

## **Executive summary:**

AQUAFIT4USE has brought innovative solutions to increase the sustainable use of water in industry in four important water intensive sectors: chemical, paper, textile and food.

By the development and piloting at industrial level of new, reliable cost-effective technologies, tools and methods, big steps have been made towards the sustainable use of water resulting in significant reduction of the fresh water needs, closing the water cycle, mitigation of the environmental impact and the production and use of water in accordance with the specifications of industry for the different processes and applications, 'water fit-for-use'. Supporting the industry to secure this vital resource and to decouple the water use and the related environmental impact from economic growth are important issues to support EU policy on competitiveness, resource efficiency and environment.

The key challenge of this EC FP7 project was to give answers to the next questions:

What is the water quality that is really needed for a process or other application?

How can this water quality be produced and maintained?

How can the water quality be monitored and controlled?

What are the effects of using other sources of different water quality?

With the new software tool WESTforINDUSTRY, developed within AQUAFIT4USE, industrial water systems can be simulated and optimized, resulting in alternative systems, with reuse options and treatment technologies for water loop closure. Part of the software tool are data bases for treatment technologies and for water quality demands.

For the fast and specific monitoring of micro-organism over 140 new probes are developed, to monitor water systems and (treatment) processes.

New solutions were explored for a number of common problems in industrial water use. Filtration Assisted Crystallization Technology (FACT) and Multiflo-Softening were successfully tested for hardness removal, Denutritor for biofouling prevention and capacitive deionization (CapDI) and evapo-concentration for treatment of saline streams. Attention was also paid to selective removal of recalcitrant substances and disinfection technologies.

The main aim however was the development of complete 'custom made' solutions - treatment trains of different technologies for the paper, textile, chemical and food industry. The combination of biological systems (Membrane Bioreactors - MBR, Anaerobic BIOMAR®ASBx), Advanced Oxidation Processes (AOP), membrane processes and evapo-concentration were tested in different industrial pilots. Examples of successful results are the further removal of COD and recalcitrant contaminants in the paper industry, the reuse of effluent of the biological treatment for cooling water in the chemical industry, the use of rain water for container cleaning in food industry and closing the water cycle in textile industry by combined membrane filtration and advanced oxidation.

By showing the solutions at location and by intensive dissemination of the results via website, posters, brochures, papers, presentations and a video, the industries were confident that the solutions offered, are

really applicable in their situation and will lead to a more sustainable and competitive enterprise.

## **Project Context and Objectives:**

By consuming several billions m<sup>3</sup> of water a year, European industry has a significant impact on available water sources. Legislation, stringent discharge standards, higher process and product demands and increasing water scarcity force industry to ensure higher water quality, resulting in higher costs.

'For the water consuming industry, water is no longer regarded as a commodity, consumable or utility but as a highly valuable resource, a vital element used in close conjunction with the production processes. Industries want to become more and more independent of public and private parties for the supply of process water and the treatment of wastewater. Furthermore, they want to use water qualities according to their own specifications, fit-for-use'.

This vision on sustainable water use and the great economic importance of water for the industrial sector was already defined in the first vision document and Strategic Research Agenda (SRA) of the European Water Platform (WssTP) in 2006. In the updated SRA in 2011 more emphasis was put at water quality definition and water-fit-for-use, closure the water cycle and a more integrated approach.

The securing of sufficient water of good quality and resources for urban, industrial and agricultural use is a major driving force behind the current EC policy on water. In the 'Resource Efficient Europe' flagship initiative, under the Europe 2020 strategy and its related roadmap, water is recognized as a basic resource that supports our ecosystem and biodiversity and is acknowledged to be a vital element in various economic sectors. The link with energy and other resources with water is also important within this framework. Also the policy towards decoupling of natural resource use and the environmental impact from economic growth gives direction to sustainable water use in industry and to the EU FP7 project AQUAFIT4USE. Within this framework the contribution of AQUAFIT4USE to support the Water Framework Directive objectives is also important to mention.

AQUAFIT4USE is ahead in the way the EC wants to tackle the societal challenges, as mentioned in the new Horizon 2020 program, where the testing, demonstration and scale up of promising solutions is specifically mentioned.

## **Four pillars for sustainable water use in industry**

### **AQUAFIT4USE is built on 4 pillars:**

#### **1. Water Fit-for-Use**

Water fit-for-use is the basis of the project. In the current situation most of the industries are not able to answer the question 'What water quality is really needed?' Knowledge on the effect of the water quality on process, product quality and health issues is not available. This makes that industries often choose for far better water qualities than needed. However, sustainable water use means: use water of right quality, 'fit-for-use'. AQUAFIT4USE gives not only an answer to the question 'What is the minimum water demand and water quality' that is really needed, but also to questions like:

How can these water qualities be produced and maintained?

How can these water qualities be monitored and controlled?  
What are the effects of using lower water quality?

These questions first need to be answered, before the industries can take further steps in sustainable water use, closing the water cycle and use of different water sources.

## **2. Integrated sustainable water use in Industry**

Sustainable water use requires an holistic approach. Within AQUAFIT4USE there is a strong focus on the integration of all water related aspects, like new sources, energy, waste(s), process, product and environment. This is carried out in a systematic and structured approach, that ensures that all important mass and energy flows are taken into account, and scenarios for optimization are developed including the use of process integration and energy and water pinch techniques. Environmental impact assessment is part of this approach.

## **3. Strong industrial participation**

Within AQUAFIT4USE, 22 of the 34 partners are industries, 9 industrial suppliers and 13 end users. The end-users, representing the four major EU water consuming industries, paper, chemistry, food and textiles, were selected on their annual volume of water consumption and are complementary towards water research needs and know-how.

Representative companies include suppliers of drinking and process water, processes equipment, water treatment technologies, water chemicals and services.

Special attention is given to SME's by their active involvement as end-user or as supplier in research activities, such as pilot testing.

The industrial partners are involved in all the parts of the project: the development of new models, tools and technologies, the pilot testing in practice on site and the dissemination and exploitation of knowledge. This active involvement of industrial suppliers and end-user companies is chosen to strengthen the applicability in practice and ensure the follow-up implementation in full practice of the AQUAFIT4USE developments.

## **4. Cross-sectorial solutions**

The four target industries are dealing partly with similar problems and challenges, but do have also their own specific questions. From previous European and national research projects, the developed knowledge can be summarized as follows:

Chemical industry: The focus in the past was on treatment of strongly polluted effluents; removal of trace elements; treatment of water, with emphasis on reuse in cooling systems, i.e focus on corrosion problems and chemical conditioning. Water loop closure for other applications got limited attention.

Paper industry: a lot of effort is given to water saving and closing of water circuits, thereby substantially reducing the environmental impact; process modelling and automation and Kidney technologies as internal process water treatment are essential elements in this strategy. A number of problems around the removal of substances are not solved yet and

further closing the water cycle causes new problems. As still some 2 billion cubic meter of fresh water is currently used, there is still a challenge.

Food industry: the main point of attention is water quality in relation to product quality and health. Therefore water reuse is only implemented to a limited extent. Besides, water treatment measuring and monitoring is a major concern.

Textile industry: it pays already a lot of attention to sustainable water use. Because most textile companies are SME's, handling small orders, the composition of water streams is highly variable and therefore often a problem.

## **Objectives**

The overall objective of AQUAFIT4USE is: The development of new, reliable, cost-effective technologies, tools and methods for sustainable water supply, use and discharge in the main European water consuming industries

in order to

- reduce high quality water consumption and fresh water needs
- mitigate environmental impact
- produce and apply water qualities in accordance with industrial own specifications (fit - for - use) from all possible sources
- Contribute to a far-going closure of the water cycle while
- Reducing water relating costs (intake, treatment, re-use, closed loops, discharge)
- Improving product quality and process stability
- Increase independency and flexibility
- Better management of health and safety risks relating to water use.

This overall objective resulted in the following S&T objectives:

### **Objective 1: Water Quality Definition for processes, utilities and environment**

As mentioned before, a good insight in the existing water system and especially in the water quality demands are basic requirements for sustainable industrial water systems. A systematic generic approach for the definition of the specific water quality demands (methodology) should be developed. Additionally a detailed definition of specific water quality demands in the various industrial process steps of the four sectors is needed, as well as cross-sectorial parameter definition on scaling, biofouling, safety and health. The methodology should become available as a user-friendly software tool, together with the results of the modeling (see objective 2), for simulation and optimization of water systems.

### **Objective 2: Integrated modeling and control of Industrial Water Systems**

Models of water treatment technologies and the various process steps (Unit-Process models) are the building blocks for modeling total water industrial systems, with the support of a newly developed Integrated Water Modeling methodology. The final model will be used in the case studies for simulation and optimization. Also insight needs to be gathered in uncertainty and risks associated to alternative the water systems.

Another part of water quality control is measuring and monitoring. The main objective in this area was the development of specific monitoring tools for the fast detection of relevant micro-organisms. The use of existing sensors was also evaluated, because a lot of sensors are inappropriately used (validation and cleaning routines, process conditions).

### **Objective 3: Practicable innovation in Water Treatment Technologies**

The development of new water treatment concepts and technologies is from the main technological objective of AQUAFIT4USE. The focus was on two levels:

- The development of new technologies to solve specific problems in the area of selective removal of specific substances, biofouling and scaling prevention, improved disinfection, desalination.
- The development and testing of different treatment concepts in practice. Combination of several treatment technologies (treatment trains) are needed to find solutions for the complex water issues and to produce water 'fit-for-use'.

Demonstration of the solutions at pilot scale is most important to convince end-users that the technologies are really applicable in practice. Around 30% of the project budget was spent on piloting and demonstration of the proposed (combinations of) technologies.

### **Objective 4: Integration and Validation of solutions:**

The interaction with other water-related issues, like process stability, product quality and energy and wastes, is regarded in more detail. Validation of the solutions, also using methodologies for environmental assessment are part of this objective.

Innovation in sustainable water use is more than the development of new technologies and/or combinations of technologies. Showing the applicability of the solutions in practice by testing these at location of an end-user is an important part of it. Not only the treatment technology should be demonstrated, but also the tools and sensors that has been developed. In this way and supported by all types of knowledge transfer and training activities different stakeholders can be convinced of the possibilities and remove prejudices.

## **Project Results:**

AQUAFIT4USE has brought about a lot of innovative and applicable results, that were very helpful to convince industries in different sectors on the feasibility of sustainable water use in their specific situation. Real steps forward have been made, giving answers to the main challenges of sustainable water use and finding solutions for the reuse of water, further closing of the water cycle and using alternative water sources without making concessions to product quality, health and safety.

In AQUAFIT4USE three types of results can be distinguished:

### **1. Solutions to improve water management**

In this area a broad list of results can be mentioned: tools for simulation and optimisation of water systems, models, knowledge on water quality demands for processes and other water applications, measuring and monitoring equipment and assessment tools. Within this framework also other water related issues and integration aspects were explored.

### **2. Technological solutions to solve common industrial problems**

Specific technological problems and challenges that are common for different sectors have been addressed by developing new technologies and adapting existing technologies for the specific situation.

### **3. Treatment trains as custom made solutions**

Existing and new technologies were combined leading to innovative solutions to improve the water systems of different industries. These solutions were tested at pilot scale at various industrial locations, leading to a lot of practical experience.

## **Water Quality Management: The key to sustainable water use**

Sustainable water use in industry starts with a good insight in the industrial water system, with a strong emphasis on the water quality issues. Answers to the next questions should be provided:

- What is the water quality that is really needed in a process or other application?
- How can this water quality be produced and/or maintained?
- What will be the effect of using a different water source?
- How can we monitor and control the water quality?

### **Water Quality Management (WQM) methodology**

For answering the above mentioned questions a systematic step-by-step approach has been developed, the WQM methodology. This methodology is coupled with newly developed software tool for industrial water management WESTforINDUSTRY.

The steps of the WQM methodology are listed below:

1. Definition of existing water system (information gathering)
2. Setting up the water network
3. Parameter selection, assessment and standardization (Water Quality Definition)
4. Supplying WESTforINDUSTRY with input
5. Simulation of the water network
6. Optimization of the water network
7. Output analysis + processing
8. Water Quality control

It is very important to determine the system boundaries in the system definition phase, deciding also at what level a water management project will be carried out and which parts of the system (factory, site) will be involved.

