Solvents are widely used in process industries such as the chemical, petrochemical, pharmaceutical, food and electronics sectors. Strategies for dealing with industrial liquid waste involve reducing both the quantity produced and its environmental impact. Recent EU statutory provisions require consideration of waste minimization and environmental impact. Innovative, efficient and cost-effective solutions are required for reduction, purification and recycling of hazardous liquid waste streams. The recovery of spent solvents containing all types of impurities presents a vast market targeting thousands of small to medium sized companies in Europe that are generating highly as well as marginally polluted solvent streams. Many
current solvent manufacturing and recycling practices rely on traditional, energy-intensive distillation based technology. This method of processing is costly, inefficient and produces large volumes of hazardous waste.

SOLVER aimed at debottlenecking current distillation based solvent purification practices with cost-effective, nano-selective membrane processes. The general objective of SOLVER was thus to strengthen the market positions of European solvent purifiers/recyclers by offering them access to innovative, advanced membrane technologies enabling them to provide more competitive products and services. The two membrane techniques studied in SOLVER were organic solvent nanofiltration (OSN) and pervaporation (PV) enabling separation of solvent based mixtures at the molecular level by applying a pressure gradient across or a vacuum at the permeate side of a selective membrane, respectively. Such chemical process intensification in the solvent recovery and purification business is expected to lead to significant reductions in production costs, materials usage, energy consumption, waste production, as well as risks and hazards.

The scientific objective of SOLVER was to acquire a better insight into the solute transport through nano-selective OSN/PV membranes. This was pursued by the use of advanced on-line monitoring techniques and was coupled to the development of a model enabling description of transport across the membranes. The consortium, comprising seven partners from five different nations, was built around four innovative SMEs at different positions of the value chain in the solvent purification and membrane production business, each building out new business lines to stay ahead of competition. Thanks to the interdisciplinary and complementary skills of the consortium members, SOLVER was able to demonstrate the practical feasibility of advanced membrane technologies in solvent purification and reclamation practices through lab-scale and pilot testing, but also to better understand the processes at the molecular scale. The SME partners are expected to continue to leverage on the technological head-start after the project, take the results of the work directly to the market, find new applications for their products, and be the market leaders in their respective sectors.

Project Context and Objectives:
The current global recession has brought the cost of chemical and pharmaceutical manufacturing into very sharp focus, with the world’s leading companies actively seeking innovative pathways to ensure efficient production and sustainability for the future. There is widespread acceptance that the next phase of global wealth will be generated in the ‘green’ economy. Governments and businesses alike are preparing for investments in technologies of the future, which are focused on providing environmentally friendly and renewable solutions to the energy issues society and business faces. Development programmes with a solid scientific basis, focused on the core values of the green economy, have a significantly better opportunity to flourish in the next 10 years than at any other time in history. There is an appetite for industry to embrace new greener technologies as part of the industries’ strategy for continual improvement and growth, as well as securing market share. A perceptible move by larger corporations towards ‘factories of the future’ and innovative pathways for sustainable chemical production is clearly visible. In many cases, the focus of chemical research does not only lie in the search for new compounds and structures, but in the optimization of production processes and usage of basic chemicals, intermediates and fine chemicals already known to society for many decades. The chemical factory of the future will be faster, more flexible and environmentally benign. It is imperative to be able to produce chemical products and intermediates that have minimal environmental impact with cost-effective nanotechnologies. This will lead to a significant reduction in the overall carbon footprint of chemical processes by dramatically reducing the energy requirements for chemical plants, allied to significant cuts in waste streams.
reducing the energy requirements for chemical plants, allied to significant cuts in waste streams. This of course will encompass the area of solvent reclamation and recycling, which opens up a large market. Solvents are widely used in process industries such as the chemical, petrochemical, pharma, food, biotechnology and microelectronics sectors. In the pharma industry, for instance, the average mass contribution of solvents to the production processes of Active Pharmaceutical Ingredients (APIs) amounts to 85-90%, with the energy, health & safety, and environmental aspects of such processes being overwhelmingly determined by solvent production, use and disposal. Strategies for dealing with industrial liquid waste streams involve reducing both the quantity produced and their environmental impact. Recent EU statutory provisions require consideration of waste minimization and environmental performance. It is illegal to discard used solvents due to grave ecological implications, and it also does not make economic sense to do so due to their high cost. On the other hand, sustainable solvent recovery poses a most difficult challenge to many industrial applications. Innovative, efficient and cost-effective solutions are required for reduction, purification and recycling of hazardous liquid wastes in particular. The recovery of spent solvents, waste streams as well as process streams, containing all types of impurities presents a vast market, targeting thousands of small to medium sized companies in Europe that are generating highly as well as marginally polluted solvent streams.

Many current solvent purification and recycling practices rely on an energy intensive vaporization technology. This method of processing is costly, inefficient and produces large volumes of hazardous waste. Since up to 70% of production costs in the process industry are associated with downstream separation, industry aims at more efficient, clean and reliable separation processes. Current separation technologies often show major drawbacks such as a high energy demand, a limited selectivity, difficulties to be integrated in production processes and limitations with respect to thermally labile products. Besides simple incineration, solvent recovery has traditionally been achieved through distillation, and when impaired by azeotrope formation, azeotropic distillation with entrainers. At the turn of the 21st century, distillation technology was, and still is, the preferred technology for the purification of most liquid chemicals. The reason for its widespread use is the lack of viable cost-effective alternatives. Distillation is intrinsically robust and highly effective, and has succeeded in supplying the pharma, microelectronics and related industries with raw materials for many years. The last 20 years has brought an efficiency drive on this technology, which has seen manufacturers gain increased product throughput without large scale investments. This increase has enabled them to stay apace of the increasing global demand, but development of new distillation technology has now reached the point of virtual exhaustion. The limitations of distillation technology are indeed being felt today by global solvent manufacturers, with increasing energy costs, high manual intervention requirements, high levels of hazardous waste being generated, and increasing pressure on competitiveness from emerging economies, primarily due to lower capital and labor costs. Contamination of the products by entrainers is for instance a very significant problem in the pharmaceutical and microelectronics industries where high solvent purity is mandatory.

