compared to the two forest wood chips samples. The results from all five laboratories were within the same range (Figure 7).

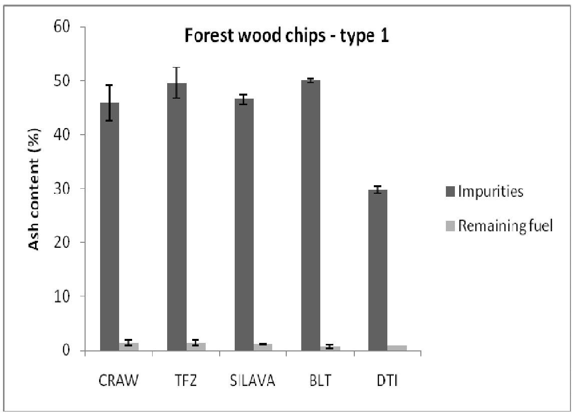

Figure 5: Ash content (%) in the solid fraction and the fraction with a NTS < 2 mm containing the mineral matter and fine biofuel parts.

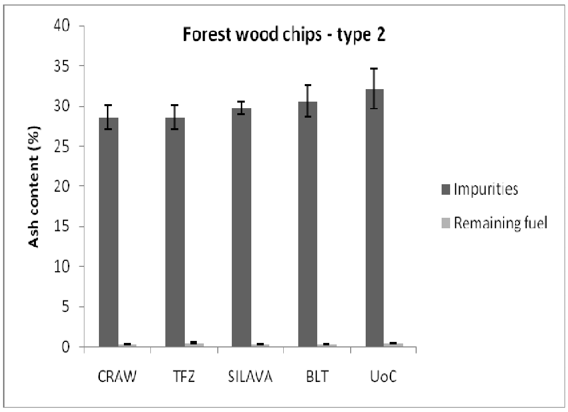

Figure 6: Ash content (%) in the solid fraction and the fraction with a NTS < 2 mm containing the mineral matter and fine biofuel parts

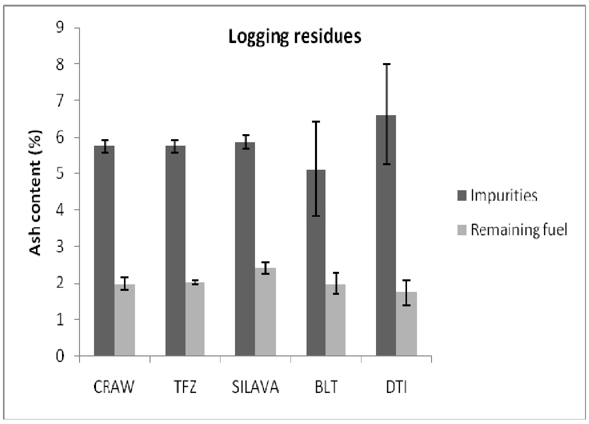

Figure 7: Ash content (%) in the solid fraction and the fraction with a NTS < 2 mm containing the mineral matter and fine biofuel parts

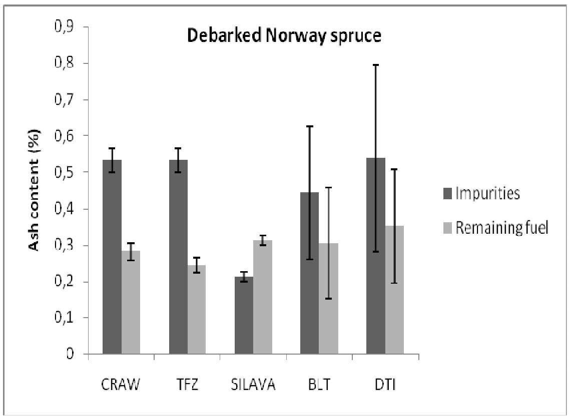

Figure 8: Ash content (%) in the solid fraction and the fraction with a NTS < 2 mm containing the mineral matter and fine biofuel parts

The second group containing the debarked, chipped Norway spruce contained very low amount of ash forming elements – as expected for this type of biofuel. All five laboratories found ash content within the same range (Figure 8).

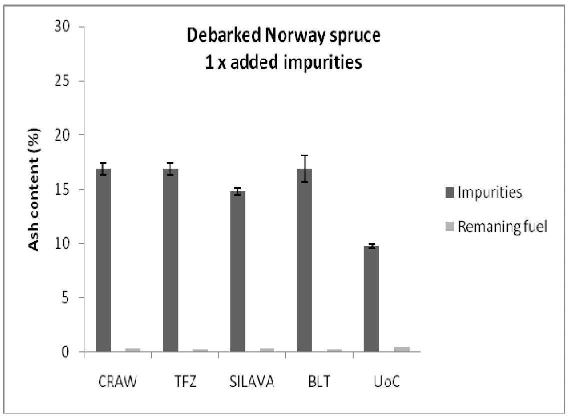

Figure 9: Ash content (%) in the solid fraction and the fraction with a NTS < 2 mm containing the mineral matter and fine biofuel parts

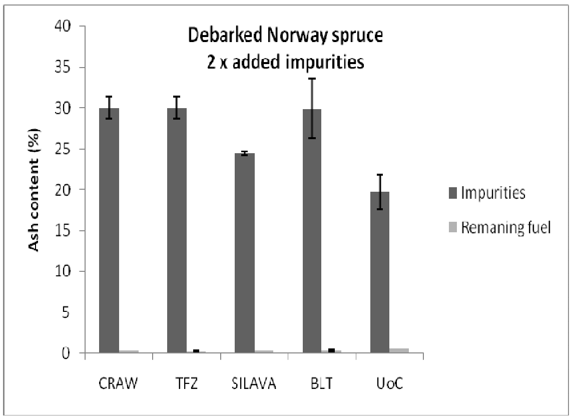

Figure 10: Ash content (%) in the solid fraction and the fraction with a NTS < 2 mm containing the mineral matter and fine biofuel parts

The two fuel types with added mineral impurities corresponding to 1 time and 2 times the original ash content of the debarked chipped Norway spruce varied moderately with respect to the measured ash content (Figure 9 and 10). It is not possible to determine whether the variation within the results is due to variation from the sample reduction procedure or the ash determination procedure. However, when the results of the analysis with the debarked Norway spruce are compared to the results of the original forest wood chips and logging residues - and disregarding the one extreme result obtained with the logging residues - it seems reasonable to conclude that the main reason to the variation within the results from the artificial fuel samples is due to problems with the sample reduction procedure.

The final calculation model for the impurity level is not definitively established, but two possible routes is suggested (Table 3). The first model calculates the fraction impurities as the ash content in the mineral divided by the ash content in solid fraction based on the figures obtained in each laboratory. This calculation gives a simple figure that makes comparisons between laboratories and fuels possible on a relative level. The second model calculates the impurity level as a fraction between result for a given fuel type divided by the same fraction for the pure wood chips from the same laboratory. By introducing this calculation a comparison to a specific fuel with a known, small influence on the ash melting behaviour is established.
Which of the two models or even other models that should be used will become evaluated during the process of transforming the present work into European standards.

5 CONCLUSIONS

The method described above for identification of coarse impurities with a NTS > 2 mm based on sieving and visual inspection of the impurities is found suitable for solid biofuel types such as wood chips and hog fuel. In the present test series the nominal top size of the smallest particles that could be identified was to 2 mm. This limit could be raised to a higher size class and also the selected sieves classes could be changed without altering the quality of results obtained with the method. However the selected aperture sizes and the minimum size of the smallest identifiable impurities reflect the results obtained by Blok and Wever [4] and the recommendations for size classes given in CEN/TS 15440 [10]. The results obtained using the method are clear and consistent and the repeatability and reproducibility is of all three tested biomasses are acceptable and the method can therefore be recommended for European standardisation.

The method for the identification of fine impurities with a NTS < 2 mm based on wet sedimentation of the impurities is found suitable for solid biofuel types such as wood chips and hog fuel. Although the results vary within fuel samples among laboratories, the differences between the solid biofuel and the fraction containing the impurities are significant and consistent and the method can therefore be recommended for European standardisation.

By combining both here recommended methods (sieving and visual determination of impurities with a nominal top size above 2 mm and wet sedimentation of mineral impurities with a nominal top size below 2 mm) the complete range of all particle sizes of impurities can be detected in solid biofuels such as wood chips and hog fuel. Thus the requirements for a comprehensive European standard procedure for determining mineral impurities can be met. The two methods are proposed for the European standardisation process.

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[8] ISO 3310-2. Test sieves – Technical requirements and testing – Part 2: Test sieves of perforated metal plate

[9] CEN/TS 14775: Solid biofuels - Method for the determination of ash content - 2004

[10] CEN/Ts 15440”Solid biofuels – Specifications and classes – 2004.

Table 3: Suggestion of two calculations modes for the ash content in the solid fraction and impurity fraction of the washed solid biofuel.

Lab	**Forest wood chips type 1**	**Forrest wood chips type 2**	**Logging residues**	**Pure wood chips**	**Debarked Norway spruce 0,5% impurities**	**Debarked Norway spruce 1,0% impurities**
Impurity level calculates as fraction of ash content in mineral fraction/ash content in solid fraction						
CRA-W	33,3	80,4	2,9	1,9	54,1	94,5
TFZ	36,0	60,1	2,8	2,2	68,1	117,2
SILAVA	39,6	80,0	2,4	0,7	46,0	69,1
BLT	67,2	89,5	2,6	1,4	74,9	15,0
DTI	33,8	73,1	3,8	1,5	20,3	37,6

Lab	**Forest wood chips type 1**	**Forrest wood chips type 2**	**Logging residues**	**Pure wood chips**	**Debarked Norway spruce 0,5% impurities**	**Debarked Norway spruce 1,0% impurities**
Impurity level calculates as fraction between figure for the fuel type / figure for pure wood chips from the same laboratory (all figures from calculation above)						
CRA-W	17,6	42,4	1,5	1,0	28,5	49,8
TFZ	16,5	27,5	1,3	1,0	31,1	53,5
SILAVA	58,3	117,6	3,6	1,0	67,6	101,5
BLT	46,6	62,1	1,8	1,0	51,9	10,4
DTI	22,1	47,7	2,5	1,0	13,3	24,5

3.2 DETERMINATION OF BRIDGING PROPERTIES - EXPERIMENTAL AND MODEL APPROACH

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A test apparatus enabling to determine the length of a fuel bridge over an opening was developed in order to describe the physical-mechanical fuel characteristic of bridging. Two such testers were applied by 6 European partners who applied a uniform test procedure on a total of 85 large biomass fuel samples of 2 m^3 volume. All samples were further analysed for angle of repose, moisture content, bulk density and image analysis parameters (e.g. particle size distribution, sphericity, length-diameter-ratio). The opening width (bridge length) of all tested fuels ranged from 0.8 cm (grain kernels) to 96 cm (unchopped grass). The mean coefficient of variation of the replications was between 5.4 % (wood pellets) and 20 % (sawdust). The angle of repose showed no satisfying correlation to the bridging measurement. But other influencing parameters for bridging were regarded in a multiple linear regression model. It was shown, that for wood samples (chips, hog fuel), the parameters mean particle size, mean sphericity and mean length-diameter-ratio are highly capable of explaining or predicting any unfavourable bridging phenomenons. In this model, there was no significant influence of bulk density in bridging.

Keywords: angle of repose, bridging test method, bulk material, image analysis, particle size distribution, physical-mechanical properties, sphericity, wood chips, wood fuels

1 INTRODUCTION AND OBJECTIVES

The building of a fuel bridge over an opening ("bridging") is a generally unfavourable phenomenon of particulate biofuels. It is particularly severe with fuels whose capability of flowing or conveying is also low. With such fuels, interruptions or failures particularly during a vertical transport can occur. This can lead to clogging of silo outlets, down pipes, funnels or screw conveyors. The bridging property of a fuel is therefore a summary characteristic for several unfavourable phenomena: Apart from the tendency to build a stable bridge over an opening, it is also synonymic with clogging hazards during conveying or with inhomogeneous horizontal distribution where this is required (e.g. in the fire bed of a boiler).

A generally applicable method for characterizing bridging as a fuel property is currently not available. Additionally, the general knowledge on influencing factors for bridging and on means for any fuel improvements in this context is still low, particularly when shape characteristics are regarded.

An objective of this project task was therefore to develop a suitable and repeatable method for determining the bridging properties of solid biofuels. This includes a reference test method and a prediction model based on the physical-mechanical fuel parameters as input variables.

2 MATERIAL AND METHODS

2.1 Bridging test principle

A test method was developed for the direct determination of the bridging properties of a biomass fuel sample. By using a special apparatus the sample is forced to form a bridge over an opening slot under controlled conditions. The opening width of the slot at the moment when the fuel bridge collapses (see bridge length *l* in Figure 3) is taken as a measure for the bridge building properties of the sample. This requires a frictionless opening of the bottom slot. It is achieved by the fact that a flat steel bottom is sliding away underneath a PVC mat on which the fuel sample is resting (see Figure 3).

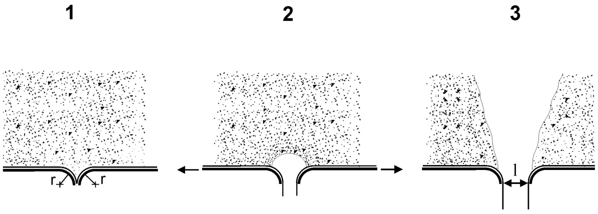

Figure 3: Functional principle of the TFZ bridging test apparatus

2.2 Bridging test apparatus

The apparatus was built with a split floor which is fully horizontally movable. Both halves of the floor are mounted on an undercarriage which is travelling on wheels. The opening mechanism ensures a fully parallel movement to both sides. It is propelled either by a crank handle or via an electric motor with adjustable speed.

The idea to the principle was based on an apparatus which had already been applied by MATTSSON [4], [5] in the early 1990ies. This apparatus had been applying a hanging bottom, sustained by steel cables, and the opening was performed via two large electrically driven rollers onto which the rubber mat bottom was rolled on. The dimensions of this previous instrument were retained in the newly built apparatus, but there were several changes: The bottom was kept completely flat, a smooth opening drive was applied and the previously large roller radius, which formed the opening slot, was replaced by a slim deflection edge of only about 26 mm radius at the opening gap (see radius *r* in Figure 3).

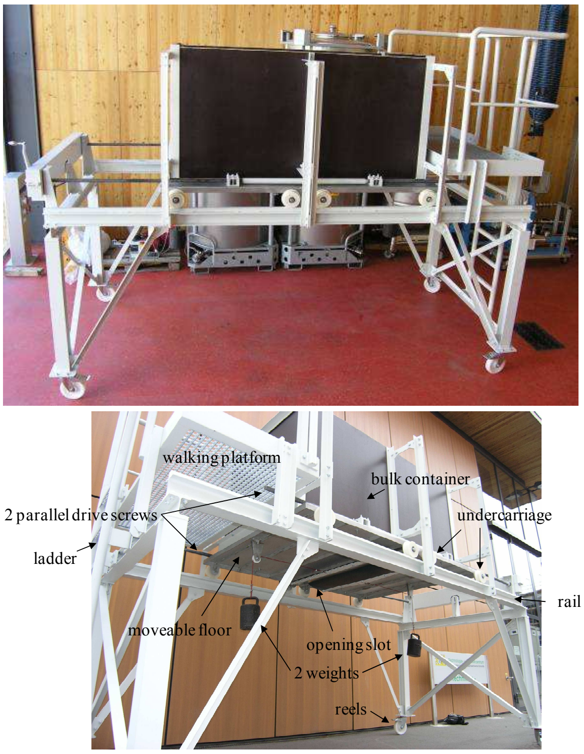

Figure 4: The TFZ bridging tester

The apparatus was built with a walking platform in order to allow a comfortable filling and levelling of the sample (Figure 4). The technical data of the bridging tester are as follows:

Inner length of bulk container: 2.0 m
Inner height of bulk container: 1.0 m
Inner width of bulk container: 1.1 m
Bulk container bottom above ground: 1.5 m
Materials of side panels: film coated plywood
Material of rope belt; PVC-mat
Opening mechanism: crank handle or motor
Opening distance per crank rotation: 12 mm
Total weight: 850 kg

The test apparatus was designed for a filling volume of 1.65 m^3, the required filling level (0.75 m) was marked on the inside walls of the bulk container. The reading of the slot opening was taken from a steel tape measure which is expanded during the slot opening process (Figure 5). Two bridging test apparatuses were built in order to ensure that all 6 project partners could perform a sufficient amount of tests within the given time schedule.

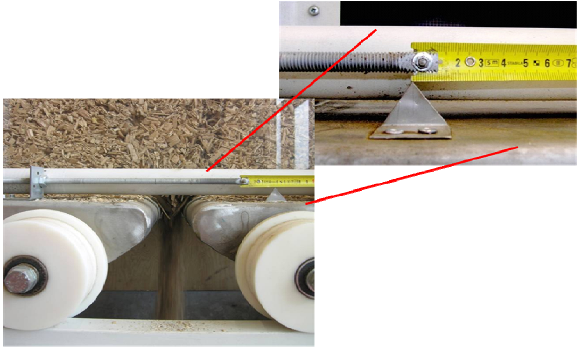

Figure 5: Measuring unit for the opening width

2.3 Procedure of a bridging test

For filling the sample into the tester, a special tilting container was modified in order to allow a hydraulically driven discharge operation of the sample into the box (Figure 6). The tilting container has a capacity of 1.7 m^3 and it was consistently used by all partners in all trials.

Figure 6: Filled tilting container mounted on a fork lifter before dropping the sample into the tester

The sample volume was fixed to 1.65 m^3 for all tests. The sample was homogenized before the tests; this was for example done by transferring the fuel by hand or by wheel loader 5 times from one place on the floor to another. The bridging tester was placed in a planar position using the installed water level. Its bottom was fully closed and it was assured that the pointer position of the measuring tape was zero. Before filling, the bulk container was completely emptied. The fuel sample was then filled into the bridging tester by using a fork lifter and the above described tilting container. This is done by filling from a height of about 140 cm above the movable floor of the bulk container without applying any compaction to the sample. The sample surface in the bulk container was then levelled by the use of a rake and the empty tilting container was placed below the slot, so that the entire falling sample could be collected again.

Then the opening procedure was started applying an opening speed of about 15 crank rotations per minute (180 mm/min). This opening speed was continued until enough material had started percolating through the slot to create a dent on the sample surface. Then it was waited until a second observing person verified that no more fuel was percolating through the opening slot. When one or several complete vertical shaft(s) in the sample layer were formed the opening width for the first measuring point ("first bridge collapse") was recorded to the nearest 1 mm (Figure 7). On a special data entry sheet it was also recorded, in which of five segments along the box width the bridge had first collapsed and whether the bridge was still in contact with the walls of the container.

The opening process was then continued until more than 50 % of the bridge width had collapsed ("OW50", Figure 8). The opening procedure finally ended at the point of a 100 %-bridge collapse (Figure 9). Sometimes one, two or all three of the measuring points were observed together and no differentiation was possible, then a uniform value was recorded for the events. This happened for example with wood pellets or grain. In the case that the bridge had fully collapsed except that a single particle was lying across the opening this position was regarded a 100 %-bridge collapse.

Figure 7: Point of first bridge collapse ("OW1") with a wood chip fuel sample

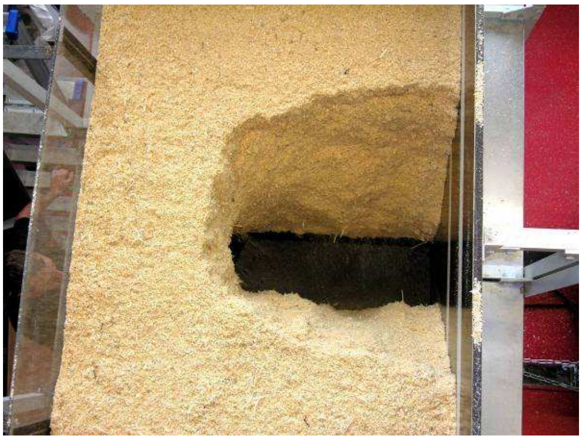

Figure 8: Point of 50 %-bridge collapse ("OW50") with a saw dust fuel sample

Figure 9: Point of 100 %-bridge collapse ("OW100") with a wood chip fuel sample

After reading the opening width at 100 %-bridge collapse point ("OW100") the whole bridging apparatus was emptied and the movable floor was closed for the next measurement. The process was then repeated 9 times and the mean value for the sample was calculated from a total of 10 measurements. An exception from this requirement was only made for tests with grain kernels and pellets; here the tests on repeatability had justified to reduce the number of measurements to five.

The series of 10 measurements can be performed in around 3 hours; this was calculated as a mean value from the time recordings of all involved partners.

2.4 Further measurements

Along with each bridging test series further fuel parameters were determined for the sample.

2.4.1 Angle of repose

After the bridging test the sample was also used for the determination of the angle of repose. This was done by percolating the sample from a height of 1 m above ground onto the floor and by measuring the height and the diameter of the cone [3] (Figure 10). The angle of repose was then calculated using Equation (1).

$$\alpha = arc\tan\frac{4*h}{D_1+D_2} \tag{1}$$

The measurement was repeated twice by forming new sample cones so that a total of three measurements were achieved per sample. The cone was formed by slowly dropping the material from a hanging big bag through an opening hose at the bottom.

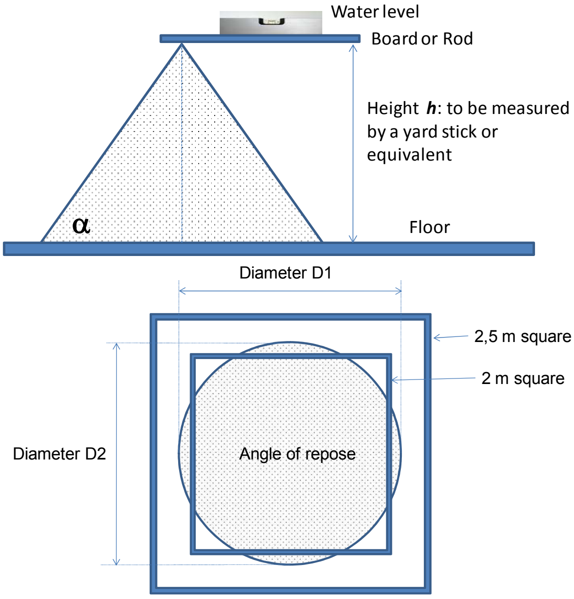

Figure 10: Method to determine the height (top image) and the diameter (bottom image) of the sample cone for calculating the angle of repose according to FEM 2581 [3]

2.4.2 Bulk density

The bulk density determination followed the European Technical Specification CEN/TS 15103 [2]. The applied uniform bulk density containers were provided by TFZ to all partners.

2.4.3 Moisture content

The moisture content determination followed the European Technical Specification CEN/TS14774-2 [1]. It was performed directly after the sampling. The mean value of three determinations was recorded.

2.5 Image analysis

2.5.1 Equipment used

For the photo-optical analysis a commercially available classifier was used ("Haver-CPA 4 Conveyor", manufactured by Haver&Boecker, Germany). In this set-up the sample is fed into a container, which serves as a feeder hopper discharging the particles to a vibrating feed canal. At the end of the canal the particles are dropped onto a conveyor belt which singularises the particles by transporting them at an approximately 22 times higher speed than on the vibrating feed canal (belt speed: 0.9 m*s^{-1}). Before the particles are falling off the end of the conveyer belt, they pass a linear light source whose light is continuously registered by a digital CCD horizontal line camera on the opposite side of the light source (see Figure 11). This camera records 4096 pixels over a width of 400 mm, thus the resolution per pixel is 98 µm. The camera processes 40 million pixels per second, whereby the matrix-equivalent resolution is conform to 24 mega pixels. When a particle passes the projection plane of the camera, the incoming light is disturbed at a width, which is proportional to the particle's momentary horizontal expansion. From the retention time within the camera's scope and the recordings for the varying horizontal expansion, the size of each particle's shadow is recorded and calculated by a computer. This calculation assumes a constant particle velocity at the measuring plane. Overlapping particles are identified as one particle of respectively larger dimension. Therefore the singularization as well as the horizontal spreading of the particles on the conveyor belt needs to be controlled carefully.

In principle there are many differences to a size classification by screening and the results are not directly comparable [6]. A major difference is that in image analysis the share of each size class is not defined by the mass of the particles but by the projection area. Consequently, the calculated fractions are the aggregated area of all particles in a size class related to the total aggregated particle area which is set as 100 % sample amount. The total area can be interpreted as volume. A determination of a particle size distribution by image analysis is thus independent from any possible inhomogeneous particle densities in the respective size fractions.

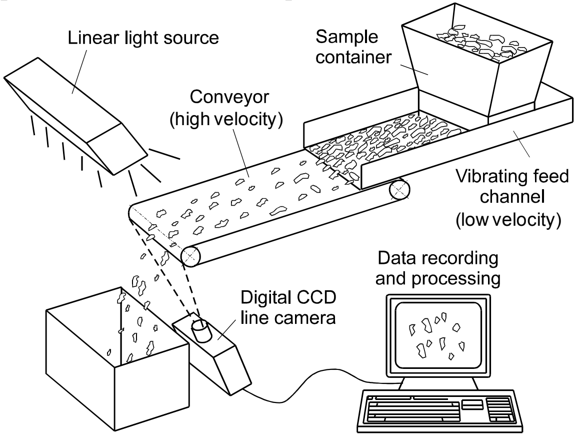

Figure 11: Functioning principle of image analysis classification method of the Haver-CPA 4 Conveyor

2.5.2 Measuring procedure

All sub-samples which had been collected by the partners in the bridging tests (between 10 and 30 litres) were divided to a final analysis volume of 1 to 3 litres, depending on the texture of the material. A full image analysis was performed over a time of 0.5 h (coarse fuels) to 4 h (saw dust). Due to higher risk of double or overlapping measuring positions the sample stream was kept low for samples with a high number of particles. No further replication was made, except for specific trials on repeatability.

2.5.3 Parameters from image analysis

From the image analysis several parameters were simultaneously recorded (dimensions marked with asterisk* are related to the measuring direction; the numbers in brackets give the position of the parameters in Figure 12):

(A) Parameters of length:
    Feret diameter* (1)
    Maximum length (4)
    Length (7)
(B) Parameters of width:
    Martin diameter* (2)
    Maximum chord* (3)
    Maximum width (5)
    Minimum Feret diameter (6)
(C) Ratio-parameters:
    Maximum length/maximum width (4/5)
    Sphericity
    Roughness

The dimensions of (A) and (B) are described in Figure 12, while the parameters of (C) are derived from the measured values of (A) and (B) or they are directly determined during the analysis.

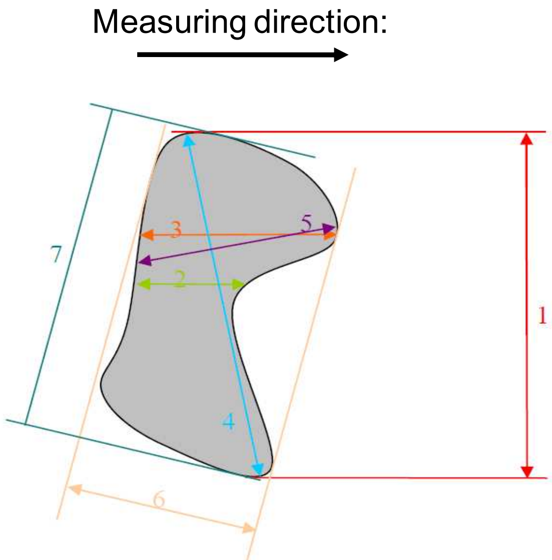

Figure 12: Directly determined size parameters by image analysis using the Haver-CPA 4 Conveyor

The sphericity is a parameter which describes the shape of a particle. Its calculation follows Equation (2).

$$\psi = \frac{C}{2 \cdot \sqrt{\pi \cdot A}} \tag{2}$$

There is

ψ Sphericity, $\psi \geq 1$

C Circumference of the projection area of the regarded particle

A Projection area of the regarded particle ("shade") in image analysis

Here, the sphericity is the measured circumference of the projection area of a particle divided by the circumference of a circle equal in size. In the case of a perfect sphere shape (round projection area) the sphericity of the particle is $\psi = 1.0$. The more a particle deviates from a round shape, the higher is the sphericity.

The roughness is a similar parameter. It characterises the surface texture and it is calculated as the sphericity divided by the length-width ratio. The length-width ratio is calculated from the maximum length and the maximum width (dimension 4 and 5 in Figure 12).

2.5.4 Calculation

Prior to starting the image analysis procedure the instrument's settings had been adjusted to perform the sorting according to useful size classes. Here the maximum length was defined as the principal sorting criteria and the length classes were chosen analogical to the R20 screen set of ISO 565 [7] and ISO 3310-1 [8], respectively (1-125 mm); furthermore some smaller size boundaries analogical to the sieves from the R20-set and also some large size boundaries were also chosen. A total of 47 classes were thus finally selected for sorting, they ranged from 0 to 300 mm.

To this principal sorting criteria all other image analysis parameters were automatically allocated by the instrument's software. This means, for example, that the sphericity was automatically recorded as a mean value for each of the 47 size classes, if applicable. In order to gain a representative mean value from all these classwise listed sphericities, the weighted mean value was then calculated *(SPHT);* this was done according to the share of fuel as determined for the principal sorting criteria, the length. These calculations were accordingly also performed for the parameters *LtoD* (mean length-to-diameter-ratio) and *RGHN* (roughness). It was done in a separate Microsoft Excel sheet in which the data set for each sample was imported.

In this spreadsheet the calculation of the mean particle size (here: length) was also made. The mean particle size ($\bar{x}_r$) of a collective is the sum of products from the shares in the single fractions and the mean fraction boundaries (e.g. the mean size of the fraction 8 to 16 mm is 12 mm). Hereby the mean size of the single fractions is weighted with their respective shares. Therefore this value is the arithmetical mean value of the particle size, as given in Equation (3).

$$\bar{x}_r = \sum_{i=1}^{n} \frac{x_i + x_{i-1}}{2} \cdot \frac{\Delta v_i}{v_{tot}} \tag{3}$$

There is

$\bar{x}_r$ Mean particle length in mm

x_i Maximum length of the fraction in mm

x_{i-1} Maximum length of the fraction of the next lower class in mm (= lower limit of x_i)

Δv_i (Area =) volume of the fraction in %
v_{tot} Total sample (area=) volume in % (=100)
n Number of fractions

According to the mean particle size, Quartile 1 (*Q1*), Quartile 3 (*Q3*), Quantile 0.9 (*Q0_9*) and the Interquartile-Distance (*IQD*) were determined. At the particle size Quartile 1 (=Quantile 0.25), 25 % of the volume is below and 75 % is above this point. Pursuant to this definition, Quartile 3 (Quantile 0.75) is this size, were 75 % of the volume is below and 25 % above. The quantiles were calculated by Equation (4):

$$Q_p = x_{uk} + \frac{n*p - F_{k-1}}{f_k} * b \qquad (4)$$

There is
x_{uk} Lower limit of the fraction, in between n*p is located
n Sample size (here 100)
p Dezimal of quantile (e.g. 0.25 or 0.75)
F_{k-1} Cumulative percentage up to the related size class (here cumulative volume = area)
f_k Frequency of the size class (here volume = area)
b Class size

The interquartile distance (*IQD*) is defined as the distance between *Q*1 and *Q*3 (Equation (5)):

$$IQD = Q_3 - Q_1 \qquad (5)$$

2.6 General procedure and test plan

2.6.1 Common guidelines

In order to ensure that all collected data from the applied fuels are comparable, common guidelines for the tests were elaborated. They extensively describe and illustrate all steps of the measuring procedure as briefly outlined in Chapter 2.3 and 2.4. The guidelines had been tested and finalised during a partner's meeting in order to avoid misunderstandings and to fine-tune the procedure.