#### **Water-fit-for-use**

A procedure for Water Quality Definition was developed, as part of WQM, to determine the actual water quality needed for a specific process or treatment step. Important steps are the selection of the relevant parameters, the parameter assessment and standardisation, all related to the five defined quality aspects: product quality, product safety, process water function, machinery and pipes and health and environment. Based on this procedure the water qualities for selected processes in the four sectors have been determined and summarized in public reports and in the data base of WESTforINDUSTRY. Also data are gathered and reported on scaling, fouling and corrosion parameters. Going through these steps of water quality definition, the user only has to check if the final result is in agreement with the company specific situation and demands.

The Water Quality Definition, Water System Modelling and Water Quality Control are the main elements of the methodology. The outcome of the Water quality definition is brought into a Water quality library as part of the software tool WESTforINDUSTRY together with the models for network editing and optimisation. Water Quality Control is a separate HACCP (Hazard Analysis and Critical Control Points) based management system.

#### **Water Quality Control (WQC)**

For water quality control a quality system for the complete water system was developed. The method uses the principles of HACCP, the quality system for safe food production that is applied for years in almost every Food company. For water quality control, the approach is extended with other quality issues, like product quality, process water function, and health and working conditions, all being part of the water quality definition.

The Water Quality Control (WQC) starts with a number of preliminary steps, more or less the same as for a general Water quality Management approach. A good definition of the scope, system boundaries and an inventory and description of the water system are also important in this phase. The core of the approach however is the identification of the Critical Control Points (CCPs) carried out according to the three steps outlined below:

Step 1 - Identification of all Potential Critical Control Points (PCCPs) taking the Water Quality Definition as the starting point - going backwards through the supply system.

Outcome - List of PCCPs with indication of process / point of concern and components / limits in focus.

Step 2 - Assessment of each PCCP for its use as CCP according to the HACCP principles - in particular assessment of existence of applicable control method.

Outcome - List of CCPs with description of point of concern, components / limits in focus and method of control.



Step 3 - Describe for each CCP the way of control, the limits to be met and the corrective measures to be applied if unforeseen performance is found.

Outcome - List of CCPs with description of control points and components and limit values, the surveillance/monitoring system and the corrective measures to be applied in case of deviations.

A CCP is a point in the water network that needs routine control in order to manage the identified risks. A decision tree for the identification and assessment of the critical control points was developed. For the CCP monitoring, procedures are described and a set of corrective actions are established to be taken when a particular CCP is not under control.

The WQC methodology was successfully applied at the water system of two companies, as part of a complete Water quality Management project.

An example of a part of the outcome of a WQC application is given in Annex 2.

The methodology can be used independently but can also easily be incorporated in existing HACCP based quality management systems. Procedures of verification and administration are part of the system.

### **Modelling in Industrial Water Systems**

The development of models and the verification/validation of these models on real scenarios provided by the industrial partners was very important for the availability of simulation tools able to reproduce the water circuits. A mathematical model library that reproduces the most relevant wastewater treatment technologies studied within the AQUAFIT4USE project and other treatment technologies has been developed. These treatment technologies compiled in the model library, are classified in three categories:

- 1) solid-liquid separation units such as membrane technologies, settlers and electrochemical separation processes
- 2) biological units like activated sludge units and membrane bioreactors
- 3) chemical processes like disinfection units, coagulation-flocculation processes.

Optimum solutions for the design or operation of an entire water circuit should consider all mutual relationships among the different unit process elements involved in this circuit, and this solution will frequently differ from the compilation of solutions achieved for the design or operation of specific sections of the water circuit. Therefore, the different models used to analyse the entire water circuit have been constructed under the Integrated Water Modelling (IWM) methodology developed here. The IWM enables the construction of unit processes in a standardized way based on three principles:

- (i) definition of a common state vector,
- (ii) definition of investment and operational cost for all unit process models, and
- (iii) upper and lower boundaries of the components at each treatment step.

All mathematical models describing unit processes are steady-state models, written as a function of design and operational parameters. To

obtain the optimum water network layout, besides mass and energy balances, all the process units have investment and operational costs and restrictions to ensure that machinery requirements, product quality, workers safety and legislation limits of the water quality parameters used are guaranteed. Different unit process models have been calibrated by using real data obtained during experimental test carried out at pilot scale in the AQUAFIT4USE project and experimental data provided from technology suppliers.

From the existing unit process library a complete water network can be built and calibrated. The construction of a mathematical model describing a whole water network follows four steps:

1. the development of process models for each individual unit included in that circuit (Unit Process models);
2. the implementation of the mathematical models in the simulation platform (WESTforINDUSTRY);
3. the connection of these Unit Process models to simulate the behaviour of the entire water circuit (building up the network in WESTforINDUSTRY);
4. the calibration of the integrated model based on the experimental data available.

### **WESTforINDUSTRY: A software tool for model based optimisation of water systems**

#### Description of the tool

In WESTforINDUSTRY knowledge about Water Quality Management, Water Treatment and Modelling is brought together in a model-based software framework. The tool allows for defining, simulating (including various forms of graphical visualization) and optimizing industrial water networks in a very intuitive way. The user is able to define the boundaries of the optimization window by choosing the treatment technologies and the place where these technologies should be positioned. The software can then calculate the effect of each scenario and give a range of results which can be compared and evaluated based on different criteria (e.g. costs, fresh water use and discharge).

The optimization module is based on the definition of an objective function in which the previous mentioned factors are being considered and a mathematical procedure using Scenario Analysis and Monte Carlo techniques. The optimum solution will be the combination of randomly generated parameters that minimizes the previously defined objective function.

In this way the tool allows for computing the effect of various changes in the water system, such as a change in water quality, the rate of recycling, a change in operational parameters or the volume of process water that will be treated by a newly introduced specific treatment technology. Changes can be made in all parts of the tool (e.g. process models, components, units). Furthermore, the tool makes use of an extensive model library and also allows for adding additional user-written process models.

#### **Tool development**

A review of recent literature on industrial process modelling showed that the use of process integration tools has increased especially over the past two decades, and is mainly applied to focus on resource conservation, pollution prevention and energy management. Mathematical models to simulate industrial processes are used in a wide range of

industrial sectors, but have especially emerged within industrial sectors with large scale production facilities, such as Pulp and Paper, Petrochemical, Synthetic Chemical and Pharmaceutical. Numerous commercial software packages are available, most of them based on object (unit processes) libraries where relevant objects are connected in hydraulic and energy networks. The models typically also include steady state as well as dynamic simulation capabilities. Few existing software tool offer the possibility to actually optimise industrial water networks in a systematic, user-friendly way. Following this analysis, the modelling and simulation software package WESTfor INDUSTRY has been developed as one of the main objectives of AQUAFIT4USE.

### **Testing and benchmarking WESTforINDUSTRY**

#### **Bench mark Water Network**

The optimization methodology proposed and implemented in the WESTforINDUSTRY software has been first verified and tested in the Benchmark Water Network (BWN), that was defined in the scope of the project. The theoretical 'benchmark case-study' has been created to be used as a simple and common example of a water network subject to optimization and that is assumed to contain the most important features typically found in industrial water networks. It has been successfully used as a common platform for discussion of water network visualization, optimization tools capability, process modelling methodology, needs for development of the simulation platform, cost analysis, optimization methodology, etc.

It has been demonstrated that the mathematical models within the tool are capable of reproducing the water network costs when a change is made to a water network without violating any restriction defined to it. With more complex models and a proper calibration of them, as it has been made in SP2, the results can be even more realistic than in this first case and didactical example

#### **Case studies with WESTforINDUSTRY**

Holmen was used as a benchmark case. The information and the results obtained during the pilot trials were used for the development of mathematical models. These models, the data and the application of the WESTforINDUSTRY at Holmen's circuits were an important starting point for the future development of the tool.

At CHS the whole water system was modelled and optimised. The analysis of several scenarios in the factory of CHS in Villafranca allowed on one hand to detect inconsistencies in the measured parameters based on mass balance analysis and in the other to establish two water reuse options with water savings up to 13% without major changes in the actual production setup.

At the chemical plant of Perstorp, WESTforINDUSTRY was applied on two levels: at a specific factory and at site level. At the specific factory level the main outcome of the application of the model was a scenario for combined product recovery and water reuse for cooling water. Further pilot scale tests are being undertaken to establish sufficient data for a full feasibility evaluation of the solution. At site specific level the model outcome was a scenario where biologically treated and membrane polished effluent was used for boiler feed water - thereby reducing significantly the needed water supply and effluent discharge by 30-40%, the reduction in effluent discharge being the most interesting for this industrial site.

At the Ice cream factory of Unilever Hellendoorn, the outcome of WESTforINDUSTRY resulted in a number of interesting options, like the use of rainwater for box cleaning and effluent for cooling tower applications. This will reduce the total water use with 40% and the groundwater use from 40.000 m<sup>3</sup> to zero.

At ALPRO the improved insight in the water system led to options for separating water flows, thereby preventing scaling in the whole water system and water treatment. Alternative softening will lead to a large reduction of the salt concentration in the treated water ( greater than 90%) and the effluent. Finally it was decided to use an alternative water source with lower hardness. Another option was the reuse of effluent for cooling tower applications. The proposed options have been tested at pilot scale.

From these studies, it has been shown that the optimization module and the procedure proposed are able to achieve optimum solutions in real water networks.

In general the tool showed to be very user-friendly, the results in general useful and reliable, in line with data from practice.

## **Measuring and monitoring**

### **Evaluation of sensors**

To manage wastewater treatment processes in the industry, devices for measuring water quality parameters are needed. The choice of an instrument for a given process is not easy and is often made according to the basic metrological characteristics given by the suppliers. However these characteristics are not always sufficient and may even lead to a wrong choice: most suppliers do not take into account the real operating context with varying process conditions. This is why a procedure for the evaluation of sensors and analysers of water quality was developed. The method is aimed at obtaining "true" data on sensors performances which can be compared to the datasheets from the suppliers. In addition, time data processing methods have been developed and implemented as a software tool to improve the user information levels. These methods allow to classify information, data reconciliation and to detect sensors faults. A remote access tools was added to the software in order to provide remote web access to the high-level information module software.

### **Fast monitoring micro-organism: 145 new gene probes**

As microbial growth and its relationship with health related issues is a key point in industrial water reuse policies, specific activities in this project were devoted to biofilm and corrosion detection methods and devices.

Another key aspect within the project was the assessment and definition of the actual microbiological water quality required for each process, as it is needed for the successful implementation of new treatment technologies for water reuse.

Together with the application of cultivation-based methods for microbial detection, a strong focus was given to the development of molecular methods based on Fluorescence in situ hybridisation (FISH) for microbial analysis in order to:

- (i) describe various population communities completely and not only partial
- (ii) Improve the current microbiological knowledge in the industries
- (iii) increase the speed of analysis in order to quickly evaluate newly implemented processes
- (iv) develop new tools to monitor micro-organisms which are process relevant, but difficult to cultivate or non-cultivable .

Among the different industries, process complexities, process conditions and potential toxicities of substrates of investigated processes as well as of water treatment management vary enormously. As a consequence, the microbiology and/or potential microbiological problems may differ. In order to develop cultivation-independent microbiological monitoring tools a comprising catalogue of micro-organisms supposed to be relevant in terms of biofilm/ biofouling, hygiene and pathogenicity per industry sector was constantly updated by literature research. A total of 279 micro-organisms were compiled and selected as being relevant in relation to water streams in the four sectors.

In parallel to the establishment of the micro-organisms catalogue, the corresponding 16S rDNA sequences were collected and added to a 16S database which served as a basis for the development of 145 specific gene probes for relevant micro-organisms. Relevance of the micro-organisms was confirmed and assessed by application of the newly developed gene probes on industry samples. Finally, based upon these results a collection of easy-to-use test-kits for the monitoring of relevant micro-organisms are being established and marketed for general use.

Molecular methods used in AQUAFIT4USE project have been based on FISH, but using a standardized industrial format developed by partner vermicon and called VIT® (Vermicon identification technology). By simplifying, shortening and standardising all steps, a very handy and effective molecular biological method was developed for fast and specific analysis of micro-organisms.