SOLVER proposed to debottleneck or even eliminate the thermal steps involved in distillation-based solvent purification, and replace them with cost-efficient, nano-selective membrane processes. When successful, these advanced membrane processes would provide a sustainable alternative for simple incineration of solvent waste streams that can potentially be recycled and re-used within factories after usage. Such chemical process intensification in the solvent recovery and purification business is expected to lead to significant reductions in product costs, materials usage, energy consumption, waste production, as well as risks and hazards. This way, it was the consortium’s ambition to contribute to a paradigm shift away from the current, distillation-based solvent purification practices and simple disposal by incineration. As a mild, non-destructive, and energy efficient separation technology, membrane processes have
As a mild, non-destructive, and energy-efficient separation technology, membrane processes have experienced a significant growth over recent years, spurred by increasing environmental concerns, rising energy prices and the need for higher product quality. This technology has set the new standards for treating aqueous streams, which still form the major membrane market. Meanwhile, new technical achievements and a growing acceptance of membrane technology in industry have recently increased interest in using membranes to separate organic solvent streams as well. As new membranes are able to separate organic mixtures at the molecular level things get particularly exciting. Organic solvent nanofiltration (OSN) and pervaporation (PV) are two emerging membrane techniques enabling separation of solvent based mixtures at the molecular level by respectively applying a pressure gradient across or a vacuum at the permeate side of a selective membrane, and could offer a sustainable alternative for classical distillation-based solvent purification.

Due to their modularity and scalability, these membrane technologies can not only be used for end-of-pipe recycling of solvent waste streams, but they could also be easily applied within the chemical processes themselves and thus contribute to solvent loop closure. Solvent recovery and re-use can indeed greatly enhance the atom economy and thus the efficiency and sustainability of chemical production processes. Despite their huge application potential and the clear ecological and economical benefits, PV and particularly OSN cannot be considered mature technologies yet. Despite the steadily increasing number of implementations worldwide, the huge potential of both membrane techniques in solvent recovery and purification remains largely unexploited and the present market is relatively small. This limited penetration in industry can amongst other reasons be explained by the limited know-how in industry of this kind of innovative membrane processes. SME solvent producers, end-users and recyclers in particular often have no access to such emerging technologies. The fact that solvent filtration is much more complex than standard aqueous membrane processes which have become state-of-the-art, makes the technology even less accessible to the non-specialist. Furthermore, several technological/scientific challenges remain, such as the small number of robust, high performance membranes on the market, the lack of straightforward membrane selection guidelines, the poorly understood transport mechanism, and the lack of suitable pilot scale test facilities.

However, it is expected that membrane-based solvent filtration will acquire a significant market share in Europe in the near future. This especially holds for OSN which has since its emergence at the beginning of the nineties been an almost exclusive European research focus and technology, with all major membrane suppliers – mainly SMEs – being present in Europe. This stands in clear contrast to the water membrane market, which is dominated by large non-European corporations that have much more resources than European players and can thus benefit from economies of scale. In the solvent filtration market, however, this is not the case and European SMEs can be competitive by offering innovative products with excellent performance. In view of the expected market growth of OSN in solvent intensive processes across a wide range of industries, there is a perceptible need for the development of more membranes with even better separation performance and solvent stability, for demonstration of OSN technology at an industrially relevant scale, and for a better insight into the separation mechanism of the membranes at the molecular scale.

The general objective of SOLVER was to contribute to the establishment of modern, sustainable purification practices in solvent intensive chemical manufacturing processes by developing and demonstrating advanced, nano-selective membrane processes at both lab and pilot scale. The use of membranes could create a substantial drop in capital expenditure for new plants, primarily through retrofitting high performance membrane separation units into existing solvent purification facilities. This represents a key step in developing sustainable chemical processes since solvent purification using...
represents a key step in developing sustainable chemical processes since solvent purification using membranes would allow recycling of solvents within closed loops, which would significantly reduce the volumes of solvents consumed and of waste streams produced.

The scientific objective of SOLVER was to acquire a better insight into the transport of solutes through OSN/PV membranes and into their behaviour during filtration, thus contributing to a better fundamental understanding of these emerging membrane processes. This was pursued by using advanced on-line, real time process monitoring tools and the development of a predictive model describing solute transport across the membranes.

The consortium aimed at transferring the know-how acquired within SOLVER into new, more competitive products and services offered by the SME end-users, so as to reinforce their respective market positions. Apart from the obvious environmental and economical drivers, the importance of technology leadership in Europe in the areas of solvent manufacture/recycling and membrane production must be emphasized, especially in view of the commercial threat from Asia where chemical, pharmaceutical and electronic products are being manufactured at much lower costs than in Europe. SOLVER could contribute to ensuring that the EU chemical production remains competitive at global scale and that production facilities are not lost to emerging low cost base economies. Moreover, the project would reinforce the European OSN/PV technology leadership and boost the global membrane market for solvent filtration.

The soundness of the project objectives was based on the experience, track record and complementary nature of the project partners. The project consortium consisted of experienced SMEs at different positions of the value chain in the solvent purification and recycling process. SOLVER joined two types of innovative SME end-users building out new business lines to stay ahead of competition. GKA Technologies Ltd (GKAT) and De Neef Chemical Processing (DNCP) are both solvent manufacturers/recyclers with a large experience in distillation technology and a vast client portfolio in the process industry. They challenged OSN and PV membranes in their processes, focusing on the high purity (GKAT) and technical grade solvent market (DNCP). On the other side of the strategic supply chain, two innovative SME membrane suppliers participated, namely SolSep BV (SOL) and Pervatech BV (PERV), having wide expertise in the development and production of polymeric OSN and ceramic PV membranes, respectively.

The format of the proposed research in SOLVER matched very well the interests and expertises of the research partners since there was a clear need for, on the one hand side practical expertise with OSN/PV processes and access to test units, and on the other hand scientific understanding of complex membrane processes. VITO, The Flemish Institute for Technological Research, coordinating this project, has a large know-how and practical expertise with OSN and PV, mainly through extensive contractual research for companies in the process industry, including SMEs. The other research partner, Fundação da Faculdade de Ciências e Tecnologia da Universidade Nova de Lisboa (FFCT-UNL), has an international reputation in advanced characterisation and process monitoring tools allowing the study of membranes and membrane processes at the molecular scale and in real time, as well as in membrane transport and process modelling. The last project partner was Packo Inox NV (PACKO), a world renowned manufacturer of stainless steel tanks and process vessels, CIP units and centrifugal pumps for the pharmaceutical and other industries.

Project Results:

1.3.1. Background on membrane technology, particularly OSN and PV

1.3.1.1. Introduction
A membrane process can be defined as a separation process where two bulk phases are physically separated by a third phase, the membrane. The membrane is a semi-permeable barrier between these two phases and may comprise either a nonporous, or a nano-, micro- or macroporous solid film. A schematic representation of a basic membrane process is shown in Figure 1. A separation is achieved because the membrane selectively transports components from the feed mixture to the permeate. The transport mechanism and driving force for this selective transport can be diverse and depend on the type of membrane process. Different membrane processes can be classified according to their separation principle which is determined by the physical or chemical characteristics of the components to be separated. The four main properties of molecules relevant to membrane separations are size, charge, vapor pressure and affinity. Furthermore, membrane processes can be subdivided according to the driving forces required to initiate the separation.

Figure 1: Schematic representation of a membrane process.