2.6.2 Round robin tests

Two sub-samples from two different round robin fuels were shipped to the partners together with each of the two bridging testers. The usual testing routine was applied on these fuels as described in the guideline (see Chapter 2.6.1). This included the bridging tests as well as the determination of bulk density, angle of repose and moisture content. The round robin sample was then packed and delivered to the next of maximum 3 partners. The final partner returned the round robin fuels to TFZ for a repeated final determination in order to identify any material changes during the various applications.

2.6.3 Fuel test plan

Along with the two round robin fuels a number of regional fuels were selected for the practise trials in order to cover a wide range of European fuel types and to provide enough measuring experience on a typical variability of fuel characteristics. The list of applied fuels is given in Table 1. According to the fuel availability during the trials slight modifications were accepted. Each partner tested a total number of 10 to 14 (TFZ 19) fuels.

Table 1: Fuel selection for bridging tests by partners

Fuel type	TFZ	UoC	CRA	VTT	BLT	Silava
Wood:						
Round robin fuel 1	X	X	X	X	X	X
Round robin fuel 2	X	X	X	X	X	X
Wood chips, hard	X		X		X	
Wood chips, soft	X	X	X	X	X	X
Hog fuel	X	X	X	X	X	X
Sawdust	X	X	X	X	X	X
Shavings	X	X	X	X	X	X
Bark	X	X			X	X
Pellets	X	X	X	X	X	X
Short rotation coppice		X				
Wood in several moisture steps	X	X	X	X	X	X
Agricultural crops:						
Herbaceous, chopped	X		X	X	X	
Grain kernels	X					
Agricultural pellets					X	

2.6.4 Shipping of sub-samples and data submission

Each partner collected a 10 l sub-sample from each of the applied regional fuels, using the cone and quartering method. The sub-sample was air dried (below 15 % moisture) and labelled, following a duplicate labelling scheme. They were then sent to TFZ for image analysis. For data recordings from the partner's measurements a common laboratory sheet and a data reporting file were elaborated. All data files and lab sheets were sent to TFZ for final evaluation.

3 EVALUATION OF BRIDGING METHOD

3.1 Pre-tests on influencing factors

During the phase of developing and optimising the bridging method a series of pre-tests was performed by TFZ. The main conclusions from these tests are:

– Layer depth: The sample layer depth (filling height) is of significant influence. Thus it is essential to perform bridging tests with the recommended and required filling height only. Only little variation on the required sample volume is tolerable.
– Fuel moisture: A significant impact of fuel moisture content was determined. Therefore this parameter shall always be reported along with the opening width data.
– Residence time: The bridging tests shall best be conducted immediately after the filling. A time gap of a full day or more between two subsequent replications should be avoided.

3.2 Reproducibility (round robin test results)

By the application of two different standard fuels (fine and coarse wood chips) the reproducibility (variation between laboratories) was determined where each of the two bridging test apparatuses was successively applied within the group of three partners per tester. The results are given in Table 2. With the fine material the reproducibility was significantly higher as indicated by a coefficient of variation (CV) between 3.4 and 10.8 %. Here also the two apparatuses provided similar bridging results. With the coarse fuel the measuring uncertainty rose to a CV between 12.1 and 17.2%. Also the mean values of the opening width deviated between the two testers in the order of 5 to 10 cm (Table 2).

Table 2: Results from round robin tests with two bridging test apparatuses and two wood chip fuels

		Fine wood chips (RR1)		Coarse wood chips (RR2)	
		Tester1[a]	Tester2[b]	Tester1[a]	Tester2[b]
1st bridge collapse	Mean (cm)	3.7	3.8	24.2	18.5
	St.dev.	0.2	0.1	3.6	2.2
	CV (%)	4.8	3.4	14.6	12.1
>50 % bridge collapse	Mean (cm)	3.9	4.0	27.1	22.7
	St.dev.	0.2	0.3	4.1	3.9
	CV (%)	5.2	7.0	14.7	16.6
>100 % bridge collapse	Mean (cm)	4.4	4.3	37.1	26.6
	St.dev.	0.5	0.2	6.3	4.6
	CV (%)	10.8	5.3	16.5	17.2

CV: coefficient of variation between laboratories; St.dev: standard deviation; Mean: mean value of opening width, RR: round robin

[a] Bridging tester 1 (electric motor drive),

[b] Bridging tester 2 (crank handle drive)

3.3 Repeatability

Each of the mean values in Table 2 was calculated from a set of 10 measurements. This is also true for all the other tests which were performed with the fuels chosen by the partners according to the test plan in Table 1 (except for grain and pellets: only 5 replications). From each individual measurement the coefficient of variation could thus be calculated. The results from all tested fuel samples (n=85) are given as follows:

1st bridge collapse:	CV = 11.9 %
>50 % bridge collapse:	CV = 13.0 %
100 % bridge collapse:	CV = 14.1 %

Due to the fact that the measurement at the 100 % bridge collapse point ("OW100") can most clearly and unambiguously be defined, this point was chosen for all further evaluations. For practical reasons this seemed most feasible although both other measuring points had shown a slightly lower coefficient of variation. Furthermore, the 100 % bridge collapse enables a much wider measuring amplitude and in practice thus it allows the highest range of differentiation between the applied fuels.

It was also observed that some fuels are more critical in view of the repeatability: Saw dust and wood chips had proven to be the most problematic fuels in this respect. Thus they require the highest number of replications for a sound fuel characterisation (Figure 13).

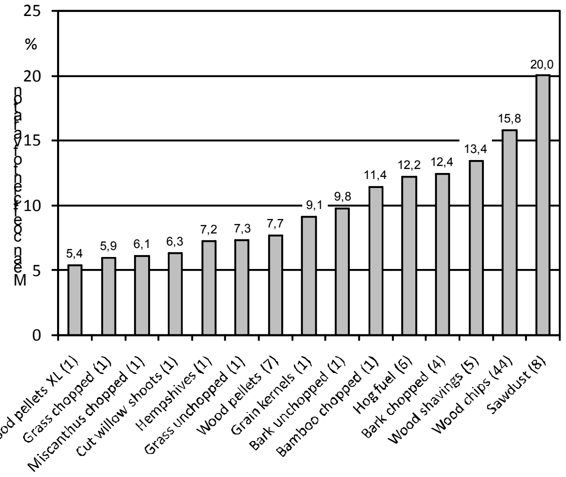

Figure 13: Ranking of the mean values of the coefficient of variation for the 100 % bridge opening width (OW100). (In brackets: number of fuels tested)

4 EVALUATION OF THE ANGLE OF REPOSE

Compared to the bridging test (Chapter 3) the angle of repose appeared to be a significantly higher repeatable test parameter. This is demonstrated by the low coefficient of variation in Table 3.

Table 3: Results from round robin tests for angle of repose using two round robin wood chip fuels

	Coarse wood chips (RR2)		Fine wood chips (RR1)	
Institute	Mean (°)	CV (%)	Mean (°)	CV (%)
1	36.0	2.2	39.4	2.6
2	37.2	1.4	37.7	4.2
3	36.9	8.5	39.2	1.4
4	33.6	7.0	36.4	6.2
5	41.0	1.4	40.2	1.2
6	39.2	2.3	39.6	1.8

However, the angle of repose proved to be only little sensitive towards variable fuel properties. Therefore a differentiation between the coarse and fine round robin fuel was hardly possible (Table 3). This observation also becomes evident when the full range of the 85 applied biomass fuels is regarded. Here the range of the measured values was between 27° and 50°, while with the same fuels the measured opening width from the bridging trials displayed a more than 4 times higher range between 1 cm and 96 cm (Figure 14).

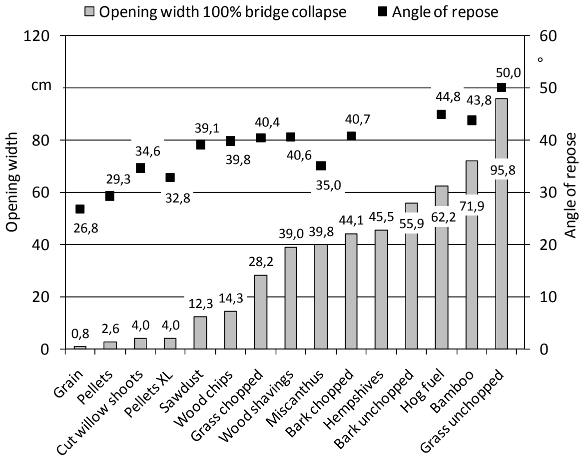

Figure 14: Angle of repose of selected biomass fuels, sorted in the order of the bridging measurements (OW100)

It was also found, that the correlation between the two parameters is rather poor, as demonstrated by the low regression coefficient (R^2) of only 0.42 in Figure 15. Therefore it can be concluded, that the determination of the angle of repose can provide only little estimate on the expected bridging hazards with biomass fuels. Furthermore, the repeatability of these measurements was also low, the mean coefficient of variation as calculated from 79 tested fuels was 2.6 % (3 replications per fuel).

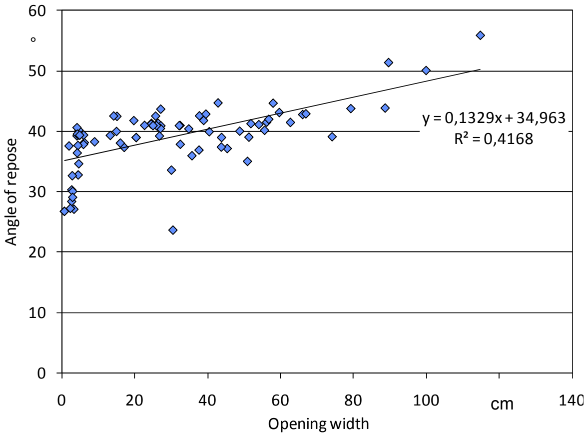

Figure 15: Correlation between angle of repose and bridging results (OW100)

5 CHARACTERISATION OF TYPICAL FUELS

Due to the fact that a large number of conventional and unconventional European biomass fuels had been selected for the tests, a fuel inventory concerning the here regarded physical-mechanical parameters was created. Table 4 shows the compilation of the determined fuel properties for 9 selected fuel types.

Table 4: Overview of typical physical mechanical fuel properties in the fuel selection

	Fine wood chips (RR1)	Coarse wood chips (RR2)	Hog fuel	Sawdust	Shavings	Grain	Bark chopped	Bark un-chopped	Wood pellets
Parameter*									
SPHT	1.51	1.73	4.18	2.09	1.50	1.12	2.67	2.79	1.24
RGHN	0.68	0.56	1.39	0.58	0.74	0.61	0.87	0.90	0.56
LtoD	3.0	4.9	7.0	6.1	2.5	1.9	4.6	5.6	2.7
MPS (mm)	21.5	67.0	125	18.0	8.2	6.6	31.5	42.8	18.1
IQD (mm)	14.1	21.8	80.0	18.4	5.9	0.8	28.1	38.8	6.5
BD_{15} (kg/m³)	214	195	149	153	66	856	209	306	773
OW100 (cm)	4.4	35.8	114.8	24.7	51.9	0.8	39.0	67.7	2.8

* SPHT=Sphericity, RGHN=Roughness, LtoD=Length/Diameter-Ratio, MPS=Mean Particle Size, IQD=Interquartile distance, BD_{15}=Bulk density at 15 % MC, MC=Moisture content, OW100= Opening width of bridging tester at 100 % bridge collapse

6 MODEL FOR BRIDGING PROPERTIES

6.1 Procedure

In order to allow an evaluation of the impact of other parallel determined fuel characteristics on bridging properties a multi-linear regression model was generated. This was done by the use of the statistical software package SAS 9.1.3. The following SAS-procedures were applied: REG/Selection= Backward, MaxR, Stepwise, Forward and AdjRsq. The primary goal of the model approach was to achieve a high adjusted regression coefficient (R^2_{adj}) instead of the “simple/normal” R^2. This was done to avoid that any useless input variables of minor importance would be considered. The adjusted R^2 would in such case be increasing as an effect of the lower degree of freedom. The level of error probability was set to $\alpha = 0.05$. Finally also a non-linear approach was considered and tested, but the achieved improvements in the adjusted R^2 were low and this strategy was thus not pursued any further.

In the model the following input variables were finally regarded:

- Bulk density, *BD*, related to 15 % moisture content (in kg/m³)
- Moisture content, *MC* (in %, wet base) (Due to the results of the pre-tests the *MC* was also regarded as an exponential influence, *MC²*)
- Mean particle size, *MPS*, calculated from the maximum length (mm)
- Mean length-diameter-ratio, *LtoD,* calculated from the max length and max width of each particle (dimensionless)
- Sphericity, *SPHT* (dimensionless)
- Roughness, *RGHN* (dimensionless)
- Interquartile distance, *IQD* (mm)

For the above input variable all possible pair wise interactions were also considered. In all regressions it was also decided not to force the regression through zero in order to achieve a better data prediction. Therefore, there is a possibility that for the opening width slightly negative model results are possible. This can however only happen with fuels where the flowability and bridging is anyway uncritical.

6.2 Selection of input data sets

For the modelling the collected data base was additionally sorted. Saw dust was eliminated from all further calculations, because the image analysis did not allow to determine the most important fuel characteristics at a sufficiently high accuracy (too many fine particles). The remaining data set (n=76) was then applied for the calculations (“all biomass fuels”). Additionally, all data for chipped or crushed wood were also selected as a second data set (n=51). Bark, wood pellets and again saw dust were not included in this set, but hog fuel remained in this group, which is subsequently denoted as “wood chips”.

6.3 Model for limited data set (“wood chips”)

With the limited data set (wood fuels, see Chapter 6.2) a first model was created. In the initial approach a linear regression without interactions was applied. It was found, that the influence of sphericity (*SPHT*), length-diameter-ratio (*LtoD*), mean particle size (*MPS*) and moisture content (*MC*) on the opening width (*OW100*) was the highest among all tested parameters; except for SPHT they were all significant at the 5 % error level (Equation (6)). SPHT was still regarded in the model because the mean error was significantly lowered and this parameter was also important in the model for “all fuels” (Chapter 6.5). In this model an adjusted regression coefficient (R^2_{adj}) of 0.85 is achieved. Other parameters (bulk density, interquartile distance, roughness, MC²) were not significant at the 5 % error level. As they only lower the adjusted regression coefficient they were eliminated from the model.

$$
\begin{aligned} OW100 = &-31.648 + 5.292 * SPHT + 3.0005 * LtoD \\ &+ 0.5005 * MPS + 0.5274 * MC \end{aligned} \tag{6}
$$

However, it could be assumed that a presence of interactions between the regarded parameters is very likely in such a complex system of causes and effects. Therefore the linear model was further developed to include any significant pair wise interactions. This is described in Equation (7).

$$
\begin{aligned} OW100 = &-4.5251 + 7.0553 * SPHT + 0.7031 * MPS \\ &- 0.2832 * MC - 6.4037 * LtoD + 0.2427 \\ &* (MC * LtoD) - 0.0058 * (MC * MPS) \end{aligned} \tag{7}
$$

With this model for wood chips an R^2_{adj} of 0.88 is achieved. The mean error between measured and predicted OW100-measurement was 32 %; it is demonstrated in Figure 16. However, this error of 32 % has to be interpreted in view of the already given reproducibility of a bridging measurement. For the round robin fuel 2 (RR2), for example, the coefficient of variation for the OW100 measurement was around 18 % (Table 2). The RR2-fuel is here best suitable for comparison as it had an OW100-value which was close to the average for all other wood fuel samples applied.

The model error of 32 % is therefore only by 14 percentage-points higher than the variation in the round robin. This suggests that a relatively high explanation of all variation is given by the model.

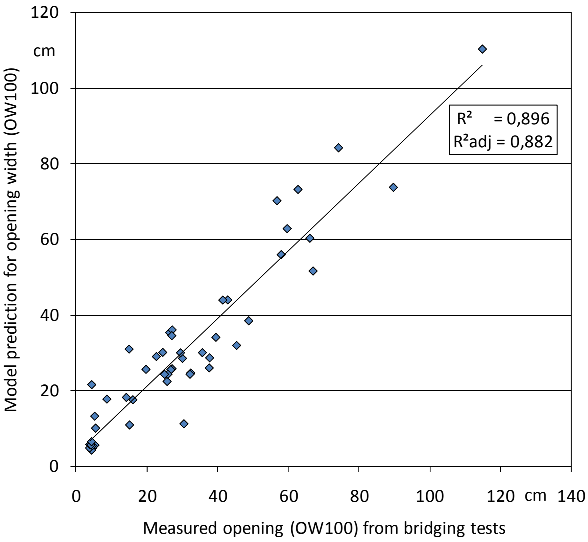

Figure 16: Model for bridging of wood fuels (n=51): Correlation between measured and predicted values (OW100) following Equation (7)

6.4 Sensitivity analysis for the wood chip model

In order to evaluate the influence of each of the parameters a sensitivity analysis was performed using the mean value of parameter over the full data set as basis. This mean value was varied up and down to gain a new data input to the data model (Equation 7) while all other parameters were kept constant at their mean values. This procedure was repeated with the other three parameters. The results are given in Figure 17.

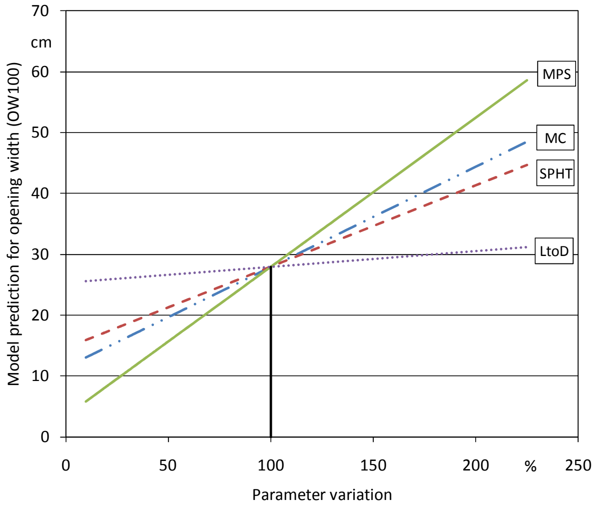

Figure 17: Sensitivity analysis for the wood chip model following Equation (7) (n=51)

It can be seen that the mean particle size has the highest influence on the bridging tendency (Figure 17). A doubling of the mean particle size would cause a nearly twice as high opening width. In order to reduce the bridging tendency of wood chips it is therefore most effective to perform a more intensive comminution in order to reduce the mean particle size. This can also be enhanced by the parameters sphericity and length-diameter-ratio: The lower the sphericity of a particle is, the lower is the predicted opening width. Therefore the application of sharp cutting knifes is important for chipping.

Following the *MPS*, the moisture content has the second highest influence on the opening width. Consequently the flowability properties of a fuel can be improved by drying.

The lowest influence on the opening width (using wood chips) here was discovered for the length-diameter-ratio. Nevertheless a higher length-diameter-ratio will lead to a higher predicted opening width and thus increase the bridging tendency.

6.5 Model for full data set ("all biomass fuels")

With the introduction of other fuel types than only natural wood new sources of variation are becoming effective in modelling. This becomes obvious when Equation (8) is regarded. The mean prediction error is here 89.4 % (75.9 % if all negative values are set to zero). However, this is due to a substantial number of measurements with fuels that showed an extremely low bridging tendency (such as for grain kernels: (OW100 = 0.81 cm) or wood pellets (OW100 = 2.8 cm). The accuracy is then quite low, because the nominal error of few centimetres can here lead to significant relative deviations, which strongly influence the mean error of the calculation for all fuels.

Figure 18 also shows that the model can here predict negative bridging values, but the relevance of such measurements is generally low when uncritical fuels are regarded.

$$\begin{aligned} OW100 = {} & -30.762 + 0.0728 * BD - 8.1106 * LtoD \\ & + 59.064 * SPHT - 0.5819 * MPS + 0.0049 \\ & * MC^2 + 0.0016 * (BC * MPS) - 0.0948 \\ & * (BD * SPHT) - 0.3058 * (MPS * SPHT) \\ & + 0.2124 * (MPS * LtoD) \end{aligned} \tag{8}$$

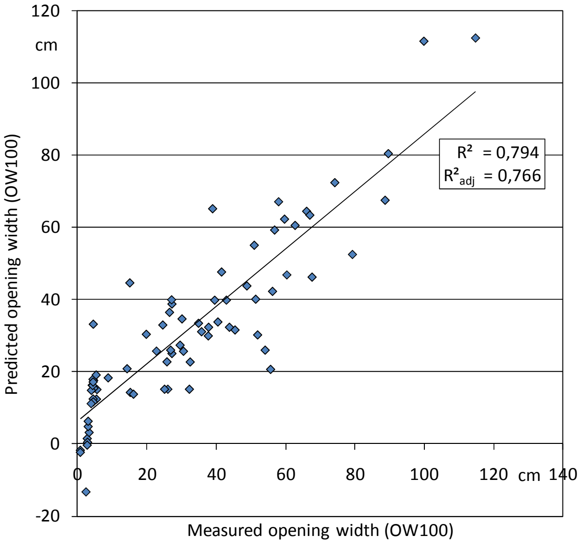

Figure 18: Model for bridging of all tested fuels (except sawdust) (n=76): Correlation between measured and predicted values (OW100) following Equation (8)

6.6 Sensitivity analysis for "all-biomass"-model

As described in Chapter 6.4, a sensitivity analysis was also performed for the "all biomass" model. The results are given in Figure 19.

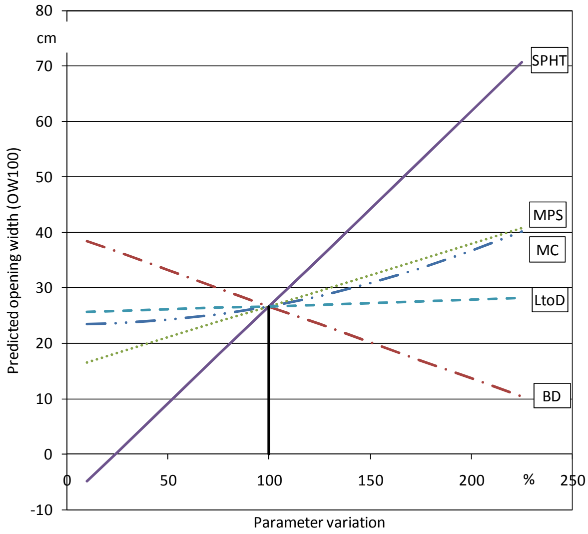

Figure 19: Sensitivity analysis of the model for "all biomass fuels" following Equation (8) (n=76)

In this model the highest influence on the opening width was given by the sphericity of the fuel particles. High sphericities lead to a high bridging tendency. As shown in Figure 19, a doubling of the sphericity will lead to a 2.5 times higher opening width.

The next important parameter is the mean particle size, which had already proved to be highly relevant in the "wood chips" model (Chapter 6.4). Further importance is also given for the moisture content. In this case the there is even an exponential impact, which means, that it becomes increasingly important at higher MC levels.

The results in Figure 19 suggest that the length- diameter-ratio (*LtoD*) is of relatively lower importance, but it should be marked that it strongly correlates with the sphericity (coefficient of correlation: 0.65), which here represents the most important influence on the bridging tendency. Therefore the length-diameter-ratio is a factor which should not be ignored.

In the model for "all biomass" an influence of the bulk density is also shown. The higher the bulk density is, the more it lowers the bridging tendency. This finding is strongly influenced by the tests with high density fuels (e.g. grain kernels or wood pellets), here the opening width was between 0.8 cm and 2.6 cm. The positive influence of pelleting (e.g. for chopped Miscanthus) therefore becomes obvious.

7 CONCLUSIONS

The suitability of the bridging test method for the determination of the flowing and bridging properties for solid biofuels is generally given. The here described technical apparatus and the developed test procedure can therefore be recommended for European standardisation.

However, due to the high complexity of the method it is desirable, that the bridging tester shall in the long run be replaced by an image analysis method. The parameters from image analysis (mean sphericity, mean length-diameter ratio and mean particle size) have proven to be highly capable of explaining or predicting any unfavourable bridging phenomenons. The relatively fast determination and the required low sample volume make this approach strongly favourable.

Other parameters such as the angle of repose cannot be seen as an alternative to indicate any bridging hazards.

8 REFERENCES

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9 ACKNOWLEDGEMENT

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4 Working Package II.2

- Test methods for chemical properties -

Coordinated by: Martin Englisch, ofi

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4.1 EVALUATION, INFLUENCES AND IMPROVEMENT OF CEN-TS FOR CHEMICAL TEST METHODS FOR UPGRADE TO EUROPEAN STANDARDS.

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ABSTRACT: In Bionorm II, WP2, Task II.2 "Test methods for chemical properties" work has been done on improvement and validation of the technical specifications from CEN TC335 WG5 (Chemical test methods). Thanks to this work the upgrade of the technical specifications to European standards could proceed. A number of technical specifications were almost ready for the upgrade, as was demonstrated in the ruggedness tests performed in the BIONORM I program. In some cases extra validation work had to be done. Results of this work are presented in this chapter. Finally the performance of all draft standards were demonstrated in a round robin. These results are presented elsewhere in this work package report.

Keywords: Chemical test methods, performance characteristics, ruggedness tests.

1 INTRODUCTION

For the upgrade of the technical specifications that were produced in CEN TC335WG5, extra validation work and determination of the performance characteristics was necessary. In this chapter the validation studies are presented. Work was carried out by all partners of this work package and reported together. For each standard the validation studies, including a summary of the most relevant obtained results, are presented in the next paragraphs.

2 WORK ON THE STANDARDS

2.1 EN 15104: CHN determination

For this standard the technical specification only needed an upgrade in terms of minor technical changes. One of the findings of the preceding Bionorm I program on nitrogen determination was confirmed in Bionorm II. For low nitrogen concentrations certain types of element analyzers give less reliable results as the measurements are taking place near the detection limit of these instruments. In general the analyzers working with higher sample intakes give the best results in terms of reproducibility. The so-called Kjeldahl analysis for low level nitrogen concentrations is a good alternative.

2.1.1 Work done: Influence water content on C and H

Dependent on the chemistry involved in the measurement of C and H, effects of the amount of water present in the sample were observed in some cases. With high water contents the hydrogen determination of some instruments was deviating from results in dried samples. In freshly dried samples some analyzers deviated as well for the carbon content. The recommendation resulting from these findings is that analysis should be done on dried lab samples (105 °C) that are in equilibrium with the lab air humidity, usually ending up with 2-5% water content.

2.2 EN 15290: Major elements

For this standard the technical specification only needed an upgrade in terms of minor technical changes.

2.2.1 Comparison with CEN 343 prEN 15410 : Solid recovered fuels - Method for the determination of the content of major elements

The performance of this method was compared with the method that is used for solid recovered fuels. The only difference of importance between these methods is the use of hydrogen peroxide in EN 15290 instead of perchloric acid in prEN 15410. For the comparison our validation samples were used. (Wood with bark, straw, bark, rapestraw, cyanara, fir without bark, orujillo, wood with glue, seaweed, coconut shells, almond kernels, and palmpit kernels)

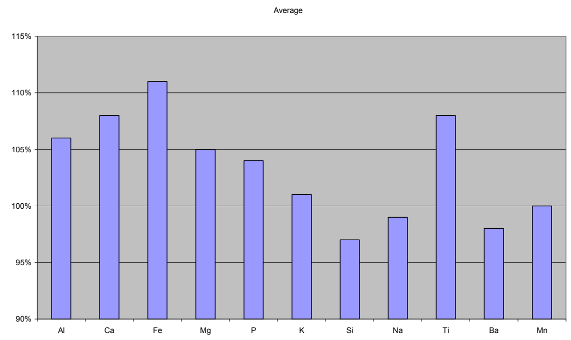


Figure 5: prEN 15290 compared with prEN15410 (expressed in terms of recovery)

As the comparison results are obtained from biomass samples that cover the whole range of concentrations that usually are found the results are correlating very well.

2.3 EN 15297: Minor elements

For this standard the technical specification only needed an upgrade in terms of minor technical changes.

2.3.1 Comparison with prEN 15411 Solid recovered fuels - Methods for the determination of the content of trace elements.

The performance of this method was also compared with the method that is used for solid recovered fuels. And also in this method the only difference of importance between these methods is the use of hydrogen peroxide in EN 15297 instead of perchloric acid in prEN 15411. For this comparison our validation samples were used as well. (Wood with bark, straw, bark, rapestraw, cyanara, fir without bark, orujillo, wood with glue, seaweed, coconut shells, almond kernels, and palmpit kernels)

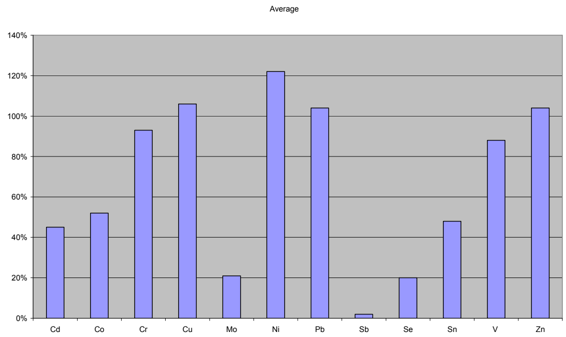


Figure 2: prEN 15297 compared with prEN15411 (expressed in terms of recovery)

In general the agreement is acceptable for these low levels. For a number of elements all results were around the detection limit, there large differences are observed, but as the perchloric acid an hydrogen peroxide is only needed to remove the organic matrix during digestion, we expect the agreement to be good for all elements when they are measured in the working range of the instruments, similar to the results obtained for the major elements.

2.3.2 Milling and sieving tests

The Bionorm II biofuel samples were analyzed with a grain size < 1 mm as delivered to the laboratories and as specified in the CEN/TS (Technical Specification) for major and minor elements. For some of the more special biofuels bad repeatability was observed compared to normal results. Therefore milling the samples to < 0.25 mm using a centrifugal mill was performed in order to get more homogenous sample material for digestion resulting in expected better repeatability. The problematic biofuels were: Olive residue, Coconut shells, Almond kernels, Palm bit kernels and Seaweed. It was observed that milling of the samples was difficult. Almond kernels and Palm bit kernels were the most problematic samples. The mill became very, very hot – even if the dosing of sample was very, very low. The following trace metal analyzes gave very amazing results, with significant higher values for Cr, Ni and Cu, than in the < 1 mm samples although the mill did not contain these elements. Also for other elements the results were higher in the milled samples. Up till now no explanation for this phenomenon was found. Suppliers of milling equipment are informed and asked for a possible explanation and work-out.

2.4 EN 15289: Cl and S

The results from investigations and robustness tests performed in the Bionorm I program indicated that more investigations were needed. The most practical method (oxygen bomb combustion followed by ion chromatography determination) gave low recovery results in the higher region, especially in combination with high ash content. Therefore several investigations on improvements were done.

2.4.1 Work on bomb combustion

Influence of oxygen pressure

Experiments with combustion in different gas mixtures were performed. Investigations in Bionorm I indicated slightly higher contents of chlorine and sulphur in Cynara, when using 10 bar in the bomb instead of 30 bar. Experiments for Cynara with 100 %, 50% and 33% oxygen, and for Orujillo with 100% and 33% were performed.