This method offers several advantages compared with other molecular methods as PCR. On one hand, it presents a similar sensitivity and specificity degree and on the other hand, it offers a robust detection method without the need to have experienced personnel or very clean (and extra) rooms for performing the analysis. Another big advantage of FISH/VIT versus other molecular methods is represented by the fact that only viable cells are detected by the FISH/VIT method. FISH uses rRNA as a target versus rDNA which is mainly used by PCR for microbial identification. This advantage is even more important if environmental samples are to be analysed in situ where pre-cultivation of the target cells is excluded to prevent cultivation-based population shifts.

Besides the overall analysis of water quality and its hygienic aspects several probes were developed to directly answer distinct microbiological questions, which arose during pilot trials of the project e.g. in the chemical sector. Other probes were developed for general microbial aspects in the industries, like fast and specific detection and elimination of early biofilm-forming bacteria in paper mill process water. For this purpose easy-to-use test kits were developed for routine use in paper mills harbouring neutral to alkaline process conditions.

Moreover, by comparison of four different clone libraries a suitable set of early warning monitoring probes with a strong cross-sectorial impact for process stability in anaerobic pellet sludge reactors was developed.

For the textile industry, the deepest survey on microbiology data so far has been performed and a surprising diversity of different microorganisms was found by molecular biological methods. Microbiological data achieved and specific probes developed might help to clarify possible production problems in the future, once water circuits get more and more closed.

For the food industry, a summary of 58 specific tailor-made gene probes are available for microorganisms important as pathogens or involved in hygienic or biofilm-forming aspects.

Inadequacy of microbial community analysis in food industry solely based on cultivation methods could be demonstrated. Some of the newly developed molecular tools are successfully used via vermicon's service portfolio for fast and specific monitoring of certain uncultivable water-borne species, which are directly connected to water quality of bottled mineral water.

### **Holistic environmental assessments**

To achieve the sustainability objective of this project, it was essential to check whether new technologies and reuse options allowed reducing environmental impacts of industrial water treatment. Two levels of scales are investigated. Firstly, the effect of water treatment processes on the actual toxicity of effluent was evaluated with the Whole Effluent Assessment (WEA) methodology.

Regarding effluents toxicity assessment, the substance-by-substance approach presents shortcomings, such as limited number of substances analyzed, combined effects of substances not considered, etc. For this reason, WEA is recognized as the most accurate approach for effluent toxicity assessment. WEA was applied to three case studies: chemistry (Perstorp), textile (Tekstina) and paper (Hamburger Rieger). In all three sectors, WEA showed that the evaluated treatment sequences typically resulted in a reduction in overall toxicity. In the chemical sector, the total toxicity of the conventional activated sludge effluent sample was already quite low and it was thus difficult to quantify toxicity reduction in comparison with other tested technologies in the treatment train.

In the textile sector, particularly the MBR treatment resulted in a strong toxicity reduction, while AOP treatment has a slightly opposite effect. Contrary to the results for the textile sector, anaerobic treatment had a major positive effect on wastewater toxicity in the paper sector. It needs to be emphasized that these results require further investigations because the number of samples analysed was rather low.

Secondly, a more macro-scale assessment was performed in order to compare the water treatment options planned in different factories, considering different environmental impacts (water pollution but also carbon footprint or impact on human health) in the whole life cycle of water treatment processes. Methodologies used for this macro-scale assessment were Life Cycle Assessment (LCA) and water footprint and were applied to three case studies: food (CHS), textile (Tekstina) and paper (Hamburger Rieger). Results show that implementation of reuse generally leads to a

reduction of the water footprint, but on the other hand to an increase of other environmental impacts (carbon footprint, impacts on human health, etc.) because of additional energy and chemicals consumption and sludge production.

In order to mitigate environmental impacts of water treatment processes, it would be more efficient to reduce energy and chemical consumptions of processes, or to valorise sludge. Ensuring that the water finally discharged into the environment reaches an acceptable quality, is also essential regarding environmental footprint. Results also remind us that reducing the volume of water abstracted makes sense in areas under water stress. Generally, water reuse increases indirect water consumption due to additional energy and chemicals usage. These indirect water consumptions might be located in areas facing higher water stress than the place where reuse is implemented. Finally, this study demonstrated the applicability and the usefulness of these recent environmental assessment methodologies. Both could help decision makers to better understand implication of water treatment system improvements in term of environmental impacts mitigation, at a local level (through WEA) and global level (LCA and water footprint). Particularly, WEA and LCA / water footprint should be considered as complementary tools. Although WEA demonstrates the usefulness of water treatment solutions for toxicity reduction, it is essential to consider these water treatment solutions improvements in a broader context of environmental impacts through LCA. On the other hand, LCA and water footprint present clear limitations for effluent toxicity assessment that could be addressed through WEA.

### **Technological solutions to solve industrial common problems**

Water recycling and reuse present common concerns, independent from the industrial sector. These are biofouling, scaling, treatment of concentrate and saline streams, membranes management and processes to increase retention rate and limit membrane fouling, enhancement of bio-treatment, removal of COD and recalcitrant contaminants, effluent polishing for suspended solids removal and disinfection. The AQUAFIT4USE project proposed solutions that could be applied to solve these common problems.

The focus was on these specific topics to get a clear view on the advantages and bottlenecks of current processes and by trying to minimize the gap by new technological developments. On the basis of wastewater characterization and definition of required water quality for each industry, different technologies and processes were tested and/or evaluated, in order to define:

- New treatment lines to obtain the water quality target, considering cost effectiveness, reliability, minimization in waste and concentrate production. These new treatment lines are focused more particularly on internal recycling;
  - New technologies that could be included in these treatment lines.
- Most of these studies constituted preliminaries for pilot trials at location.

### **Biofouling and scaling prevention technologies**

In the area of biofouling and scaling prevention AQUAFIT4USE focused on three technologies: Denutritor, Nanosilver and FACT.

Denutritor - a biofiltration based biofouling prevention technology - was successfully tested at the effluent of the Waste Water Treatment Plant

(WWTP) of a chemical plant in Sweden, resulting in an about 8-fold reduction of the biofouling potential, increasing the possibilities of reuse of the water for cooling and other industrial applications.

Denutritor was also tested at the Ben&Jerry's ice cream factory for the removal of (pathogenic) micro-organisms from rain water. It showed that Denutritor can reduce but not fully remove coliforms, E. coli and pathogenic bacteria present in collected rainwater. The amount of viable bacteria can additionally be reduced with 90-99% efficiency by exposure to UV doses between 50 and 2500 J/L. During these laboratory tests, three new types of biofouling monitors were evaluated. The increase of protein concentration on the monitors appeared to be a good indicator of the biofouling potential of the synthetic WWTP effluent water tested. Biofouling monitors made from silicon or polyethylene (PE) tubes were finally selected for use during the further pilots.

The effect of silver nanoparticles, which are used at different paper products, has been analysed using model systems for anaerobic and aerobic processes of treating wastewater. The impact of nanoscale silver on the respiratory activity of aerobic activated sludge was traced continuously. Furthermore, trials were conducted at lab-scale and on-site of the mill to study the effect of surface-bonded Ag nanoparticles on the prevention of slime formation.

It was found in model experiments concerning anaerobic and aerobic biodegradability that concentrations below 30 mg nanoscale silver per litre did not cause any impairment of these processes. Contents above this value, however, reduced process efficiency, but these values do usually not occur.

Hardness removal in the Pulp and Paper industry is a very important step for further closing of the water cycle and reducing the use of process chemicals.

Filtration Assisted Crystallization Technology (FACT) is a fast chemical crystallization process, based on accelerated precipitation, using a selected seed material, and separation of the precipitate by a filter. Results of the pilot tests at the paper mill of SAPPI in Maastricht showed that the FACT pilot worked well on both white water streams tested (white water and/or clear white water), both with and without the installed Cricketfilter.

The calcium concentration was reduced with lime from around 80 to 26 mg/l  $\text{Ca}^{2+}$ . The carbonate concentration showed to be limiting for further calcium depletion. Laboratory tests with addition of sodium bicarbonate resulted in a final concentration of 5 mg  $\text{Ca}^{2+}$ /l.

The obtained calcium concentration with sodium hydroxide was 10 mg/l. Seed addition had no effect on the final calcium concentration obtained. Apparently, both white water flows contain already enough fines to accelerate the calcium carbonate precipitation.

Filtration is a bottleneck in many tests. Plugging of filter pores by fines was a problem, especially at flow rates above 2 m<sup>3</sup>/h. The formation of a precoat with 'virgin cellulose' might be an option, but could only be successfully tested in laboratory scale filtration tests.



The filter can possibly be replaced by another solids separation process or discarded when the 'ACT' (i.e. FACT without the filter) is positioned between the paper machine and the present poly disc filter.

The use of precipitate will lead to maximal 2% reduction of filler material in paper making.

Increased process water reuse will avoid the need for heating fresh river water to the process temperature of 40°C. The FACT treated process water requires approx. 10%-20% less chemical additives for the same drainage than the untreated process water, and the quality of paper produced with the use of FACT softened water and FACT-precipitate is good.

### **Selective removal of substances**

In a first part of the work, the potential of a new reductive technology was evaluated for the removal of brominated compounds and organic halides (AOX) in general. The reductive agent investigated was zerovalent iron (ZVI), which is extensively applied in groundwater treatment, but is an emerging technology for wastewater treatment.

Batch screening tests were performed for a broad range of wastewaters and ZVI types. For AOX, removal efficiencies were usually below 50%, even at high iron concentrations. Removal efficiencies were best with nano-scale iron. For the removal of brominated compounds, efficiencies were on average higher than for AOX and reached up to 99%.

At the level of reactor concepts, granular iron was considered for treatment of lower concentrated streams in continuous column tests and in a magnetically stabilized fluidized bed set-up. Column tests showed that conventional activated carbon treatment in fact performed better than granular ZVI. Removal efficiencies were similar, time till breakthrough shorter and there was no increase in pH or released iron which may be problematic for discharge or reuse. The use of a magnetic field assisted fluidized bed set-up could be energetically interesting to obtain a fluidized bed system with powdered ZVI (clogging column set-ups) at reduced upflow velocities. In view of this concept, powdered ZVI was immobilized on magnetite to increase particle density and reduce washout. Particles aggregation in a high ratio magnetite:iron improved the reactivity of the mixtures. Unexpectedly, the concept was found to be too expensive for wastewater treatment because the cost of magnetite rose very strongly during the months of testing.

Nano-iron was applied in sequencing batch systems for concentrated wastewaters. For AOX in a textile wastewater, treatment of more concentrated water was more efficient, with at least 1600 ppm AOX being eliminated with 5.8 g of nanoZVI in 10 repeated sequences. The eliminated amount of AOX increased in an almost exponential way with the nanoZVI concentrations.

A second part of the work addressed the destabilization and removal of recalcitrant organic colloids in paper mills. During the last years, it was shown that there is an intrinsic limit in the removal of organic colloids with chemicals, estimated to be around 20%, measured as soluble COD. This was confirmed in the present work. In all cases a maximum removal of 20% of soluble COD has been obtained, even with high dosages of coagulant and flocculant. This is considered too costly by the mills.

These facts suggest not continuing working on this issue until new chemicals will be available as the available traditional products from the main chemical suppliers and also new chemicals (in developing stage) have been already tested.

As an alternative, removal of microstickies was investigated in an industrial ultrafiltration (UF) unit and the results compared with data from an industrial dissolved air flotation unit (DAF). Results show that up to 90% of these compounds may be removed by UF but the fibres and fillers present in the process water erode the active layer of the membranes which highly reduces the membrane life. Therefore, there is a technical limitation for this application. The short membrane lifetime also leads to an economically non-viable process.

### **Disinfection**

The state-of-art study on innovative technology for the disinfection of industrial reused water and reclaimed water, showed that monochloramine has been found to be more effective than free chlorine in controlling biofilms and coliform bacteria in systems with long detention times due to the lower decay rate of chloramine.

Innovative disinfection technologies are mostly physical technologies (pulsed electric field, radiofrequency power, ultrasound (US)) and photosensitization of nano-particles (TiO<sub>2</sub>). They seem to be efficient against micro-organisms depending on many parameters.

Though they are not used a lot at this time to treat industrial water, these technologies can improve classical disinfection processes when used in association. The efficiency of US when used alone is not very high, but in association with other disinfection processes, US can be very interesting to reduce dosage or contact time.

There are only limited literature data on disinfection processes for industrial water reuse. Physical processes could be an interesting alternative to industries in order to reduce their chemicals consumption and to limit the by-product formation.