As a mild, non-destructive, waste- and energy-efficient separation technology, membrane processes have experienced a significant growth in recent years, spurred by increasing environmental concerns and rising energy prices. The technology has set the new standards for treating aqueous streams which still form the major membrane market, for instance in the production of drinking water and process water, and in waste water treatment. Meanwhile, new technical achievements and a growing acceptance of membrane technology in industry have recently increased interest in using membranes to separate process and waste streams containing organic solvents as well. Especially organic solvent nanofiltration (OSN) and pervaporation (PV), two promising, nano-selective membrane processes, can offer a sustainable and economically attractive alternative for energy-intensive distillation, waste-generating extraction or solvent-intensive chromatographical separation processes. Despite their huge application potential, the potentially large cost savings and the clear ecological benefits, these emerging membrane processes cannot be considered mature technologies yet. Even though both techniques have shown to be feasible for solute and/or solvent recovery from various industrial, solvent based process and waste streams, the present market is relatively small and the number of implementations at industrial scale is so far limited.

1.3.1.2. Organic solvent nanofiltration (OSN)

Molecular separations in aqueous solutions have been possible since the latter part of the 20th century with the advent of reverse osmosis (RO) and nanofiltration (NF) membranes enabling the separation of ions and small organic molecules from water. Until recently, both separation techniques have been almost exclusively implemented at industrial scale in the treatment of aqueous streams, e.g. for desalination of sea and brackish water, production of process water, waste water treatment, and processing of milk and beverages. Pressure-driven membrane separations in organic solvents have emerged as a new area of membrane technology during the last decade, with huge potential across chemistry-related industrial sectors. Especially OSN that allows separating solvent based mixtures at the molecular scale and in a non-thermal way by simply applying a pressure gradient over a selective membrane offers great perspectives towards wide application in solvent intensive processes in the process industry. Commercial NF membranes were initially developed from RO membranes. The first NF membranes on the
Commercial NF membranes were initially developed from RO membranes. The first NF membranes on the market were essentially “loose” RO membranes, which allowed monovalent salts to pass through. While these membranes perform very well in aqueous environments, they lose their integrity and/or provide poor separation performance in most organic solvents. Solvent-stable OSN membranes came on the market around the beginning of this century, even though pressure-driven solvent separations had been occasionally mentioned already before. For the last 10-15 years, there has been considerable academic activity in developing solvent-stable membranes, and in the last 5-7 years, an increasing number of commercial high-performance OSN membranes have been brought to the market. Today there are 4-5 suppliers offering both polymeric and ceramic OSN membranes and modules, enabling molecular separations in virtually all solvents.

During the last years, OSN is finding application across a wide range of separation problems. With a molecular weight cut-off (MWCO) between 200 and 1000 Da, a range covering a large number of industrially relevant product molecules, OSN membranes can for instance be applied to recover homogeneous catalysts from their reaction mixtures, to isolate, concentrate or purify pharmaceutical products or transfer them from a high boiling to a low boiling synthesis solvent, to purify and fractionate natural extracts containing thermally labile molecules or to process edible oils extracted with organic solvents. Research on OSN has so far been mostly focusing on the recovery and purification of high value product molecules.

However, OSN obviously also allows to purify and recycle spent solvents as well. Such solvent recovery could contribute to solvent loop closure and is particularly attractive for solvent-intensive processes such as the extraction of vegetable oils. The first and so far largest industrial OSN plant, successfully running already for more than 10 years, is the Max-Dewax™ process at the ExxonMobil refinery in Beaumont (Texas) for the recovery of spent dewaxing solvents from lube oil filtrates. The key to successful operation of this large-scale solvent recovery process, handling 11 500 m³ of dewaxing solvent per day, was the integration of the OSN unit with the existing process. OSN allowed debottlenecking the conventional, distillation-based solvent recovery process the expansion of which was needed but practically limited by the refrigeration and solvent recirculation capacity. The capital investments for the OSN installation only amounted to about one third of the costs for the same capacity increase using conventional technologies. Moreover, the membrane-based solvent recovery process only required 25% of the heat consumption, 10% of the refrigeration capacity and 20% of the size of the conventional unit, overall resulting in a payback time of less than one year. SOLVER aimed at further exploring the potential of OSN in solvent recycling but also in the high end purification of solvents for exacting applications.

Thanks to its mild character, low energy consumption, scalability and ease of operation, OSN is rapidly becoming a valuable part of the toolbox of separation specialists and process engineers in the fine chemical, pharmaceutical and related industries. Its capability to efficiently separate and purify target compounds, remove post-reaction residues and recover spent solvents, provides its users with a competitive advantage in producing high quality products and closing material loops. The industrial implementation of OSN is however hampered by a few challenges, which include the complex and very case-specific interactions between solvent, solute and membrane, the lack of clear membrane selection guidelines, the poorly understood transport and separation mechanism and the lack of suitable pilot test facilities. The inherent complexity of this kind of membrane processes where discrimination occurs at the molecular level stems from the wide range of solvents, solutes and membranes used, and from the associated interactions at the molecular scale between the permeating species and the membrane material. In OSN, these interactions start to play a key role, in addition to and often even more dominant than mere molecular size. This makes OSN less accessible for the non-specialist and makes prediction of
than mere molecular size. This makes OSN less accessible for the non-specialist and makes prediction of membrane performance difficult. Extended experimental screening is therefore almost a prerequisite to marginally optimize OSN processes.

1.3.1.3. Pervaporation (PV)

‘Pervaporation’, being the contraction of ‘permeation’ and ‘evaporation’, is a membrane separation technique that involves the use of a non-porous polymeric or nanoporous inorganic membrane in contact with a liquid feed. It involves the separation of a feed mixture from which target compounds are removed by selective vaporization through the membrane. The driving force for transport is a chemical potential gradient across the membrane. In practice, a gradient in partial vapor pressure across the membrane is achieved by establishing a vacuum or applying a sweep gas at the permeate side. This partial vapor pressure difference can be further enhanced by heating the feed mixture.

Although PV involves a phase change, the energy consumption is much lower than for distillation, since the separation efficiency mainly depends on the affinity of the target compounds for the membrane. PV offers most potential in those cases where classical distillation fails, for instance when the liquids to be separated form an azeotrope, have similar boiling points or contain heat-sensitive (bio-)molecules. In contrast to distillation which is governed by thermodynamic vapor-liquid equilibria and thus by relative differences in volatility, the separation in PV is determined by differences in sorption of the feed constituents in the membrane and in their diffusion through the membrane. Transport through nonporous polymeric PV membranes can be described with a three step mechanism, involving selective sorption of the target components from the bulk feed in the membrane, followed by selective diffusion through the membrane, and finally desorption of the permeate in the vapor state at the downstream side of the membrane. Basically, the transport through PV membranes can thus be described in terms of the solubility and diffusivity of the target compounds. Solubility is a thermodynamic parameter, providing information on the quantity of penetrant absorbed by the membrane under equilibrium conditions, while the diffusivity is a kinetic parameter indicating the transport rate of the penetrant through the membrane.