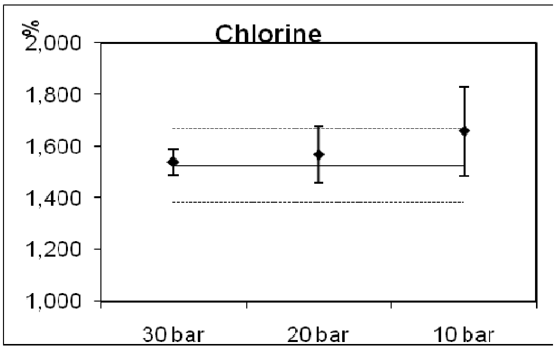


Figure 3: Chlorine results at different oxygen pressures

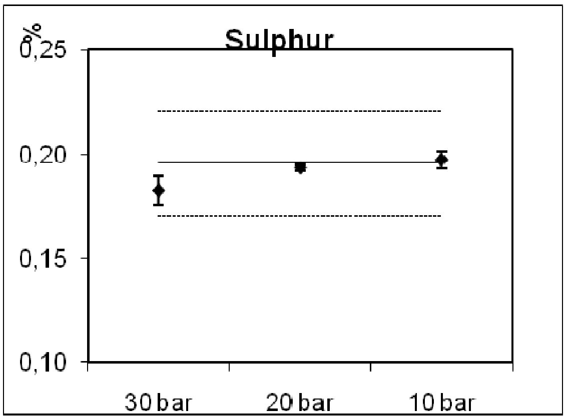


Figure 4: Sulphur results at different oxygen pressures

Oxygen/Argon mixtures of 33%/66% and 50%/50%. 100 % oxygen at 10 bar, and 33 % oxygen at 35 bar seem to give a little higher results for Cl and S, but no differences in calorific values. From these results it can be concluded that lower oxygen concentrations will lead to higher recoveries.

For an explanation it was speculated, that lower oxygen partial pressure results in lower combustion temperatures and less sintering or melting of the combustion residues trapping some of the chloride (see below).

In close relation to this investigation, dependency of sample intake in relation to recovery was performed. Experiments with smaller sample intakes, using polymer combustion bags, revealed that at lower sample intakes (0.2 g instead of 1 g) recoveries near 100% were obtained.

A possible explanation for these results is again the reduced possibility of forming larger slag agglomerates trapping chlorine.

Losses in released gases after combustion
The gas phase after combustion was investigated in order to assess the losses during pressure release after bomb combustion.
Description of experiment:
Samples: Cynara (ash=10.7%) and seaweed (ash=34.4%).
Process: filtration of exhaust vent using a filter and a bubbler containing ultrapure water. Two positions of the filter: A and B (see picture).
Filter: mixed cellulose esters (Millipore SMWP, 5 microns).
Analyses: filter weight, SEM-EDX of filter, and Cl and S of bomb and bubbler solutions.

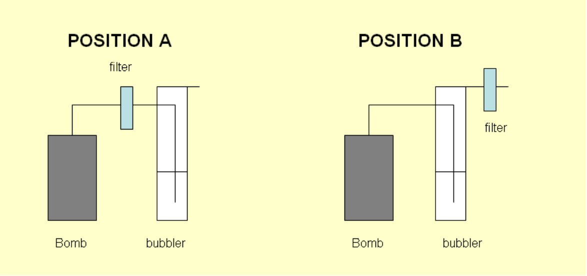
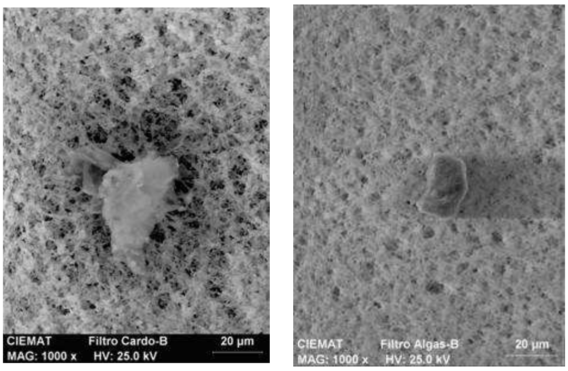


Figure 5: Experimental setup

The recovered chlorine and sulphate in the bubbler was negligible. Thus, the dissolution of hydrochloric acid and sulphuroxides in the aqueous phase in the bomb is quantitative.

On the filters only some traces of deposit were found that were carried out of the bomb with the gas stream (aerosols).



Figures 6 and 7: SEM recordings of deposits

EDX analysis of the deposits revealed the presence of mainly Na, Al, Si, Cl, Ca, S, P, Mg and K. The size of particles is higher in seaweed than in cynara. Conclusions: The filter recovery of particles, which come from the exhaust vent of bomb, is considered negligible. The percentage of chlorine and sulphur recovered in the bubbler is negligible compared with the recovery in the bombs

Investigation of remains inside oxygen bomb
After combustion of the problematic Cyanara sample, with high chlorine content and high ash content, the remainings after combustion in the oxygen bomb were collected and analyzed with IC after destruction with NaOH fusion or SEM analysis of the ash in combination with spectral chlorine determination. In the remaining ashes almost all missing chlorine was found , and the SEM recording showed that chlorine was present in the ash, mostly concentrated in some spots.

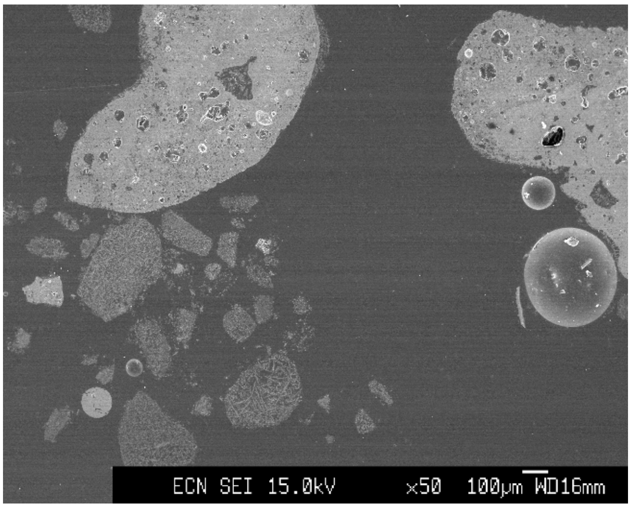


Figure 8: SEM picture of ash remaining, some spots with high chlorine contents.

Conclusion: A modification of the draft standard is recommended for fuels with high chlorine content and either high ash content or were ash sintering is observed in bomb combustion. In these cases, the combustion residues must be analyzed for chlorine and sulfur. Alternatively, other methods may be used (e.g. EN 15105 for Cl and EN 15290 for S)

Determination of Sulphur in biofuel by isotope dilution.

Isotope dilution is an analytical technique that is known for its high accuracy and precision, and is used for certification purposes. For 2 bionorm samples this technique was applied, in order to get an impression of the performance of the standard method.

Experimental:

Combustion: About 1 g of undried biofuels were dissolved by combustion in a calorimetric bomb according to CEN/TS 15289:2006 and diluted to 200 ml. The blank from digestion was subtracted from the result.

Isotope dilution and sulphur measurement: Isotopic spike was added to the digested sample and measurements were performed with a high resolution ICP-MS instrument *Element2* in medium resolution. The differences in sensitivity for different masses were calibrated with a mass bias solution prepared by mixing the isotope spike with natural sodium sulphate. The samples were run in the following order: acid-blank, mass bias, mass bias, acid-blank, sample, sample, acid-blank, mass bias, mass bias, acid-blank, acid-blank. After a preflush of 2 min the ion intensities of mass 30 and 34 were measured for a period of 3 minutes. The average value of mass bias and sample was used for the concentration calculations. The value of the acid-blank, determined before and after the measurement was subtracted before ratio calculations. The result were corrected for dry mass basis (105 °C, 2 h)

Table 1: Results for S in % in the biofuels dry basis (105 °C, 2 h)

	S %	U, K=2 %
Rape straw	0,188	±0,007
Sea weed	1,87	±0,04

The sulphur results for biofuel Rape straw is 0,188 % ± 0.004 % and for Sea weed 1,87 % ± 0.04 % is found. After combustion of the sample in a calorimetric bomb the measurements were performed using isotope dilution with inductively coupled plasma mass spectrometry (ICP-MS). The calibration is based on an isotope spike of ^{34}S - IRMM646. These results are however slightly lower than the results of the ICP methods (0.195 for Rape straw and 2.05 for sea weed). Losses during oxygen bomb combustion might explain these differences.

Determination of Bromine in Fuels

In order to evaluate the applicability of ion chromatography (IC) for the determination of bromine in solid fuels, experiments were made where booth ICP-MS and IC was used for the determination of the bromine concentration in the solution from the bomb combustion. A summary of the results follows below. Since the concentration of bromine is very low in most biofuels solid recovered fuels were used in these experiments. The best results were obtained when samples were combusted in a calorimetric bomb with 5 ml 1M NaOH as the receiving liquid. This was then diluted to 1 l and analyzed with inductively coupled plasma mass spectrometry (ICP-MS) and IC.

Two groups of samples in different concentration ranges were analyzed using these conditions, the results are shown in Figure 9.

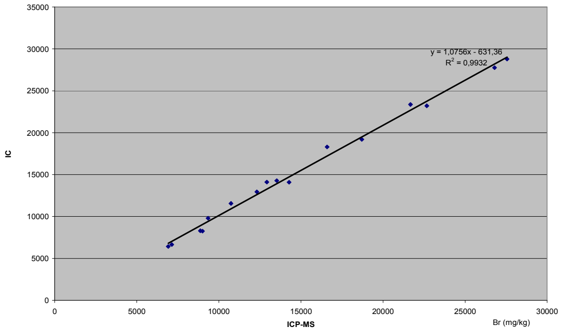


Figure 9: IC versus ICP-MS

Conclusion; In this working range the agreement between IC and ICP measurements for bromine is good.

3 CONCLUSIONS

All validation work on the draft standards resulted in robust methods for the chemical characterisation of solid biofuels. For all problems that were encountered in the Bionorm I project, practical solutions could be found. The problems with halogen losses during oxygen combustion were solved. Milling of biofuel samples can cause contamination problems for trace elements.

4 REFERENCES

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4.2 ROUND ROBIN FOR VALIDATION OF TEST METHODS AND THE IMPLEMENTATION IN THE STANDARDS

[a]Fernández, M. J.; [a]Barro, R.; [a]Carrasco, J.E.; [b]English, M.; [b]Wojcik, M.; [c]Bakker, F.P.; [d]Ranta, J.; [e]Haroldson, C.; [f]Thomsen, E.A.
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Analytical procedures developed in the BioNorm project provide a complete set of methods for the determination of most frequently required chemical parameters in solid biofuel characterization (C, H, N, major and minor elements, Cl and S). Although they are optimised for solid biofuels, respecting the specific characteristics of these fuels, the applicability of the methods and a respective validation based on international round robin was missing. Based on the precision limits shown in this paper and by comparing them with the precision limits set up in the edited technical specifications, it can be demonstrated that the majority of the precision limits obtained in the round robins are higher than those established in the edited technical specifications. The work presented in this paper provides a picture on the performance of the methods and also shows the level of current chemical analysis quality.

Keywords: biofuels, BioNorm, chemical analysis, round robin, standards.

1 INTRODUCTION

Analytical procedures developed in the BioNorm 1 project provide a complete set of methods for the determination of most frequently required chemical parameters in solid biofuel characterization (C, H, N, major and minor elements, Cl and S). Although they are optimised for solid biofuels, respecting the specific characteristics of these fuels, the applicability of the methods and a respective validation based on international round robin was missing.

The knowledge of the content of chemical elements is very important. For example, chlorine, sulphur and other elements such as potassium, sodium, silicon, aluminium, calcium, magnesium and phosphorous are involved in corrosion and ash melting behaviour. Nitrogen, sulphur and chlorine form toxic gaseous compounds in thermochemical processes, which are partially emitted by stack together with other volatile or semivolatile minor elements such as mercury, lead, copper or zinc.

The accurate determination of chemical element contents and other analytical tests applied to solid biofuels, with regard to physical, chemical and energy considerations, is an important factor to determine the quality as a fuel, and to predict possible problems such as corrosion, formation of sinters and slags, environmental contamination by stack emissions or ash disposal, re-utilisation of the ash, etc., that these materials could present in some energy applications. The above mentioned properties are factors that are taken into account in the design of energy conversion plants.

In order to fulfil the purposes of the upgrading of the different technical specifications to reach European standards, two round robins were carried out during the project BioNorm 2. The main purpose of the first round robin was to investigate the performance of the different analytical methods on a wide variety of biofuels available on the European market. The aim of the second or final round robin was to determine the precision of each method in terms of repeatability and reproducibility. The present paper summarises the results of the two interlaboratory comparison tests and includes a statistical evaluation of these tests, which was carried out in accordance with the provisions of ISO 5725 [1].

2 MATERIALS AND METHODS

2.1 Biofuels and laboratories of the first round robin

Six laboratories (OFI, SP, ECN, VTT, DONG and CIEMAT) took part in the first round robin. Twelve biofuels, which mainly came from different countries of European Union, were analysed in the first round robin. Eight of them were analysed in the previous project BioNorm I in order to obtain the current technical specifications. Selected Biofuels include woody biomass such as wood chips, bark, fir without bark and hardwood with glue, and herbaceous biomass as cereal straw, rape straw and thistle. Thistle "*Cynara carduncculus*" could have an important future application as an energy crop due to its adaptation to semiarid climatic conditions, e. g. Mediterranean areas.

A third group of solid biofuel, which come from the agro-industrial processes, was also represented in this work: exhausted olive residue, almond shells, coconut shells and palm bit kernels. Exhausted olive residue is a by-product of the olive oil three-phase extraction process. Coconut shells and palm bit kernels came from non European countries but they could be burnt in European countries in very large quantities. At least, palm bit kernels are already shipped to Europe to be used as fuel in co-combustion power plants.

Although aquatic biomass is not included in the scope of the future European standard 14961-1 [2], seaweed was also considered in this paper. The seaweed sample was collected on a beach in Gothenburg.

The samples were dried and milled, often in two steps with manual removal of impurities (if necessary), to pass a 1 mm sieve. Careful mixing and sampling was carried out manually. The number of individual tests per laboratory and per sample was limited to three.

2.2 Biofuels and laboratories of the final round robin

The participation was open only for invited laboratories, preferring those laboratories accredited according to EN 17025 [3]. A total of 37 laboratories from 11 countries participated in this validation round robin.

Two biofuel samples were selected from the first round robin in order to be compared by a large number of laboratories and obtain the precision of the future European standards. The chosen samples were woodchips and exhausted olive residue. The wood chip sample represents a biofuel with low contents of most of the elements and the olive residue samples with high amounts of most of the elements. The sample wood chips were derived from German coniferous wood chips. The "exhausted olive residue" samples were obtained from the olive oil industry in Spain.

The number of individual tests by a particular participant was equal or higher than five.

2.3 Methods of analyses

A total of 12 samples were investigated for ash content, C, H, N, Cl, S, major elements: Al, Ca, Fe, Mg, P, K, Si, Na, Ti, minor elements: As, Cd, Co, Cr, Cu, Hg, Mn, Mo, Ni, Pb, Sb, V, Zn and the water soluble content of K, Na and Cl. The analyses were performed during the year 2007 according to the edited technical specifications presented in Section 5.

Moisture content in the analytical sample [4] was determined at the same time as the chemical analysis so that an appropriate correction to calculate the data from wet basis to dry basis (d.b.) was made by using, e. g., the CEN technical specification 15296 [5].

The laboratories of the first round robin decided to carry out this extended program because it was expected to obtain all necessary information to make amendments to the CEN/TS before sending them out for inquiry and the final round robin, which was carried out during the year 2008.

Additionally to the previous chemical methods, the net calorific value was determined in the final round only.

2.4 Statistical analysis

Statistical parameters were calculated by OFI according to ISO 5725 [1]. Some of them are shown below:

Repeatability limit (r):

A value less than or equal to what the absolute difference between two test results obtained under repeatability conditions may be expected to be with a probability of 95%:

$$r = 2{,}8 * s_r$$

s_r = repeatability standard deviation

Repeatability conditions set up test results obtained with the same method on identical test items in the same laboratory by the same operator using the same equipment within short intervals of time.

Reproducibility limit (R):

A value less than or equal to what the absolute difference between two test results obtained under reproducibility conditions may be expected to be with a probability of 95%:

$$R = 2{,}8 * s_R$$

s_R = reproducibility standard deviation

Reproducibility conditions set up test results obtained with the same method on identical test items in different laboratories with different operators using different equipment.

Outlier according to Cochran (applied to the final round robin only). With this test, the within-laboratory variances are tested for homogeneity ("outliers regarding standard deviations"):

$$Cochran\ \ criterion\ \ C = \frac{s_{\max}^2}{\sum_{i=1}^{n} s_i^2}$$

$s_{\max}$ = highest value of all s_i

C-values for statistical (probability 99%) and possible (probability 95%) outliers are listed in relevant literature.

Outlier according to Grubbs (applied to the final round robin only). With this test, the extreme values of x_i ($x_{extr} = x_{max}$ or x_{min}) are tested to be an outlier ("outlier regarding the mean value")

$$Grubbs\ \ criterion\ \ G = \left| \ \bar{x}_p - \bar{x}_{extr} \right| \ / s_p$$

x_p = general mean incl. test result which is tested according to Grubbs (x_{extr})

x_{extr} = extreme value of x_i

s_p = general standard deviation (tested result is taken into account as in x_p)

G-values for statistical (probability 99%) and possible (probability 95%) outliers are listed in relevant literature.

3 RESULTS AND DISCUSSION

The limits of repeatability (r) and reproducibility (R) from the published CEN technical specifications (TS) are shown in Table I.

Table 1: Limits of repeatability (r) and reproducibility (R) from the published CEN technical specifications.

		Repeatability		Reproducibility	
		r abs [%]	**r rel [%]**	R abs [%]	**R rel [%]**
	< 0,1%	0,1		0,2	
Ash	1-10%	0,3		0,5	
	>10%		2		4
Carbon		0,5		1,5	
Hydrogen		0,25		0,5	
Nitrogen	< 0,5%	0,05		0,1	
	> 0,5%		10		20
Chloride	< 0,05%	0,005		0,01	
	> 0,05%		10		20
Sulphur	< 0,05%	0,005		0,01	
	> 0,05%		10		20
Chloride	< 0,05%	0,005		0,01	
water soluble	> 0,05%		10		20
Na and K	< 0,01%	0,001		0,002	
water soluble	> 0,01%		10		20

The limits of precision in C, H, N, Cl, S and water soluble content of Cl, Na and K were obtained in the previous project BioNorm I by applying the standard ISO 5725 to relevant data of each method and previous investigations of participating laboratories. The ash values given in Table I are based on precision data for coal and coke taking the standard ISO 1171 [6] into account. Conversely, r and R values in major and minor elements were neither calculated nor estimated from bibliography.

3.1 Interlaboratory comparisons for carbon, hydrogen and nitrogen

The carbon results for arithmetic mean and precision (r and R limits) obtained in the first and final interlaboratory study are shown in Table II. No laboratory is eliminated in the first round robin due to the fact that five laboratories participated only and no result given for these laboratories was found as aberrant. The overall carbon results for wood chips obtained in the final round robin are shown in Figure 1. By applying the tests of Cochran and Grubbs, which were commented in Section 2.4, three laboratories are eliminated and subsequent statistical parameters such as arithmetic mean, standard deviations: repeatability, between-laboratories, reproducibility and limits of repeatability and reproducibility are calculated. This statistical process is repeated for each chemical element or test analysed in the final round robin.

It is important to note that no biofuel fulfils the criteria of carbon precision shown in Table I, with the exception of wood chips analysed in the final round robin (see Table II).

Table 2: Mean (wt% d.b.) and precision (%) of carbon results.

	1st round robin				Final round robin			
	Mean	r rel.	R rel.	Nº lab.	Mean	r rel.	R rel.	Nº lab.
seaweed	32,8	5,3	8,2	5				
thistle	43,8	1,5	4,5	5				
straw	45,9	1,4	5,5	5				
rape straw	46,1	1,4	5,1	5				
bark	46,2	4,4	4,1	5				
olive residue	46,8	1,6	6,7	5	48,1	1,6	3,2	26
almond shells	49,3	1,2	2,6	5				
fir without bark	49,4	2,3	6,9	5				
wood chips	49,5	2,7	7,3	5	50,3	0,8	3,1	27
coconut shells	50,7	1,8	4,5	5				
palm bit kernels	51,1	3,4	6,1	5				
hardwood	51,3	1,2	2,1	5				

Results in dark background fulfil the precision given in CEN/TS [7].

Similarly to carbon, hydrogen results are shown in Table III. In contrast to reproducibility, repeatability criterion is fulfilled by most of biofuels. In order to reach the reproducibility criterion, the absolute R should be higher than 1,2%, which differs considerably of one defined in Table I (0,5%). This absolute R (1,2%) is calculated, e. g., from a relative R around 20% (Table III) in a 6,0 wt% d.b. hydrogen mean content.

Table 3: Mean (wt% d.b.) and precision (%) of hydrogen results.

	1st round robin				Final round robin			
	Mean	r rel.	R rel.	Nº lab.	Mean	r rel.	R rel.	Nº lab.
seaweed	4,0	6,2	19	5				
bark	5,2	6,0	16	5				
coconut shells	5,4	4,1	21	5				
olive residue	5,6	4,7	17	5	5,7	3,1	16	24
palm bit kernels	5,6	3,0	23	5				
thistle	5,7	3,4	16	5				
rape straw	5,8	4,4	17	5				
almond shells	5,9	3,2	25	5				
straw	5,9	3,4	17	5				
hardwood	6,0	2,8	13	5				
wood chips	6,1	6,0	17	5	6,1	3,3	17	28
fir without bark	6,1	3,1	16	5				

Results in dark background fulfil the precision given in CEN/TS [7].

Table IV provides the nitrogen results. Data seem to fulfil precision at very low nitrogen contents much better compared with high nitrogen results.

Table 4: Mean (wt% d.b.) and precision (%) of nitrogen results.

	1st round robin				Final round robin			
	Mean	r rel.	R rel.	Nº lab.	Mean	r rel.	R rel.	Nº lab.
fir without bark	0,05	27	89	5				
wood chips	0,10	25	30	5	0,12	21	83	18
coconut shells	0,24	19	49	5				
hardwood	0,31	18	45	5				
almond shells	0,31	50	85	5				
rape straw	0,39	14	36	5				
palm bit kernels	0,43	18	51	5				
bark	0,63	20	36	5				
straw	0,70	8,4	24	5				
thistle	1,01	11	15	5				
olive residue	1,46	13	23	5	1,39	9,0	23	25
seaweed	2,65	5,7	19	5				

Results in dark background fulfil the precision given in CEN/TS [7].

Carbon, hydrogen and nitrogen results obtained in the final round robin seem to be higher than those obtained in the first round robin, especially carbon content for olive residue, which increases from 46.8 to 48.1 wt%. In general, the laboratories should gain experience and/or an improve their own methodology while working with these hygroscopic samples in continuous instrumental equipments. The samples may lose moisture or adsorb it while waiting in the automatic sampler of the elemental analyser. The loss and adsorbing of moisture depends on the own characteristics of automatic elemental analyser. These considerations related to moisture have been added to the European technical specification for the determination of C, H, and N by instrumental methods CEN/TS 15104 [7].

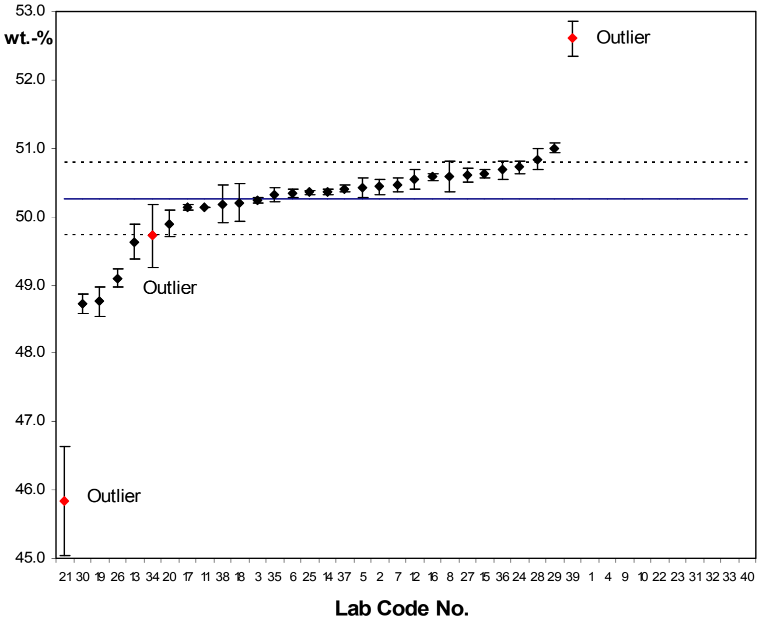

Figure 1: Results of carbon determination in wood chips sample in the final round robin.

3.2 Interlaboratory comparisons for chlorine and sulphur

Results of chlorine and sulphur are shown in tables V and VI, respectively. Contrarily to sulphur, for chlorine analysis an important number of biofuels fulfils the established precision, mainly for values lower than 0,05%.

It is important to note that r and R limits are higher at low contents of sulphur and chlorine. The lower the mean content is, the higher the limits for precision. This is a consequence of the very low concentrations of Cl and S of the woody biomass, fruit shells and fruit kernels, which are near the detection limits of the instruments.

Olive residues do not reach extreme or high values in chlorine and sulphur test methods. Thistle presents the highest value for chlorine and seaweed for sulphur.

Olive residues do not reach extreme or high values in chlorine and sulphur test methods. Thistle presents the highest value for chlorine and seaweed for sulphur. In this technical specification no relevant technical changes are necessary with respect to round robin results. Improvement and changes in texts were necessary as a consequence of the large number of methods and equipments that can be used to determine Cl and S concerning CEN TS 15289 [8]

Table 5: Mean (wt% d.b.) and precision (%) of chlorine results.

	1st round robin				Final round robin			
	Mean	r rel.	R rel.	Nº lab.	Mean	r rel.	R rel.	Nº lab.
fir without bark	0,004	52	186	6				
wood chips	0,004	32	58	4	0,006	36	145	17
bark	0,009	34	48	6				
almond shells	0,012	69	70	6				
hardwood	0,014	31	57	5				
palm bit kernels	0,015	31	46	5				
straw	0,109	13	24	6				
coconut shells	0,169	14	31	6				
seaweed	0,198	13	29	5				
olive residue	0,180	13	16	5	0,200	7,7	22	16
rape straw	0,289	8,6	38	6				
thistle	1,65	7,6	9,1	5				

Results in dark background fulfil the precision given in CEN/TS [8].

Table 6: Mean (wt% d.b.) and precision (%) of sulphur results.

	1st round robin				Final round robin			
	Mean	r rel.	R rel.	Nº lab.	Mean	r rel.	R rel.	Nº lab.
fir without bark	0,006	110	146	5				
wood chips	0,009	103	136	4	0,009	34	95	20
hardwood	0,018	90	108	6				
almond shells	0,028	123	141	6				
palm bit kernels	0,030	34	39	6				
coconut shells	0,041	29	38	6				
bark	0,062	24	32	6				
straw	0,101	8,8	27	6				
olive residue	0,109	28	35	6	0,120	15	48	23
thistle	0,169	25	39	6				
rape straw	0,184	14	24	6				
seaweed	2,11	11	25	6				

Results in dark background fulfil the precision given in CEN/TS [8].

3.3 Interlaboratory comparisons for water soluble content of chloride, potassium and sodium

The obtained data are shown in the Table VII. By comparing the data of this Table VII with data from total chlorine (Table V) and potassium (Table VIII) and sodium (Table XI) can be concluded that the water soluble contents tend to be similar or a slightly lower than those obtained under preparation based on combustion or digestion, previous to the determination of the elements.

Table 7: Mean (wt% d.b.) and precision (%) of sulphur results.

		1st round robin				Final round robin				Mean dif.
		Mean	r rel.	R rel.	Nº lab.	Mean	r rel.	R rel.	Nº lab.	%
Cl	wood chips	0,008	30	28	2	0,003	53	136	8	-208
	olive residue	0,218	19	43	3	0,207	6,6	21	7	-5
K	wood chips	0,051	nd	nd	1	0,063	4,0	8,7	6	20
	olive residue	1,98	nd	nd	1	2,24	6,6	13	7	12
Na	wood chips	0,0019	nd	nd	1	0,0016	151	269	4	-19
	olive residue	0,018	nd	nd	1	0,010	24	45	8	-82

Results in dark background fulfil the precision given in CEN/TS [9]. Nd: non determined.

Contrarily to potassium, sodium precision results do not seem to reach the criterion shown in table I. Important mean differences and low precision are found in wood chips sample for chloride ion as happened in chlorine determination (see Section 3.2).

3.4 Interlaboratory comparisons for major elements

Interlaboratory exercises for potassium, silicon and calcium are shown in Tables VIII, IX and X , respectively. According to the values shown in Tables VIII, IX and X, silicon data offer the highest r and R results and the widest variations when comparing mean values between the first and final second round robin. Probably, problems in the determination of silicon could be due to an incomplete dissolution of the silicon using the digestion methods given in the technical specification CEN/TS 15290 [10]. One point that corroborate the previous comment was the apparition in some laboratories of ash deposits, with higher proportion in silicon, within important parts of spectrometers, when the ash (550º C) digestion method was used for a long period of time. After the first round robin, it was decided to increase the amount of HF from 1.0 ml to 2.0 ml in biomass digestion if the sample is expected to have an ash content above 10 % and from 0.75 ml to 2.0 ml in ash (550º C) digestion to overcome these problems. Therefore, the silicon results of the final round robin are considered more representative and more accurate.

Table 8: Mean (mg/kg d.b.) and precision (%) of potassium results.

	1st round robin				Final round robin			
	Mean	r rel.	R rel.	Nº lab.	Mean	r rel.	R rel.	Nº lab.
fir without bark	353	32	35	5				
wood chips	700	8,9	22	5	691	5,0	31	13
hardwood	994	10	26	5				
palm bit kernels	1333	16	27	5				
bark	2214	8,6	13	5				
coconut shells	3822	14	27	5				
almond shells	4326	10	22	5				
rape straw	9363	5,6	35	5				
seaweed	10869	10	23	5				
straw	11156	30	41	5				
thistle	13527	8,1	26	5				
olive residue	23152	19	45	5	24524	5,3	18	11

Table 9: Mean (mg/kg d.b.) and precision (%) of silicon results.

	1st round robin				Final round robin			
	Mean	r rel.	R rel.	Nº lab.	Mean	r rel.	R rel.	Nº lab.
fir without bark	455	47	247	3				
rape straw	459	62	288	4				
wood chips	609	17	311	3	320	31	92	8
hardwood	931	91	328	4				
thistle	1634	52	169	4				
almond shells	1698	81	117	5				
coconut shells	2938	74	98	6				
bark	3138	19	84	4				
palm bit kernels	7304	55	80	5				
straw	8885	68	149	5				
olive residue	10859	19	21	5	10043	21	34	10
seaweed	55262	62	100	4				

Table 10: Mean (mg/kg d.b.) and precision (%) of calcium results.