From above discussed experimental results the following conclusions were stated:

- Spores of *Bacillus subtilis* are extremely resistant and no significant improvement in reduction of spores was observed with ultrasound treatment at low and high frequency,
- In comparison to ultrasound treatment of *Bacillus subtilis* spores, the reduction of *E.coli* is achieved in a very short treatment time,
- The higher the frequency, the better the inhibition efficiency for *E.coli*,
- The longer the treatment time, the better the inhibition efficiency for *E.coli*,
- The highest efficiency of inhibition was observed at 817 kHz for both 5 and 10 min
- The treatment at 817 kHz and 5 minutes presents optimal conditions that allow a decrease of *E.coli* K12 concentration for  $2.97 \pm 0.58$  Log in demineralized water for the ultrasound equipment used in this study.

### **Saline streams**

One of the objectives of AQUAFIT4USE was the development of new sustainable technologies for the treatment of saline streams. Three types of technologies have been considered:

a) CapDI (Capacitive Deionisation)

In a CapDI system salts are removed from the saline stream by the principle of capacitive deionization.

**Main results achieved are:**

- Developed modular CapDI system - A CapDI system based on unit modules has been developed that is now in production. Size of the system can be easily adjusted by installing more modules.
- Improved coated electrodes - Carbon coated electrodes showed to have excellent salt removal capacity, when compared to good quality commercial electrodes.
- Improved spacers: a low pressure drop spacer allows higher flow rates, resulting in the possibility to effectively treat a larger volume of water. This has been verified in the field.
- Improved membranes - PC Cell is able to produce ion exchange membranes at significantly reduced cost compared to those of Tokuyama (the industry standard), without loss of desalination performance.
- Better understanding of performance relations for different types of water. It is beneficial to increase the total salt flux presented to the CapDI system, when the total amount of salt removed is of most interest. For cooling tower applications, where the salt concentration of the treated stream is very low, this means that the flow should be maximized.
- Use of constant current operation to enable energy re-use. More than 75% of energy can be recovered at very low current densities. At practical conditions more than 40% of energy can be recovered.
- Experience of running a pilot system and full scale system on a cooling tower at two different sites
- Knowledge on filtering requirements for pretreatment for CapDI for cooling tower application.

**b) MDC (Membrane distillation-crystallization) for highly concentrated saline streams**

**In MDC the salts are concentrated and crystallized by a membrane distillation process:**

- Salt solutions can be concentrated up to saturation by MD (membrane distillation) without loss of specific flux (flux corrected for driving force)
- During crystallization a rapid flux decline is observed in the MD operation. It is expected that this is typically the case with salts like NaCl, but not with salts like CaCO<sub>3</sub>, because the lower temperature near the membrane due to temperature polarization is favorable for this type of salts with an inverse solubility-temperature profile.
- Applying OD (osmotic distillation, a type of membrane distillation) instead of MD gives a less sudden flux decline during crystallization, but has to be optimized e.g. by adding seeds to prevent crystallization on the membrane which still occurred in the experiments performed.

**c) Evapoconcentration**

Evapoconcentration is a technology commonly used to treat salty waste waters and results in relatively clean water (potentially suitable for reuse) and highly concentrated brine or precipitates. However this technology is energy consuming and it is necessary to design/develop small scale units which are thermally optimized (way of evaporation and the heat recovery system), and use standardized compounds. The design must as well take into consideration the corrosion aspect. The objective of the work done was then to define such type of units for the treatment

of industrial waste waters for the Pulp&Paper, Textile and Chemistry sector.

A state-of-the-art of evapoconcentration only dedicated to the part concerning the choice of an evaporator on basis of the effluent to be treated was done: evapoconcentration and its fundamentals were described together with the existing technologies to undertake evapoconcentration and basics for heat transfer.

A new concept developed and proposed by VITO for thermally optimized evapoconcentration units/combinations was as well described. It is based on a Combined Heat and Power (CHP) turbine linked to a mechanical vapour compression for a primary falling film evaporator and a secondary concentrate evaporator.

Evapoconcentration has been applied at lab scale to treat five different "concentrated" streams from the Pulp&Paper sector (i.e. membrane concentrates) and two from the Textile industry with high COD, chloride and salt contents:

- On basis of the characteristics of the flux to be treated, and the corrosion and scaling risks, the final global concentration factor which can be reached has been described together with the best configuration of evapoconcentrators to be applied at industrial scale.
- The evaporation technology has to be chosen considering the flux to be treated:

High flux: A falling film evaporator seems to be a well-adapted technology for this type of effluent combined with a forced circulation evaporator for an over-concentration step; this approach minimizes the total energy consumption.

Low flux: The effluent can be directly treated by a forced circulation evaporator.

#### **Use of micro-turbine:**

- The combined heat and power concept with mechanical vapour recompression and a micro-turbine proved to be an interesting and energy efficient approach for evaporation.
- However, it could not be tested in practice because the chosen scale for demonstration was too small. Therefore, it was evaluated in a case study corresponding to landfill leachates treatment for which industrial data were available. Three different configurations were evaluated by calculations. For such an application (landfill leachates), the micro-turbine could be an interesting technology to produce heat and electricity from biogas.

#### **Treatment trains as custom made solution - reliable and cost-effective**

The objectives in this area was the identification of the best new (combinations of) water technologies to reduce environmental impacts by advanced closure of the water cycle and produce the required water quality for re-use. The work concerned the laboratory and preparations of pilot trials in the two industrial paper mills and two textile mills and case studies in all 4 sectors

#### **Laboratory trials Pulp and paper sector**

In the Pulp&Paper industry a lot of effort is put into water saving and closing water circuits, also reducing substantially the environmental impact, both by process modelling and Kidney technologies as internal

process water treatment. Kidney technology aims at removing specific substances from the recirculating process water. However a number of problems around the removal of substances are not solved yet and further closing of the water cycle causes other problems.

Challenges for water reuse in the Pulp&Paper industry are the following:

- The elimination of residual (soluble) COD and BOD which can both affect the production process and the paper quality;
- The removal of sticky solids and suspended solids, which can induce plugging of pipes and showers, deposit formation, abrasion, loss of tensile strength;
- The treatment of concentrate streams containing calcium, sulphate, chloride and organics which can lead to salt accumulation in case of water loop closure, corrosion, scaling of pipes and showers in the paper production process. The removal of calcium carbonate is crucial in the last case.

Therefore there is a need to find new and reliable (combinations of) technologies to solve this challenges to achieve the water quality target for water re-use and which are tailored to suit product demands and standards.

Based on the waste water characterization and the defined water quality requirements for paper mills, new treatment lines focused on internal recycling were defined to reach the water quality target including effectiveness, reliability and minimization in waste and concentrate production.

The emphasis was on different key steps in the overall treatment train:

- Biological treatment: anaerobic processes and MBR;
- Filtration processes: 3FM high speed technology and nanofiltration;
- Tertiary treatments to reduce recalcitrant COD and scaling: advanced oxidation processes, coagulation, precipitation;
- Integration of processes (evapoconcentration, electrodialysis and softening) in the treatment line to treat the concentrate streams containing calcium, sulphate, chloride, organics, to minimize the waste production and enhance internal recycling.

Technologies were tested at lab scale on the wastewaters from two different paper mills: Hamburger Rieger (HRT), producing high quality coated and uncoated board from recycled paper and Holmen Paper Madrid (HOL), producing standard newsprint, improved newsprint (higher brightness) and lightweight coated paper (for magazines).

The most important findings for corrugated board paper mills are:

- A softening step is needed to assure steady/good performance of membrane processes (MBR and NF):

Stable MBR operation is not possible at calcium concentrations greater than 400 mg/l due to scaling problems. Softening upstream of the MBR is then absolutely necessary.

Intensive pre-treatment or conditioning is needed to obtain steady NF membrane performance and high recovery rates due to the high scaling tendency (membrane blocking) of aerobic effluents. Softening of wastewater allowed higher recovery up to and lower chemical consumption for conditioning (no-use of hydrochloric acid).

In this view, the Multiflo™ softening technology is well adapted to remove calcium carbonate.

- 3FM technology showed good performances at lab scale regarding TSS removal and turbidity reduction.
- Further treatment of NF concentrates is needed for liquid discharge in municipal waste water to comply with the legislation. A further treatment step is needed.
- Evapo-concentration is well adapted to treat membrane concentrates: the amount of final waste to be disposed is reduced (down to 0.5-0.7% in volume of waste water treated by the global treatment line) and the produced water meets the highest water quality criteria for reuse in the paper production.
- Re-injection of NF concentrates upstream biological treatments has a negative impact on anaerobic degradation rate in pellet sludge reactors.
- AOPs applied to membrane concentrates did not improve biodegradability.

**The most important findings for newsprint paper mills are:**

- Anaerobic pre-treatment showed very good performance treating a low organic load wastewater as the effluent of a 100% recycled NP/LWC paper mill, and assisting the aerobic stage on removing organics and sulphates; besides it produced enough biogas for being considered as cost-effective. Wastewater quality after biological treatment was suitable to perform a posterior membrane treatment.
- Membrane treatment by UF + RO is able to generate permeates of high water quality, fulfilling all the requirements for being used in critical points of the paper machine that require a very high water quality.

However, silica is a main issue for reuse as it leads to a reduction of recovery for RO treatment and scaling in UF treatment:

Different chemicals have been tested to achieve the silica removal efficiencies necessary to avoid silica scaling on RO membranes in the advanced treatment of the paper mill effluent: 80-90% silica removal was achieved by coagulation at different pHs. Coagulation treatment can be as efficient as required (greater than 95% removal), however, the costs are relatively high due to high dosages required and/or the use of complex chemicals.

Softening at high pH with the addition of magnesium compounds can also be used and efficient removal of silica is achieved down to the required values.

- Further treatment of RO concentrates is needed for liquid discharge in municipal waste water to comply with the legislation. A further treatment is needed:

Evapo-concentration is well adapted to treat membrane concentrates: the amount of final waste to be disposed is reduced and the produced water meets the water quality criteria for reuse in the paper production. AOPs applied to membrane concentrates allow to reduce the recalcitrant COD, TOC and colour. The water can then be sent to the (closest municipal) WWTP.

In general:

- Ozone and AOPs were validated for both type of paper mills as final polishing treatment for water to be reused concerning disinfection, color removal, TOC and COD removal and biodegradability improvement.

**Laboratory trials Textile sector**

The current situation reflects that the typical textile company prefers to use fresh high quality water in all production operations processes after softening to preserve the quality of their final products.

Wastewaters are mostly discharged without any pre-treatments into the sewer, since sophisticated wastewater treatment technologies are mainly not affordable for small to medium-sized textile companies. Water reuse in the textile finishing industry needs the development of very specific processes (which can also be chosen among the already available ones) in order to produce the "fit-for-use" water for a specific process (or a few processes).

In addition, the challenge for implementing treatment of textile waste waters for re-use purposes stands in the characteristics of processes' textile waste waters, which have an extremely high variability with frequently changeable composition (seasonally, even daily, due the use of numerous different dyes and auxiliary chemicals), that could substantially differ from company to company. Then, the selected concept in the AQUAFIT4USE project is based on segregation of very complex textile streams regarding their pollution level and their separate treatment. This concept increases the applicability of conventional treatment technologies, as the ones described above, and also increases the possibility for real water reuse implementation in textile sector.

The emphasis was on different treatment technologies applied to treat separately low, medium and high concentrated textile waste streams from two Slovene textile finishing companies Tekstina and Svilanit.

Tested technologies were mainly based on:

- For low and medium concentrated streams:

As single treatments: Membrane processes (UF, NF), coagulation, oxidation processes (UV/H<sub>2</sub>O<sub>2</sub>, UV/H<sub>2</sub>O<sub>2</sub>/catalyst, UV/H<sub>2</sub>O<sub>2</sub> (in situ electro synthesized H<sub>2</sub>O<sub>2</sub>)), A<sub>2</sub>O-MBR (Anaerobic biofilm/Anoxic/Oxic-MBR) and SAMBR (Submerged Anaerobic Membrane Bioreactor);

Combinations of some of these technologies: "UF + NF", "UF + AOP", "coagulation + UF", "A<sub>2</sub>O-MBR + NF" and "A<sub>2</sub>O-MBR + AOP".