Thanks to the use of highly selective membranes, PV offers an efficient solution in many liquid separation problems. Water can for instance be preferentially removed from a solvent bulk by using a water-selective membrane, irrespective of the presence of other components. Moreover, in contrast to distillation where the entire feed is subjected to repeated heating-condensation cycles, in PV the membrane is chosen in such way that only the minor component consumes the latent heat of vaporization. PV is performed in the absence of additives such as entrainers, thereby avoiding the need for extra recovery columns and minimizing the risk on product contamination. As distillation processes are driven by vapor-liquid equilibria, the columns need to operate with a high reflux ratio to achieve the desired separation whenever volatility differences become small. Again, selective PV membranes can efficiently separate close-boiling point mixtures at a much lower energy cost and without the need for additional chemicals.

However, in many cases hybrid distillation-PV processes can be the most economical solution. Standalone PV may not be able to supply products with the required quality that are suitable for further processing or waste disposal in accordance with environmental standards. Hybrid membrane based separation technologies are regarded as one means of overcoming these limitations, and are currently gaining momentum. Within such hybrid processes, PV can be integrated either upstream or downstream of the distillation column. It can be used as the process which achieves the final concentration of the chemical to be purified. This is for instance industrially applied in the final dehydration of organic solvents, for instance
be purified. This is for instance industrially applied in the final dehydration of organic solvents, for instance bio-ethanol, using hydrophilic PV membranes. Alternatively, the possibility of positioning PV in front of a distillation column to overcome the azeotropic point and thus debottleneck the distillation process, can be considered.

The term ‘pervaporation’ was coined by Philip Adolph Kober already in 1917 in the course of some dialysis experiments. Until the late 1950s, no major progress was made, although few researchers published their findings on dehydration by PV. The first industrial dehydration PV unit, using polymeric membranes, was put into service in 1983 in Brazil by GFT (Germany). Despite the promising results and realizations in the 1980s and the large number of published papers on the development of PV membranes and processes in the last decades, the developments in PV technology stagnated somehow and dehydration of organic solvents still remains the only well-established PV applications. Other potential PV applications like for instance organic-organic separations, removal of VOCs from waste waters, and aroma recovery from natural extracts have been extensively studied, but no industrial breakthrough has been achieved in these areas so far. In contrast to solvent dehydration which relies on the use of hydrophilic PV membranes, removal of organics from dilute aqueous streams and organic-organic separations respectively require hydrophobic and target-organophilic membranes, which are not (widely) available on the market. In view of the increasing energy costs associated with conventional separation processes, more stringent environmental legislation and expected breakthroughs in membrane and process developments, PV is expected to gain a foothold in more and more industrial processes in the coming years. Industries moving to incorporate pollution prevention principles into their production processes consider it to be an attractive alternative to conventional thermal separation methods.

1.3.2. Work package 1 - Identification and classification of solvent streams and impurities

1.3.2.1. Objectives

- Identify and classify relevant solvent streams;
- Provide insight into waste/solvent generation processes;
- Identify impurity types and define purity standards.

1.3.2.2. Methodology and results

In line with the objectives of this work package, potential end markets for the solvent streams to be studied have been identified by the SME solvent manufacturers/recyclers at the commencement of the project. The production cycles from which the solvent streams originate have been studied to ensure that the developed membrane processes would be compatible with existing infrastructure, enabling easy integration at the manufacturing site. Both potential SME end-users selected two cases directly relevant to their businesses where the conventional, distillation based separation process either failed, or was inefficient or expensive. In this selection process, the know-how and practical expertise on OSN/PV of the two SME membrane suppliers and of the two research organizations helped to identify cases that appeared at the same time technologically feasible and attractive from a membrane business point of view. The selection was thus entirely based on the interests of the four SMEs in the consortium, and aimed at keeping a balance between, high purity and technical grade solvents. The suitable membranes/modules available from the SME membrane suppliers, and the dedicated OSN and PV test units within the consortium, were listed. For each of the cases, the key impurities (if known) were identified, purity
GKAT, having wide expertise in the production of ultrapure solvents, proposed to investigate the potential of OSN and/or PV in purifying solvents for the high purity market. Purification in this case requires the removal of organic and/or inorganic impurities down to ppb levels. Such solvents are typically used in ultrasensitive applications requiring the highest solvent purity standards to ensure consistency and accuracy. It is a growing market which is however difficult to penetrate with market prices being dictated by purity and layers of complexity being added as one moves towards the highest purity end. GKAT’s area of expertise is with organic solvents in their purest form, which is the single biggest market (Figure 2).

Figure 2: Distribution of GKAT’s high purity solvent sales.

A first case study proposed by GKAT involved the purification of acetonitrile for HPLC applications, which came down to the removal of small organic contaminants present in ppm and sub-ppm levels. As the nature of these organic impurities was unknown, this was considered to be a very challenging, and moreover very critical case. GKAT’s second case focusing on the purification of solvents for the electrochemical market was however more concrete and it could be imagined that OSN would enable partial removal of the key metal impurities. To be applicable in the production and processing of, for instance, integrated semiconductor circuits, photovoltaic cells and flat panel displays, these metal impurities need to be removed down to ppb levels. A wide range of solvents is used in semiconductor processes, typically in wet chemical applications such as cleaning of electronic wafer stacks. This range includes water, acids and organic solvents such as acetone, methanol and isopropanol. The semiconductor industry demands solvents of the highest quality with metal concentrations below 100 ppb, more typically even 10 ppb.

The electrochemical solvent market has a value of approximately 1 billion €. This market has seen a considerable growth over the past 15 years due to the growth of the micro-electronics industry. The presence of metals in solvents is not consistent, with metals founds in ligands, clusters and single atoms. As for other high purity solvents, the limitations of the current distillation-based purification technology are today being felt by global manufacturers of electrochemical solvents. This technology is indeed slow and expensive, while generating large volumes of residues and wastes and requiring significant capital investment to increase capacity. The electrochemical solvent market would truly be revolutionized if a simple, modular OSN unit could be developed which would simply allow manufacturers to pump solvents through the module thereby removing the metals to the requisite level. Technical challenges of such alternative OSN based purification process include maintaining the integrity of the solvent, particularly its assay, water content and acidity, avoiding leaching of preservatives from the membranes/modules and assuring sufficient capacity (i.e. flux) to be economically viable. The key enabler here is a process that can not only deliver the required quality, but also the necessary capacity to service the market.