	1st round robin				Final round robin			
	Mean	r rel.	R rel.	Nº lab.	Mean	r rel.	R rel.	Nº lab.
fir without bark	831	6,7	15	5				
coconut shells	847	18	31	6				
wood chips	1432	5,6	12	5	1502	4,5	19	11
straw	2912	8,1	19	5				
almond kernels	2922	19	32	6				
hardwood	4183	11	43	5				
palm bit kernels	4880	21	37	5				
rape straw	12572	9,3	27	5				
olive residue	14461	7,2	13	6	14216	12	20	13
thistle	14501	6,5	13	5				
seaweed	20369	14	19	4				
bark	47146	8,7	16	5				

The rest of major elements (Mg, P, Al, Fe and Ti), which usually have a lower content than the above mentioned elements, are presented for wood chips and exhausted olive residue in Table XI.

The values of the rest of major elements of the first round robin (Table XI) seem to correlate well with the results of final round robin. In the case of Na, an important difference is observed in wood chips sample. An explanation of this fact is attributed to the very low contents of sodium, next to the quantification limits of the equipments.

Regarding the precision obtained in most of the major elements, an important number of repeatability and reproducibility limits are lower than 20 % and 30 %, respectively.

Table 11: Mean (mg/kg d.b.) and precision (%) of magnesium, sodium, phosphorous, aluminium, iron and titanium results.

		1st round robin				Final round robin				Mean dif.
		Mean	r rel.	R rel.	Nº lab.	Mean	r rel.	R rel.	Nº lab.	%
Mg	wood chips	182	4,0	9,0	5	194	4,7	20	12	6
	olive residue	2847	21	62	5	3143	13	22	13	9
Na	wood chips	19	112	138	5	13	40	134	9	-46
	olive residue	176	18	19	5	171	15	65	10	-3
P	wood chips	73	6,0	56	5	74	9,4	19	11	1
	olive residue	1523	19	20	5	1488	11	24	13	-2
Al	wood chips	50	11	55	5	47	12	50	12	-6
	olive residue	2350	13	19	4	2364	13	20	11	1
Fe	wood chips	49	26	64	6	54	18	33	15	9
	olive residue	1561	12	27	7	1598	14	29	15	2
Ti	wood chips	5,6	80	140	4	5,5	17	21	9	-2
	olive residue	130	18	20	4	136	12	22	11	4

No relevant comments are found in the results of Mg, P, Al, Fe and Ti for the rest of the biofuels (results not shown).

3.5 Interlaboratory comparisons for minor elements

The minor element concentrations show significant differences among biofuels, e. g. in lead, cadmium and mercury results, Tables XII, XIII and XIV, respectively, as also happened in major elements, Cl and S. These differences between minimum and maximum averaged values can exceed a factor of 100.

As expected, the limits of r and R increase as the content of minor elements decrease. The precision results obtained in the final round robin are poorer than those achieved in major elements, especially at very low contents of trace elements such as As, Cd, Cr, Hg, Mo, Sb and V (see Table XV). The low precision obtained could be a consequence of the reduced number of participating laboratories or the utilisation of equipments with low analytical sensitivity. Further research is required to investigate this statement and to improve CEN/TS 15297 [11].

Table 12: Mean (mg/kg d.b.) and precision (%) of lead results.

	1st round robin				Final round robin			
	Mean	r rel.	R rel.	Nº lab.	Mean	r rel.	R rel.	Nº lab.
rape straw	0,10	74	178	2				
fir without bark	0,13	119	144	3				
straw	0,34	172	200	3				
wood chips	0,52	42	35	4	0,75	27	44	9
thistle	0,52	129	145	5				
palm bit kernels	0,53	61	132	6				
almond shells	0,76	155	154	5				
bark	2,62	33	95	5				
hardwood	2,88	113	250	4				
olive residue	5,03	97	122	6	3,83	26	42	13
seaweed	7,10	46	75	5				
coconut shells	22,5	138	187	6				

Table 13: Mean (mg/kg d.b.) and precision (%) of cadmium results.

	1st round robin				Final round robin			
	Mean	r rel.	R rel.	Nº lab.	Mean	r rel.	R rel.	Nº lab.
almond shells	0,008	111	122	3				
hardwood	0,023	250	229	3				
olive residue	0,034	99	113	3	0,025	55	64	8
palm bit kernels	0,051	92	80	3				
thistle	0,059	14	19	3				
coconut shells	0,084	61	78	4				
fir without bark	0,106	15	19	4				
straw	0,108	45	58	4				
bark	0,257	8,3	14	4				
rape straw	0,292	18	34	4				
wood chips	0,317	12	24	4	0,320	8	18	13
seaweed	0,767	11	25	5				

Table 14: Mean (mg/kg d.b.) and precision (%) of mercury results.

	1st round robin				Final round robin			
	Mean	r rel.	R rel.	Nº lab.	Mean	r rel.	R rel.	Nº lab.
hardwood	0,0025	33	237	2				
almond shells	0,0033	153	135	2				
coconut shells	0,0033	28	58	4				
palm bit kernels	0,0034	42	58	3				
fir without bark	0,0035	5	71	3				
wood chips	0,0056	22	26	2	0,0072	37	64	6
rape straw	0,0065	21	34	3				
thisle	0,0080	20	42	3				
olive residue	0,0088	66	81	4	0,0120	61	111	10
straw	0,0123	23	23	3				
bark	0,0153	18	47	3				
seaweed	0,0321	33	61	4				

Mean differences between the first round robin and the second one (Table XV) are considered significant. Obviously this can be due to many factors, however, the number of laboratories implied in the first round robin is rather small and any erroneous result given by one of these laboratories can add an important bias in the measurement.

Table 15: Mean (mg/kg d.b.) and precision (%) of arsenic, cobalt, chromium, copper, manganese, molybdenum, nickel, antimony, vanadium and zinc results.

		1st round robin				Final round robin				Mean Dif.
		Mean	r rel.	R rel.	Nº lab.	Mean	r rel.	R rel.	Nº lab.	%
As	wood chips	0,021	35	Nap	1	0,036	94	180	6	43
	olive residue	0,720	108	100	4	0,600	17	27	8	-20
Co	wood chips	0,36	12	21	4	0,34	8,0	27	9	-6
	olive residue	1,01	9,0	12	6	1,04	15	34	11	3
Cr	wood chips	0,49	112	139	3	0,37	57	89	9	-32
	olive residue	15,5	30	27	7	14,3	21	63	15	-8
Cu	wood chips	1,70	55	185	3	1,29	20	33	12	-32
	olive residue	25,0	15	18	6	25,0	9,5	24	15	0
Mn	wood chips	267	4,0	10	5	261	4,1	19	14	-2
	olive residue	39,0	5,0	15	5	40,2	10	18	15	3
Mo	wood chips	0,064	44	267	3	0,028	115	145	5	-129
	olive residue	0,28	38	49	3	0,22	23	70	8	-27
Ni	wood chips	0,68	44	63	4	0,60	20	49	10	-13
	olive residue	13,0	15	32	7	12,5	15	18	11	-4
Sb	wood chips	0,059	195	291	3	0,013	30	87	5	-354
	olive residue	0,162	38	194	3	0,094	30	42	5	-72
V	wood chips	0,053	36	149	3	0,076	34	65	9	30
	olive residue	4,65	15	24	5	4,26	14	30	11	-9
Zn	wood chips	13,4	17	38	4	13,8	14	39	15	3
	olive residue	17,8	21	23	5	18,2	13	30	15	2

3.6 Interlaboratory comparisons for ash

According to the limits of precision given in Table I, very low ash contents fulfil only (see Table XVI) the criteria set up in the CEN/TS 14775 [12]. These results are opposite to what could be expected because other fuels such as coal and coke maintain a good precision.

The ash composition strongly depends on the temperature. Insufficient control of the temperature or different heating rates, among other factors, may lead to a change in the ash content, and therefore, in a bias of the measurement method. It is known that biomass ash contains more volatile inorganic compounds at temperature at which the ash is obtained (550º C) than solid mineral fuels, e. g. alkaline and alkaline-earth carbonates, compounds and species of chlorine and sulphur, which begin to release in the furnace at temperatures below 550º C [13-14]. Therefore, data of precision based on standards from other solid fuels could not be reached in interlaboratory studies using biomass.

Table 16: Mean (wt% d.b.) and precision (%) of ash results.

	1st round robin				Final round robin			
	Mean	r rel.	R rel.	Nº lab.	Mean	r rel.	R rel.	Nº lab.
fir without bark	0,25	25	42	6				
wood chips	0,58	14	42	7	0,59	9,2	18	28
hardwood	1,35	13	27	6				
almond kernels	1,99	12	37	8				
coconut shells	2,13	14	29	8				
palm bit kernels	3,44	25	34	8				
rape straw	5,14	6,6	9,5	6				
straw	5,39	3,4	12	8				
thistle	9,88	7,1	18	8				
olive residue	10,8	7,7	17	8	11,1	5,0	11	30
bark	12,9	7,4	24	8				
seaweed	32,9	3,1	10	8				

Results in dark background fulfil the precision given in CEN/TS [12].

3.7 Interlaboratory comparisons for calorific value

In addition to chemical characterisation, calorific values were also determined in the final round robin and their results are presented in Table XVII. The precision limits published in the CEN/TS 14918 [15] were mainly based on other standards related to solid mineral fuels. The limits of repeatability and reproducibility are 120 and 300 J/g, respectively, taking CEN/TS 14918 into account.

Table 17: Gross and net calorific values and precision in J/g d.b.

		Mean	r abs.	R abs.	Nº lab.
GCV	Wood chips	20092	117	346	28
	Olive residue	19401	138	356	24
NCV	Wood chips	18764	105	369	22
	Olive residue	18146	138	335	20

The limits obtained are similar to those established in the technical specification. A slight correction should be carried out in solid biofuels with respect to solid fuels with an increase in repeatability limit from 120 to 150 J/g and an increase in reproducibility limit from 300 to 400 J/g.

4 CONCLUSIONS AND RECOMMENDATIONS

Based on the precision limits shown in the tables for ash content, calorific value, carbon, hydrogen, nitrogen, chlorine, sulphur and water soluble contents of chloride, sodium and potassium and by comparing them with the precision limits set up in the edited technical specifications, it can be demonstrated that the majority of the precision limits obtained in the round robins are higher than those established in the edited technical specifications.

It is difficult to establish precision limits of repeatability and reproducibility in major and minor elements as well as in other chemical elements as a consequence of the variability of the precision results found among biofuels. A solution may be to state the performance characteristics of the methods, which present statistical results obtained in interlaboratory comparisons, in stead of giving definite repeatability and reproducibility values for the methods.

The accurate determination of mercury, molybdenum, antimony and other minor elements could require more sensitive instruments and/or more laboratories participating in the interlaboratory comparisons.

The results of these round robins can not be used to guarantee that all options described in the standards deliver perfect results for all biofuels available. The methods are new and experiences are still growing.

However the work presented in this paper provides a picture on the performance of the methods and also shows the level of current chemical analysis quality.

5 REFERENCES

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6 ACKNOWLEDGEMENTS

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4.3 RAPID TEST METHODS FOR LABORATORIES, TEST METHODS FOR FIELD AND INDUSTRIAL USE

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Reliable field and fast laboratory methods are needed to generally improve possibilities for quality measures regarding chemical properties. The current development of the market for solid biofuels aims at using biomass resources with often unknown or widely varying chemical compositions. Since the chemical composition of solid biofuels is important for thermal conversion processes, analytical results are required to classify solid biofuels. Based on a mandate given by the EC, CEN TC 335 "Solid Biofuels" standards for reference methods to assess required chemical properties were developed and will soon be published. Experiences with field methods and rapid test methods for laboratories are scarce, a screening and development of methods was part of BioNorm II project. The research on XRF performed in this project initiated a new work item in European Standardization, a first draft of a standard will be available soon. The evaluation of field methods showed, that it is possible to assess chlorine and potassium content of biofuels with minimal equipment directly during harvest. This article provides a brief summary of both methods.

Keywords: BioNorm, solid biofuels, chemical test methods, rapid test methods, field methods

1 INTRODUCTION

Successful research in BioNorm I and II and committed work in CEN TC 335 working groups for physical and chemical test methods resulted in method development and method evaluation that will be finished in the near future with the publication of European Standards (EN's). The research results made it possible to develop these standards starting from (i) very different individual traditions in European laboratories and (ii) from a very wide variety of different chemical analysis methods. Due to limitations in budget the development of methods focussed on reference methods for professional laboratories with best available precision and accuracy.

In practise often fast but approximate results are required instead of comparable slow and expensive lab analysis. Rapid test methods are necessary to decrease analysis costs and to obtain necessary information much faster. For the young and still small market of modern solid biofuels, like pellets or agricultural residues, rapid test methods are scarcely available as commercial methods. To decrease market barriers and to support trade with highly sophisticated quality assurance systems on well defined critical control points the growing bioenergy markets need fast and cheap analytical methods for the characterisation of biofuels. Methods are needed for laboratory and for field use. Rapid test methods in laboratories aim at a reduction of analysis time and costs, but shall still provide high reliability and a maximum of information with respect to the investigated samples. For field use, methods are required for the use by non-chemists providing quick information about the most critical element concentrations like chlorine and alkali metals. Within few minutes information shall be available to decide if a given biomass can be used for combustion in a specific plant. Finally, for the production of processed, high quality biofuels, online measurements of physical and chemical parameters are necessary for a stable product quality.

Except for moisture content, rapid methods are not widespread, neither in laboratories, nor in field use or for process control. In BioNorm II a limited budget was reserved for collecting and summarizing available technologies, for testing of field methods and for the development of rapid test method for laboratory use.

2 PROJECTS' RELATION TO THE EUROPEAN STANDARDISATION COMMITTEE

The project is in continuous contact with the CEN TC 335 "Solid Biofuels" team to ensure a close connection and interaction between the standardisation bodies and the scientific work in this area. Based on research in BioNorm II, a new work item was created on Oct. 9th 2008 [i] "Solid Biofuels – Determination of the chemical composition by XRF (X-ray fluorescence)". Other results and work done is jet not developed enough to be standardized. The work done is presented within the CEN TC working groups and is spread by these working groups in the respective European countries for exchange of know-how, for testing and evaluation. It is very likely, that these methods presented in the next paragraphs will be the basis for new work items and standards in further development of solid biofuel characterization.

3 RAPID TEST METHODS FOR LABORATORIES

3.1 Objectives

Currently, a full chemical characterisation of solid biofuels is fairly time-consuming and therefore quite expensive. Rapid test methods for routine analysis and process control are yet neither developed nor evaluated. From an industrial point of view, a method is required that provides results in a short time. An instrumental analysis method is necessary that provides all parameters of interest at once: the major or ash forming elements (K, Na, Ca, Mg, Si, Al, Fe, Mn), elements that contribute to environmentally harmful emissions especially Cd, Zn, Cu, Ni, Hg, Pb, and elements causing corrosion, fouling and slagging, especially Cl, S, Na and K. A method based on X-ray fluorescence (XRF) is most promising. XRF analysis is already widespread in other fields like waste analysis or in the cement industry but up to now rarely used for biofuels.
The research work focused on the development of a simple sample preparation method and on the evaluation of the possibilities and experimental limitation of available XRF systems.

3.2 Results and discussion

To apply XRF to solid biofuels, some aspects have to be considered:

- Sample preparation
- Calibration
- Calculation of results from spectra
- Limitations (precision of method)

For sample preparation different possibilities exist which were evaluated in the BioNorm II project. Five different sample preparations were used on the sample in powder form, see **Table 1**.

Table 1: Sample preparation for XRF, investigated

Sample preparation	**Sample amount**	**Comment**
None – direct measurement	0.5 g	40 mm sandwich in a liquid disposable sample cup – sample placed between two polypropen films, one 6 µm thick nonporous film and one microporous film
Pellet pressed using 12.5 % wax	3.5 g	Sample + 0.5 g wax pressed into 32 mm discs in an aluminium cup at 15 ton for 3 minutes
Pellet using heat – hot melt	7 g	Only two samples were available – sample 1 and sample 7
Ashing at 550 °C	20 g	Two hours or more of dry ashing
Glass Bead – $Li_2B_4O_7$	20 g	5 gram of flux, available ash of sample up to 0.5 g and 20 mg of LiBr

The first three – direct, briquette and warm briquette can be regarded as simple and fast. The last one, preparation of a bead from the ashes can be regarded as quantitative for all elements except the volatiles.

For biofuels the pellet is the preferred simple sample preparation technique for both light and heavy elements. Direct analysis (0,5 g sample) gave similar performance for light elements but higher LOQ for heavier elements due to less sample amount. The best preparation method is a hot melt pellet that provides high long term stability [[ii]]. To obtain best hot melt pellets, following procedure is suggested:

- For samples with water content above 10% pre-drying at 105°C is necessary
- Milling of dried sample to a particle size < 500 µm, press power: 50 kN
- temperature 140°C, duration: 4,5 minutes
- sample amount: ca. 7 g to get sufficient thick pellets

In theory, a smaller particle size as < 1mm (general analysis sample) is required because assuming a XRF measurement area of 2 cm² and a penetration depth of 2 microns, a sample amount of 5-10 mg is analyzed. For this small sample amount, a particle size of < 40 µm is preferable. The penetration depth depends on the power of the XRF-tube which of course favours instruments with high power. However, milling of solid biofuels may lead to contamination, see [[iii]]. Additionally, milling of solid biofuels to below 500 µm leads to an uneven element distribution in different size fractions of the sample. A higher concentration of ash forming elements is found in the smallest particle size fraction as shown for sieve fractions in Figure 1.

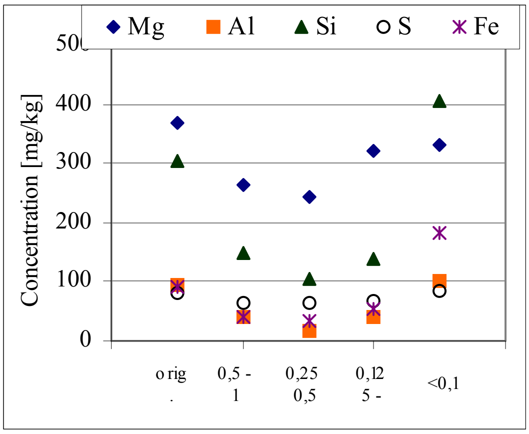

Figure 1: Element distribution in the original 1 mm sample and in the respective fractions after milling

To investigate the repeatability of XRF measurements for given samples, hot melt pellets were produced and measured with the same method in the same instrument according to the procedure described above with a particle size < 500 µm, see Table 1. The standard deviation is very small, the repeatability very good and much better compared with the standard method (EN 15290). Thus it can be concluded, that a particle size < 500 µm is sufficient.

Table 2: XRF-Analysis on sample olive residues, 6 different pellets [mg/kg]

	Mg	Al	Si	P	Cl	K	CA
1	2415	1626	7990	1496	1508	23352	11652
2	2442	1593	8043	1496	1510	23344	11236
3	2470	1706	8331	1496	1518	23563	11646
4	2567	1708	8527	1545	1517	23857	11960
5	2399	1578	7830	1479	1506	23173	11060
6	2431	1588	7915	1457	1529	23285	10820
mean	2454	1633	8106	1495	1515	23429	11396
RSD	60	59	268	29	9	245	428

Independent if energy dispersive X-ray fluorescence (EDXRF) spectrometry or wavelength dispersive X-ray fluorescence (WDXRF) spectrometry is used, quantification can be done by standardless software provided by most instrument suppliers or using a calibration with matrix-matched standards. The second method is much more accurate, see e.g.

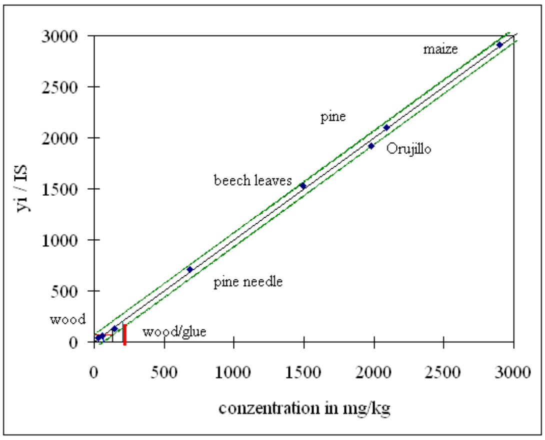

Figure 2: Example of a XRF calibration for chlorine based on CRM's and BioNorm samples; the mark at 200 mg/kg in the graph is the limit for wood pellets according to EN 14961-2

For XRF calibration of all relevant parameters, a minimum of 15 reference materials seems necessary. Since only a few CRM's (e.g. from BCR, GBW and Wageningen) were available in the past, calibration was very difficult to establish. With the help of BioNorm I and II well characterized materials, calibration is now possible. In BioNorm II, a laboratory comparison test was carried out with two samples, olive residues and woodchips. From the five participating laboratories, most did not use a biomass calibration. Examples of results are shown in figure 3 and figure 4.

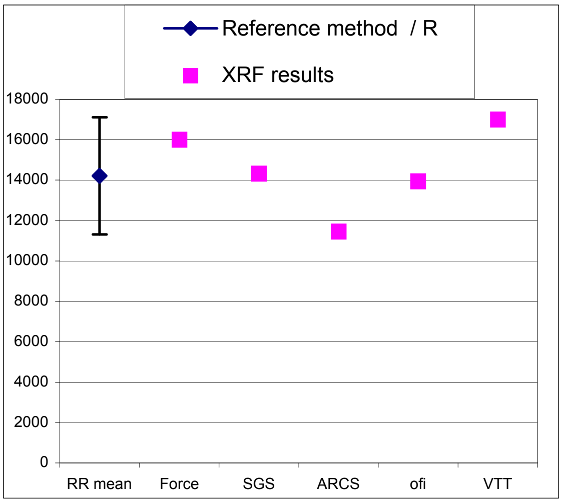

Figure 3 XRF results of different laboratories, calcium content in olive residues in mg/kg and the reference value and bias obtained in the validation round robin for EN 15290

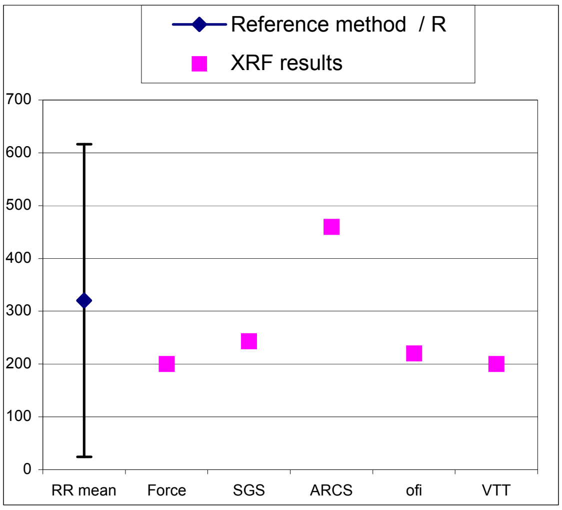

Figure 4: XRF results of different laboratories, silicon content in woodchips in mg/kg and the reference value and bias obtained in the validation round robin for EN 15290

For minor elements, there is a limitation for applying XRF. Depending on instrument, calibration and sample preparation, detection limits can be as low as a few ppm which is for most elements enough to detect impurities.

Conclusion: With proper sample preparation (pellets) the following components can be determined in most solid biofuels specified in EN 14691: Al, Ca, Cl, Fe, K, Mg, Mn, P, S, Si, Zn. For the other tested elements only major impurities can be assessed with XRF e.g Cu, Na.

4 RAPID TEST METHODS FOR FIELD USE

4.1 Objectives

Chlorine, sodium, and potassium are of outstanding importance for solid biofuels combustion behaviour with respect to corrosion, slagging and fouling. These three elements are found in widely varying concentrations in the same type of solid biofuel. Thus, testing of raw materials at a very early stage of fuel processing may sometimes be advantageous. Cheap methods for outdoor use are required and should be developed and evaluated. The methods should be especially applicable for biofuels with usually high contents of these elements and should be applicable by non chemists after a very brief training.
The methods investigated in BioNorm II utilise the high solubility of Cl, K and Na in water, a principle that is already used in the laboratory method [iv]. Possibilities were investigated in a Danish research project [v], a method was published in 2008. The principle is to cut the fuel into an appropriate fineness. A portion of this material is extracted with pure water or 2 % acetic acid solution by shaking. The amount of chloride, sodium, potassium in the extract is determined by quick tests, such as test kits for water analyses.

4.2 Experimental

The method should be applicable by non chemists after a very brief training. Household equipment should be used for sample preparation according to the guideline, see Table 3.
Extraction, quantification and calculation are described in the guideline. Due to the different suppliers of test kits in the participating countries and since the method should not rely on certain trade mark items, it was intended to use different test kits for evaluation.

Table 3: Sample preparation recommendation [v]

Form of solid biofuel	Sample preparation
Straw and straw-like materials	Cut the material into pieces of maximum length 3 cm
Grains, seeds and shells	Mill the material in a coffee mill for about 10 seconds
Pellets	Blend the pellets in a blender for 10 – 30 seconds, depending of the durability of the pellets
Chips and shavings	Run a larger portion of the material, e.g. ½ kg, through a garden shredder

5 SERIES OF EXPERIMENTS WERE PERFORMED:

Table 4: Test parameters for field tests [v]

	Test fuels	Field test method
Series 1	Field samples Straw and woodchips	Cl^-: HACH Quantab Low Range 30-600 mgl/l Na: flame test K: only lab-finish
Series 2	10 BioNorm samples	Test –kits for Cl^-, SO_4, K^+ and Ion-selective electrode for Na^+
Series 3	7 BioNorm samples and 5 field samples	Cl^-: HACH Quantab Low Range 30-600 mgl/l K: Visocolor Macherey-Nagel, Range 2-15 mg/l and Quantofix Macherey-Nagel, Range 200-1500 mg/l
Series 4	12 field samples	chloride test Aquamerck (titrimetric determination with a titration pipette), Range 2-200 mg/l

Series 1 was performed by Dong Energy [vi], samples were extracted alternatively in deionized water and in 2% acetic acid. As reference methods quantification was done by Ion Chromatography (Cl) and ICP-OES (Na) and the samples were analysed according to CEN/TS 15105. For the experiments well characterized and homogenized samples from BioNorm II project were used, experiments were carried out in laboratory.

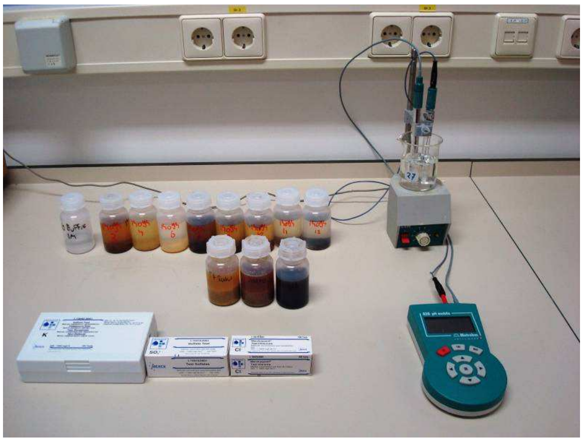

Figure 5: equipment used in series 2

Series 2 was performed by ECN [vii] using all samples prepared for BioNorm I and II for method development to cover a wide range of concentrations, see figure 5. Thus, sample preparation (cutting, grinding) was not evaluated. 1 g sample was mixed with 100 ml deionized water, similar to CEN TS 15105, but the slurry was only shaken, not stirred and no heating was applied. The quicktest kits were applied according instructions.

Series 3 was performed by CIEMAT [viii] using selected BioNorm I and II samples and field samples such as pine chips, wheat straw, rape pellet, almond shells and exhausted olive residue. Thus, sample preparation (cutting, grinding) was not evaluated in the BioNorm samples. Conversely, the amount of material tested and biofuel particle size was evaluated in field samples. 2,5 g of sample was mixed with 100 ml deionized water (for wood 10g was used) in a preserving jar, shaken 25 times and rested for 30 minutes. Quicktests were used for Cl and K (2 types) determination.

Series 4 was performed by ofi [viii]. The aim was to get experiences in sampling of different agricultural plants. Sampling of eight different biomass materials was done by students in September 2009 east of Vienna. The samples were taken on-site with secateurs and were packed airproof. Sampling was partially problematic, because the plants were not always easy to remove from the field. The individual sample amount was at least 1,5 kg.

Figure 6: sampling of reed, series 4

The high inhomogeneity of the biomass samples made it necessary to divide several samples in two fractions. Fraction (a) consisted of the halm, stem etc. of the taken sample, the second fraction (b) contained the corns or blossoms. The size reduction was done by scissors and/or similar household equipment. In the laboratory approximately 50 grams of every sample was weighed and cut in small pieces of 3-5 cm.

Three different extraction methods were tested:
(extract 1) the sample was mixed with one litre of deionized water and was shaken 30 times to wet the sample completely. After half an hour resting, a sample was taken for analysis.
(extract 2) the sample was also mixed with one litre of deionized water. Then, the slurry was stirred with a mixer. A sample for analysis was taken after 30 min segregation of the slurry.
(extract 3) similar to (extract 2) but with an extended extraction time (resting overnight).
Samples were filtered and analysed for chlorine with the Aquamerck quicktest; in parallel they were analysed by IC.

5.1 Results

The results of **series 1** are summarized in Figure -3. Concerning the extraction, the field method delivers results that comply quite well with the reference method for all three elements. For chlorine, the quantification with the test kit supplies acceptable results, the flame test is not sufficient.

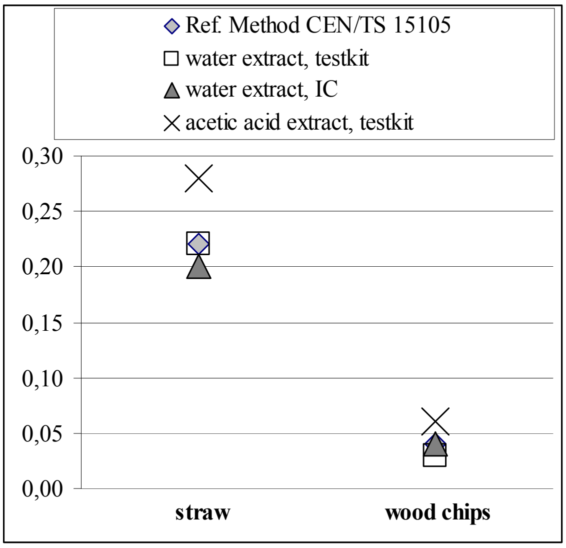

Figure 7: Cl determination using field- and reference methods

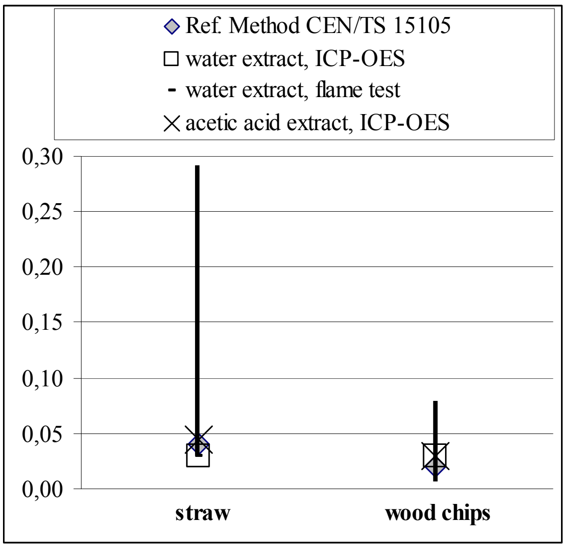

Figure 8: Na determination using field- and reference methods

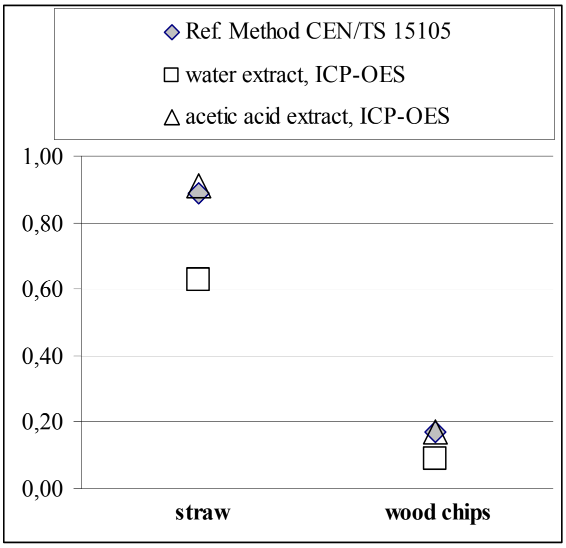

Figure 9: K determination using field-preparation and lab-finish and reference methods

For **series 2** only results for Na and K could be obtained. The concentrations of Cl and SO4 were below lowest measurement range. Especially the colours of the biofuel extracts disturbed the readings.