- For high concentrated streams and concentrated streams produced from NF treatment of low concentrated streams: evapo-concentration.

From the performed experiments following conclusions were drawn:

- Treatment of low concentrated streams:

The combination of "UF + UV/H<sub>2</sub>O<sub>2</sub>" seems to be efficient in producing the right water quality for subsequent reuse in dyeing processes. Reuse experiments indicate the lowest colour difference.

The combination of "coagulation +UF(HF)" gives comparable removal of colour and COD even though the values are a bit higher than the guide values for reusability and treatability. The best filterability was observed after the coagulation treatment. Reuse experiments indicate exceeded values for allowed colour difference.

- For medium concentrated textile streams MBR in combination with UV/H<sub>2</sub>O<sub>2</sub> or NF is also a possible treatment train combination.

- Evapo-concentration proved to be well adapted for the treatment of high concentrated textile streams and of membrane (NF) concentrates produced from non-concentrated streams. The produced distilled water has a quality which fulfils water quality requirements for re-use purposes: no impact on the coloured textile was observed in reuse experiments in cotton dyeing processes.

In addition, it should be possible to have a cross treatment between non concentrated and concentrated streams to produce water for re-use and to decrease final waste to be treated.

- Reuse experiments are necessary and useful, but their results are not always in agreement with expectations regarding the recommended values for water reuse criteria. Performed reuse experiments suggest that some deviations in recommended maximal values of reusability criteria are possible.

The proposed treatment system pays attention to maintain the technology train as simple as possible:

- In this view, the ultrafiltration is applied for water pre-treatment (with the exception of very low polluted wastewater as described above) for nanofiltration or AOP.

- Moreover, UF is also the proposed filtration process for the application of MBR for medium-concentrated wastewaters.

- In addition, a possible cross-treatment between low-concentrated and high concentrated is proposed in case the distillate produced by evapoconcentration has a too high COD content or absorbance value (e.g. case of Stenter distillate):



## **Case studies: The proof of applicability**

### **Paper sector**

Based on laboratory work where a range of new and reliable (combinations of) technologies were evaluated, the most promising concepts have been scaled up successfully in different pilot trials. The focus has been on water quality targets for water reuse in-line with product demands and standards whilst avoiding any negative impact on paper machine runability and paper quality. As a result, customised pilot plant trains have been implemented.

### **Tests at Hamburger Rieger**

Extensive tests involving seven simultaneously operated water treatment technologies were run at a German paper and board mill from February to September 2011. The dimensions of the trials were extraordinary in terms of both volume flows and the variety of treatment lines. Various processes could be operated in parallel as well as in series.

The technological and economic benefits of tailor-made water treatment lines have been investigated by comprehensive pilot trials. The trials were aimed at increasing the water reclamation rate of paper mills. Individually, each of the chosen treatment technologies represented the state-of-the-art in the paper industry in a particular case. By combining these advanced technologies to customized treatment trains, innovative research enabled breaking new ground in forward-looking water treatment.

The special composition of paper mill wastewaters calls for tailor-made treatment concepts. Consequently the following main results have been derived:

- Water softening has been proven as an essential pre-treatment whenever membrane processes are to be used.
- Recirculation of nanofiltration concentrates in the anaerobic reactor after pre-treatment by ozone oxidation turned out to be unreliable in some cases.

Using evapo-concentration for NF concentrate disposal has been evaluated as an alternative treatment technology.

- Two treatment lines including biological and physico-chemical processes have been proven to be technologically as well as economically viable:
  - (1) Anaerobic treatment -greater than Conventional activated sludge process -greater than Softening -greater than Pre-filtration -greater than Nanofiltration -greater than Evapo-concentration of concentrates
  - (2) Anaerobic treatment -greater than Softening -greater than MBR - greater than Nanofiltration -greater than Evapo-concentration of concentrates

### **Holmen**

This paper mill, producing recycled paper from 100% recovered paper, has optimized its water circuits for a longer period. Nowadays, they are consuming about 8 m<sup>3</sup>/T, which is lower than the estimated consumption in the BREF document for papers mills manufacturing newsprint paper. The required quality for any further closure of the circuits is very high, close to potable water quality.

During AQUAFIT4USE, after a deeper study of the current situation, two pilot plants with a capacity of 1 m<sup>3</sup>/h were installed in parallel to compare two different types of pre-treatments before the RO units.

The treatments tested were:

- Solids filtration
- Anaerobic treatment (UASB/EGSB)
- Aerobic treatment (activated sludge/membrane bioreactor)
- Ultra-filtration (pressurized hollow-fiber)
- Reverse osmosis

These trials had the objective of increasing effluent reclamation and substituting fresh water in this paper mill. The main results in AQUAFIT4USE were the following. Anaerobic treatment was able to remove 50% and 70% of the initial COD and BOD5, respectively, in both pilot plants, even though the presence of sulphates in the effluent is high in relation to COD values. Furthermore, these treatments produced enough biogas to be considered as cost-effective (valuated at 700,000 Euros/year).

As part of the (biodegradable) COD and BOD5 was not removed in the anaerobic step, the aerobic stage was necessary before feeding the membrane treatments. COD and BOD5 removals were acceptable after the aerobic treatment to further perform a posterior membrane treatment. Although both pilot plants showed similar organic and sulphate removal rates, the pilot plant which holds the MBR treatment, showed a more stable operation behaviour regarding the removal of organics and fouling, but it was more expensive in terms of energy consumption.

Nevertheless, the effluent from the UF step showed similar characteristics and the RO treatment of both plants also showed a similar behaviour. Silica content at the exit of the UF steps was about 150- 210 mg/L. As a consequence scaling phenomena could not be totally avoided at the silica levels present in this mill. Therefore, considering these results, and in order to avoid an irreversible damage to the RO membranes, the tested systems could only achieve a maximum recovery of about 30-50%. Additional laboratory research for removing the silica content showed that coagulation and precipitation could be feasible options.

In all the cases the permeate from the RO stages fulfilled the quality requirements for the critical points where fresh water is consumed in the paper mill, and hence, its use could be replaced by the recovered effluent. Finally, RO rejected fractions also met the requirements set by the legislation in force for accepting the final effluent in the closest municipal wastewater treatment plant.

The final economical evaluation comparing the use of reclaimed water from the closest municipal wastewater treatment plant (mWWTP) and the regeneration of the effluent within the mill, in this particular case, showed that nowadays, particularly due to silica content in this mill, the most economical option for substituting fresh water is the use of reclaimed water from the closest mWWTP, although both initiatives are sustainable and economically viable. Therefore, nowadays, Holmen Paper is using for its production reclaimed water and it is the first mill in Europe producing 100% recycled paper using 100% reclaimed water.

### **Case studies Chemical sector**

The pilot work with the chemical industry focused on three major aspects:

- The application of membrane bioreactor technology (MBR) on variable and harsh chemical wastewater
- Piloting of various treatment trains for total effluent reuse

- Investigating technologies for trace organics removal combining valuable product recovery and local water reuse.  
The tests were executed at two sites of BASF and Perstorp.

### **BASF**

The integrated production site of BASF in Antwerp, Belgium, was considered a worst case scenario within the BASF-group to evaluate MBR process stability for total effluent treatment because it combines 54 production units in 4 production sectors.

During 1 year, 3 different submerged MBR pilots were tested in a set-up consisting of membrane filtration tanks coupled directly to the full-scale aeration basin, thereby taking into account site specific constraints. The 3 selected technologies represented different membrane module configurations and different membrane materials. It was concluded that membrane separation of mixed liquor from the BASF Antwerp treatment plant was technically feasible at flux ranges of 8 - 20 l/m<sup>2</sup>.h. However, membrane permeability was clearly influenced by wastewater composition, with several simultaneous upsets in all pilot units. More scaling and fouling was observed than originally expected and this caused the need of frequent cleanings. In addition to the rather high cleaning frequency, membrane recoveries were sometimes unpredictable. The test results thus underachieved the expectations for a stable and reliable long-term full-scale application at the BASF Antwerp site.

In view of water reuse, MBR is generally considered an excellent pre-treatment step for application of reverse osmosis (RO). In our test trials, the Silt Density Index already gave an indication that the permeates would lead to severe fouling of RO membranes, and were unacceptable as a feed without pre-treatment. Simulations at various recoveries and with or without the addition of antiscalants confirmed that many ions were present in too high concentrations to allow for RO treatment. Thus, (part of) the hazardous scalants must be removed before the MBR permeates can be fed to a RO installation.

Complementary to the pilot tests, the MBR-VFM (VITO Fouling Measurement) monitoring sensor was adapted for tests on chemical wastewater. Over a period of around 1 year, various fouling indicators were determined weekly in parallel with MBR-VFM measurements on mixed liquor from the three running MBR pilot units at BASF Antwerp.

Measurements with various types of membranes and measurement protocols gave similar results. Significant fluctuations were observed over the test period for both reversible and irreversible fouling propensity.

Because fouling in MBRs on chemical wastewater was mostly linked to variability in the wastewater composition, it seemed promising to test various fouling reducing additives to counter the negative impact of substances in the wastewater on fouling propensity.

Out of 8 tested fouling reducers, the cationic polymers Nalco MPE50 and Adifloc KD452 and 2 novel cationic polymers, developed by BASF, were found to have a positive effect and high effectiveness on mixed liquor filterability. The MBR-VFM was found to be a suitable tool to screen novel additives and benchmark them against existing ones.

### **Perstorp**

The second test site was Perstorp Specialty Chemicals AB in Sweden. Here, various treatment trains were assessed in view of total effluent reuse. Emphasis was put on the possibility to find the minimum water quality fit-for-use for cooling purposes and other reuse options for low grade water quality, while keeping good cooling or process water practise by reducing the biofouling and hygienic risks. The tested treatment trains included MBR, RO, Denutritor and AOP technology in various combinations.

As opposed to the results obtained at the BASF Antwerp site, MBR operation was reliable and membrane performance recovered well after cleanings. MBR performance on chemical wastewater is thus clearly site dependent.

The results also showed that while the effluent from the MBR pilot was not improved as compared to the CAS facility with respect to conventional dissolved wastewater parameters, the biofouling potential of the MBR effluent was significantly lower and comparable to effluent from the combination CAS/Denutritor. The low biofouling potential being a highly important quality improvement for most reuse options, it can be concluded that the effluent from MBR or CAS/Denutritor is significantly better suited than CAS effluent for reuse purposes at Perstorp.

The tests demonstrated that RO treatment on CAS and MBR effluent was a suitable technology for production of high quality water/process water/cooling water for reuse in the chemical industry. All three water qualities could be produced when RO was used as final treatment. Cooling water make up for evaporative cooling towers could be of lower water quality than the permeate from the RO except that the treatment is needed for removal of salts. Other technologies removing salts (NF, ED) could also be used. The permeate from the RO has better quality than the present process water and could be used as such without further treatment. Make up water for the steam generation plant is considered a high quality water application. Today normal process water with extra treatment by ion exchange is used to produce this water. As the permeate from the RO has better quality than the present process water it can replace the process water but still needs polishing by the ion exchange or electrodeionization. When used with the present ion exchange technology, the life time of the ion exchange resin is expected to increase significantly.

Based on economic feasibility the suggested reuse scheme looks reasonably attractive for Perstorp AB as a step towards increasing production capacity without increasing the effluent discharge volume. This is important for Perstorp Specialty Chemicals AB as the effluent discharge volume today is the bottle-neck once production capacity increase is needed.

In addition to total effluent treatment and reuse, local reuse scenarios were investigated as well. Polishing technologies to reduce trace organics to very low levels are essential to a successful water recovery on a local level. Organic substances with low molecular weight, e.g. methanol, formaldehyde, are in general a barrier for reuse in the chemical industry.

A tailor-made technology concept for product recovery by RO, supplemented by additional polishing treatment of the RO permeate for reuse as cooling tower make up and/or low-grade process water was investigated.

However, the retention of trace organic compounds in the waste process streams is low thus resulting in a RO permeate contaminated with relatively high concentrations of these trace organics. These and in particular formaldehyde can be easily degraded by AOP but not to any significant degree by conventional oxidation using ozone.

High formaldehyde removal efficiencies can also be obtained with MBR technology but attention has to be paid to reduce stripping, and to avoid shortages of nutrients.