The electronics industry uses large volumes of electrochemical solvents in their processes. These spent solvents enter the chemical waste, and are normally disposed of through incineration. To date the only real form of waste recovery in these industries is the removal of water from solvent streams. This allows increasing the calorific value (heat of combustion) of the waste, which has in turn a positive impact on waste handling costs. Water removal is however applied on very few fronts only, and the preferred method of operation is absorption. Several multinational as well as small to medium scale companies active in the electronics generate every week thousands of litres of waste which is taken away to incineration. These...
electronics generate every week thousands of litres of waste which is taken away to incineration. These waste solvent streams contain known contaminants and have the potential to be purified and recycled in a similar way as in the solvent manufacturing process. There is thus a significant opportunity to lower the volumes of waste solvents generated in the process industry through the development of facile, efficient and scalable membrane processes that can purify them to a level that will allow them to be re-used in the production chain. This of course encompasses the area of solvent reclamation and recycling, which opens up a large market. In many solvent-intensive processes in the micro-electronics, chemical and pharmaceutical industries, the average mass contribution of solvents to the manufacturing of products can indeed be very large, with the footprint in terms of energy, health & safety, and environmental aspects of such processes being overwhelmingly determined by the use, but also by the production and disposal of solvents.

The other potential SME end-user in the consortium, DNCP, is specialised in the recycling and upgrading of liquid chemicals and solvents, mainly through distillation. DNCP’s recycling activities are mainly directed to technical solvents, to be used for instance in the production of paints and lubricants, as well as in the automotive, chemical and pharma industries. DNCP’s focus is turning more and more to alternative, sustainable solvent purification processes which are not (solely) based on distillation technology. Membrane technologies could offer significant opportunities here, for instance in the separation of challenging organic-organic waste streams that cannot be straightforwardly separated by distillation, for instance because of the occurrence of azeotropes. DNCP proposed three such cases, including the separation of a tetrahydrofuran/cyclohexane mixture with azeotropic composition, i.e. 84.8 wt.% THF, representing a volume of more than 500 MT per year. Because of this azeotrope, this stream cannot be further enriched in THF by classical distillation. The target here was to remove cyclohexane from this mixture so as to obtain THF with the required purity. As PV is not governed by vapour-liquid equilibria, the azeotrope could be broken whenever a cyclohexane-selective membrane would be found. Besides, DNCP proposed also a case where a heat-sensitive biopolymer had to be transferred from its solvent environment to water. To be applicable in food and cosmetic products, solvent removal down to below 0.5 wt.% was required. Standard technologies such as evaporation and extraction failed in reducing the residual solvent, N-ethylpyrrolidone (NEP), below this threshold. It was anticipated that the solvent transfer of this bio-product could be carried out in a facile, non-thermal way by operating a selective, solvent-stable membrane in diafiltration mode.

1.3.3. Work package 2 - Membrane selection and lab-scale filtration tests

1.3.3.1. Objectives

- Select suitable membranes and membrane processes;
- Screen selected membranes at lab-scale.

1.3.3.2. Methodology and results

In WP2, an experimental plan was set up, involving membrane filtrations at lab scale (WP2), first on flat sheet membranes (WP2) and later on small commercial modules (WP4), eventually evolving towards pilot scale testing on larger modules in case of a proof-of-concept (WP6), following the general approach schematically outlined in Figure 3.
With respect to GKAT’s electrochemical solvents case, five different commercial flat sheet OSN membranes were provided by SOL and tested by VITO on technical grade methanol, isopropanol and acetone, three solvents that are typically used in semiconductor manufacturing processes. While adequate cleaning of the test set-ups and accurate analysis of the metals in their solvent environment by Inductively Coupled Plasma – Mass Spectrometry (ICP-MS) proved to be critical, these screening tests showed that OSN enabled (partial) rejection of the metals to various extents, depending on the metal, solvent and membrane screened (Figure 4).

After these initial screening trials, the most suitable membranes for each of the solvents were selected and further tests were carried out on flat sheets to marginally optimize the metal rejection rates. This involved for instance testing of the membranes under various operational conditions. A proof-of-principle of an OSN based purification process was thus developed for the three solvents, enabling to bring the concentrations of most metals within specification. Inspired by this success, tests on small commercial membrane elements were planned (WP4) with the ambition to proceed towards pilot testing (WP6).

GKAT’s second case, the removal of small organic impurities from acetonitrile so as to meet the specifications for the HPLC market, proved to be much more challenging. As the impurities were in this case unknown, FFCT-UNL carried out a thorough characterization on technical grade acetonitrile typically used as raw material in purification processes. Mass Spectrometry (MS) analysis revealed the presence of propionitrile and acetamide, molecules that are very close to acetonitrile in terms of molecular weight, functionality and volatility.

As no NEP-stable membranes, nor the feed stream itself were available at the start of the project, lab scale tests on DNCP’s biopolymer-NEP stream could only be performed at a late stage of the project. NEP is indeed a good solvent for most polymers, necessitating development of tailor-made, NEP-stable membranes. Preliminary screening tests on this mixture using modified flat sheet membranes provided by SOL showed the potential of OSN, with almost complete retentions of the biopolymer. This is a suitable starting point for a purification process where the OSN membranes are operated in diafiltration mode so as to progressively wash out the NEP until below the specified threshold by adding water to the feed mixture during filtration.

1.3.4. Work package 3 - Membrane characterisation and process monitoring

1.3.4.1. Objectives

- Characterize membrane properties at the nano-scale;
- Study membrane processes via real time, in situ monitoring techniques;
- Enhance fundamental understanding of membrane structure and transport.

1.3.4.2. Methodology and results

In this work package, FFCT-UNL developed, implemented and validated an on-line, in situ mass...
In this work package, FFCT-UNL developed, implemented and validated an online, in situ mass spectrometry (MS) technique allowing to acquire one data point every second, thus making it possible to monitor PV/OSN processes and learn about the short and longer term effects of permeating compounds on the membrane behaviour under different operating conditions. In order to characterise quantitatively the composition of a sample, the MS converts each molecule present into ions so that they can be moved about and manipulated by external electric and magnetic fields. Ions are produced in the ionisation chamber of the MS by electron impact due to the difference in potential between the filament and electron collector. Positive ions are separated according to their mass-to-electric charge (m/z) in the mass filter and converted to a corresponding electric signal in the detector. A MS characterises compounds according to their specific m/z ratio and the intensity of the electric signal, providing a fingerprint of mass fragments for a specific compound.

The tests were conducted on an experimental PV-MS system composed of a PV test cell the permeate circuit of which was connected with a splitting line to the MS ionisation chamber, enabling analysis of the permeate composition in real time (Figure 5). This technique was validated for monitoring the permeation of different organic compounds, using binary and multi-component mixtures. The calibration of the MS response for different target compounds was carried out by measuring the MS intensities of characteristic fragments, produced from generated gas/vapour streams for each individual compound in the downstream circuit, in a large range of concentrations. The technique was developed, optimized and validated in SOLVER for a PV case study, but it can be readily applied for monitoring the permeation of organic species during OSN processes as well.

Figure 5: Experimental set-up of PV – condensation system monitored by MS: (1) recirculation pump, (2) vacuum pump. The splitting system consists in a fixed bore.