The conclusion of this series was that the applicability of standard quicktests on water extracts of biofuels is limited. Due to the colour of the extract colour tests are not very reliable. Dilution is no option, as the sensitivity of these methods is limited.

Table 5: Results of Na and K determination, series 2 in [mg/kg]

sample	**Na**	**Na_{ref}**	**K**	**K_{ref}**
Bark	10	120	25000	2100
Rapestraw	10	660	< 250	9000
Fir without bark	50	5	< 250	310
Orujilo (1:100)	530	170	25000	24000
Orujilo (5:100)	260	170	14000	24000
Seaweed	4900	12000	< 250	10300
Coconut shells	2200	1950	< 250	3500
Almond kernels	350	50	< 250	4000
Palm pit kernels	80	90	< 250	1200
Cyanara	8800	12000	9000-14000	13000
Straw	1200	70	5000-9000	11000

Some results for series 3 are compiled in Table 6 and Table 7. More results can be found in the original report [viii].

Table 6: results of Cl determination, series 3 in [wt-%]

	field method	CEN/TS 15105
Wood chips	< 0,03	0,012
Almond kernels	0,02	0,033
Rape straw	0,29	0,24
Cynara	1,53	1,60
Coconut shells	0,26	0,18
Exhausted olive residue	0,37	0,18
Seaweed	0,45	0,14

Table 7: Results of K determination, series 3 in [wt-%]

	field method, Visocolor	field method, Quantofix	CEN/TS 15105
Wood chips	0,10	0,20	0,076
Almond kernels	n.d.	0,10	0,38
Rape straw	0,80	0,80	0,71
Cynara	0,80	1,60	1,20
Coconut shells	0,16	0,80	0,21
Exhausted olive residue	2,40	1,60	1,98
Seaweed	0,12	0,80	0,32

Biomass with high contents of other halogens such as fluoride and bromide, e. g. exhausted olive residue and seaweed, can cause interferences in the determination of the chloride content. Analytical quicktest-kits are of different applicability for biofuel analysis (e.g. the Quantofix method is a faster method to determine potassium than Visocolor method, although inaccurate at low contents of potassium (< 0.5%).

The following conclusion can be deduced according to fast tests with field samples: herbaceous materials and pellets are easily mixed and extracted in water and, consequently, it is not necessary to grind them before analyzing. Contrarily, materials such as wood chips, fruit kernels and other agro-industrial biomasses need a grinding before analyzing.

Some results for **series 4** are compiled in Table 8. For higher concentrations, testkit and IC results correspond very well, for lower concentrations the test kit performance is limited. Extraction seems to be completed after 30 minutes but extensive mixing (with a household mixer) liberates chlorine. The extraction of chlorine depends strongly on the type of biomass.

6 CONCLUSIONS

In general, the rapid test methods for field use, especially the Danish PSO 5297 method, are suitable to determine soluble chlorine and alkali metals. However, in all experiments or series of experiments, limitations of applicability of the methods were found. Currently it seems, that it is not possible to use one rapid test method for all solid biofuels and for all possible element concentrations. It is recommended to validate a procedure for one kind of biofuel and the corresponding experimental procedure (sample preparation, sample amount, test kits, etc.).

Table 8: Results of Cl determination, series 4 in [mg/l extract]

	extract 1		extract 2		extract 3	
	kit	IC	kit	IC	kit	IC
Maize (halm)	15	6,8	20	17	22	15
Maize (corncob)	5	6,2	15	12	20	12
Beet	10	4,6	105	62	105	60
Legume	25	21	77	56	77	50
Reed	57	47	65	47	-	46
Weed	110	38	110	85	110	83
Sorgum (halm)	40	28	57	47	60	47
Sorgum (corn)	10	2,8	10	47	40	47
Pulse	10	2,2	10	3,1	10	4,4
Sunfl.(stem)	70	60	150	135	150	142
Sunfl. (blossom)	100	108	100	79	105	79
Sunfl. (stem)	130	109	130	99	135	114
Sunfl. (stem)	42	38	50	21	55	21

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5 Working Package III
- Quality Measures -

Coordinated by: Janet Witt

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5.1 ELABORATING QUALITY MEASURES FOR SELECTED BIOFUELS

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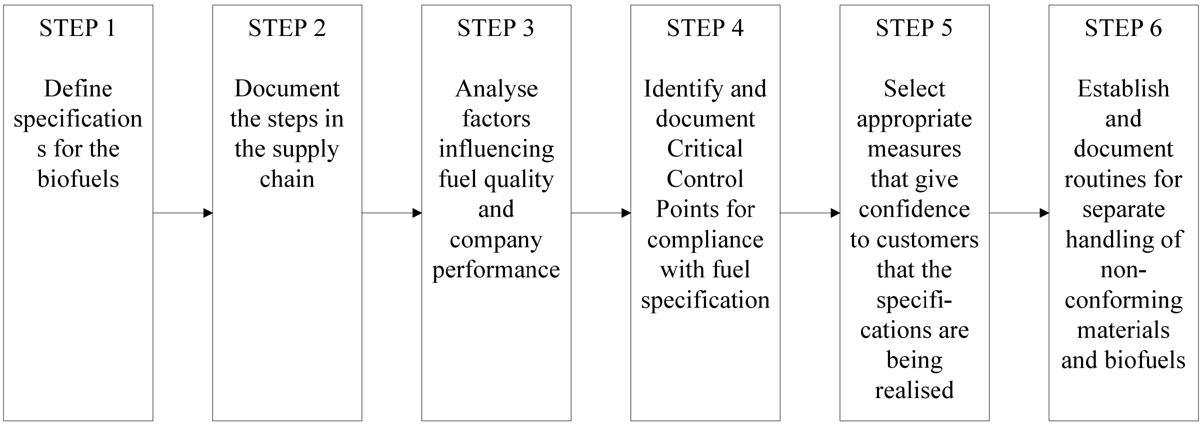
ABSTRACT: To increase the use of solid biofuels and permit a long term use of different available biomass a consistent and homogenous fuel quality has to be guaranteed. Identification of fuel supply chains, of customer requirements, biomass potential and aggravating circumstance e. g. different or missing environmental laws are the first steps of work to elaborate quality measures which are described by Quality Planning (QP). The assessment and evaluation of this information gives an overview about the demand of the quality assurance and quality improvement to ensure the planned fuel quality. This paper provides details about the quality terms, the evaluation of the actual state of different biofuels and the quality influencing factors defined by different supply chain players. Moreover an example is presented of supply chains of different biofuels with a description of the most important critical control points. Based on these results of WP 3, each fuel producer should recognise the advantages that accrued of Quality Planning.

Keywords: Biofuels, Critical Control Point (CCP), Quality Planning, Quality management, supply chain

1 INTRODUCTION

The research work in BioNorm II is partly directed to fundamental work to complete the description of Quality Management for solid biofuels. The development of a procedure for Quality Planning and Quality Improvement is also integrated to the standardisation work of CEN TC 335 WG2 for the upgrade step of the Technical Specification to European standard.
A "Procedure for Quality Planning" will give an overview about relevant quality measures within the supply chains of different biofuels. The following biofuels: wood pellets, wood chips, hog fuel, straw and olive residues cover the most common and also new and potential biofuels. Therefore relevant information about end-user requirements, quality influencing factors, Critical Control Points, appropriate test methods and the description of the supply chains was compiled by four national teams. This work was performed on basis of the 6-step-procedure (described in figure 1).

Figure 1: 6-step-procedure for Quality Planning (QP) [1]



2 THEORY OF QUALITY MANAGEMENT AND THE DEFINITION OF QUALITY TERMS

Quality Management (QM) is based upon 4 quality elements Quality Planning (QP), Quality Control (QC), Quality Assurance (QA) and Quality Improvement (QI) which are key stages for the PDCA-Cycle. PDCA is an abbreviation for Plan, Do, Control and Act. First, the desired quality improvements are planned, afterwards the planned actions are applied (Do). The product quality is assessed (Control) quality improvements are planned. In the "Act" stage, the effects of the applied changes are assessed. Based on these results, further improvements are planned, so the cycle continues iteratively. [2]

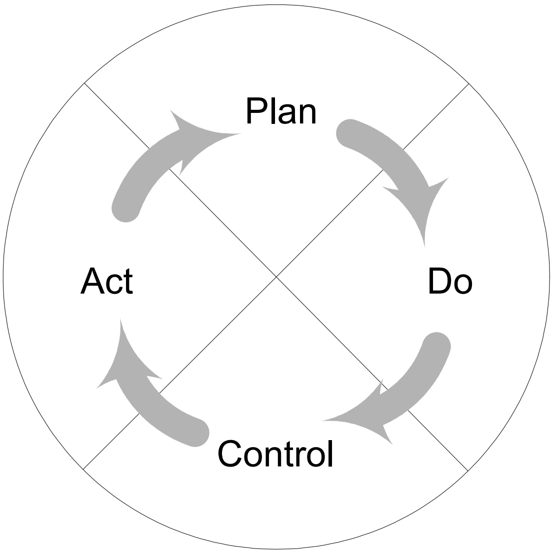


Figure 2: PDCA-cycle

In practice the PDCA-cycle is often adopted to the company conditions and the planning part is carried out by the management along with resource management. Then the product is realised (Do). Measurement and analysis are the "Control" part. The management responsibility represents the "Act" part.

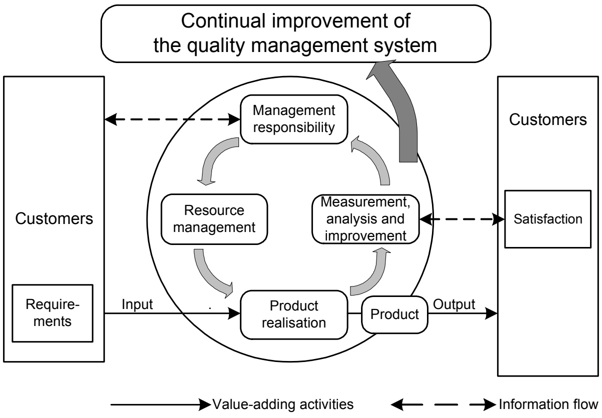


Figure 3: PDCA-cycle [2]

According to ISO 9000:2006 Quality Planning is "the part of quality management focused on setting quality objectives and specifying necessary operational processes and related resources to fulfil the quality objectives". [2] For this Task the procedure for quality planning includes the status quo analysis, product requirements along the supply chain, Critical Control Points, most appropriate test methods, costs as well as necessary arrangements for provision, transport and storage along the whole supply chain.
Potentials for quality improvement are identified and quality objectives (e. g. ash content) are set to meet customer requirements. Necessary steps to fulfil them (e. g. change the raw material) are defined. [3]

Quality Planning also includes the discovery of hidden customer needs and the translation of these needs into the production process, as well as the design of the process in the most efficient way.
In a simplified manner, Quality Planning is the first step for implementing quality measures in a company. QP describes the actual facts about the fuel (or raw material), helps to describe the supply chain and define the critical control points. QP also allows to set control mechanisms and improvement tools to guarantee a required fuel quality.

3 METHODOLOGY

3.1 Status quo analysis

For the evaluation of requirements and the description of the different supply chains it was decided to collect relevant data with the help of a poll. To get an overview about the current situation within the supply chains different actors, mainly end user, were interviewed by all 4 national teams in different ways. Depending on the fuel some interviews were performed by phone calls e. g. for wood pellets because of the widespread and common fuel, or by visiting the site owner in foreign countries e.g. for olive residues in Spain because of the rare use combined with translations problems or by visiting the fuel producer nearby the own institute.
End-users like power plant operators or boiler owners who use biofuels for combustion purposes were interviewed about used techniques, demands on and to which standard the used fuel quality complies. In addition to this information the interviewees were asked about logistic issues, storage management, quality improvement devices like sieves, etc. The questionnaires also included questions about the size of the combustion unit and the consumption of fuel per year.
Besides of the fuel producer and user, also boiler producers were interviewed in order to provide an overview about current boiler techniques and specifications of biofuels from their point of view. Additional interviews with fuel distributors and also information from other projects and literature completed the data collection.
For the realisation of the poll questionnaires considering the chosen biofuels, wood pellets, wood chips, straw, hog fuel and olive residues were developed for boiler producers and for end-users. They were tested by some of the interviewees. After this first test-run the two types of questionnaires for different solid biofuels were adapted. For harmonisation and optimisation, the questionnaires were exchanged between the different workgroups. The results of this poll with about 80 interviewees (see table 1) was the basis for all subsequent tasks of WP 3.

Table 1: Number of performed interviews [4]

	end user	**boiler producer**	**others** (e.g. fuel producer, fuel distributor)
performed interviews	45	17	7
data from earlier project works	2	1	4
data from literature			2

The assessment of data was performed and evaluated separately by each national team for each fuel. Some general and specific information was gathered out of this poll. For the most interviewed supply chain players (fuel producer, but also boiler producer and fuel user) there are two very important parameters influencing the fuel quality of all kind of fuels: the moisture content and the impurities. The interviewees often could not explain what kind of problem a high content of impurities means, but out of their experience this could cause disturbance during the combustion process.

There also exists special quality influencing fuel parameters just for special fuels e. g. moulding or fungi of straw, so a complete supply chain with quality influencing factors cannot be described in general

Also the comparison of these results with results of the work packages: chemical and physical analysis shows that these two parameters can cause serious troubles. The following figure shows the results of the poll for end users of wood pellets.

The assessment and evaluation of these data was a milestone in the project delivering information for description of supply chain, for the definition of quality influencing factors, for definition of appropriate test methods etc. But also information about laws and the amount of biomass was very helpful to understand and explain why the use of new biofuels could be relevant for future. For example, the global acreage of olive trees is 8,6 million ha with over 17 million tons of olives per year. About 30% of these olives remain after the olive-oil-production as waste, which could be used e.g. for heat production. So this is a very potential biofuel source. [6]

3.2 Description of supply chain and definition of quality influencing factors

The description of a supply chain as part of Quality Planning for a fuel is an essential element to get an overview about the actors within the supply chain, the material flow and the single process steps. After that the definition of quality influencing factors should be performed. These are points within the supply chain, where the quality can change because of changing raw material, or potential sources of impurities (e.g. storage on sand), or not optimised process steps (e.g. insufficient drier) etc.

This information can be completely incongruent for different solid biofuels, but general quality influencing factors are often similar within the different sorts of fuels and include factors like storage, transport, processing of raw material and final product as well

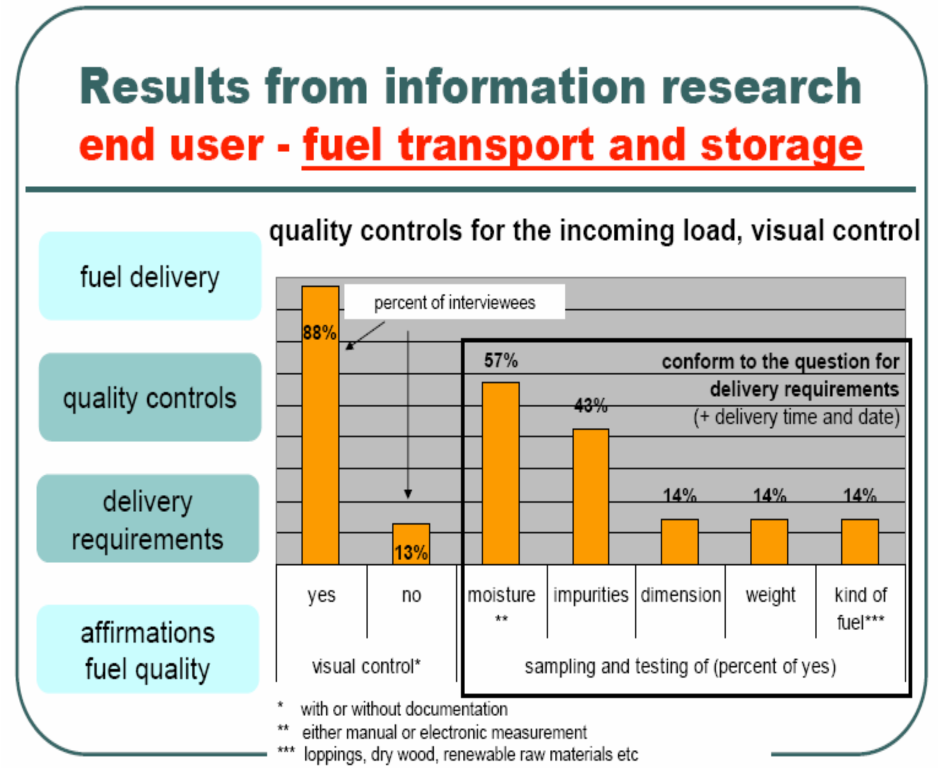


Figure 4: Results of the pool for pellets end user [5]

as selection and collection of raw material. Quality influencing factors could be the weather and weather conditions, the type of storage, raw material, drying, etc.

After identification of quality influencing factors the definition of Critical Control Points (CCPs) should be performed. CCPs are points within the supply chain were the fuel quality can be influenced and should be controlled.

A good description of supply chain allows a good identification of quality influencing factors and of CCPs. In principal quality influencing factors can be generalized to the single process steps, depending on the respective biofuel with varying focus.

3.3 Critical Control Points and appropriate test method

With the definition of CCPs the quality planning process is nearly closed. The CCPs describe the points within the supply chain were the quality influencing factors define the parameters which should be controlled to guarantee a special fuel quality.

The QP should also include the appropriate test methods. Depending on the fuel parameter and the CCP one or more test methods can be used.

The appropriate test method for moisture content is the method according to prEN 14774 Determination of moisture content [7], but the analysis time is up to 10 hours or more. This is, why this method is often not the appropriate one for on site quality control. Therefore a lot of alternatives, faster and often cheaper methods exist. The analysis accuracy of this method is not as good as the accuracy of the standardised method, but often sufficient for on site quality control.

Alternative test methods do not exist for all other fuel parameters, some are under development.

Table 2: Test methods for moisture content measurement [8]

Principle	**Technique**	**Instrument (example)**
Thermo gravimetric analysis	Drying oven Duration ca. 6-24h	Memmert UF series
		Nabertherm TR series
	IR drying Duration up to 1h Working range 1-100% Sample Mass below 100 g	Sartorius MA series
		Kern ML series
		Mettler-Toledo HG63, MJ33
		A&P Ultra X series
	Microwave drying Duration few minutes Working range 8-100% Sample mass below 70 g	Sartorius LMA200
		Anton Paar Multiwave 3000
Electric analysis	Conductivity Duration a few seconds Working range 5-50%	Gann Hydromette Series
		Testo 606
	Capacity Online measurement possible Working range 0-100%	Pandis FMG 3000
		Schaller Humimeter pellets analyzer
		Liebherr Litronic FMS II
	Microwave absorbance/reflectance Online measurement possible Working range 0-100%	hf-Sensor MOIST series
		Sartorius PMD 300
	Time-Domain-Reflectometry Online measurement possible Working range 0-100%	Imko Trime-GW or Trime-TDR
Optical analysis	IR reflectance Online measurement Working range 0-90%	Sartorius LMA500
		Moist-Tech IR-3000
		Pier Reflexionsphotometer

Besides of the moisture content especially impurities were mentioned in the polls to cause combustion problems. For impurities no appropriate test method exists up to now. Experienced fuel producer know the CCPs were impurities can have bad influence of fuel quality, but new fuel producer has no possibility to measure the impurities. There are no methods to analyse impurities, because impurities can also be snow or sand and it is difficult to measure something that is naturally part of the biofuel. To guarantee a good biofuel quality it is important to protect the fuel from weather effects, also from contamination with earth, sand or stones and to make aware of the danger of impurities. Other kind of impurities like nails or metals in log wood can be easily removed by a magnetic separator, but these impurities are not a natural part of biomass.

The contamination of material can happen everywhere, beginning with the transport of log wood from the forest, where soil can cling on the log and the bark; up to old loads on trucks and lorries or old material in the storage room. If the delivered raw material is already contaminated, the quality of the biofuel can not be really enhanced. Some producers of biofuel try to wash the raw material to get the grains of sand and earth out of the material, but this method is very expensive and not really effective.

So to guarantee a good fuel quality, the raw material should already have a high quality and all kinds of contamination should be prevented. Precautions like good and protected storage rooms, clean transport machines, carefulness during the harvesting and use of properly maintained material can strongly influence the fuel quality.

The description of CCPs was one of the milestones of the work package 3, but this information is just applicable with data about the analysis costs. So in addition to the planed and proposed work a research about the average laboratory- and equipment costs completed the evaluation of the QP-task (see BioNorm II- WP3- DIII.8 Procedure for Quality Planning).

4 CONCLUSION

Quality Planning is one of the 4 elements of quality management and represents the first step to a living quality system. QP can give an overview of the Critical Control Points and the fuel influencing factors. With the estimation of analysis costs the costs of the quality control system to ensure a special quality can be calculated.

Additionally, the results of this work can be integrated into the Quality Assurance standard prEN15234 for solid biofuels.

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6 ACKNOWLEDGEMENTS

This work has been performed as part of Work Package III of the BioNorm II project. Following institutes and companies were responsible fore the elaboration of the results Bruins & Kwast, DBFZ, Halm80, KWB, PLANT Engineering, ofi, Sparkling Projects and Teknologisk Institut. We kindly express our gratitude for their contributions.

Appendix A

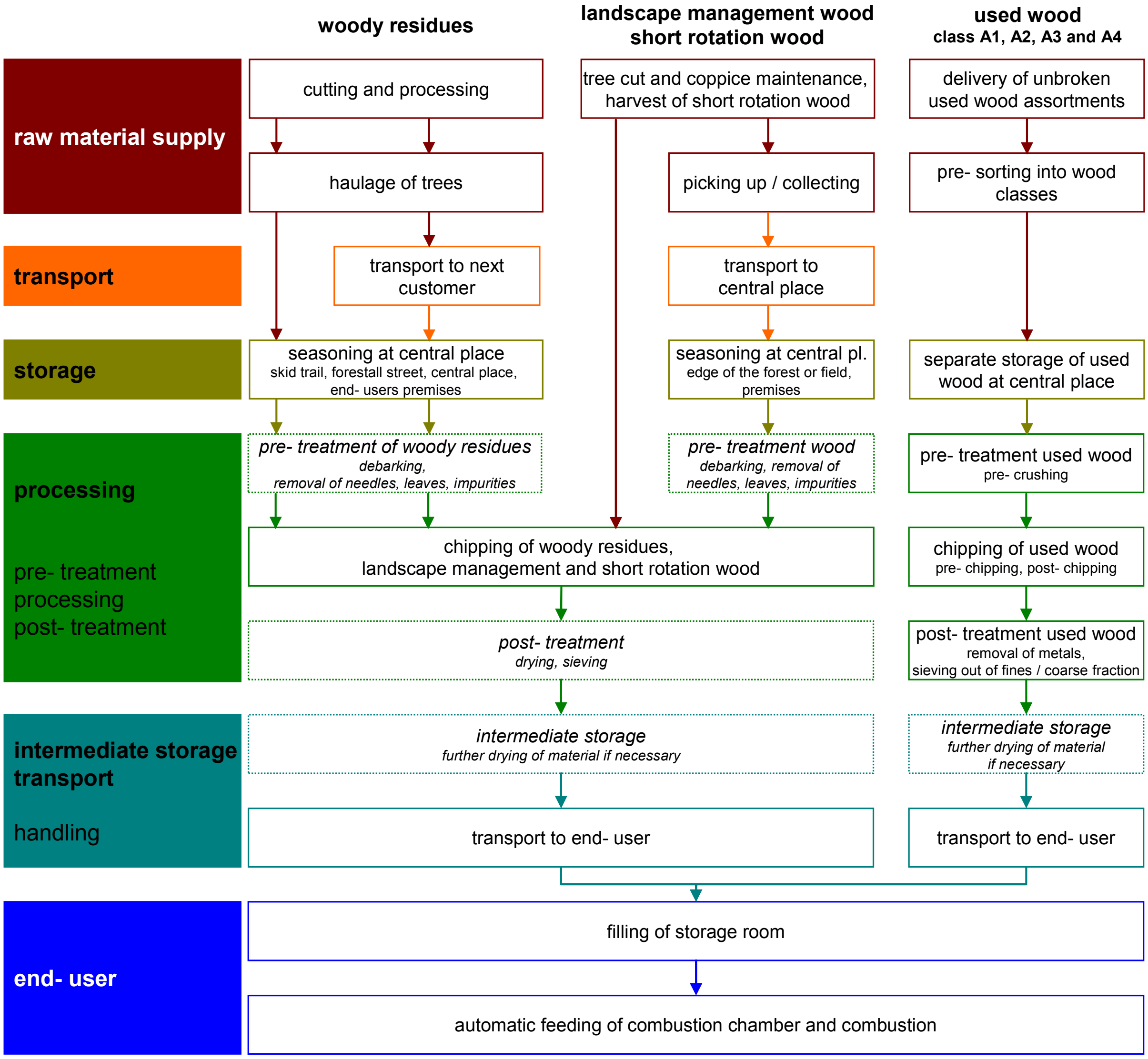

Figure 5: Supply chain of wood chips [6]

5.2 IMPLEMENTATION OF QUALITY IMPROVEMENT

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ABSTRACT: Quality improvement is one of the main issues in quality management. For the supply of biofuels, it implies that there is a continuous thrive for identifying locations in the fuel supply chain where quality is affected in a negative sense, and consequently mitigating these influences by implementing cost effective process modifications or control measures. This paper provides a number of examples that illustrate how this can be done in practise. Based on the results obtained, the challenge now is to implement the findings in an operative way; the examples elaborated (wood chips, straw etc.) are intended to serve as important tools for the standardisation work of WG2 of CEN TC335.

Keywords: biofuels, improvement, quality, supply, chain.

1 INTRODUCTION

Specific tasks within the BioNorm II project have been allocated to the issue of Quality Improvement of solid biomass fuel supply. It is investigated within these tasks how Quality Improvement is done in the most relevant biomass supply chains (wood pellets, wood chips, hog fuel, straw and olive residues) and which specific aspects related to biofuels should be considered when implementing Quality Improvement in existing biomass supply chains.
This paper reflects the results of the analysis of the Quality Improvement issues within biomass supply chains.

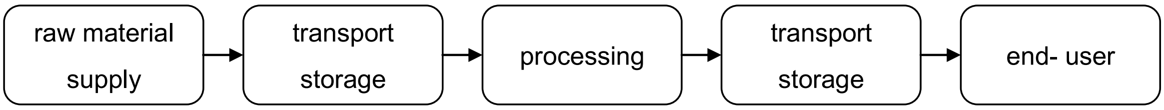

Figure 1: General description of a supply chain for solid biofuels

2 METHODOLOGY

2.1 Biomass supply chain

Single steps of a general supply chain (see figure 1) have been defined for each investigated biofuel (wood chips and pellets, hog fuel, olive residues and straw).
When considering Quality Improvement, the following questions are of main importance:

1. Which fuel parameters are considered important by the next customer in the biomass supply chain?
2. Which typical requirements exist in absolute terms and what deviations are allowed?
3. At which locations in the process these parameters can be best sampled, tested and influenced through modification of the production process?
4. What sampling and testing methods and/or other quality assurance/quality control measures should be applied at these locations considering costs and quality improvement potentials?

2.2 Critical control points

Although the biomass supply chains seem completely different, general quality influencing factors are often similar within the different sorts of solid biofuels and include factors like storage, transport, processing of raw material and final product as well as selection and collection of raw material. In principal quality influencing factors can be generalized to the single process steps, which is illustrated in figure 2.

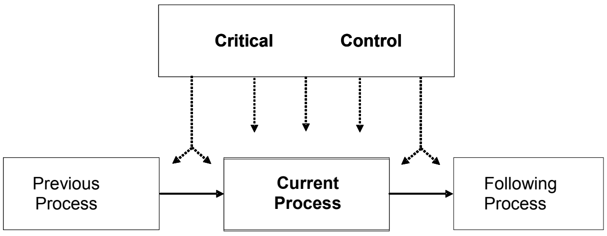

Figure 2: Possible positions of Critical Control Points within the single steps of a supply chain [2]

With the help of this supply chain structure, Critical Control Points (CCPs) have been identified in order to figure out where fuel specifications can be influenced and determined easily and with a great potential for quality improvement.

In the next step, a more detailed examination of CCPs was compiled with regard to quality influencing factors and sampling and test methods. Therefore, practical information and experiences were gathered. This information was organised in a way that made it possible to quantify the influence of the quality influencing factors in the process on normative and informative biofuel parameters given in CEN/TS 14961. This procedure should help to clarify to what extent the CCPs are useful or to clarify the possibility to reduce the amount of CCPs to the main important process steps.

Fuel parameters in the draft version have been restructured according to the normative and informative fuel parameters of CEN/TS 14961 (Fuel specifications and classes) and other relevant parameters. Furthermore the option to score the relevance of the quality influencing factor for each fuel parameter was included at each process step. Last but not least the sampling and testing methods as well as the quality control measures have been related to each fuel parameter.

This template of a CCP table was afterwards provided to all national teams of BioNorm II, WP III in order to be completed. The CCP table was regarded as a working sheet, which aims at:

- Evaluating the influence of the CCPs in the biomass supply chains on product quality
- Evaluating appropriate sampling and testing methods and optimal locations in the supply chain
- Evaluating quality improvement potentials for each biomass supply chain

The evaluation was done by filling in the CCP table for each biomass supply chain. By filling in the CCP table for each biomass supply chain, a good overview at the CCP's has been obtained of:

- The relevance of the quality influencing factors
- The frequency & costs of sampling and testing
- The current and alternative quality control measures
- Documentation, work instructions, etc.

The results of this comprehensive exercise have resulted in a detailed overview of the fuel quality improvement potential, and particularly of:

- Alternative, cost effective sampling and testing methods
- Additional available quality control measures ranked with respect to the importance of the quality influencing factors.