As several process streams in the chemical industry are loaded with recalcitrant organics, AOP treatment could be beneficial to increase the potential for local water reuse. Pilot tests performed on four single streams showed biodegradability improvement by AOP and to a much lower extent by ozonation. Ozonation is only efficient for detoxification of specific compounds, such as. phenol containing wastewater streams.

### **Case studies Food sector**

In the food industry the main points of attention in relation to sustainable water use and closing the water cycle are health and safety. Therefore, for this sector the focus was on applications where no extra risks for health and safety are introduced. This includes alternative water sources for application in no-direct contact with the product and the application of new technologies for the production of process and product water from existing and safe water sources.

Cleaning is one of the most water, energy and chemical consuming processes in the food sector. For this reason an in-depth study was carried out at Nestle Waters, Unilever and Alpro factories, covering a variety of topics. This study was carried out in two steps.

A first step focused on the optimization of existing Cleaning in Place (CIP) processes.

A second part of the study consisted of identifying new and alternative CIP technologies that could help to save water, time/labour, chemicals and/or energy, e.g. use of chemicals "2 in 1" that clean and disinfect at the same time, pigging systems, ozone and/or chlorine dioxide disinfection to disinfect equipment.

In addition, four different treatment technologies were explored at pilot scale:

- Denutritor for the treatment of rainwater, to make it applicable for washing and cleaning (Unilever's ice cream factory Ben&Jerry's, the Netherlands)
- UF-Filtration on the effluent of a biological treatment plant, to upgrade the effluent for make-up water for cooling water system (Soy Factory Alpro, Belgium)
- Filtration Assisted Crystallization Technology as alternative for the softening of drinking water (Soy Factory Alpro, Belgium)
- Evaluation of different filter media for the removal of specific chemical compounds for the production of bottled water (Nestlé Waters, France)

### **Denutritor for reduction of micro-organisms in rain water**

Unilever Ben&Jerry's ice-cream Factory in the Netherlands is aiming at the reuse of collected rainwater to minimize the amount of drinking water

in their production. This rainwater can be used for cleaning of packing and transport material (containers, pallets, etc.) and for sanitation (toilets) at the plant location. Denutritor can potentially improve the microbial quality of stored rainwater, by reducing the amount of available nutrients in the water and thus minimizing the microbial growth and biofilm formation.

Biofouling potential was reduced with 72% and the AOC content in the rainwater was depleted to concentrations below 10 µg AOC/l.

Besides the pilot tests indicated that by Denutritor treatment risk for accumulation of pathogens in the rainwater reservoir tank was reduced, and combined with relatively low doses of UV, collected rainwater can be safely applied for sanitation purpose in food industry.

#### **UF for effluent treatment for cooling water preparation**

The application of ultrafiltration for polishing a tertiary effluent stream from the biological waste water treatment was successfully tested at the soy factory of ALPRO in Wevelgem to upgrade the biological effluent for make-up water in cooling water systems. Based on the results of the pilot test it was recommended to perform the UF filtration at a gross flux of 17 l/m<sup>2</sup>.h, in presence of 1 ml/min iron dosing.

To have an indication of the suitability for use as RO feed, the potential for RO membrane fouling was evaluated and compared with sand filtration. Breakthroughs of suspended solids were sometimes detected for the sand filtration, while these did not occur in the ultrafiltration effluent.

Based on the results of the pilot study, the estimated operational cost of a UF installation for the production of 10 m<sup>3</sup>/h effluent would be 41,950 and 24,450 EUR/year, respectively with and without depreciation cost.

The operational costs of a RO installation of 7.5 m<sup>3</sup> permeate/h and a calculated conductivity below the norm of 600 µS/cm for cooling water make up, with and without depreciation cost, were respectively 23,100 and 12,100 EUR/year for the treatment of 10 m<sup>3</sup>/h pre-treatment effluent.

The total operational and water costs are 65,050 EUR/year and 0.99 EUR/m<sup>3</sup> cooling water make-up, including depreciation cost (first 10 years).

#### **FACT for softening of drinking water**

A series of FACT pilot test trials was carried out at ALPRO in Belgium for softening City Water from three sources, with average calcium concentration of 116 mg/l.

During the pilot tests at Alpro it was shown that hardness can be reduced by 80% and the final calcium concentration in the softened water is circa 20 mg/l calcium. The residual calcium concentration is limited by the dissolved carbonate present in City Water. Lower calcium concentrations are obtainable by adding extra soda to the City Water. At pH=10 magnesium is only partly (~30%) removed from City Water.

The laboratory filtration tests showed a good filtration with clear filtrate. At pilot scale this could only be repeated for short periods, due to problems with the control of the process conditions, leading to clogging of the filter. This aspect needs to be further explored.

At present ALPRO pays 0.56€/m<sup>3</sup> for costs associated with their ion exchange softening columns. If the fixed costs, which already have been made are deducted, then the price is 0.44 EURO per m<sup>3</sup> softened water. Total costs for FACT are estimated at 0.25€/m<sup>3</sup> +/- 40%.

### **Evaluation of different filter media for the removal of specific chemical compounds**

To evaluate the efficiency of different filter media for the removal of specific chemical compounds in bottled water industry, laboratory and pilot tests have been carried out with different technologies, based on adsorption and ion exchange. The focus was on arsenic, boron and bromide.

The results of the pilot tests have proven that titanium-dioxide based media are an efficient selective treatment for arsenic removal with very high adsorption capacity. TiO<sub>2</sub> is a non-regenerative media, similar to granular ferric hydroxide-based media but with higher adsorption capacity. This treatment does not consume chemicals for regeneration and water losses are therefore very limited: 21 g Fe(OH)<sub>3</sub>/m<sup>3</sup> versus 8 g TiO<sub>2</sub>/m<sup>3</sup> treated water.

No significant impact on water quality except during the 1st days after start-up of the pilot was observed.

The main results from the test for Boron removal with a strong basic Anion exchanger are:

- Boron removal efficiency was high, from 7.5 mg B/L to less than 25µg B/L.
- The impact of the ion exchanger on water composition does not allow a treatment of natural mineral and spring waters, but other applications, with less critical quality demands than these types of waters are possible.
- Water loss estimations are comparable to those of reverse osmosis.
- Ion exchange technology requires less energy than reverse osmosis but chemicals consumption is important.

For the removal of Bromide the resin Purolite Bromide Plus was selected for pilot testing as it has the most suitable characteristics for treating the water tested without changing its composition too much. The main results of the tests are:

- Efficient bromide removal, from 28 µg/L to less than 5 µg/L, allowing ozonation of the treated water without bromate formation
- Low impact on the water quality compared to a RO process, but for the treatment of natural mineral and spring waters the impact on water quality is still too high
- High chemicals consumption during regeneration step.

These experiments showed that bromide removal resins were capable to remove high quantities of bromide with little impact on the water quality and low water losses compared to reverse osmosis. However, the impact is still considered too large for its application in the production of mineral water.

### **Case studies Textile sector**

The textile finishing companies Tekstina, Svilanit and Inotex do not implement any water reuse in 2012. Fresh and high quality water is used in all the production processes, usually after softening. All the water used in the production, except for the amount that is lost (mainly

evaporation), is collected through the wastewater network and, after neutralization, sent to the municipal WWTP. Wastewater includes: cleaning water, process water, cooling water and storm water. The amount of water used varies widely, depending on the specific process operated at the mill, the equipment used and the prevailing management approach regarding water use.

The "Manual for the characterisation of textile SMEs" (PIDACS) was issued and used in Tekstina, Svilanit and Inotex production characterisation. The document was aimed at guiding partners in the data collection procedure providing a systematic factory investigation and production processes characterisation. In such a way a detailed analysis, containing all the relevant process data for further elaboration of a comprehensive mapping of all processes related to water use was obtained.

This manual and datasheets proved to be easy to use by companies and very useful for further company studies. The same procedure could be used for checking any textile finishing company interested in wastewater reuse and it can be adapted to other industrial sectors characterized by a similar (weekly, monthly and yearly) variation of effluents discharges.

A very complete database was obtained with the complete characterization of relevant textile production processes. On the one hand data concerning water use, chemicals and energy were collected for a total of 46 production processes including yarn scouring, bleaching, dyeing and fabric desizing, bleaching, mercerizing, dyeing, printing and washing. On the other hand all batch discharges from each process (in total 184 different samples) were analyzed by measuring relevant physical-chemical parameters (in total 25). An example of a batch process description is given in Annex 2.

These data have a high utility for future applications in any other similar textile finishing company, allowing to complete characterisation of textile wastewater without the necessity of repeating such a huge characterisation work. In such a way the overall data collection will require much less time and effort.

Definition of criteria for the effluent separation in low concentrated and high concentrated effluents and definition of criteria for water reuse.

The distinction between low and high concentrated effluents was based on effluents potential treatability by proposed technologies (membrane technologies, AOP) and their reusability in textile processes. Definition of both criteria was established on the basis of literature search and was refined through wastewater treatment on laboratory scale and reuse tests.

Further definition of criteria was established on the basis of literature search and was refined through wastewater treatment on laboratory scale and reuse tests.

### **Water reuse network design**

Different reuse network scenarios were designed on the basis of existing water and wastewater network analysis, from the effluents characterization and through water and contaminants balances calculations. Scenarios are based either on machinery separation or on effluents separation based on continuous monitoring of the effluents



characteristics. In these scenarios wastewater treatment technologies evaluated were different combinations of UF, NF, AOP, MBR and evapoconcentration. For Tekstina the most relevant reuse scenario based on machinery separation while for Svilanit based on monitoring separation.

Mass balances for water and pollutants (based on wastewater chemical/physical characterization) were calculated for Svilanit and Tekstina for the evaluation of the potential to reduce water use. In both companies most machinery is used to carry out different processes and their discharges are very diverse in pollution level. Investigated companies are very different in annual volume and pollution level of major waste streams. In Tekstina three machineries present less than 1% of the total wastewater volume while amounting to almost 50% of the total COD load. For this reason the separation of waste streams based on machinery would allow to segregate significant pollutant loads in a very small volume.

In Svilanit no reuse network based on machinery separation is useful as very different processes are carried out at almost every machine. As an alternative an effective way of effluents segregation based on effluents monitoring was proposed.

That makes a reuse treatment possible at viable technical and economical conditions, with very good characteristics of treated effluents. It could compensate the additional cost for the realization of the dual wastewater network and the monitoring devices.

All scenarios were analyzed in terms of the water savings allowed and quality of the reclaimed water. Cost efficiency evaluation was made based on the results of pilot scale testing.

#### **Pilot testing of proposed reuse schemes**

In Svilanit the waste stream separation was based on the monitoring of relevant parameters mainly due to the polyvalent application of different textile processes in the same equipment. The majority of the wastewater is low concentrated (approximately 60%) and treatable by conventional membrane technologies like UF, NF. For Svilanit the treatment train composed of UF and NF or UF and AOP has been verified to be effective enough to meet water reuse criteria. Moreover, for specific very low concentrated streams (e.g. the last fabric rinsing) only UF or AOP as standalone treatment technology were sufficient to provide reusable water. Estimated investment costs for the treatment train composed of UF and NF amounted to 230 kEUROS and operation costs 18 k€/a.

On the contrary, in Tekstina, equipment is much more specialized. Some machines produce very concentrated streams that contribute 48% of total COD and 19% of total conductivity although representing only 1% of overall annual discharge. Separate treatment of these high concentrated streams by evapo-concentration further reduces the amount of wastewater and produces distillate treatable with technologies for low concentrated streams and has big reuse potential. The rest of the waste streams after mixing are low to medium concentrated (99%). For these streams, combinations of biological treatment (anaerobic/MBR), membrane processes and/or advanced oxidation process were tested. MBR allowed significant colour and good COD removal, but post treatment step is necessary. As a post treatment NF is equally replaced by UV/H<sub>2</sub>O<sub>2</sub>. Proposed segregation of waste streams in this company allows the reduction of fresh water

consumption up to 85%. Estimated investment costs for MBR are 180 kEUROS, for NF 130 kEUROS -150 kEUROS for a scale of 22-26 m<sup>3</sup>/h, and 160 kEUROS for evapo-concentration when 3 m<sup>3</sup>/day of concentrated streams will be treated. The investment of proposed treatment train would amount to 490 kEUROS and corresponding operation costs to 46 k EUROS/a.