It was shown that by monitoring the composition of PV/OSN permeate streams with in situ, real time MS, transient transport studies can be accomplished and the kinetics of solute transport can be monitored. From the mathematical analysis of these data, it is possible to determine the time-dependence of the solutes’ diffusion coefficients, which is related to the evolvement of the membrane structure as the permeants progressively solubilize into the membrane. Therefore, this technique offers a unique potential for understanding the effect of the permeating species on the membrane structure and infer what type of alterations are induced. By contributing to a better understanding of the behaviour of permeating species, this innovative in situ MS technique not only contributes to the advancement of membrane science, but also enables optimisation of operating conditions when dealing with real separation problems, as well as better membrane and module design with beneficial transport properties for targeted solutes.

The capabilities of this in situ MS tool were illustrated in SOLVER with a dehydration case study relevant to the SMEs in the consortium. The membrane used was a composite hydrophilic PV membrane. First, a characterisation of the membrane was performed in terms of the sorption coefficient of water in the membrane material. A real-time characterisation of the membrane performance was obtained through online MS monitoring of the PV process under variable feed compositions. The steady-state regime was characterised in detail in terms of permeability, water diffusion and selectivity for water. The transient regime was perfectly defined in time/length and it was characterised in terms of real-time permeate composition and partial fluxes of the permeants. Time-dependent water diffusion coefficients were calculated through the whole transient regime (Figure 6), which represents a significant advancement when compared with the common analysis where either an average diffusion coefficient for the transient period is calculated or the steady-state diffusion coefficient is estimated.
period is calculated or the steady-state diffusion coefficient is estimated.

Figure 6: Water diffusion coefficients as function of time, obtained through on-line MS monitoring.

1.3.5. Work package 4 - Process design and application development

1.3.5.1. Objectives
- Adapt and optimize membrane separation to meet purity requirements of selected streams;
- Design and engineer (hybrid) separation processes and develop treatment trains;
- Redesign pilot test units.

1.3.5.2. Methodology and results

The experimental work in WP4 was mostly focused on GKAT’s electrochemical solvent case, where the proof-of-principle of an OSN based purification process was demonstrated at flat sheet scale (WP2). Further bench scale tests were conducted on methanol, acetone and isopropanol using commercial 2.5” spiral-wound modules (membrane surface area of approximately 1 m²) provided by SOL. These spiral elements were prepared from the membranes that showed the highest metal rejection rates in the screening trials (WP2). To meet the market specifications of electrochemical solvents for all 34 screened metals, the OSN process was further optimised, and the effect of operational parameters on the metal rejections was studied. As this application is all about maximisation of metal rejections, flux levels were found less important at this stage. While most metals could be removed down to lower ppb concentration levels, confirming the results of the screening tests, there were anomalies for others. Given the sensitive character of the tests, extra efforts were devoted to the cleaning of the test units where a thorough cleaning protocol was brought in place, to membrane/module pre-treatment and conditioning, and to the accuracy and consistency of the sample pre-treatment and ICP-MS analysis. This resulted in a proof-of-concept of an integrated OSN process at small module scale, allowing proceeding to the pilot test campaign (WP6), where the know-how acquired in WP4 could be directly used.

With respect to Task 4.3 – Translation of application design to engineering recommendations, which was coupled to Task 6.1 – Modification of pilot installations for targeted applications, VITO’s novel OSN pilot unit was further improved to enable pilot tests on electrochemical solvents under optimal conditions, while DNCP’s older PV pilot unit was completely redesigned and refurbished.

A couple of years ago, VITO invested in a mobile, cross-flow pilot unit to demonstrate OSN processes at an industrially relevant scale and facilitate technology uptake by potential end-users (Figure 7). In order to be compatible with and usable within strictly regulated manufacturing environments, the mobile pilot unit was designed to meet the quality requirements imposed by the pharmaceutical industry (GMP compliant), in terms of materials choice, cleanability and safety aspects. To ensure operation in all organic solvents, the materials coming into contact with the liquid (feed and permeate circuits) are limited to stainless, Kalrez and PTFE. Great care was devoted to cleanability and hygienic design of the unit, with all components having a low surface roughness and full draining and cleaning-in-place (CIP) being possible.

Figure 7: Picture of VITO’s mobile OSN pilot unit.

The unit, unique in its kind, consists a feed vessel (400 L), a circulation pump, heat exchanger and
The unit, unique in its kind, consists a feed vessel (400 L), a circulation pump, heat exchanger and pressure pump and offers the flexibility to test both spiral-wound elements and multi-tube ceramic membranes at pressures up to 45 bar and temperatures of 50°C (ATEX solvents). It is fully instrumented (flow rates, pressures, temperatures) with real-time process monitoring and data acquisition by pc. The pilot unit is transportable and available for industrial clients on a rental basis for pilot scale tests or batch productions. Figure 8 lists the main technical specifications of the pilot unit.

Figure 8: Technical specifications of VITO’s OSN pilot unit.

Within SOLVER, the OSN pilot unit was modified and improved according to the specific requirements for the planned pilot tests on electrochemical solvents. The modifications, jointly carried out by PACKO and VITO, mainly involved design, construction and/or purchase of auxiliaries required to smoothly and safely conduct the SOLVER pilot tests.

DNCP’s older PV pilot unit that fell into disuse over the years was completely refurbished and redesigned by PACKO to offer more flexibility in terms of membrane choice and operation, as well as a better process control (vacuum, sampling) and a higher capacity (permeate collection). At the start of the project, the pilot unit was found in need of thorough refurbishment as some critical parts were dysfunctional or even missing. To make the pilot unit operational again and enable flexible operation, it was first carefully inspected, after which a comprehensive redesign was proposed and the unit was then refurbished accordingly to enable project partners to demonstrate PV processes at pilot scale, within and beyond SOLVER. The focus of the redesign was put upon enhancing the flexibility of the pilot unit to enable testing of both polymeric membranes (flat sheet and small spiral-wound) and multi-tubular ceramic membranes without modifying the vacuum chamber, to upgrade the permeate collection capacity, and to enable basic vacuum control and process monitoring. The improvements determined in the engineering phase were combined in a comprehensive model and implemented. The redesigned PV pilot unit is depicted in Figure 9.

Figure 9: DNCP’s redesigned/refurbished PV pilot unit: 3D rendering of modified skid (left) and frontal picture (right).

1.3.6. Work package 5 - Process modelling and assessment

1.3.6.1. Objectives

- Develop and validate a predictive membrane transport model;
- Evaluate and benchmark purification processes from a techno-economical and sustainability perspective.