As an example, the results for wood pellets of this exercise are presented in the next chapter.

3 QUALITY IMPROVEMENT EXAMPLE: WOOD CHIPS

More or less well established wood pellet standards exist European wide on the market like e. g. ÖNorm M 7135 from Austria or DINplus from Germany. In the near future, a multipart European standard for the specification and classification of solid biofuels (prEN 14961-1), including product standards for non-industrial used biofuels, will replace national standards. Despite of production improvements in the last years, experiences and approaches of producers to optimise the pellet production process are differently distinctive. However, this is also the result of the development of national markets and the accompanied supply-demand-situation for pellets. On the one hand high-tech pellet units with high demands on the biofuel in central and northern Europe are sold; on the other hand rudimentary boilers are partly running in other European regions, which are also suitable for other solid biofuels than wood pellets. From that context partly strongly different wood pellet qualities result. There is also the fact of increasing the pellet production capacities which is accompanied by additional raw material need. With the additionally required raw material also the interest on alternative raw materials as well as the international biofuel trade in Germany, Europe and worldwide increases. Apart from this strength to strength happening European and worldwide market development for wood pellets a furthermore discussion about growing using competitions of biogene resources is held. In that context it is important to define standardised agreements about qualities to be traded on the one hand and about mechanisms for controlling required qualities on the other hand in order to fulfil quality standards in spite of broadened raw material range and widening of purchasing areas anyway. Meant by this are clear agreements, fixing of responsibilities and handling over interfaces between producer, supplier and end-user, which enable a pursuit of material flows.

Furthermore regularly controls within the supply chain at control points, where the quality of the product can be influenced significantly, are essential.

In order to fulfil sustainability standards and to exclude inappropriate raw materials it is moreover essential to document the origin of raw materials. For pellet producer it is relevant to know that the first operator is responsible for the documents being prepared the first time. The documents shall be available and provided on justified request throughout the entire supply chain. In that context the first operator in the supply chain is a body or enterprise, which operates at the beginning of the supply chain.

For optimisation of the wood pellet supply information about influencing factors, quality control points, test methods and frequencies are given (see below). For instance the use of appropriate wood materials for pellet production can consequently be influenced during supply or harvest by choosing only fresh and healthy wood. If the wood is not debarked for pellet production, mineral impurities of the bark have to be avoided, in order to prevent an increase of the ash content beyond the natural degree.

Further considerable aspects are transport and storage of all intermediate and final products within the pellet supply. The avoidance of impurities through transport and storage as well as the avoidance of moistness into the pellets are important quality criteria. As all handling processes result into the development of abrasion, which causes an increase of the amount of fines as well as the danger of dust explosion, it is essential to reduce the amount of fines regularly to < 1% by sieving.

Within the real pellet production regularly carried out product controls are a necessary and important part in order to check the wood pellets' quality and to make adaptations to the production process if necessary. Only through this procedure a continuous production of standardised quality which is required by the end-user is possible.

The following example mirrors a representative example of a pellet production chain. In individual case single process steps might differ from the ones shown in the draft. The aim of quality assurance for wood pellets is to create European wide awareness for the fact that required quality from end-user need a consequent control along the whole supply chain. For this it is not important if the required quality refers to standards or individually met agreements. It is rather about delivering demanded quality to the end-user and therefore meet transparent and fixed down in writing agreements as well as to prove customers that the production of a high quality product has highest priority in the company.

Production requirements according to prEN 15234-1 are subdivided into six consecutive steps.

Step 1: Fuel specification of the final product

Step 2: Production description (documentation of steps in the production chain)

Step 3: Quality influencing factors including company performance

Step 4: Critical Control Points for compliance with the fuel specification

Step 5: Measures to give confidence that the specification(s) is/are being realised

Step 6: Routines of separate handling of nonconforming materials and biofuels

The following information will give a general overview about documenting the requirements for the production in a wood pellets supply chain.

Step 1. Fuel specification for the final product

The fuel specification is based on prEN 14961-1 general part, table 4 in case of individually met agreements. Quality pellets are produced according to prEN 14961-2, product standard for wood pellets.

Steps 2-5. Production description → Quality measures

The flow sheet in Appendix A is a general example, which gives an overview about the whole supply chain for wood pellets. Relevant single process steps in the supply chain are illustrated. In individual cases of wood pellet suppliers, the flow sheet for one single process step should be illustrated in more detail.

Appendix B summarises steps two to five and give information about quality influencing factors (step 3), Critical Control Points (step 4) and appropriate quality measures (step 5) for each single process step (see figure 1).

Step 6. Routines for separate handling of nonconforming materials and biofuels

If wood pellets are not fulfilling the requirements, these batches have to be stored separately from conforming biofuel. All necessary information has to be filed. If nonconformity is discovered at the premises of the consumer in connection with delivery, a nonconformity report is generated and handling of the nonconforming lot is agreed with the consumer.

4 IMPLEMENTATION OF QUALITY IMPROVEMENT

4.1 Evaluation of quality improvement analysis

In summary a lot of important aspects concerning, Critical Control Points, quality influencing factors and appropriate test methods have been collected for different biomass fuel supply chains.

The example of wood pellets shown in this paper illustrates the possibility to systematically analyse a given fuel supply chain, how quality is influenced and what can and should be done to improve quality of the final product. The methodology used in this evaluation helps a fuel producer to identify Critical Control Points in the production process, where there is significant influence or opportunity to improve on one or more quality parameters of the end product. The following steps were taken in each analysis:

1. Define the process chain in different process steps
2. Define product quality in both normative and informative fuel parameters
3. For each process step, determine on a scale from 1-10 to what degree external and internal influencing factors may exist that influence each of the relative fuel parameters in the matrix
4. The result is a clear overview of what process steps and external influences are crucial in the production process.

4.2 Implementation in the CEN standardisation process

The overall aim of BIONORM II has been to carry out pre-normative research on solid biofuels in order to support the 2nd Working Group (WG2), established from CEN TC 335. Thus the work within BIONORM II – and indeed the work on quality improvement - has been continuously adjusted according to in-put and feed-back from WG2.

Based on the results obtained, the challenge now is to implement the findings into the standardisation work in an operative way; the examples elaborated (wood chips, straw etc.) are intended to serve as important tools for the WG2.

BioNorm II ends by December 31st 2009, whereas the CEN standardisation work continues in 2010. Therefore it is important, that the results of BioNorm II is presented in a way and somehow left open for WP2 to adapt it in their work.

4.3 Further opportunities for Quality Improvement

Besides forming the basis for the CEN work, BioNorm II results should be applied more directly within the biomass sector when possible.

Many experiences, which are not suitable for use in the CEN work, has been gathered in the project from actors within the sector (e.g. straw producers, transport companies, consumers). As many countries in these years make plans for the increased use of biomass in the energy sector, such hands-on experiences and results should be disseminated on another level in order to prevent the same “beginners mistakes” to be repeated. This is especially important, because there may be large differences between different fuel types as to the possibilities of quality improvement; differences which are difficult to describe in detail in standards, making the use of the standards themselves a challenge to use for the individual actor.

This dissemination could be in the form of manuals or handbooks for producers, operators and consumers of solid biofuels.

In the following Appendices A and B of this paper the reader can find a draft for the practical integration of a biofuel supply chain in accordance to the pre EN 15234-1. This example can be seen as one result of the work of BioNorm II, WP III and was sent to CEN TC 335/WG2.

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Appendix A

Example of documenting requirements for the production of wood pellets according to prEN 15234 [7]

Step 1: Fuel specification for the final product

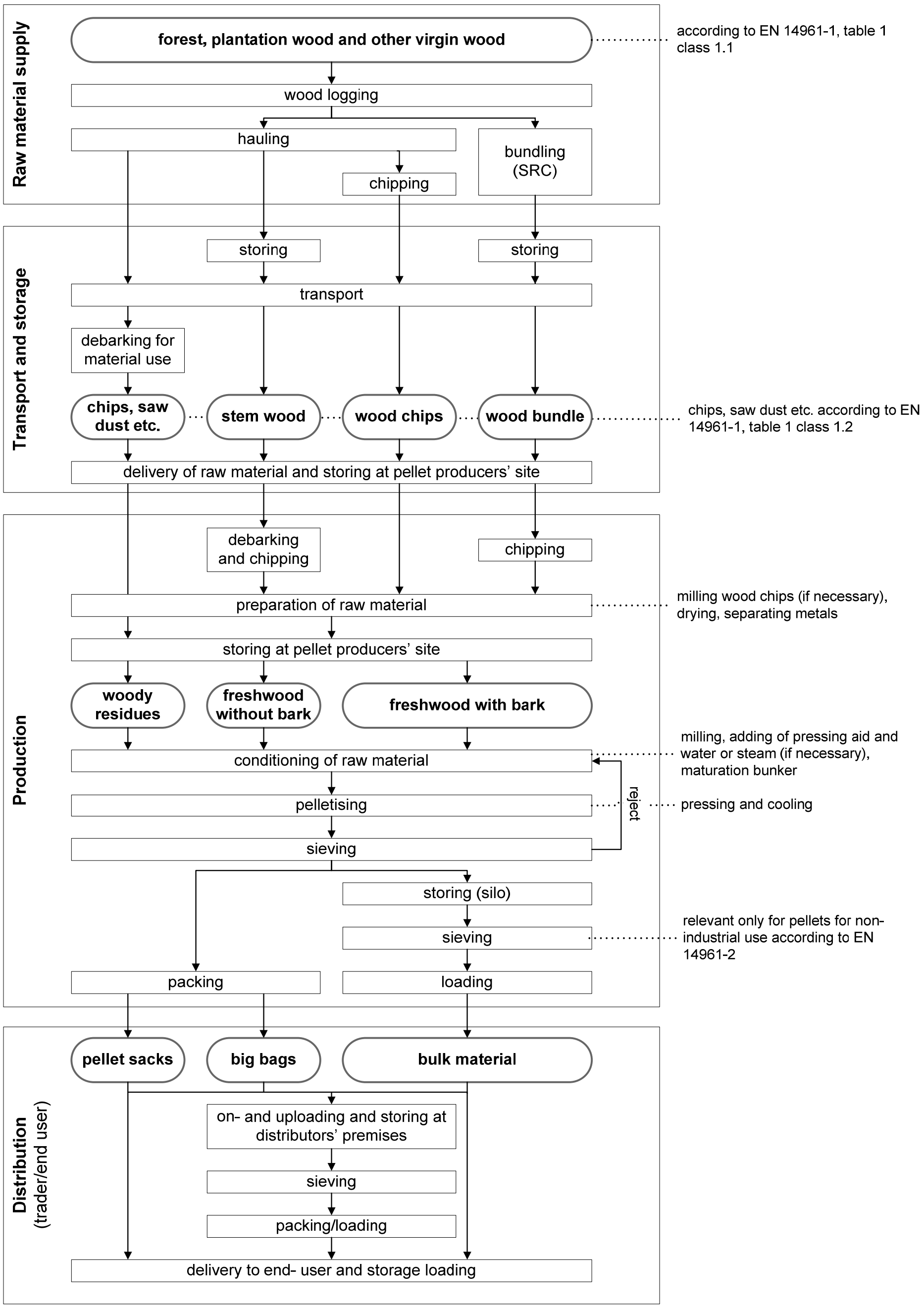
The fuel specification is based on prEN 14961-1, table 4 in case of individually met agreements. Quality pellets for non-industrial use are produced according to prEN 14961-2, product standard for wood pellets.

Step 2: Production description (documentation of steps in the production chain)

The following flow sheet is a general example, which gives an overview about the whole supply chain for wood pellets. Relevant single process steps in the supply chain are illustrated. In individual case the flow sheet for one single process step must be illustrated more detailed.

NOTE The illustration given is only a general example. The individual process chain might look differently at single process steps.

NOTE The location, where raw material for wood pellet production is harvested has an influence on single parameters. Big differences with regard to e. g. silicates, which influence the ash content, occur European wide. This has to be taken into consideration when illustrating a supply or rather production chain and its respective design.

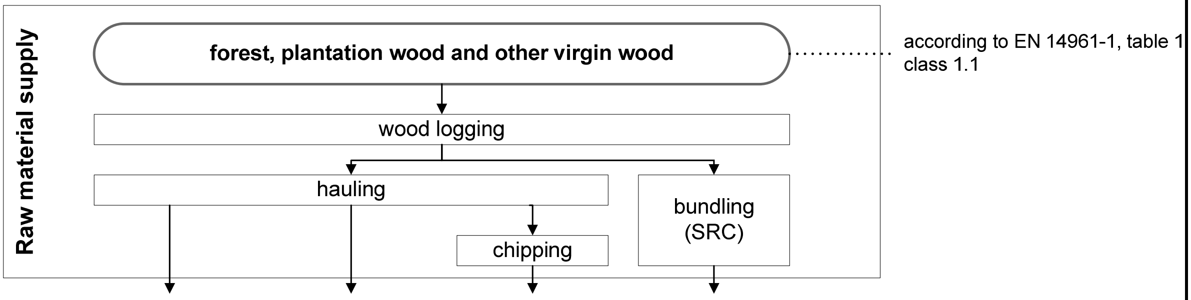


Appendix B

The following tables summarise steps two to five in detail and give information about quality influencing factors (step 3), Critical Control Points (step 4) and appropriate quality measures (step 5) for each single process step:

[9] Raw material supply
[10] Transport and storage
[11] Production
[12] Distribution

Step 2: Production description – Raw material supply



Step 3: Quality influencing factors

- species of wood / origin
- quality of wood [a]
- impurities [b]

Step 4: Critical Control Points

wood logging

Step 5: Measures to give confidence to customers that the specification(s) is/are being realised

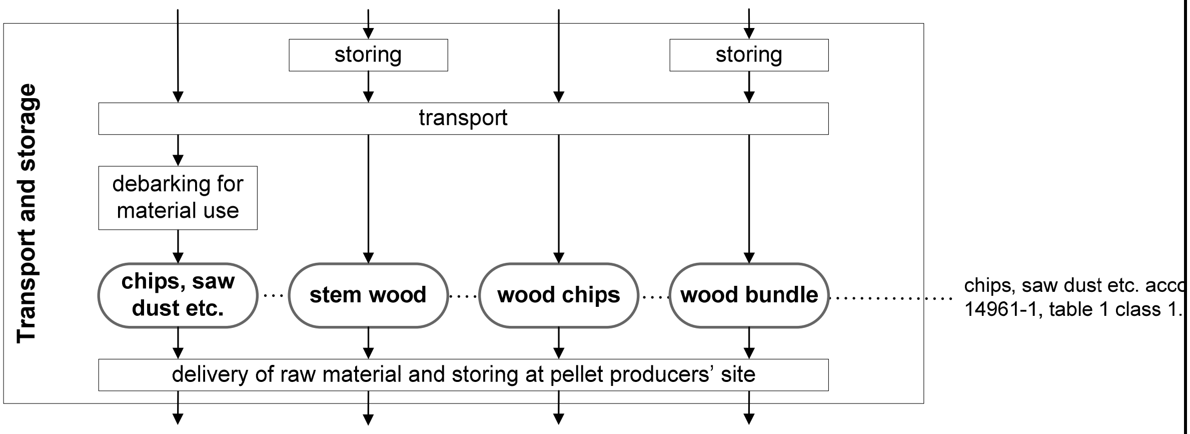
- carry out visual or other sensory inspections across the whole raw material supply process,
- singular testing of soil, where the raw material is generally harvested from, in advance gives important information about slagging elements in the wood and helps to calculate required pellet quality,

NOTE. This measure is on voluntary basis and especially useful when new pellet plants are built and the area where the raw material is mostly harvested from is known.

- only fresh and healthy wood without a beginning decomposition and without insect damage or insect attack will be used,
- contract between supplier and producer with "terms of delivery" including fuel specifications and biofuel handling.

[a] Quality of wood:	Fresh and healthy wood without beginning decomposition and without insect damage or insect attack. In mixtures also used wood can be used, which is not demolition wood.
[b] Impurities:	Material other than the fuel itself e.g. stones, soil, pieces of plastic, rope, ice and snow (see EN 14588)

Step 2: Production description – Transport and storage



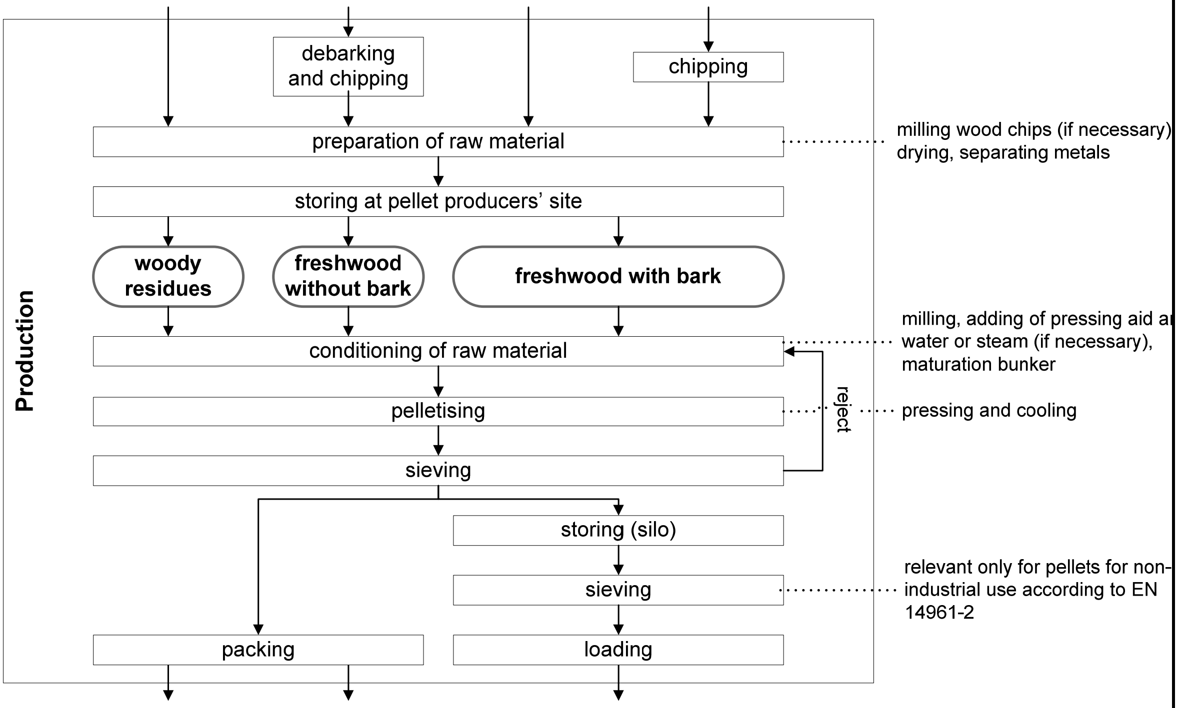
Step 3: Quality influencing factors	Step 4: Critical Control Points
• quality of wood [a)] • impurities [b)] • weather conditions [c)] • storage time • bark	storing of whole trees and wood bundles storing after delivery of saw dust, stem wood, wood chips and/or wood bundles at pellet producers' site
• cleanliness of transportation unit	transport of saw dust and/or wood chips
• checking material quality • species of wood / origin	delivery of saw dust, stem wood, wood chips and/or wood bundles

Step 5: Measures to give confidence to customers that the specification(s) is/are being realised

- carry out visual or other sensory inspections across the whole transport and storage process,
- storage areas have a clean and solid underground, which prevents an entry of impurities into the biofuel,
- storage areas are roofed or protected by tarpaulin or fleece to prevent an entry of moisture into the biofuel,
- transport equipment has to be free from impurities and has to protect the loading from rain or humidity,
- storage and transport equipment is checked regularly,
- contract between supplier and producer with "terms of delivery" including fuel specifications and biofuel handling.

[a)] Quality of wood:	Fresh and healthy wood without beginning decomposition and without insect damage or insect attack. In mixtures also used wood can be used, which is not demolition wood.
[b)] Impurities:	Material other than the fuel itself e.g. stones, soil, pieces of plastic, rope, ice and snow (see prEN 14588)
[c)] Weather conditions:	Temperature and rain influencing drying of wood and impurities like sand, stones etc., e. g. due to muddy underground after rain or humidity

Step 2: Production description – Production



Step 3: Quality influencing factors	Step 4: Critical Control Points
• quality of wood a) • impurities b) • degree of dryness	Chipper
• impurities b) • weather conditions c) • quality of wood a) • bark	storing at pellet producers' site
• quality of wood a) • impurities b)	conditioning of raw material with hammer mill
• dust formation • impurities in the transportation unit • suitability of the transportation unit	loading station
NOTE: The end user shall receive wood pellets with an amount of fines ≤ 1% and which don't cause to much dust when delivering them. Additionally wood pellets have to be clean and free from impurities. For that purpose the transportation unit must be suitable for the transport of pellets and must be kept free from impurities in case of open transportation units.	
• filling velocity	packing station

Step 5: Measures to give confidence to customers that the specification(s) is/are being realised

- carry out visual or with sensory inspections across the whole production process,
- measurement of certain properties after the raw material basis has changed on a frequency appropriate to the process requirements;
- equipment is repaired or changed when necessary; some parts will require changing regularly according to their technical life time or the production control system,

- regularly calibration of humidity measurement on dryer

NOTE: In many drying processes the online humidity measurement after the drying process is an important debit value for the Process Light System of the dryer to control the belt speed e. g. for belt dryers This humidity measurement system has to be calibrated. It starts with a weekly control. If no adjustments are necessary the control can be performed every two weeks, every four weeks etc. Vibrations influence the measurement systems and lead to wrong measurement values after a while. The result is too wet or too dry saw dust in the storage.

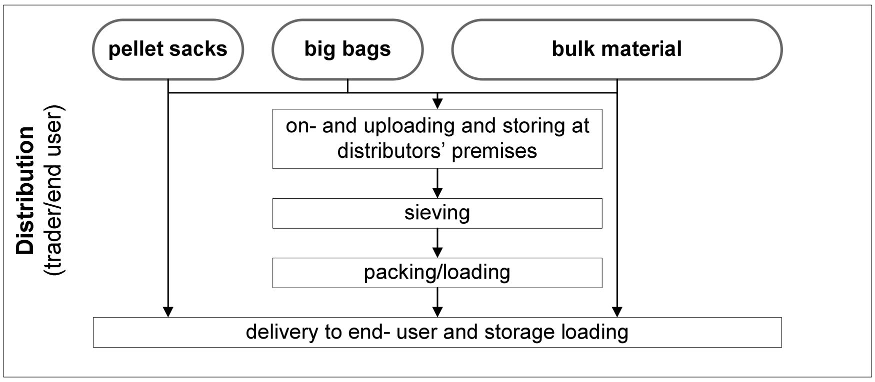
- agreements between supplier and customer with regard to biofuel handling and quality,
- wood pellets are protected from moistness caused by snow, rain humid stonework or rather condensation moistness through a suitable storage
- storage areas, silos and haulage plants where wood pellets are handled, are kept free from impurities
- determination of properties after production
  1) using typical values, e. g. laid down in annex B of the prEN 14961-1, or obtained by experience;
  2) calculation of properties, e.g. by using typical values and considering documented specific values;
  3) carrying out of analysis: a) with simplified methods if available, b) with reference methods.
  amount of fines: prEN 15149-2
  dimensions: new work item in CEN TC 335 to come
  moisture: prEN 14774-1 to -3
  mech. durability: prEN 15210-1
  ash: prEN 14775
  net calorific value: prEN 14918,
- mechanical durability, dimensions, moisture content are controlled every two to four hours,
- ash and net calorific value are checked if required,
- sieving and checking of fines every time pellets are filled into the silo; amount of fines ≤ 1% according to prEN 14961-2 unless no other agreements are met,
- pellet retain samples are taken every time pellets are loaded from silo to transportation unit,
- pellet retain samples are preserved approx. half a year at winter or spring delivery and approx. one year at summer delivery,
- production control, conditions and adjustment of the equipment (e. g. ampere of presses, temperature of koller bearings, vibration of presses).

[a] Quality of wood: Fresh and healthy wood without beginning decomposition and without insect damage or insect attack. In mixtures also used wood can be used, which is not demolition wood.

[b] Impurities: Material other than the fuel itself e.g. stones, soil, pieces of plastic, rope, ice and snow (see prEN 14588)

[c] Weather conditions: Temperature and rain influencing drying of wood and impurities like sand, stones etc., e. g. due to muddy underground after rain or humidity

Step 2: Production description – Distribution



Step 3: Quality influencing factors	**Step 4: Critical Control Points**
• dust formation • impurities in the transportation unit • air pressure in case of unloading with pressurized air • suitability of the transportation unit	on- and uploading at distributor's premises
• filling velocity	packing-station
• remaining material inside storage room • length of injection tube • material of hose (especially inside) • amount of bows • injection pressure	storage loading at end user's premises

Step 5: Measures to give confidence to customers that the specification(s) is/are being realised

- carry out visual or with other sensory inspections across the whole distribution process,
- storage areas, silos and haulage plants are cleaned completely before conveying or storing pellets if other materials than wood pellets have been handled before,
- storing areas, where wood pellets are handled, are roofed
- transportation unit is constructed in a way that protects wood pellets from moistness
- moisture content analysis before delivery to the end user after a long time intermediate storage,
- sieving of fines, amount of fines ≤ 1% according to prEN 14961-2 unless no other agreements are met,
- determination of properties after delivery from pellet plant for loose material if required and when pellet distributor changes
  1) using typical values, e.g. laid down in annex B of the prEN 14961-1, or obtained by experience;
  2) calculation of properties, e.g. by using typical values and considering documented specific values;
  3) carrying out of analysis: a) with simplified methods if available, b) with reference methods.
  amount of fines: prEN 15149-2
  dimensions: new work item in CEN TC 335 to come
  moisture: prEN 14774-1 to -3
  mech. durability: prEN 15210-1
  ash: prEN 14775
  net calorific value: prEN 14918,

NOTE This measure is on voluntary basis.

- storage and transport equipment is checked regularly,
- air pressure during unloading is to be checked and stated in the supply protocol,
- contract between producer and distributor including fuel specifications and biofuel handling

Step 6. Routines for separate handling of nonconforming materials and biofuels
If wood pellets are not fulfilling the requirements, these batches have to be stored separately from conforming biofuel. All necessary information has to be filled in. If non-conformity is discovered at the premises of the consumer in connection with delivery, a non-conformity report is generated and the handling of the non-conforming lot is agreed with the consumer.

Signature of assigned person   Place and date

NOTE This example represents an individual plant or process. The document requirements vary depending on the situation and the complexity of the process.

6 Working Package IV

- Biofuel specifications -

Coordinated by: Eija Alakangas, VTT

Involved partners:

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RTU	**Riga Technical University / Institute of Energy Systems and Environmental Protection (RTU)** Kronvalda boulevard 1, LV-1010 Riga, Latvia, www.btv.lv
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Technologie- und Förderzentrum	**Technologie- und Förderzentrum im Kompetenzzentrum für Nachwachsende Rohstoffe (TFZ)** Schulgasse 18, D-94315 Straubing, Germany, www.tfz.bayern.de
VTT	**Technical Research Centre of Finland (VTT)** P.O. Box 1603, Koivurannantie 1, FI-40101 Jyvaeskylae, Finland, www.vtt.fi

6.1 FUEL SPECIFICATION AND CLASSES – INTRODUCTION TO TASK IV AND FEEDBACK FROM INDUSTRY AND CLASSIFICATION OF USED WOOD IN FUEL SPECIFICATION AND CLASSES (EN 14961) MULTIPART STANDARD

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ABSTRACT: The work of the BioNormII on Fuel specification and classes (Task IV) has very valuable in upgrading of prEN 14961 to European standard. Testing of the draft versions of EN 14961 in companies and collecting feedback from associations has provided useful information from practise in setting threshold values for property tables in Part 1 of EN 14961 and proposal for classification of product standards. The Task IV has also made a comprehensive study of current classification of used wood including implementation of European legislations and standards. The study included also analysis of different used wood sortiments and their composition especially contaminants. It is necessary to define the impact of possible contaminants on the energy utilisation of used wood. This is helpful for categorisation, as well as for identification and characterisation of the contaminants. From an environmental point of view, the risks of used wood combustion are air pollution from flue gases or leaching of hazardous substances from ash after disposal. Operation failure, normally appearing as boiler deposit formation or corrosion, is a technical issue that also needs to be considered. Mechanically, problems in fuel conveyors may occur due to glass, fittings, and various fastening systems that are attached to used wood products. Another important aspect to be accounted for is issues related to occupational health. All these aspects were surveyed during the study. The project also made a proposal for overall categorisation of chemically treated industrial wood residues and used wood. In this classification used wood is divided in 4 categories A, B, C and D. Categories A and B are classified under EN 14961-1–Solid biofuel standard and class C under prEN 15359–Solid recovered fuel. Fuels falling into category C should be incinerated according Waste Incineration Directive (2000/76/EC). Class D wood is treated by wood preservatives and is hazardous waste.

Keywords: biomass fuel, classification, fuel specification, standards, waste wood.

1 INTRODUCTION TO TASK IV – fuel specification

The technical specification: Solid biofuels–Fuel specification and classes (CEN/TS 14961) was published in year 2005 and it is upgraded to the full European Norm (EN) for solid biofuels during 2008 and 2009. Therefore the collection of feedback from the practice, scientific institutions and other BioNorm II partners to the draft standards, their evaluation and harmonisation was the main task of WP IV. The encouraged work finished in a new draft of the prEN 14961 with the integration of more detailed specifications and for further solid biofuels. Furthermore, some modifications of existing thresholds were needed but also the extension of the prEN 14961 to multi-part standard [12-16], in which concrete fuel quality requirements for different solid biofuel products used in small-scale can be seen as a result of the feedback analysis of companies and several associations and in collaboration with the CEN/TC 335 WG 2. Also combustion tests with small-scale boilers (<50 kW) by using the most traded solid biomass fuels (pellets, briquettes, wood logs, wood chips and olive residues) were used for analysis of fuel quality effects. The work will also benefit the upgrading of CEN/TC 335 standards to international ISO standards.

The objectives of the WP IV were:

- To clarify the specification of solid biofuels to support the upgrading of the CEN/TS 14961 (to EN-standard.
- To guarantee by measurements, interviews and reviewing data the suitability of different biomass fuels for domestic consumers
- To clarify properties of biomass fuels for different supply chains
- To draft basics for conformity rules for solid biofuels

The aim of the Task IV.1 is it to define the fuel properties according by supply chain. This was realised based on the work already carried out within the "Fuel specification and classes" (prEN 14961), by interviewing 10 industrial companies and associations [2, 3, 8] and organising workshops to different stakeholders [1, 4]. Addition to this VTT collected information from literature and project partners from their own countries to define which fractions of used wood can be included in EN 14961[6, 7,17]. The aim of the Task IV.2 was to identify and assess the properties which are defined (or needed) by the combustion appliances and other end-user devices, e.g. handling equipment. This included physical-mechanical as well as chemical parameters. All possible parameters are regarded and the most important are assessed. For the important properties classes and their variations (i.e. band width) will be defined which meet the needs of industry and/or the consumer (i.e. household consumer with small scale systems). The importance of fuel properties for domestic users was studied carrying out combustion tests [5])

The aim of Task IV.3 was to establish a basis for the development of rules for conformity (for upgrading of CEN/TS 15234 – Fuel quality assurance). The work provided information about how a producer of solid biofuels can assure in a transparent way that his products are conform to the customers' needs and the defined fuel standards, respectively [10]).