In the company Inotex the proposed treatment concept composed of flocculation, biological treatment and AOP was very efficient for the tested mixed wastewater containing reactive dyestuffs; the final result of combined technologies was complete decolourization of mixed streams including elimination of organic non-coloured pollution.

### **Cross-sector benefits: real-life experiences in four sectors**

One of the important overall objectives of the project was the search for cross-sector benefits. Technologies, management strategies applied in one sector could be beneficial in other sectors as well. Instead of developing separate routes in each sector, it is also good to look for synergies between the sectors. Although specifically designed for one sector, some measures could also be of interest for other sectors. The starting point for this work was the study of 4 cases, one in each sector. Four companies were investigated and the water network was scrutinized. The goal was to characterize, analyse and compare the water networks in the four sectors and find benefits beyond the sector level. These benefits can be situated on different areas: the water network itself, process conditions of water treatment or other processes, optimization strategies. The water networks of a paper mill (SAPPI), a food company (CHS), a textile company (Textina) and a chemical plant (Perstorp) were described and visualized. The visualized water network also immediately defines the scope of the optimization. Each case was handled by a different partner to optimize. Hence, a comparison could be made in the way the optimisation problem was approached (the WESTforINDUSTRY tool was not used in the cases, as it was not available at that moment).

Due to the invariability in the applied processes and the intrinsic high water demand, the paper sector has developed several, dedicated tools to improve water networks and close water loops. In the past a specific approach was developed, which easily shows the performance of a paper mill water network, based on the calculation of three key parameters. These so-called "K-values" provide useful information on fresh water use, separation of circuits and counter-current arrangement. The major hurdles to be taken in paper industry are the removal of recalcitrant COD from the effluent, high silica and calcium levels but also chloride concentrations in the loops that cause respectively scaling and corrosion. The major bottlenecks for water loop closure are the impact of the water quality on the product quality, the formation of 'slime' in the paper machine, the control of odour and the calcium levels in the waste and process water streams.

In the chemical sector water pinch has been used for many years as a tool (amongst others) to optimize water networks, although certainly not exclusively. Water reuse has been widely applied in chemical companies, mostly for in-process applications to recover as much product as possible in subsequent purification and separation processes. This was proven by the results from the pinch analysis that was performed on one plant at the Perstorp production facilities. The main water consuming processes in the chemical sector are the cooling towers and the steam production. The

latter relate to the use of energy, the first to the dissipation of energy. Taking control of energy demanding processes and recovering energy in a counter-current process directly decreases the total amount of energy, hence the amount of steam needed, hence the amount of cooling water that is needed to cool the process down. The main bottle necks in the chemical companies are the presence of (small) organics and salts. Mostly residual concentrations of (in)organic reagents need to be removed in order for the process stream to be able to be re-used. Salts also impair certain chemical processes or equipment and need to be removed. This of course generates a new problem: the creation of a concentrated stream that has to be removed as waste or treated further (e.g. evapo-concentration). Also temperature can sometimes create difficulties for re-use, so heating or cooling is often required.

For food industries water reuse is quite difficult -but certainly not impossible- due to the stringent water quality demands. As drinking water quality is required in all 'food processes', reuse automatically involves multiple treatments to remove unwanted substances: filtration, advanced oxidation processes, reverse osmosis, disinfection technologies. All are needed to provide and ensure the water meets the drinking water standards. On the other hand, non-potable water could be used in 'non-food processes' like cooling towers and sanitary use.

Textile companies -although highly water-intensive due to subsequent washing processes- generally do not have a long tradition of water savings. In numerous cases fresh water is used to ensure product quality -colour in particular- is not affected. Batch processes also limit the practical possibilities of reusing water as large buffers need to be installed to collect the water streams. Very often ground water is used as a cheap -and chemically stable- water source, consequently economic evaluations often show that reuse scenarios are too expensive. Moreover the extensive use of salts and highly recalcitrant organic compound also aggravates the possibilities for water reuse (or at least the costs). However the pinch technology, previously used at Perstorp, revealed some interesting possibilities for reuse by segregating highly contaminated and relatively clean waste water streams and treating them with a combination of membrane bioreactor and nanofiltration.

For all sectors three important rules of thumb are used to: reduce fresh water usage, use alternative water sources, use eco-efficient water treatment.

In general, the project has developed some very interesting pieces of knowledge, experience, technology, etc. that can be used across the sector borders. Looking at water management tools the WESTforINDUSTRY tool comes to mind in the first place. But also HACCP -hazard analysis of critical control points- a specific approach used in the food industry to control product safety can be of great value for other industries. Especially when reusing treated effluent as process water, it is of prime importance to control and secure the water quality and to be able to adjust in the right way in case it is out of spec. The methodology can be used to define and control the critical control point in virtually any industrial water loop. Paper and chemical sector in particular also introduced a new way of thinking about closing water loops.

By allowing very high concentrations of a limited amount of contaminants within the loops, costly and energy-intensive treatments can be avoided and water reuse can be intensified (water is used 20-30 times before

being discharged). Another valuable principle from the food sector is called 'hygienic design'.

This is a list of rules that describe how equipment, piping and pumps should be designed in order to be able to maintain hygienic properties. This automatically involves shorter cleaning times, less product loss, hence less contaminated waste water and more possibilities for reuse. Also alternative technologies for cleaning in place (CIP) like 'pigging' could be very useful, especially in the chemical process industry.

Closing water cycles in industrial companies automatically entails monitoring of the various components of the water circuit. Several devices and techniques were developed within AQUAFIT4USE that can be deployed in all four sectors. The VIT-kit for fast and easy detection and quantification of specific target organisms, the VFM-sensor for detecting fouling in MBR-systems, WEA (whole effluent analysis) for determining real ecological impact of effluents, FBRM (focused beam reflectance measurement) for fast analysis of concentration and size of particles in a solution and on-line sensors for COD, nitrogen, orthophosphate and calcium. Also the LCA and water footprint methodology that were customized specifically for this project proved to be very useful to assess the sustainability of certain scenarios

Several cutting-edge treatment technologies were tested on lab- and pilot-scale: MBR, NF/RO, AOP, Denutritor, FACT, CAPDI, evapoconcentration, etc. Each of these had a specific role in solving reuse problems and other challenges related to sustainable water use. Tests showed good results for many of the technologies in different sectors.

Finally the developed and further explored treatment technologies showed to be all applicable in different combinations, treatment trains, that were developed for the custom made solutions for the pilot locations in the four sectors of industry. These real-life experiences were the most important results for making steps forward in sustainable water in industry as a whole.

## **Potential Impact:**

During the development of AQUAFIT4USE the expected general impact of the project was defined as follows:

AQUAFIT4USE will contribute to strongly improved sustainable water use in industries, leading to

- reinforcing the competitiveness of both the water consuming industry as the water technology suppliers,
- getting new market opportunities from the results of AQUAFIT4USE.

More specific impacts of sustainable water use on resource efficiency, product and processes are:

- Substantial reduction of fresh water needs and more efficient use of limited water resources in European Industries for the four sectors with the highest water demand by integrated water resources management, also by  
new cross-sectorial technologies and other solutions and the implementation of the integrated process technologies in the four industrial sectors
- Substantial reduction of effluent discharges to mitigate the environmental impact of water use and water treatment, by new technologies with higher recovery and/or lower wastes and by-products and a better process integration
- Better management of health and safety risks in water use by new technologies, monitoring systems and water management tools
- Improved process stability and product quality by improved and more constant water quality.

After more than four years of research it can be concluded that on both the general level and on the more specific topics AQUAFIT4USE has had a great impact on industrial water use. Besides the development of technological solutions for the main challenges, the dissemination and training activities played an important role to come to these achievements.

## **General Impact**

The main overall result of AQUAFIT4USE is that it showed that closing the water cycle (by reuse of water) and the use of water sources other than drinking water can be done on a safe and reliable way for several industrial sectors. AQUAFIT4USE showed this very convincing by setting-up pilot testing and treatment trains. The project showed it not only to its project partners but also to the broad outside world, thereby opening doors for the realization of future demonstration projects.

AQUAFIT4USE realized that, in order to bring about this change in the water use paradigm in the different industrial sectors, it is very important to create more awareness. First of all about value and importance of water for industries, but also about questions like:

- Where is the sector is not sustainable in relation to water
  - Water use
  - Emissions
- What kind of solutions are available
- Which solutions have other sectors available
- What are the real bottlenecks.

With the help of comprehensive inventories and the developed tools, like WESTforINDUSTRY, the participating parties gained a great deal of insight

in the situation of their water system and in the effect and bottlenecks of the applications of technologies to make it more sustainable. This has led to big changes in the way the participating companies will use their water in future. Thanks to the large number of dissemination and training activities, this will also achieve big changes for the water using industries especially in the four sectors paper, food, textile and chemistry, resulting in lower water and water related costs.

### **Specific impact on water use, process and product**

#### **Water (re)-use**

The improved insight of the participating water using industries in their water use and the opportunities will lead to a reduction of the use of fresh drinking water of 30% in the coming years. This decrease in the use of water will differ by sector as some sectors (like the paper one) did already close their water cycle to a great extent; for others, however, a more or less complete closure of the water cycle is possible in future, leading to a higher reduction. To give some figures from the participating partners, one paper company reduced their water and energy use with 15 -20% thanks to the better insight in his water system, a second paper industry changed to 100% use of reclaimed water. One of the textile partners made plans to go to a far going closure of the water cycle. For one of the chemical industries the reuse of effluent will lead to a reduction of water use of over 20%. Also for one of the Food companies effluent reuse is an opportunity, besides the change to a different water source, reducing the need for softening the water

#### **Mitigate environmental impact**

The environmental impacts of the project are focused on:

- Reduction of (high quality) water use and the use of other water sources. This is a result of the above mentioned water reuse and closure of the water cycle.
- Reduction of effluent discharge. The focus in AQUAFIT4USE was not on end-of-pipe treatment but water reuse, which leads to a reduction of the volume finally discharged. A disadvantage of this approach is that the concentration of pollutants or problematic species is increased (see also next point). For one company replacing the ion exchange softening by a crystallization process, the amount of salts discharged (from the regeneration) reduced with over 90%.-
- Concentrates and brines. Closure of the water cycle often leads to more concentrated streams that cannot be discharged. By the development of new desalination technologies like Flow Through Capacitor and improved evapo-concentration the volume of brines will decrease and possibly the regain of raw materials can be improved
- Less energy and chemicals use. The reuse of warm and/or cold water will lead to a reduction of the energy needed by 20 to over 50%. Besides, scaling prevention has a great impact at the functioning of heating and cooling systems. The development of new biofouling and scaling prevention technologies have reduce the use of water conditioning chemicals both for utility water and in paper processing

#### **Better management of safety and health risks**

##### **The safety and health risks are reduced by:**

- The development of a HACCP based Water Quality Control Methodology. An improved insight in health and safety risks of the water system is an important part of this tool
- The development of new probes for faster analyzing micro biological parameters

- Improved disinfection technologies, like Denutritor and better insight in the effect of nano-silver

The impact on this area is hard to measure, but is directly coupled with water reuse, and the use of other sources than drinking water, especially for the food industry. So the expected impact of AQUAFIT4USE on these topics is considerable.

### **Improving process stability and product quality**

There is a very strong link between water quality and processes and product. For all industries a better control of the water quality will lead to less production stops, less fall out of products and a better product quality. For food products this is related to the above mentioned safety issues. For textile industry it has been proven in different lab tests of dying of fabrics.

In paper industries it is proven that a better removal of disturbing components will make a more stable process. These topics have not been tested extensively, but based on the available knowledge a considerable effect of a good water quality control at the product quality might be expected.

Via the different partners, different sector organizations are involved in AQUAFIT4USE. The results of the project will be used in future to improve the IPPC documents for these sectors to define BAT.

### **Contribution to standards**

AQUAFIT4USE has also collaborated with other initiatives as in the case of the European Water Partnership.

As a response to Europe's water challenge of achieving Sustainable Water Management in Europe by 2030, according to the Water Vision for Europe, the European Water Partnership (EWP) initiated the Aquawareness Program as a strategy to move Europe towards a resource efficient and sustainable water culture, targeting political decision makers, key stakeholders and European citizens.

To reach this challenge, in December 2008, the EWP launched the Stewardship Program. The aim of this project is to provide a tool to communicate and award responsible water users through the development of a common framework for assessing, implementing and communicating Sustainable Water Management (SWM).