1.3.6.2. Methodology and results

In WP5, a model was developed for estimating transient mass transport of solutes through a membrane, more specifically for estimating the solute concentration across the membrane and over time. The model developed by FFCT-UNL opens new perspectives in understanding transient mass transport phenomena. The model uses time-dependent solute diffusion coefficients during a solute permeation process, which reflect changes in the internal environment of the membrane when the solute is permeating, enabling a
reflect changes in the internal environment of the membrane when the solute is permeating, enabling a more realistic understanding on how the solute permeates the membrane across it and over time. The system under study was a pervaporative dehydration process using a hydrophilic composite membrane, where the water mass transport coefficients in transient and steady-state were obtained by in-situ MS, as explained in Paragraph 1.3.4 (WP4). This methodology may be equally applied to OSN and to vapour permeation processes.

1.3.7. Work package 6 - Pilot testing

1.3.7.1. Objectives

- Modify pilot filtration installations to targeted applications;
- Demonstrate solvent purification and recycling at pilot scale;
- Study long term performance, stability and robustness of membranes and modules.

1.3.7.2. Methodology and results

Lab tests on flat sheet membranes (WP2) and small modules (WP4) showed the potential of OSN in the purification of solvents for the electrochemical market. A proof-of-concept of an integrated OSN process, enabling removal of most metals down to low ppb levels, was demonstrated using 2.5" (SR-1) spiral-wound elements (~1 m²). In this work package, the developed integrated OSN process was further scaled to pilot scale using 4" (SR-5) spiral-wound modules (~5 m²). The pilot test was carried out on VITO’s mobile, cGMP compliant OSN pilot unit. The features and specifications of this unit, and the modifications carried out to make the unit even more fit to the highly sensitive high purity solvent cases studied within SOLVER, have been described in Paragraph 1.3.5 (WP4). An overview of the entire ATEX zone with the experimental set-up for the SOLVER pilot test campaign is depicted in Figure 10.

Figure 10: Emplacement of OSN pilot unit in VITO’s pilot hall. Experimental set-up for SOLVER pilot tests. Frontal (left) and top view (right).

As this was the first real pilot test on a flammable solvent (i.e. methanol) on this unit and moreover the first OSN test at such scale within VITO’s premises, the commissioning and certification process of the entire set-up (skid and auxiliaries, i.e. product vessels, venting panel, transfer pumps, pipings, etc.) required significant efforts. A thorough risk assessment (HAZOP study) was conducted and safety measurements, testing and emergency procedures, and cleaning protocols were brought in place. Detailed procedures for the SOLVER tests on electrochemical solvents were compiled, covering all aspects, including initial cleaning of the skid and the product vessels, installation of membrane modules, solvent transfer from 200 L drums into the feed vessel, inertization of the system, sampling, draining of the skid, etc.

After an initial thorough cleaning of the test unit and the product storage vessels, and an adequate pre-conditioning of the membrane modules, the actual pilot test was started. It consisted of two phases (Figure 11), i.e. a parameter study involving shorter term tests on the individual modules which were operated in total recirculation mode, to evaluate the effect of the operational conditions on the achievable metal rejection rates, and subsequently the actual integrated OSN pilot test itself, using approximately 700 L of methanol as raw material and generating approximately 500 L of permeate, with the modules being run in batch concentration mode and under optimal operational conditions. A total recovery of about 71% was
Batch concentration mode and under optimal operational conditions. A total recovery of about 71% was thus achieved.

Figure 11: Phases OSN pilot test campaign on electrochemical solvents.

In both phases, a representative general purpose methanol grade was used as raw material. The selected temperature, transmembrane pressure and crossflow dP values were well within the operational window specified by the module supplier. The permeate flux was monitored and permeate and retentate samples were taken at regular intervals. All samples were analysed by ICP-MS. The detection level was for most samples and metals 0.1 ppb, and the ICP-MS instrument was recalibrated every single day to enhance accuracy and consistency of the analyses. As an example, the metal rejection rates of a particular module under various sets of operational conditions are shown in Figure 12.

Figure 12: Parameter study – Metal concentrations in raw material and in permeate samples of Module X as function of the operational conditions.

The outcome of the pilot tests was very positive and in line with, or even better than the lab tests (WP2, WP4), with all 34 screened metals in the raw material being reduced down to below the respective concentrations of the highest electrochemical grade methanol on the market (Figure 13), and the process running stably. Hence, these pilot tests demonstrates for the first time and at an industrially significant scale that OSN also works for high purity solvent purification.

As such high purity levels are challenging for any separation process, it must be emphasized that significant efforts on different levels, besides the membrane separation itself, proved crucial in achieving success. These include the material choice, hygienic design and adequate cleaning of the test facilities, membrane pre-treatment, sampling and sample storage, and accuracy of sample pre-treatment (open digestion) and analysis (ICP-MS). Especially the fact that VITO’s pilot unit was really designed for pharmaceutical and other critical production environments, allied to a considerably more sensitive, accurate and consistent ICP-MS analyses as compared to the earlier trials at bench scale, made these pilot tests a real success.

Figure 13: Permeate production test – Metal concentrations in permeate of optimised, integrated OSN process vs. Raw material and Highest commercial electrochemical methanol spec.

As the pilot tests could only be carried out at the very end of the project, an in-depth process evaluation (technical, economic, environmental) and benchmarking with conventional, distillation based solvent purification processes could not be carried out within SOLVER. While such assessment is currently being conducted by the involved project partners, it is anticipated that the developed OSN process could produce an important breakthrough in cost-effective manufacturing of high purity solvents, offering significant benefits on different levels. This way, membrane processes would contribute to debottlenecking distillation based processing of liquid chemicals, enable reduction of waste solvent volumes and solvent loop closure, and counter the reliance on volatile solvents in the chemical industry.

1.3.8. Work package 7 - Exploitation and dissemination activities
1.3.8.1. Objectives

- Enhance exploitation of project results by cross-sectorial knowledge exchange;
- Handle PR issues;
- Disseminate results of the work done within the project to potential end-users and other stakeholders.

1.3.8.2. Methodology and results

WP7 was dedicated to dissemination and exploitation activities that were targeted to enhancing uptake of the developed technology, broadening its possible application fields and to assess the business opportunities for the developed processes. Main activities performed were the launch of a project website and presentations at scientific conferences and workshops. The partners are currently exploring different routes to further exploit the SOLVER results and expand the potential of nano-selective membrane processes in the solvent manufacturing and recycling market. The objectives of this work package are reflected in the next section on the potential impact of the project and dissemination/exploitation activities.

Potential Impact:

1.4.1. Potential impact and exploitation

The current global recession has brought the cost of chemical and pharmaceutical manufacturing into very sharp focus, with the world’s leading companies actively seeking innovative pathways to ensure efficient production and sustainability for the future. Societal challenges will indeed require more innovative technologies whether seeking to address the future health needs of an aging population, the longer term energy needs as the availability of fossil fuels diminishes, or the sustainability of the chemical industry in a world of diminishing resources, increasing international competition, and increasing environmental and safety regulations. In order to become competitive and sustainable at the same time, the future chemical industry in Europe must become flexible, modular, fit to market, adapted to different processes with a very high versatility and with a seriously increased efficiency.