The following research organizations have participated in the Task IV: VTT (coordinator), FJ-BLT Wieselburg, University of Stuttgart (USTUTT), Riga Technical University (RTU) and Centre For Research & Technology Hellas, Institute For Solid Fuels Technology & Applications (CERTH) and Technologie und Förderzentrum (TFZ).

2 FEEDBACK FROM INDUSTRY TO PREN14961 MULTIPART STANDARD

Feedback was collected from 10 companies operating in Austria, Finland, Germany, Greece and Latvia [2]. These companies produced wood briquettes and pellets, wood chips, hog fuel from different wood fuel sources including also used wood, olive residue cakes and agrobiomass bales. Companies presented different type of organisations including international companies but also small local producers.

Comments were also collected from different associations: Österreichischer Kachelofenverband" (Austrian tiled stove association), Finnish Pellet Energy Association, the European Fireplace Association, the German Energy Pellet Association, HKI which represents the interests of the producers of domestic heating and cooking appliances (more than 70 manufacturers of domestic heating and cooking appliances), BDH represents the interest of 69 companies from the sector of building services engineering with focus on heating engineering, association of the German sawmill industry, German Chimney Sweeper Association, association for trade and production of firewood with around 80 members from economy and politics and Institute for firewood technology with focus on drying and moisture determination of firewood.

Companies tested different draft versions on prEN 14961-1 during six months period. Some of these companies also provided fuels for combustion tests. Results were reported to WG2 of CEN/TC335 by using their commenting template and in the summary reports [2]. Project partners also surveyed existing European wood pellet standards [3]. The classification system developed in prEN 14961–Part 1 was basic tool for most of the companies involved in case studies. Wood pellet and briquettes producers and equipment manufacturers have also commented prEN 14961 – Part 2 and Part 3.

General conclusions from feedback studies:

- Flexible classification system (Part 1) is a good basic tool for most of the companies.
- Companies involved in the case studies proposed several additional classes with threshold values for different biomass fuels based on their own products.
- Wood chips and hog fuel produced from chemically treated wood or forest wood, which may have more heavy metals should be have own tables, because this caused a lot of misunderstanding when nitrogen and chlorine is normative for above mentioned wood fuels, but not for chemically untreated wood.
- There should be enough moisture content classes (interval 5 w-% units) because pricing is based on the moisture content and net calorific value. Also more ash classes for wood chips and hog fuel is needed.
- There should be more particle size classes for wood chips and hog fuel e.g. P31.5.
- Used wood falling to the scope of CEN/TC 335 should be classified in categories A and B (Figure 2, [17, 7]).
- Southern European companies also requested the possibility to have chemically treated wood and used wood as a raw material for wood pellets. Northern European companies also proposed that raw material should be classified as chemically untreated if the amount of chemically untreated wood is at least 97w-%.
- Reed canary grass is different product compared to straw bales, and most of the bales produced for the markets are round bales, now classification is only for square straw bales. Own property table for reed canary grass bales was proposed. This table might be used also for miscanthus bales. Harvesting time is important and should be normative and also tying material should be stated.
- Exhausted olive cake needs different definition for fines, because the particle size is usually on average 5 mm. Proposal for fines is < 1 mm. Also nitrogen and chlorine is proposed to be informative.
- Domestic consumers need more simple classification and properties should be bind together to form a class. Some companies proposed that class A should be targeted for packages pellets especially to be used in stoves, class B for packaged and bulk deliveries. Class C is for such installation which do not require so high quality

fuel as in classes A and B are. Austria proposed two classes for wood pellets (wood without bark and wood-bark pellets) and no chemically treated wood should be allowed.

- There are contradictive proposals of the chemical compounds for wood briquettes and pellets. Companies situating in Baltic State area and in southern Europe do not see important to state nitrogen, sulphur and heavy metal content for wood pellets or briquettes, because virgin wood includes small amount of these compounds. On the contrary Central European companies are requesting these as normative with very strict threshold values, because of their national legislation (e.g. NO_x emission limits for small scale boilers).
- For log wood classes should be stated according diameter and length, type of wood should be stated with a definite abbreviation of the botanic name.

Addition to detailed proposals companies and associations made first draft classification tables to wood pellet standard (prEN 14961-2 [2, 12], tables for reed canary grass bales and olive residue tables for prEN 14961-1. The project partners have collected data for typical properties of olive residues, other fruit biomass and woody biomass for Annex B of prEN 14961-1[9]).

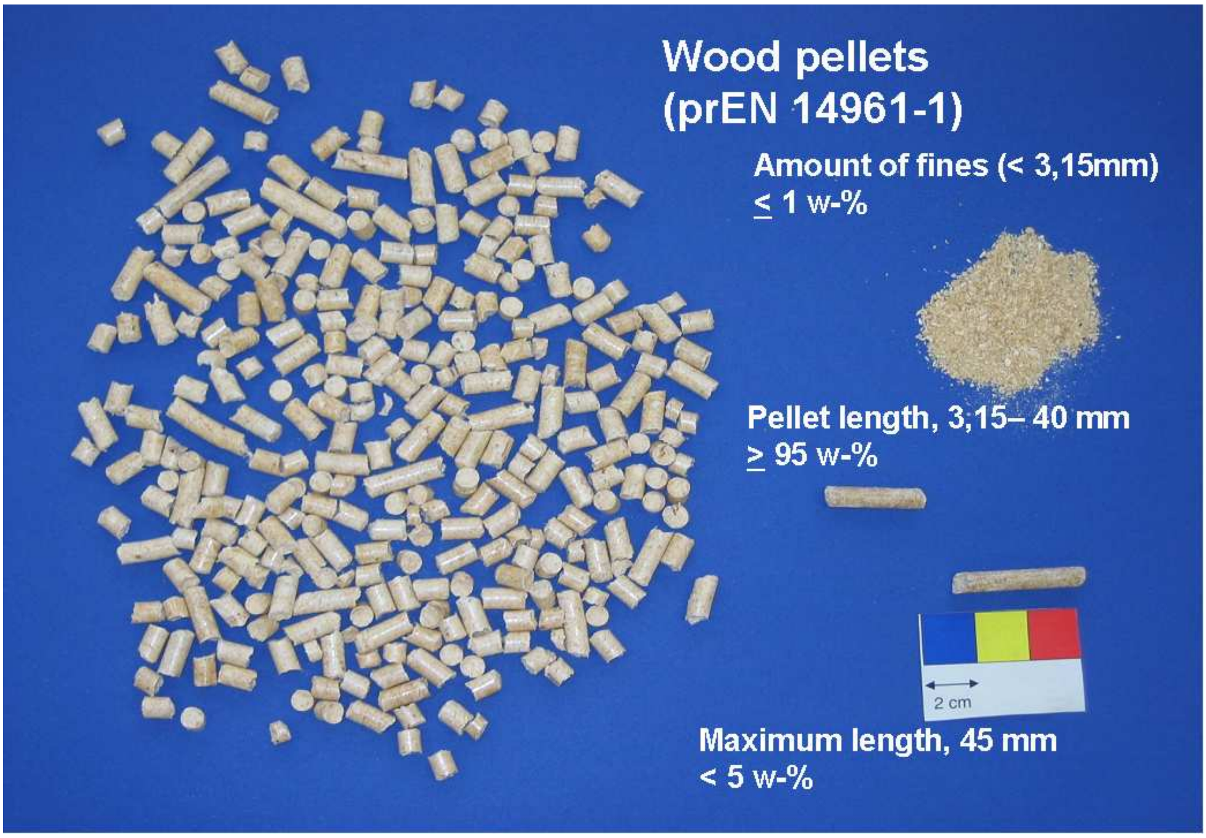

Figure 1. Wood pellets classified according to FprEN 14961-1 [9].

3 CLASSIFICATION OF USED WOOD

The prEN14961 – Part 1 [9] includes also wood waste, if it is not containing halogenated organic compounds or heavy metals as a result of treatment with wood preservatives or coating. One aim of the Task IV.1 was clarify which fractions of wood waste can be defined as solid biofuel.

VTT collected data from literature, industries and project partners of used wood. Part 1 of the report [17] includes information of existing legislation in the European Union, practises and regulations of certain member states. Part 2 includes examples of different used wood fractions and their classification [6].

The terminology used to describe the material that is derived from wood or wood products, which no longer can be used for their original intended purpose varies according to source (country, writer, point of view). The term *used wood* [11] that has been adopted by CEN is unambiguous, and clearly states the origin of the material. In some contexts also *recovered wood* or *recycled wood* has been used to describe this material. Additionally, one may come across the terms *waste wood* or *wood waste*, but these terms are somewhat problematic as the terms themselves imply that the material should be classified as waste. This is, however, not always the case even when the word waste is used as part of the term. Therefore, this paper will employ the term used wood and leave the categorisation of the material to be based on the findings of this work.

Figure 2 describes solid biofuels derived from the by-products and residues of the wood processing industry, as well as post-society used wood. Part of the woody material under the heading "used wood" (class 1.3 according to prEN 14961-1) can justifiably be classified as biomass. Due to the absence of clear guidelines and definitions, the classification of used wood into either waste or biomass remains debatable in the case of certain fractions of wood residues and wastes. Although used wood is included in the list of wastes, it should be kept in mind that it should not always necessarily be regarded or treated as waste. Used wood or used wood products may enter another life cycle, especially through material recycling and reuse but also when the material is utilised as energy.

BioNormII project has collected information on the following issues [17]:

- European legislation and standards of used wood
- composition of used wood and chemically treated industrial wood residues (chemical and mechanical contaminants)
- management of used wood (amounts, markets, risk analysis)
- combustion of used wood
- national legislation, standards and classification of used wood (Austria, Finland, Greece, Germany and the Netherlands, Sweden)
- real case products of used wood [6]

It is necessary to define the impact of possible contaminants on the energy utilisation of used wood. This is helpful for categorisation, as well as for identification and characterisation of the contaminants. From an environmental point of view, the risks of used wood combustion are air pollution from flue gases or leaching of hazardous substances from ash after disposal. Operation failure, normally appearing as boiler deposit formation or corrosion, is a technical issue that also needs to be considered. Mechanically, problems in fuel conveyors may occur due to glass, fittings, and various fastening systems that are attached to used wood products. Another important aspect to be accounted for is issues related to occupational health. It is mainly dust that causes problems, especially if hazardous contaminants are present. Regardless of what the negative impact of contaminants is, whether it is technical, environmental, or health related, the characterisation of used wood including the contaminants within it is of utmost importance. Characterisation will provide necessary information for decision making and planning of activities. When the material is adequately characterised, it should then be possible to plan appropriate sorting and quality assurance procedures. Some contaminants, like plastic, metals, concrete, gypsum, and glass, are possible and quite easy to remove mechanically. Others are more or less permanently attached to the wood, for instance paints, varnishes, coatings, preservative, glues, etc., and are thus practically impossible to separate from the wood material.

Used wood contamination can be divided into two categories: "chemical" and "mechanical". Chemical contamination is more or less permanently adhered to the wood and thus nearly impossible to separate from the wood material, whereas mechanical contaminants are possible to remove from the wood by sorting or work up. Methods for detecting contaminants are necessary for establishing and improving product quality. Moreover, characterisation of used wood is the basis of risk assessment of products made thereof. Normally, the following parameters of used wood are screened:

- "Heavy metals": arsenic, cadmium, chromium, copper, mercury, nickel, lead, thallium, zinc
- Polycyclic aromatic hydrocarbons (PAHs), e.g. napthalin, anthracene, benzo[a]pyrene
- Halogens like chlorine, bromine and fluorine
- Pentachlorophenol (PCP) and lindane
- Polychlorinated biphenyl (PCB)
- Volatile organic compounds (VOCs)

Heavy metal contaminants, originating from paints and preservatives, need to be considered in the production and utilisation of solid biofuels from used wood.

Another important issue is to control the nature and amount of pollutants formed during used wood combustion. This is normally not a problem in large scale combustion plants with efficient flue gas cleaning, but combustion of this material in the single ovens of private houses should be discouraged. Ashes, on the other hand, contain the residues of the burnt wood. Consequently, there is the possibility of high concentrations of heavy metals, and unburned or oxidised pollutants. Environmental risks related to used wood combustion in large scale plants are related to ash recycling as fertiliser.

The value of used wood products always needs to be considered when making decisions on the costs, efforts and time that can be spent on the analysis of the starting material. In addition to being a raw material for solid biofuel, used wood can also be used for wood-based panels, bedding products for horses, cattle, chicken and domestic animals, as well as landscaping and interior furnishing products.

The behaviour of used wood as a fuel is in principle similar to that of clean wood. Used wood is often quite dry, thus the net calorific value can be higher than for common fresh wood fuels (e.g. bark). Used wood fuels may, however, have a higher content of contaminants, which may accelerate the formation of deposits in the boiler as well as corrosion. Further, the composition of ash can be affected.

Deposit formation is related to the fuel and ash composition. Solid biofuels and recovered fuels are high in calcium and alkali metals. In addition, recovered fuels tend to have relatively high chlorine contents. The result of this is accelerated deposit formation in the boiler. In fuels, reactive calcium and alkali metals are either organically bound or they may occur as carbonates or salts. During combustion these react and form oxides, sulphates or chlorides. Especially chloride salts have low melting points. Therefore, it is generally acknowledged that chlorine induces ash deposition. Chips made from clean used wood and used construction wood, as well as pallets, can from a combustion point of view be regarded equivalent to wood chips. Demolition wood, containing painted wood, board residues and other non-woody construction materials, may, on the other hand, contain for instance lead. Lead is known to cause problems in combustion. The level of boiler deposits from combustion of contaminated demolition wood is similar to that of recovered fuels in general.

There are a great number of non-wood components, such as plastics and metals that add to the contamination level of used wood. It is suggested that by careful and selective demolition, as well as sorting of the resulting wood waste most non-wood compounds can successfully be separated from the actual wood. Analyses on sorted fractions indicate that the used wood contains approximately 1% non-wood compounds, mainly plastic and metal compounds, glass, dirt, concrete, bricks and gypsum. Even if the proportion of non-wood components seems relatively small, the effect of incinerating large amounts of unsorted used wood will inevitably cause problems. For example, metal objects of zinc, brass and aluminium, may plug primary air openings. High chlorine levels, with related corrosion issues, are indicative of the presence of PVC residues in the fuel.

Several countries have produced more detailed classification of used wood. These classifications have been basis of the BioNormII project to define general classification which is based on the standard prEN 14961-1. Classes A, B, C and D for used wood and industrial wood residues and by-products are proposed as a result of this study. The classification into these four classes is elucidated by examples with real used wood cases presented in the Part 2 [6, 7, 17].

Used wood is divided into 4 different categories (Figure 2); A, B, C and D. Categories A and B are classified under EN 14961-1–Solid biofuel standard and class C under prEN 15359–Solid recovered fuel.

Fuels falling into category C should be incinerated according Waste Incineration Directive (2000/76/EC). Class D wood is treated by wood preservatives and is hazardous waste. This classification is in accordance with the classification in the European Waste Catalogue (Table 1). Flow chart in Figure 3 clarifies how different used wood classes can be determined.

This classification has been tested in Finland and results of the first year's experience was be discussed in October 2009 with in representatives of used wood producers, forest industry and energy utilities [7]. Many companies has started to apply classification in their used wood business and also some environmental permission includes reference to this report [7].

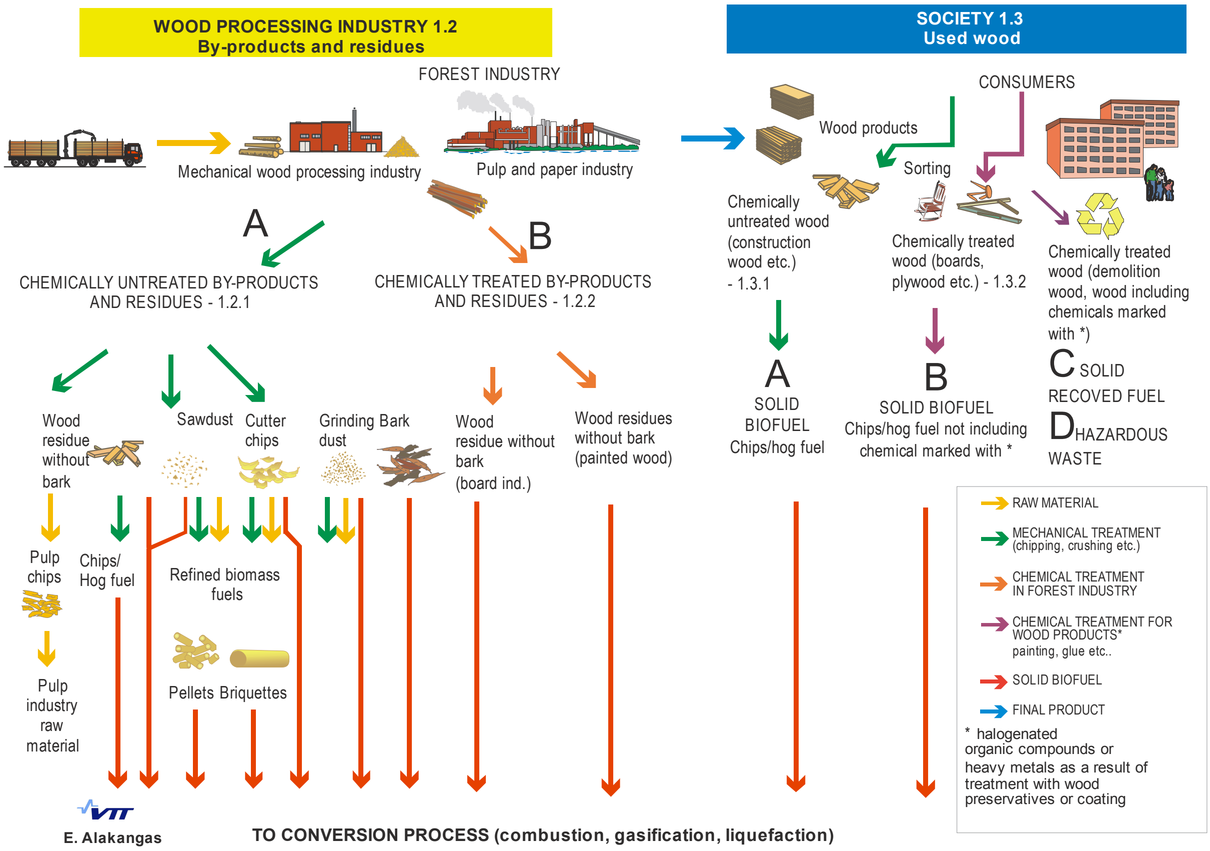

Figure 2. Classification of the industrial by-products and residues and used wood to A, B, C and D [17].

Table 1. Classification of industrial wood residues (1.2) and used wood (1.3) according European Waste Catalogue and Hazardous Waste List [17].

Definition of waste code and waste code	**Typical assortments**	**Origin according to prEN 14961-1**	**Category**	**Definition of category**
Wastes from wood processing and the production of panels and furniture 03 01 05	Waste, cuttings, shavings from solid wood in its natural state	1.2.1	A	Natural or merely mechanically processed wood, contaminated only to an insignificant extent during use by substances that are not normally found in wood in its natural state
	Waste, cuttings, shavings from derived timber products and other treated wood (without harmful contaminants)	1.2.2	B	Glued, painted, coated, lacquered or otherwise treated wood, without halogenated organic compounds in the coating and without wood preservatives
Wood waste packaging (including separately collected municipal packaging waste) 15 01 03	Pallets made from solid wood in its natural state such as: Euro-pallets, industrial pallets made from solid wood in its natural state	1.3.1	A	Natural or merely mechanically processed wood, contaminated only to an insignificant extent during use by substances that are not normally found in wood in its natural state
	Transport cases, crates made from solid wood in its natural state			
	Boxes for fruit, vegetables and ornamental plants as well as similar boxes made from solid wood in its natural state			
	Cable reels made from solid wood in its natural state (made after 1989)			
	Pallets made from derived timber products	1.3.2	B	Glued, painted, coated, lacquered or otherwise treated wood, without halogenated organic compounds in the coating and without wood preservatives
	Transport cases made from derived timber products			

Definition of waste code and waste code	**Typical assortments**	**Origin according to prEN 14961-1**	**Category**	**Definition of category**
Construction and demolition wood wastes* 17 02 01	Construction wood from building sites made from solid wood in its natural state	1.3.1	A	Natural or merely mechanically processed wood, contaminated only to an insignificant extent during use by substances that are not normally found in wood in its natural state
	Construction wood from building sites made from derived timber products, barked wood, treated solid wood (without harmful contaminants)	1.3.2	B	Glued, painted, coated, lacquered or otherwise treated wood, without halogenated organic compounds in the coating and without wood preservatives
	Boards, false ceilings, planks from interior works (without harmful contaminants)			
	Door leaves and frames from demolition* and restoration work (without harmful contaminants)			
	Profile boards for the fitting out of rooms, ceiling panels, ornamental beams etc. from demolition and restoration work (without harmful contaminants)			
	Chipboard used in construction from demolition* and restoration work			
Municipal wood wastes (household wood waste and similar commercial, industrial and institutional wood wastes) including separately collected fractions 20 01 38	Furniture made from solid wood in its natural state	1.3.1	A	Natural or merely mechanically processed wood, contaminated only to an insignificant extent during use by substances that are not normally found in wood in its natural state
	Furniture without halogenated organic compounds in the coating	1.3.2	B	Glued, painted, coated, lacquered or otherwise treated wood, without halogenated organic compounds in the coating and without wood preservatives

Waste codes marked with an asterisk (*) are excluded from the scope of prEN 14961, i.e. used wood with halogenated organic compounds in the coating OR treated with wood preservatives OR that cannot be assigned to category A or B due to its contamination.

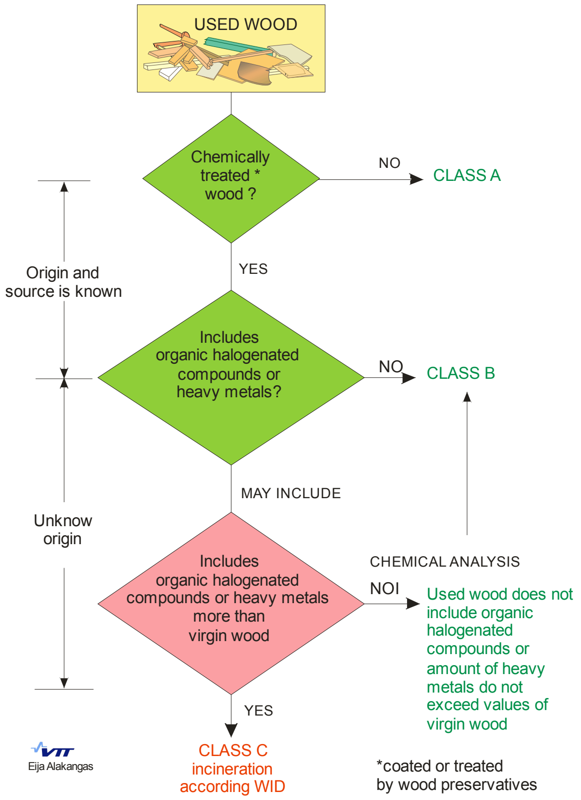

Figure 3. Flow chart how to determinate different used wood categories [17]

4 CONCLUSIONS

The work of the BioNormII on fuel specification and classes has very valuable in upgrading of prEN 14961 to European standard. Testing of the draft version of EN 14961 in companies has provided useful information from practise when setting threshold values for property tables in Part 1 [9] and proposals for classification of product standards has given the basis for WG2 for discussion.

The Task IV has also made a comprehensive study of current classification of used wood including implementation of European legislations and standards. The study included also analysis of different used wood sortiments and their composition especially contaminants. It is necessary to define the impact of possible contaminants on the energy utilisation of used wood. This is helpful for categorisation, as well as for identification and characterisation of the contaminants. From an environmental point of view, the risks of used wood combustion are air pollution from flue gases or leaching of hazardous substances from ash after disposal. Operation failure, normally appearing as boiler deposit formation or corrosion, is a technical issue that also needs to be considered. Mechanically, problems in fuel conveyors may occur due to glass, fittings, and various fastening systems that are attached to used wood products. Another important aspect to be accounted for is issues related to occupational health. All these aspects were surveyed during the study.

The project also made a proposal for overall categorisation of industrial wood residues and used wood. In this classification used wood is divided in 4 categories A, B, C and D. Categories A and B are classified under EN 14961-1–Solid biofuel standard and class C under prEN 15359–Solid recovered fuel. Fuels falling into category C should be incinerated according Waste Incineration Directive (2000/76/EC). Class D wood is treated by wood preservatives and is hazardous waste.

Class A (Virgin wood, only mechanically treated)

- Chemically untreated by-products or residues from forest and wood processing industry (1.2.1, 1.1.7)
- Chemically untreated used wood (1.3.1)
- Classification according EN 14961-1
- No Waste Incineration Directive to be applied

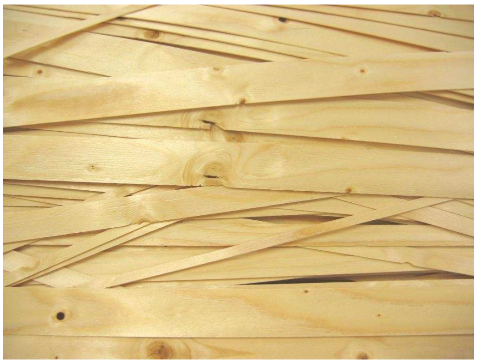

Figure 4. Example of class A wood, ply. Photo Camilla Wiik, VTT [6].

Class B (Coated, lacquered or otherwise chemically treated and coating does not contain halogenated organic compounds (for example PVC) and preservatives, no demolition wood)

- Chemically treated by-products and residues from forest and wood processing industry (1.2.2)
- Chemically treated used wood (1.3.2) excluding demolition wood

Figure 5. Example of class B wood, pallet. Photo Camilla Wiik, VTT.[6]

Class C (Halogenated organic compounds for example PVC in the coating)

- Difficult to verify the origin of the material e.g. demolition wood
- Does not contain preservatives
- Demolitions wood is mainly classified in this category
- Classification according to prEN 15359– Solid recovered fuels
- Class C wood is solid recoved fuel and waste incineration directive to be applied

Class D (Preservative-treated wood)

- Railway sleepers
- Transmission and telephone line poles
- Class D wood is hazardous waste

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6.2 COMBUSTION EXPERIMENTS WITH SEVERAL DOMESTIC HEATING APPLIANCES

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ABSTRACT: Five institutes carried out combustion tests using small scale combustion appliances. VTT with a heat retaining stove and an over-fire boiler, TZF with a modern down-draft boiler, FJ-BLT and Riga Technical University (RTU) with pellet boilers and GERT/ISFTA from Greece with an under-feed stoker boiler using wood pellets, Cynara Cardunculus Pellets, exhausted olive cakes, almond shells and peach kernels. The general aim of the combustion tests was to find out what are the most critical fuel properties that affect emissions and efficiency of different kind of domestic solid biomass burning appliances. This presentation gives a summary of conclusions of all combustion experiments. More detailed information can be found from individual combustion test reports.

Wood logs are still very widely used in domestic heating. Combustion tests with several different appliances showed the importance of fuel quality to the behaviour of the appliances. Modern wood burning appliances are not so sensitive to wood log moisture. However, in general moisture content limits were found for all types of appliance which is 20 - 25% on wet basis. It was also found that too low moisture content may cause increase of emissions. It can be the case when wood logs are artificially dried. Too dry wood pyrolyses very quickly and can cause peak in emissions.

Wood pellets are the most appropriate fuel to be used in small-scale heating boilers and thus its categorization in different classes is obligatory. Attention has to be paid on the origin of the raw wood since it influences the properties of the derived product.

Exhausted olive cake is used in small scale boilers for heating purposes. Despite its extensive use, some problems on the emitted pollutants, especially concerning the dust emissions, may arise. The properties of this solid biomass fuel vary, since they are related to the way of its production in the oil factories. As a result, its combustion behavior may be seriously affected. Based on the activities undertaken in BIONORM II project and the acquired experience from the utilization of exhausted olive cake in Italy and Spain, specifications of various by-products streams coming from the olive oil processing chain were provided to the CEN/TC 335/WG2.

The energy crop examined within the project was Cynara Cardunculus, which is typical for the climatic conditions in the Mediterranean region. Difficulties were detected when burning this biomass in a small scale boiler due to enhanced deposition tendency and high dust emissions. Further investigations should address the potential use of this energy crop in industrial combustion units. Other energy crops which are favoured for cultivation in the Mediterranean region should be also examined.

It is very difficult to classify the properties of agricultural and agro-industrial residues in specific categories. Some efforts has been undertaken to categorize the properties of fruit biomass within CEN/TC 335/WG2. Additional materials may be added in this category of solid biomass fuels, depending on the priorities and needs in the agricultural sector.

Future research efforts should focus on the verification of the limits set for the characteristics of the solid residues, especially concerning the contents of heavy metals.

Keywords: biomass fuel, small scale combustion, pellets, wood logs, boilers

1 INTRODUCTION TO COMBUSTION EXPERIMENTS

Five institutes carried out combustion tests using small scale combustion appliances. VTT with a heat retaining stove and an over-fire boiler, TZF with a modern down-draft boiler, FJ-BLT and Riga Technical University (RTU) with pellet boilers and GERT/ISFTA from Greece with an under-feed stoker boiler using wood pellets, Cynara Cardunculus Pellets, exhausted olive cakes, almond shells and peach kernels. The general aim of the combustion tests was to find out what are the most critical fuel properties that affect emissions and efficiency of different kind of domestic wood burning appliances.

The idea was to choose representative domestic scale combustion appliances for experiments. Wood logs are still the most widely used domestic wood energy. However there are different options to burn wood logs and also modern boilers are available. Wood pellets are coming more and more popular, because it is commercially available in almost every country and combustion appliances are well developed.

There is a lack of high quality wood for fuel in some countries. This is why also some agro-biomasses were chosen for combustion experiments.

2 WOOD LOG COMBUSTION

2.1 A Finnish heat retaining stove

Technical Research Centre of Finland carried out wood log combustion tests using a Finnish type of heat retaining stove and an conventional over-fire boiler. Both appliances represent batch-type of combustion of wood logs. Typically wood logs are burned in these appliances with quite high burning rate, because both store heat while combustion. Heat retaining stoves into the solid mass of a stove (1000 - 3000 kg) and over-fire boilers into the water tank (1000 - 5000 litres).

Figure 1. A Finnish heat retaining stove.

Wood is burned normally once a day for about 2 – 3 hours. The amount of wood burned is normally from 10 to 20 kg per day. Recommendation is 10 kg of wood per one ton of stove's weight. The energy of wood is mainly stored into the mass of a stove and slowly released to the surrounding room space. Heating rate is in the order of 4 kW or less.

It was quite difficult to achieve good combustion of woods. On the contrary the burning was quite lumbering. That is why the differences of different wood logs perhaps did not come out quite clearly. It should be noted that more modern heat retaining stoves use different combustion air delivery and burn much more cleanly. Typically in modern stoves CO-concentration is less than 0.1% (at 13% O_2).