This SWM tool is based on a voluntary system consisting of:

- An objective assessment of the water management of an operator, and
- A transparent, open, dynamic and representative SWM Standard on which the assessment is based and realized on.

Complutense University of Madrid, Holmen and BASF have contributed to develop the SWM Standard by selecting the indicators and criteria to be considered and by implementing the 1st draft in Holmen for its evaluation and improvement. Results have been also applied into the company to move towards a more sustainable water use. After the implementation of the standard in a second round of pilots UCM and BASF continue with the support to the development of the final standard which was recently launched.

### **Societal implication**

In 4.3 an overview is given of the societal implication of AQUAFIT4USE.

There were no specific ethical issues.

Over 200 persons were involved in the project, of which 40% women, so no specific gender equality actions were needed. Remarkable is the high number of female PhD students. In the project a lot of attention is paid to the high-level education especially at the universities. Different disciplines are involved like Engineering sciences, Chemical sciences, Mathematics, etc.

The project did generate a lot of useful output for policy makers, especially in the field of Environment, Energy, Food safety. The outcome will be used for general policy making and supports different water directives (f.i. WFD) gives input to the BREF documents, supports new initiatives, like the EU flagship initiative on resource efficiency and is very useful for the definition of the future research agenda. AQUAFIT4USE is mentioned as 1 of the 15 featured projects of the FP6 and 7 Framework Program, in the document 'Horizon 2020 Investing in Success'.

### **Socio-economic impact**

The economic impact and the effect of AQUAFIT4USE on the competitiveness of both the water using industries and the suppliers is difficult to estimate but will in most cases be considerable.

For the end-users sustainable water use and closing the water cycle can lead to a substantial cost reduction. In general the savings on the water costs itself will not be the most important part. For most industries the costs for water intake are not more than a few percent of the total costs, but the water related cost can be plural. The saving of energy by reuse of warm water streams, reduction of losses of raw materials and lower discharge and treatment costs can result in a saving of five to over 25% of the total production costs.

Additionally the reduction of water use can give the company opportunities to grow, which sometimes is not possible due to legislation on water withdrawal and discharge.

Finally the increased knowledge and better control of the water quality will often lead to a better product quality and less fall-out.

For the suppliers of (technological) solutions new market opportunities are created, both for new technologies and combinations of technologies developed in AQUAFIT4USE. WESTfor INDUSTRY as a new Water management tool applicable in different sectors, Capacitive Deionisation for the treatment of cooling water and Multiflo™-softening are examples of new developments that are available for the industrial water market now, but also technologies for biofouling and scaling prevention (Denutritor, FACT) are close to implementation.

Yet, a greater impact on the market for treatment technologies can be expected from the application of combinations of existing and emerging technologies in the so-called treatment trains for custom made solutions studied and tested in our initiative. Finally the development of new monitoring and analyses tools, like the around 150 new developed specific probes for in-situ analyses of micro-organisms will lead to new market opportunities.

### **Dissemination and training**



## **General**

To create real impact a very active dissemination of the project findings and further transfer of the knowledge by training and exploitation activities is essential. Due to the strong applied character of the project the focus of the dissemination was on the industrial suppliers and end users, but also other stakeholders, like policy makers, researchers and students were informed about the results. The knowledge transfer was partly carried out inside the project, between the partners, and between the different sectors. This will be elaborated further below.

## **Types of activities**

Different types of dissemination activities were performed, like oral communications at congresses and workshops, articles, scientific papers, posters, newsletter and the website

Dissemination activities were classified in general and specific ones. The former are those aimed for a wide audience not having a deep knowledge on water science and technology. The topics of general dissemination activities could be specific developments of the project but these were then always presented in an accessible manner.

The target audience of specific dissemination activities was assumed to be familiar with scientific and engineering aspects of industrial water systems. The focus of this type of activities consisted of detailed project findings or developments.

It can be observed that in the case of general dissemination, the first and the last year were the most prolific ones in terms of activities carried out. Both, oral communications and articles were of almost equal importance. The majority of specific dissemination activities was concentrated in the last two years of the project, which is coherent with the progression of knowledge generated.

## **Dissemination Activities**

### **AQUAFIT4USE Website - <http://www.AQUAFIT4USE.eu>**

The AQUAFIT4USE project portal was set up from the beginning and upgraded several times in the course of the project. The website offers public access contents as well as a private section for AQUAFIT4USE partners. The public section is updated periodically with water related news and information on industrial water use events. The project newsletters are posted on the home page to enable easy access by the users. The AQUAFIT4USE website differs from other funded project websites in that the project portal offers a menu from which the visitor can download valuable scientific contents, such as the presentations given in the different congresses organized in the frame of the project as well as those articles considered publicly available.

### **AQUAFIT4USE newsletters (AquaFit4NEWS)**

Given the light style in which they are written and the synthetic way of presenting the information, the AQUAFIT4USE newsletters denominated "AquaFit4NEWS" were conceived as a key tool for the project consortium to communicate with the public, not only regarding the hard technical or scientific results achieved but also considering the social implications of the project. . The consortium issued a total of 15 newsletters. A few newsletters were dedicated to general topics, like the general announcement of the project start, the midterm and End congress, the participation in other congresses, the collaboration between AQUAFIT4USE and EWP, and the participation of future researchers. Other were more

dedicated to a specific sector (paper, food, textile chemistry) or a group of specific results (tools and modeling, monitoring, fouling and scaling) and one on the scientific publications

### **Congresses**

Partners of AQUAFIT4USE have participated in a lot of congresses on different levels, from individual presentations of project results, the organisation of a special session in a congress to the organisation of two specific AQUAFIT4USE congresses, the Midterm Congress and the Final Congress. The latter two mentioned were the main events in which the visibility of the project was given a boost.

### **The AQUAFIT4USE Midterm Conference**

The AQUAFIT4USE Midterm Conference took place in Oviedo (Spain) between 13th -16th June 2010 and was organized in collaboration with The Integral Water Cycle: Present and Future, 7th International Congress of ANQUE (Spanish National Association of Chemistry-related professionals). A total of 26 oral communications were given in the event presenting the results achieved till then, from the assessment of a sustainable water use in industry or the modeling of different water stages, to the application of advanced treatments and the results obtained in the project industrial cases.

The presentations were divided in four technical sessions:

Session 1.- Sustainable Water Management

Session 2.- Waste Water Treatments

Session 3.- Advanced Treatments

Session 4.- Good examples of Industrial Cases

In addition, an AQUAFIT4USE-Poster session was organized. The session was the platform to show additional project results not covered in the presentations and also to present technical offers from project partners to the conference audience. The conference also included two open AQUAFIT4USE-Seminars, with the titles:

Membrane Bioreactor Seminar and Workshop (organized by Veolia and VITO).

Chemometric methods as useful tool for characterization of different environmental samples (organized by University of Maribor).

Finally, also framed within the event, a demonstration session of the Water Quality Management Tool developed within AQUAFIT4USE was held. The demonstration served to improve the tool with feedback from the conference participants.

### **The AQUAFIT4USE End Congress**

The Congress was celebrated in cooperation with the I-SUP Conference, that was held from 6th to 10th May 2012 in Bruges (Belgium). Since the i-SUP event focused on sustainability, it was considered the perfect platform to achieve a good visibility and audience for the AQUAFIT4USE End Congress.

There were 5 sessions during the 2 day conference also including a number of external presentations:

- Session one was an integrated part of the i-SUP program, starting with a plenary speech of Durk Krol of WssTP and a key-note presentation of the EC on future perspectives for innovation in the water sector, Horizon

2020. This session was followed by presentations about sustainable water use in the chemical industry.

- Session 2: Industrial water loop closure: technologies

Keynote Thomas Wintgens, FHNW: Technological solutions in sustainable water use

- Session 3: Challenges and solutions for paper industry

Keynote: Angeles Blanco, UCM: Sustainable water use in the paper sector

- Session 4: Challenges and solutions for textile industry

Keynote: Walter Lutz, Euratex: Vision for a sustainable water use from EURATEX

- Session 5: Challenges and solutions for food industry

Keynote : Renaud Sublet, Nestlé: Ensuring water quality and safety in sustainable water use programs in the food and beverage industry.

In addition to the oral session, the consortium organized a poster session in which the technological offers of a large number of project partners were exhibited.

Sessions in other congresses

AQUAFIT4USE was invited to organize a special session in different congresses:

- MIDW2010 Congress Trondheim (June 2010)

- TNAV workshop AQUARAMA, Mechelen, November 2010

- IWA Water and Industry Conference in Valladolid (1st - 4th May 2011)

- ECOMONDO Rimini Italy, November 2011

### **Papers and articles**

Over 50 papers, articles and other news items were written and published in different types of Journals and magazines, both national and international.

The great majority of specific dissemination items is constituted by oral communications and related papers. The number of peer-reviewed scientific papers produced in the project was 19. This rather low number can be explained by the application focused research in the project.

### **Exploitation**

The knowledge generated has helped the industries of the sectors considered in the project to improve their water systems either by reducing their fresh water consumption or by increasing the efficiency of their water related processes. This knowledge is in most of the cases not suitable for intellectual property protection. The following project results however, can be understood as complete, distinct blocks of knowledge and can be subject of clear commercial exploitation. For each one of the results below, a plan for exploitation, dissemination and training was prepared and updated in the course of the project:

The Water Quality Management Tool (WESTforINDUSTRY)

VIT-kits

FACT

CapDI

Denutritor.

These exploitation, dissemination and training plans were used to support the developers in the next steps towards marketing of the results.

### **Training activities**

From the project proposal phase, it was decided that training would be organised in three different levels, namely:

- Learning-from-each-other activities. These activities entail the transfer of knowledge, from one sector to another. The initial idea of transferring project generated knowledge was expanded to consider the transmission of any type of technical or scientific knowledge that

especially the industrial partners may require Through knowledge  
prospection, in total 46 learning-from-each-other sessions were organized  
in the course of the project, some of which were also open to audiences  
from outside the consortium.

- Lifelong learning activities. This type of training consisted mainly in updating the technical knowledge of the staff of industrial partners. The typical training scheme associated to this concept involved an RTD performer training companies of its own sector. In some cases, training was also open to external companies of the same sector. At the end of the project. 19 lifelong learning had been carried out by the project partners.

- Education of young researchers. Education of young researchers was considered of crucial importance for the project, since they are the ones who will ensure that our project results generate a lasting value, enabling the transfer and consolidation of existing and newly acquired know-how in the organizations hosting them. The activities dealing with education of young researchers took several shapes in the project. Some activities involved the short stay of young researchers from a project partner in the facilities of another for intensive training (complementary to the learning-from-each-other concept). In other cases, the researchers collaborated directly in the hosting organization using the knowledge generated in the preparation of a master dissertation or a PhD thesis. 71 activities concerned with the education of young researchers have taken place in the course of AQUAFIT4USE.

## **Special Dissemination activities**

### **The AQUAFIT4USE movie**

With support of EC dissemination project STREAM a video was made of the project.

The AQUAFIT4USE video provides an overview of the reasons for initiating a project focusing on a more sustainable water use in the European industry. It includes the general background, and more detailed technical aspects of the projects explained by consortium members. The collaborative approach of the project, the AQUAFIT4USE learning-from-each-other motto, is emphasized. The video is available on the project website and has been shown at different occasions.

The AQUAFIT4USE final Brochure: 'Bringing innovation to sustainable industrial water use in Europe'

A brochure of the project has been made summarizing the main results in an accessible way for a broad audience. The brochure was presented at the end congress and 1000 copies were distributed afterwards to interested stakeholders

Horizon 2020 Investing in Success. Research and innovation to boost growth and jobs in Europe

AQUAFIT4USE is mentioned at the Horizon 2020 website and in the report 'Horizon 2020 Investing in Success' as one of the 15 featured projects of the FP 6 and 7 programme.

According to the Commissioner for Research, Innovation and Science, Máire Geoghegan-Quinn, the results from the mentioned projects have shown great strides in innovation and will make a positive change to our daily lives.

Cooperation with EU dissemination project Innovationsseeds

AQUAFIT4USE has cooperated with the project Innovationsseeds for extra dissemination activities on the project results. Fact sheets have been made of a number of successful results for the virtual library and AQUAFIT4USE attended different meetings organised by this project.

### **List of Websites:**

<http://www.aquafit4use.eu>