However, it was quite clearly found out that

- When the moisture content of fuel is high, gaseous emissions are usually higher.
- CO and OGC concentrations were higher when using larger logs (D2), except when the moisture content was the lowest. In modern heat-retaining stoves also large logs normally burn with low emissions.
- OGC and dust concentrations follow very well changes in CO-concentration. Correlation of OGC and CO is better than dust and CO. It should also be noted that we did not repeat individual tests. Wood log combustion is very sensitive process and to get really statistically proven results, same test should be repeated more than one time.
- Different wood species did not have very much differences in emissions. Dry small pine wood resulted in highest dust emissions, but gaseous emission were quite the same as with spruce and birch. So the result could have been only coincidence. The amount of fuel burned was about 15 kg in every test. However the fuel moisture varied. Thus the amount of energy also varied. Burning times were between 153 minutes and 383 minutes. The latter indicates that burning of wood was very slow. In this appliance, burning of moist and large logs was quite poor.

Recommendations to prEN 14961

Based on these combustion tests it is recommended that

- moisture content should be M20 for this kind of appliance. Too dry wood results in high emissions because of quick pyrolysis and too high moisture content for very low burning rate and high excess air ratio
- to be used in this appliance, particle size of the wood should be quite small. Large logs did not burn well. However, this is controversial to

earlier experiences from other heat retaining stoves This means that operation instructions of heating appliances should clearly say what size of fuel should be used in different burning phases in different appliances. Fuel standard should allow different particle sizes for wood logs.

2.2 A Finnish over-fire wood log boiler

Figure 2: A Finnish over-fire boiler for wood logs.

These kinds of boilers are still quite common in Finland and also in many other countries of EU. Usually the boiler is connected to an accumulator tank of 1 – 5 m^3. It allows the boiler to be used at nominal heat output and only when excess heat is required. In Finnish weather conditions it means that in summer time the boiler is used once a week and in the winter time once or twice a day.

50 cm wood logs were used in the test boiler. Its nominal heat output is 20 – 40 kW. A drawing of the combustion principal is shown in figure 3.

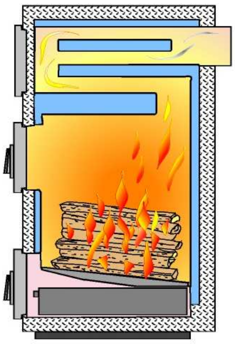

Figure 3: The combustion principle of an over-fire boiler.

When the moisture content of fuel is between 15 and 20%, it is recommend to use large diameter logs. Then emissions of CO, OGC and particulates seem to be then a little lower. However, power output is then also lower, because burning of wood logs is slower. When the moisture content of fuel was near 30%, there was a higher risk for higher emissions. However the trend is not clear.

Birch and pine do not differ when taking emissions in consideration. It is safer to keep fuel moisture content between 20 to 25%. Then it is easier to avoid high peaks of emissions, which may be caused from burning too dry and small diameter or too wet and too large diameter logs.

Recommendations to prEN 14961

- Fuel moisture should be limited to 25% or it must be clearly stated that delivered fuel has to be further dried if the moisture content is higher.
- Fuel moisture content should not be also too low. Lower limit should be around 15%. This is especially important for thermal dried wood logs.
- It should be clearly marked if wood is seasoned wood or thermal dried wood.
- High quality wood logs have lower bark content. Bark results in higher NO_x-emissions and for higher risk of particulate formation.

2.3 A modern down-draught wood log boiler

Figure 4: Wood log boiler FHG Turbo 3000-30 (Fröling)

A downdraft log wood boiler by Fröling (Grießkirchen, Austria) of the type "FHG Turbo 3000 (Lambda)" with 30 kW nominal heat power output was used (Figure 4). As it is common for wood boilers in Germany and Austria the boiler was equipped with turbulence spirals in the heat exchanger pipes. Their use leads to increased combustion efficiency and through a mechanical moving system a de-ashing and cleaning of the heat exchanger pipes is achieved. The boiler applies a lambda controlled air flow and it represents the actual state of technology in Germany and Austria. Furthermore, due to the high market share of this boiler it is highly representative for the described regions.
The boiler has a large feeding hopper which is fitting to the use of half metre logs. This enables a long burning duration. The combustion chamber and the heat exchangers are spatially separated. The high temperature turbulence chamber and the electronically controlled suction fan allow an

adaptation to variable fuels, while the combustion air flow is optimized via lambda signals and via an adjustable flue gas temperature. The range of heat output is between 9 and 30 kW, which is typically for rural single house heating demands.

3 CONCLUSIONS

Moisture content of fuel

- For modern wood log boilers an increased risk of rising CO and particle emissions is given at moisture levels higher than M = 25%.
- Power output of the boiler is gradually declining with higher fuel moisture content between 10 and 35%, associated with a slight loss of efficiency.
- These conclusions can not be applied for stoves where experiments performed in other similar projects show a larger response of pollutant emissions towards moisture contents above 20%.

Concerning the log dimension the following conclusions can be drawn:

- Modern downdraft wood log boilers (with ventilation and lambda control) are largely tolerant towards variable log diameters between 60 and 250 mm, this means that
  - no influence on efficiency is expected,
  - a slight decline in power output may be possible for larger log diameters
  - no significant effect on pollutant emissions can be expected if the moisture content of the fuel is in the required range.
- These conclusions do not apply for older boiler types (e. g. updraft combustion) and for stoves, where experiments performed in similar projects by TFZ show that a clear optimum is given for medium log sizes (this applies for chimney stoves and tiled stove inserts).

Recommendations to the prEN14961

From the here presented results and with reference to other similar or relevant experimental trials performed at TFZ the following recommendations can be made to future definitions and to fuel product requirements in the frame of EN14961 (part 5: non industrial fire wood).

Maximum fuel moisture

- For wood log combustion the use of fuels with higher moisture content than 20% implies an increased risk of poor combustion quality.
- This risk is lower for modern lambda controlled downdraft boilers, but such technology cannot be compared to updraft furnaces such as wood stoves.
- In order to avoid any excessive pollutant emissions and to prevent a decline in combustion efficiency and power output the fuel moisture content should be limited to a maximum of 20% at the time of energetic use but also already at the time of delivery, because such users often buy their fuels for immediate use.

Minimum moisture content

- An extremely low moisture content of wood logs, as achievable by technical drying with hot air, may also be problematic, although no advantage or disadvantage was shown in the here performed tests using a modern lambda controlled log wood boiler.
- However, from ongoing research at TFZ recently some evidence arose, that such low moisture contents may be highly problematic for single stoves (e.g. with fuel moisture around 7%).
- For the definition of log wood quality requirements it should therefore be considered to establish a minimum fuel moisture limit (e.g. at 10%).
- Additionally there should be a normative indication, which informs the customer whether the drying to oven ready fuel was achieved under atmospheric conditions (by ambient air during long term storage) or by ventilation with hot air.

Hot air drying becomes more and more popular in Germany due to the necessity to make use of the residual heat from agricultural biogas plants and due to the requirement to increase the flexibility towards unexpected market developments.
In order to allow a differentiation between stoves and boilers (updraft) and modern downdraft boilers it should be considered to define the minimum (M_{min}) and maximum M_{max}) fuel moisture requirements as follows:

P1000 and 500: M_{min}: no limitations,
M_{max}: 25% (at delivery to customer)
to be stated: method of drying (ambient air / hot air)

P250 and P330: M_{min}: 10%,
M_{max}: 20% (at delivery to customer)
to be stated: method of drying (ambient air / hot air)

Log diameters

For modern wood log boilers which commonly use logs of 500 or 1000 mm length the diameter limitations as given in prEN14961 are suitable, as these boilers are largely insensitive towards variable wood log diameters. But there is a difference between split and unsplit (round) wood, which however was not tested here, but it was observed during experiments performed by TFZ in similar other project: Round wood is associated with a higher risk of forming a bridge over the fire bed (so-called “hollow fire”) which consequently increases the pollutant emissions. Round wood should therefore be treated as a separate fuel quality class.

For individual stoves, where the updraft combustion principle is commonly applied, a more narrow range of diameters seems useful. Previous research by TFZ shows, that benefits are given for medium size wood logs of the 330 and 250 mm length. The highest quality class should therefore be defined by a slightly stricter diameter range than the one mentioned in prEN14961 (Table 9).

4 WOOD PELLET COMBUSTION

4.1 A modern wood pellet boiler

For the test series the KWB Easyfire 20, typ KWB USP 20 from KWB (Kraft und Wärme aus Biomasse GmbH) was used. The boiler has a rated heat output of 20 kW and consists of the pellets reservoir, the fuel handling device, the burner, the heating exchange device and the ash box.

Figure 5: KWB Easyfire 20 pellets heating boiler

The pellets come from the reservoir by a rotary feeder and a feeding screw on to the burning plate. At the burning plate, the wood pellets get inflamed by an electrical ignition. The combustion air blower aerates the combustion air through the fuel as primary air and through an afterburning ring as secondary air into the combustion gas. After the flame pipe, the combustion gas gets to the heat exchanger. Beneath the combustion chamber is an automatically ash retrain. The ash gets by a screw into the ash box in front of the boiler, where it is ready to dispose.
The KWB Easyfire 20 is a state-of-art product and technically matured. It was important to take a boiler, which is able to burn different kinds of pellets from different origin.
To get comparable results, the boiler was operated with the same settings and without a lambda oxygen sensor section.

Conclusions

In the line of the combustion test seven different pellets types were burned. Wood pellets with varieties in ash content and size were used as well as pellets made from miscanthus and hemp straw.
Principally every fuel of the test series except hemp pellets were able to be used as fuel. The appliance used for the tests was a high quality product. With modifications in the settings it would be possible to optimise the burning process with each fuel, but to ensure fully comparable results, it was important to fix settings, that every selected fuel could be burned. A special case was hemp pellets. The quality of these pellets was so bad, that a combustion test under these fixed conditions was not possible.

The most significant parameter, which affects the burning process and especially the emissions, is the ash content. Results of the combustion test show that hemp pellets and miscanthus pellets have the highest content of dust, CO, OGC and NO_x. These two fuels have also the highest ash content of all seven fuels.

Different wood species did not have significant differences in emissions. Wood pellets with higher bark content and a compound of hardwood and softwood did not show any specific trend.
Another variable is the pellet boiler. The size of the combustion chamber and the setting of the boiler parameter change the results.

Recommendations to the prEN14961.

On the basis of the combustion tests the following recommendations for pellets could be drawn:

- For small scale combustion high quality pellets are recommended to avoid masses of ashes and to reduce emissions.
- A clear differentiation between wood pellets and non-wood pellets has to be made.
- Generally the Austrian proposal of the ON-K 241 for product standards for wood pellets for domestic use and the CEN/TS 335/WG 2 N143 proposal for solid biofuels – fuel specifications and classes – Part 2: wood pellets for domestic consumers are endorsed.

4.2 A Latvian pellet boiler

The boiler tested in the framework of the Bionorm II project has a nominal power of 15 kW and it is produced by a Latvian manufacturer (see Figure 6).

The boiler has a semiautomatic operational system; the user manually ignites the fuel in the furnace and regularly has to clean the boiler from ashes.
After the manual ignition, once the fuel has a stable combustion, the burning process is switched to automatic regime, where the boiler automatically supplies pellets and control on/off sequences in function of the water temperature in the boiler.

When the boiler reaches the maximum set temperature, it switches off: the air fan and fuel feeding system are switched off and the pellets remain in the burner in a no-air environment. Then, when the temperature goes below a minimum set temperature, the combustion starts again. If the time between an off phase and an on phase is very long, it may happen that the combustion process is completely extinguished and consequently the user should ignite the fuel again. A modulation of the heating power is not possible.

Figure 6: A Latvian pellet boiler used in the tests as installed in the tests rig.

Fuel feeding from the pellet storage is carried out with a fixed supply screw. The boiler has a bottom fed burner and is equipped with an alarm system that warns when the pellet store is empty.

The boiler has an internal circulation pump and is equipped with a three-way valve for regulation the supply water temperature to the heating system.

Conclusions

- The boiler used for tests is produced by a Latvian manufacturer and it could represent a classic model easily findable available on the Latvian market.
- Boiler efficiency varied in a range between 83.9% and 86.4%. Difference in boiler efficiency values is connected with both parameters of boiler operation conditions (different oxygen content in furnace, temperature of flues gases) and the different types of pellets.
- The 3 different pellet qualities used in the test were rather close to each other in terms of pellet quality parameters and therefore the separate effect of pellet quality parameters on boiler efficiency is not clearly detectable. For example ash content of pellets in range of 0,39 – 0,55 % did not affect combustion and boiler efficiency.
- Emission level of nitrogen oxides in flue gases depends on temperature level. Some features are observed which need additional testing and research. For example lowest NO_x emissions have been measured are obtained for the pellets sample with higher moisture content. Higher nitrogen content compared to the other pellet samples could lead to higher concentration of NO.
- Two of the samples used in these tests have very similar durability values while the third one had an higher value. From this few data it was not possible to make relevant conclusion on the effect of mechanical durability on boiler efficiency and emission level.
- Ash content of pellets in range of 0.39 – 0.55 % did not affect combustion efficiency. Therefore higher ash content has to be tested to find influence to boiler efficiency.

Emission levels of NO_x should be further analyzed in particular in relation to nitrogen content in the pellet sample and moisture content of the pellets.

5 AGRICULTURAL RESIDUES

5.1 An underfeed stoked boiler

A heating boiler, classified to Mile П 50/2008 type, burning biomass was selected for the tests, Figure 7.

Figure 7: Mile Π Boiler on the test facility.

Eight (8) combustion experiments were performed in a heating boiler burning biomass at the nominal heat output, according to EN 303-5. These boilers are manufactured in the range of 35-1000 kW and CERTH/ISFTA representatives chose to use a boiler having heat output about 60 kW in the tests. This is mostly preferred for domestic heating purposes. They comply with the present legislations and specifications, classified as Class 2 heating Boilers for solid bio-fuels.

As regards the constructive configuration, it consists of the cast iron hearth, the three different flue gas passages and the cylindrical combustion chamber, ensuring the optimal thermal combustion efficiency at low operating cost and reduced cleaning demands. The doors are designed to allow the unproblematic entrance of even large pieces of wood, while they are easily converted into diesel boilers by attaching an extra cast iron door and extra insulating material to block the standard wood-chip burner.

The fuel is inserted into the combustion chamber, by using a cast iron screw feeder (Figure 8). A second cast iron screw feeder with blades is also available, in particular for hard material i.e. kernels/shells in order to avoid potential implications with the fuel supply. A motor is used to regulate the rotation velocity (set at 1370 rpm) and the fuel supply.

Recommendations to the prEN14961

- Wood pellets are the most appropriate fuel to be used in small-scale heating boilers and thus its categorisation in different classes is obligatory. Attention has to be paid on the origin of the raw wood since it influences the properties of the derived product.

Figure 8: Cast Iron Screw Feeder.

Different classes of solid biofuels were tested, i.e. pellets from agro-biomass (cynara cardunculus or cardoon), pellets of woody biomass as well as various qualities of exhausted olive cakes (Greece, Cyprus and Spain), peach kernels and almond shells.

Conclusions

Eight (8) solid biofuels were burned in a biomass boiler at the test facility, which is certified for heating boilers accreditation. Specifically, wood pellets, almond shells, peach kernels, pellets of cynara cardunculus and different qualities of exhausted olive cakes were tested. The whole test procedure was carried out according to the European Norms EN 303-5:04/1999.

Wood pellets and almond shells are shown to be the most suitable for domestic boilers. Especially, wood pellets were proven to have the higher efficiency and also the emissions (CO, SO_2, NO_x, OGC) were kept in very low levels. Almond shells exhibited sufficient operational behaviour, regarding the efficiency and the emissions. Combustion of peach kernels is characterised from low emission levels and comparatively low efficiency. Exhausted olive cake combustion was resulted in a moderate efficiency and emission levels, also depending on the origin of each fuel. Only almond shells and peach kernels exhibited dust concentrations below the limits, while this value was a little bit higher for wood pellets. On the other hand, combustion of cynara cardunculus led into high dust concentrations and enhanced deposition tendency.
Based on the fuel characterisation and the results of the combustion tests, recommendations to the prEN14961 have been formulated as follows:

- Exhausted olive cake is used in small scale boilers for heating purposes. Despite its extensive use, some problems on the emitted pollutants, especially concerning the dust emissions, may arise. The properties of this solid biofuel vary, since they are related to the way of its production in the oil factories. As a result, its combustion behavior may be seriously affected. Based on the activities undertaken in BIONORM II project and the acquired experience from the utilization of exhausted olive cake in Italy and Spain, specifications of various by-products streams coming from the olive oil processing chain were provided to the CEN/TC 335/WG2, Appendix 17.

- The energy crop examined within the project is cynara cardunculus, which is typical for the climatic conditions in the Mediterranean region. Difficulties were detected when burning this biomass species in small scale boilers due to the enhanced deposition tendency and the high dust emissions. Further investigations should address the potential use of this energy crop in industrial combustion units. Other energy crops which are favoured for cultivation in the Mediterranean region should be examined.

- It is very difficult to classify the properties of agricultural and agro-industrial residues in specific categories. Some efforts were undertaken to categorise the properties of fruit biomass within CEN/TC 335/WG2, Appendix 18. Additional materials may be added in this category of solid biofuels, depending on the priorities and needs in the agricultural sector.

- Future research efforts should focus on the verification of the limits set for the characteristics of the solid residues, especially concerning the contents of heavy metals.

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6.3 CONFORMITY RULES FOR SOLID BIOFUELS – RESULTS OF SURVEY AND PROPOSAL OF CONFORMITY DECLARATION FOR FUEL QUALITY ASSURANCE (EN 15234) MULTIPART STANDARD

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An important issue for the further development of the fuel quality assurance (EN 15234) multipart standard is the question, how conformity with the fuel specification and classes (EN 14961) multipart standard shall be demonstrated. Therefore, the aim of the BioNorm II Task IV.3 is to establish the adequate basis for the development of corresponding rules for conformity. Based on the findings of an extensive survey of conformity assessment systems across Europe and abroad, a specific conformity and certification scheme, respectively, for solid biofuels was drafted and put for discussion among experts in the field of conformity assessment and solid biofuels. As a result of the numerous feedbacks and in consideration of the previous work on quality assurance for solid biofuels, a conformity declaration for solid biofuels is complementary proposed for an implementation in EN 15234, which commits the producer/supplier of solid biofuels to officially declare the conformity of his product with the corresponding part of EN 14961. Beyond that, the standard can not require an independent control of the fuel quality by a third party in any way as it is done e.g. in certification schemes. This can only be the result of the market mechanisms and additionally developed rules for conformity independent of the fuel standards. However, the proposed conformity declaration in the context of the fuel quality assurance (EN 15234) multipart standard is considered to force the corresponding market mechanisms and to provide confidence in the applied European fuel standards.

Keywords: biofuel specification, conformity declaration, quality measures, solid biofuels, standards

1 INTRODUCTION

The aim of the BioNorm II Task IV.3 “Basics for conformity rules” is to provide information on the question, how a producer of solid biofuels can demonstrate and how a customer of solid biofuels can check that the products conform to the requirements laid down in the multipart standard EN 14961 “Solid biofuels – Fuel specification and classes”. This task is closely connected to the scope of the multipart standard EN 15234 “Solid biofuels – Fuel quality assurance” which defines procedures and describes measures in order to ensure adequate confidence that the biofuel specifications are fulfilled [1]. The findings of Task IV.3 shall, therefore, provide additional information on conformity rules for solid biofuels in the above-mentioned meaning, which is needed for the further development of EN 15234.

Starting point was the consideration of a general conformity assessment system consisting of the elements standardisation, accreditation and certification [2-3]. This system can be sub-divided into two stages as outlined in Figure 1. (1) The quality of products and services that is controlled by conformity assessment bodies (e.g. certification bodies, inspection bodies, laboratories) in order to build up customer confidence. (2) The quality of the conformity assessment bodies that again is controlled by superior and neutral accreditation bodies in order to build up producer confidence.

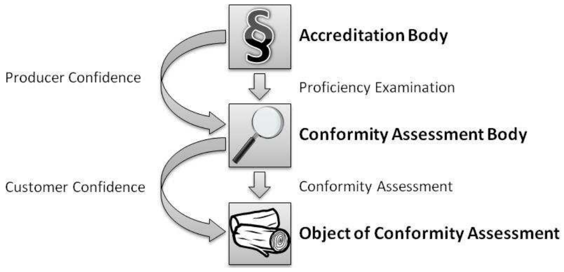

Figure 1: Conformity assessment system [3]

Standardisation goes along with this conformity assessment system as it provides on the one hand rules for the specification of quality requirements (as defined in EN 14961), and on the other hand rules for the demonstration and traceability of conformity with these specified requirements (as shall be provided by EN 15234).

A survey of conformity assessment systems was carried out to provide a basis for the development of a specific conformity scheme for solid biofuels. This includes the development of a proficiency scheme for conformity assessment bodies in order to address uncertainties introduced through sampling and testing and to refer to both data repeatability and reproducibility [4-5].

Furthermore, feedback was collected from conformity assessment bodies, associations, producers and research institutions in the field of solid biofuels, and a proposal for conformity rules for an implementation in EN 15234 was deduced from this feedback in close cooperation with CEN/TC 335 WG 2.

2 SURVEY OF CONFORMITY RULES

The survey of conformity rules that are included in standards and other specifications for particulate materials covers information from the EU, Austria, Denmark, Finland, Germany, Greece, Italy, Latvia, the Netherlands, Russia, Spain, Sweden and the USA. The survey was focused on conformity rules in terms of product certification schemes which include at least the following tasks:

- Application for certification
- Conformity testing (feasibility check)
- Conformity assessment
  - Initial type testing
  - Initial inspection of production control
- Product certification
- Continuous conformity control
  - Continuous in-house production control
  - Regular external control of product an in-house production control

Amongst others, the following questions were addressed:

- What institutions are in charge of certification and conformity assessment?
- How is the certification and conformity assessment scheme built up?
- How to apply for certification? What information is requested by the conformity assessment body?
- How does the conformity assessment body check, if a conformity assessment can be carried out (feasibility study)? What are the rules for acceptance/rejection of an application?
- What are the rules for acceptance/rejection of conformity based on the applied assessment methods (e.g. sampling and testing)?
- How is the production control assessed? What are the rules for acceptance/rejection of conformity based on the assessment of the production control?
- What are the requirements for the assignment of a product certificate? What are the rules for acceptance/rejection of a product certificate? How long is a product certificate valid?
- How is a continuous conformity control carried out? What are the rules for continuous in-house production controls and regular external controls?

Moreover, questions concerning the accreditation of conformity assessment bodies were also addressed.

As a result of the survey, it can be concluded that product certification schemes generally follow a common approach which is exemplarily illustrated by the following flow chart of the Danish certification body DANCERT [6] in Figure 2.

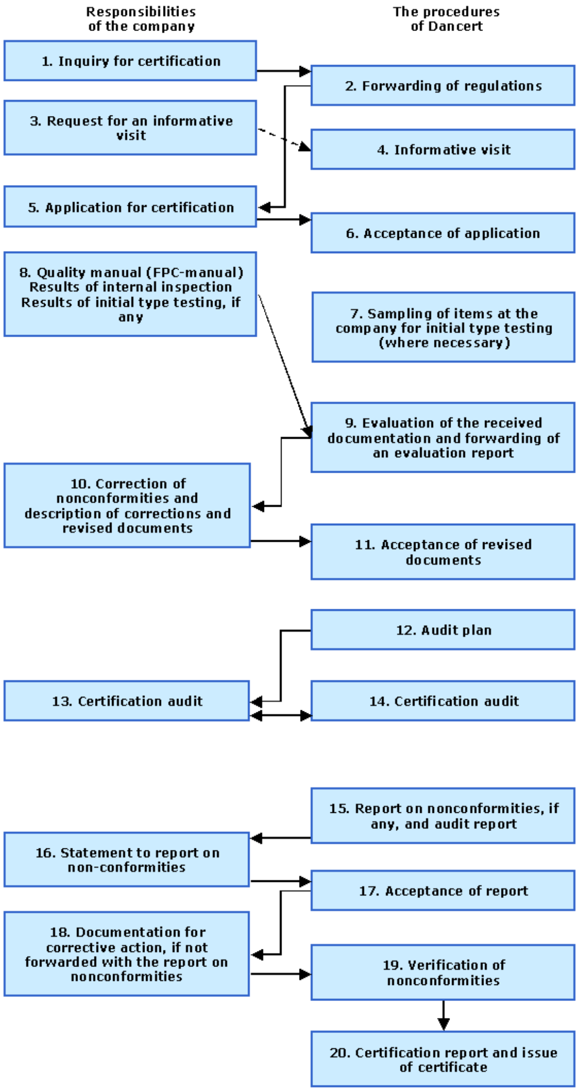

Figure 2: Exemplary product certification scheme [6]

For the elaboration of a conformity scheme for solid biofuels, the scheme rules of the European voluntary third-party certification mark, the Keymark (Figure 3), were applied and adapted to the specific scope.

Figure 3: The Keymark

The Keymark scheme rules substantially follow the above-mentioned process steps of a product certification. Moreover, regulations for the accreditation of certification bodies as well as co-operating testing laboratories and inspection bodies are covered by the Keymark scheme rules. The objective of the Keymark is to demonstrate to customers that a product complies with relevant European standards. It involves regular product testing and inspection of the factory production control by a third party. The Keymark can only be granted by certification bodies that have been empowered by the Certification Board of CEN for a specific European standard or a group of European standards. To improve the confidence in the Keymark, it will only be granted in combination with marks of existing national certification systems which are based on the conformity with the considered European standards [7].

In addition to the adapted Keymark scheme for solid biofuels, a proficiency scheme for laboratories was outlined. The proficiency scheme shall refer to both repeatability and reproducibility of sampling and testing data in order to provide confidence to the manufacturer that the results are not misinterpreted due to errors introduced through sampling and testing. Therefore, regular round-robin tests of solid biofuels were proposed.

3 FEEDBACK ON CONFORMITY SCHEME FOR SOLID BIOFUELS

The drafted conformity scheme for solid biofuels, including the proficiency scheme for laboratories, was up for discussion among different experts in the field of conformity assessment and solid biofuels. Interviews were carried out with conformity assessment bodies, solid biofuel associations, single producers and research institutions. Furthermore, an international workshop was organised in Riga, Latvia, to discuss the findings. The drafted conformity scheme was also put to the vote of CEN/TC 335 WG 2. The main feedback can be summarised as follows:

The main idea of accreditation and certification is to provide confidence to producers and customers by an independent third-party assessment. However, standards are considered to describe (technical and other) product (and service) requirements only, i.e. they predominantly define the specifications of a product (dimensions, chemical and physical properties, etc.) and, if necessary, basic sampling and test conditions or whole procedures. However, beyond the determination of technical sampling and test specifications, standards shall not determine responsibilities, i.e. if it is up to the producer/supplier (first party), the customer (second party) or any conformity assessment body (third party) to carry out sampling and test procedures. This should finally be the result of market mechanisms.

Furthermore, certification should not be the only way to attest conformity. From the view of standardisation, conformity assessment should be broken down from a general level in order to take the manifold supply chains and applications of solid biofuels into account. Certification might be a good way to attest conformity for a widespread small-scale use of well-defined solid biofuels like e.g. wood pellets. On the other hand, certification might not be appropriate for small actors on the market or industrial applications. In these cases, there should be alternatives like a reliable self-control or mutual agreements between producers and customers.

4 PROPOSAL OF CONFORMITY DECLARATION FOR EN 15234

With the findings from the survey of conformity rules and the feedback on the drafted conformity scheme, a proposal of conformity rules for solid biofuels for an implementation in EN 15234 was elaborated. The proposal is limited to the proposal of a conformity declaration in order to prevent overlapping, since quality assurance and control measures as well as procedures are already described in the current draft of the quality assurance standard.

According to the proposal, the fuel properties shall be specified in the conformity declaration according to the requirements of the appropriate part of EN 14961. With the conformity declaration of fuel quality the producer or supplier confirms that the properties of the end-product are in accordance with the requirements of the relevant part of EN 14961.

Conformity declarations shall be issued for both solid biofuels handled as bulk material and for packaged solid biofuels, in any case for each delivery lot. For packaged solid biofuels, the quality information given in the conformity declaration shall be labelled on the packaging. The supplier shall date the declaration and keep all relevant records for a minimum of one year after the delivery. The conformity declaration shall state the fuel quality in accordance to the relevant part of EN 14961.

The conformity declaration of the fuel quality shall as a minimum include the following information:

- product name;
- applied standard (relevant part of EN 14961);
- traded form (acc. to Table 2 EN 14961-1);
- origin and source (acc. to Table 1 EN 14961-1);
- country and location where the biomass is harvested or first traded as biofuel;
- fuel specifications (according to the relevant part of EN 14961)
  - normative properties;
  - informative properties (if applicable);
- supplier (body or enterprise) including contact information;
- conformity declaration stating compliance to the relevant part of EN 14961;
- signature (assigned person), name, date and place.

An example of a conformity declaration is given in Figure 4.

CONFORMITY DECLARATION

Product name: SB Pellets
Applied standard: prEN 14961-2
Traded form: Class A1 wood pellets for non-industrial use
Origin and source: 1.1.3 Stemwood
Location and country: Anylocation, Anycountry

Normative properties:

Diameter, D and Length, L:	D06 6 ± 1; 3,15 ≤ L ≤ 40	[mm]
Moisture, M:	M10 ≤ 10	[w-% as received]
Ash, A:	A0.5 ≤ 0,5	[w-% dry]
Mechanical durability, DU:	DU 97.5 ≥ 97,5	[w-% as received]
Fines, F:	F1.0 ≤ 1,0	[w-%]
Additives:	none	[w-%]
Net calorific value, Q:	Q16.5 ≥ 16,5	[MJ/kg as received]
Bulk density, BD:	BD600 ≥ 600	[kg/m³]
Nitrogen, N:	N0.3 ≤ 0,3	[w-% dry]
Sulphur, S:	S0.05 ≤ 0,05	[w-% dry]
Chlorine, Cl:	Cl0.02 ≤ 0,02	[w-% dry]
Arsenic, As:	≤ 1	[mg/kg dry]
Cadmium, Cd:	≤ 0,5	[mg/kg dry]
Chromium, Cr:	≤ 10	[mg/kg dry]
Copper, Cu:	≤ 10	[mg/kg dry]
Lead, Pb:	≤ 10	[mg/kg dry]
Mercury, Hg:	≤ 0,1	[mg/kg dry]
Nickel, Ni:	≤ 10	[mg/kg dry]
Zinc, Zn:	≤ 100	[mg/kg dry]
Ash melting behaviour, DT:	DT1200 ≥ 1200	[°C]

The manufacturer herby declares that the product described above complies with the provisions and current amendments of the European Standard prEN 14961-2.

Done at: Solid Biofuels Ltd., 123 Main Street, Anytown, 12345, Anycountry
Date: August 12th, 2009
Name and position of signatory: Mr. John Q. Public, General Manager

Signature:

Table 1: Example of conformity declaration referring to prEN 14961-2 [8]

5 CONCLUSIONS

With the proposal of a conformity declaration for solid biofuels, minimum requirements for conformity rules are introduced providing a generally applicable tool for the demonstration of conformity of solid biofuels with the corresponding European fuel standards. With regard to an implementation of these rules in the fuel quality assurance (EN 15234) multipart standard, an independent third-party assessment of the fuel quality can not be specified. This can only be the result of the market mechanisms and additionally developed rules for conformity independent of the fuel standards. However, the mandatory issuing of a conformity declaration for traded solid biofuels is considered to force the corresponding market mechanisms and to provide confidence in the applied European fuel standards.

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