SOLVER addressed some of these issues by developing and demonstrating nano-selective membrane processes enabling solvents to be purified and waste solvent streams to be valorized and reused in the market place in an efficient, non-thermal way. Solvent recycling and recovery is a highly complex activity in its current form, with the use of thermal processes being the proven route to success in this area. SOLVER has shown that advanced membrane technology can offer an alternative pathway for this thermal processing, offering significant benefits to potential solvent manufacturers, users and recyclers. Nano-selective membrane processes could indeed make current solvent recovery and purification practices significantly more efficient, cleaner and safer. Applying this technology to solvent-intensive production sites across the chemical, pharmaceutical, microelectronics and related industries would have a large impact in reducing chemical waste volumes. This way, the carbon footprint of the entire value chain, incorporating solvent production, solvent use within factories and disposal of solvent waste streams by incineration or thermal recycling could be significantly reduced.

Solvents are widely used in everyday life by all spectrums of society. Varying levels of solvent purity can be achieved, and the market prices are dictated by a combination of purity and solvent type. The domestic solvent business, e.g. for nail varnish removers and paints, are high volume and low quality. For exacting conditions, like laboratory use, where precise results are mandatory, the correct equipment and...
Conditions, like laboratory use, where precise results are mandatory, the correct equipment and consumables must be selected to ensure consistency and accuracy. This is the environment where high purity solvents are utilized. These have a wide range of applications, which have evolved over the course of the past 40 years with the acceleration of drug discovery, developments in analytical techniques and equipment, and the emergence of a diversity of industries striving to follow the levels of excellence achieved in the pharmaceutical industry. High purity solvents have a major role to play within several of the world largest manufacturing sectors, most importantly the pharmaceutical and electronic industries which have a chemical market value in excess of $1 billion each. These industries are at the cutting edge of technology, and demand the highest standards in the products they use. The target market for high purity solvents is predominantly the High Performance Liquid Chromatography (HPLC) market and the electrochemical market. The high purity solvent business has been in a state of constant growth (average 8%) over the past 20 years, and this growth is expected to accelerate in the coming 5-10 years due to the development of emerging economies such as India, China, South East Asia and Eastern Europe.

The OSN process for electrochemical solvents developed and validated at pilot scale within SOLVER is expected to achieve an important breakthrough in cost-effective manufacturing of high purity solvents, with the potential of being able to dramatically change the face of chemical manufacturing in the future. The pilot tests showed for the first time and at a significant scale that OSN not only offers potential in product isolation, concentration and purification where it is usually applied, but that the technology also works in much more critical applications requiring impurities to be removed from solvents down to (sub-)ppb levels. The key advantages of an OSN based purification process for high purity solvents are summarized in Figure 14.

Figure 14: Key advantages OSN based purification process for high purity solvents.

OSN based purification processes would not only contribute to more efficient manufacturing/refining of (high purity) solvents, but also offer a sustainable alternative for incineration or thermal recovery of waste solvents. This way, non-thermal membrane processes would also enable reduced waste solvent volumes and solvent loop closure, and counter the reliance of the chemical industry on volatile solvents. Such process intensification with modular membrane units in the solvent reclamation and purification business is expected to reduce energy consumption by 50-90%, and exponentially increase process efficiencies and reduce process costs by 50-85%. The potential economic impact of SOLVER is immense, with the proposed technology being suitable for the purification and recycling of numerous different solvents and industrial liquid waste streams.

Apart from the environmental and economic drivers, the importance of technology leadership in Europe in the area of solvent manufacturing and recycling must be emphasized, especially in view of the commercial threat from Asia where chemical, pharmaceutical and electronics products are being manufactured at much lower costs than in Europe. The EU, Asia and USA account for more than 80% of the world chemical sales, with the EU still being the world's leading exporter and importer of chemicals, accounting for more than half of the global trade. However, the chemical industry in Europe is under great pressure from India, China and other emerging, low cost economies to manufacture fine chemicals at lower costs, whilst ensuring that environmentally acceptable processes are used in order to comply with EU legislation. This situation puts more and more pressure on companies to exploit sustainable processes which are usually more expensive. In addition, the costs of chemical waste treatment are increasing. The innovative membrane processes studied and developed in SOLVER bear the potential to enable chemicals to be manufactured more economically, at the time and point of use and in an environmentally friendly manner.
manufactured more economically, at the time and point-of-use and in an environmentally friendly manner, thereby strengthening the competitive nature of Europe’s chemical industry and avoiding that too many production facilities are lost to emerging low cost base economies. Energy-efficient and environmentally sound processing of solvents is indeed expected to be a key ingredient for ensuring the viability of European process industries in the years ahead. As membrane technology is modular and scalable, it offers the flexibility to be configured to many different solvent producing processes, in a wide variety of manufacturing arenas. This would mean that SMEs could develop a wide assortment of products without the need for dedicated pilot plants for individual operations and processes.

The value chain approach adopted in SOLVER, involving membrane suppliers, system integrators, R&D organizations and end-users, allowed synergy effects to be tapped in and ensured that the economic benefits of the project could be enjoyed by all companies involved. The SMEs formed a strategic supply chain and will therefore be able to take the results of SOLVER directly to the market. They are expected to continue to leverage on the technological head-start after the project, take the results of the technological and scientific work directly to the market, find new applications for their products, and be the market leaders in their respective sectors. The competitive edge will have to be further fed from the performance enhancement brought about by built-up application know-how. The results of this project will definitely provide further business opportunities and stir interest among the potential customers of the SMEs. It is envisaged that the market volume achieved by their application will bring in the revenues to foster the further growth of the involved SMEs, and create additional jobs. This will ensure the further exploitation of the project outputs and the entrance for the developed membrane separation concepts in the strongly growing solvent manufacturing/recycling markets. Next to the European ‘home market’, several SME partner are already worldwide players, active in the US, but also in the Middle East and Asia. Apart from the direct use of the developed technology in the processes of the involved SME solvent manufacturers/recyclers, the four SMEs as well as VITO and FFCT-UNL have extensive links with end-users in the process industry which significantly broadens the valorisation potential and impact of the project results.

SOLVER also strengthened the European OSN/PV technology leadership and boosted the membrane market for solvent filtration. The global membrane market is steadily growing with an annual growth rate of 10% to 20%. At this rate, membranes will quite likely become ubiquitous in the future and will be implemented in the majority of the tertiary treatment plants. The total world market for membranes is expected to exceed 9.2 billion Euros in the next decade. The current membrane filtration market is dominated by large non-European players (e.g. GE-Zenon, Kubota, Koch Membrane Systems). For the large water market, they have much more resources then the European players and can benefit from economies of scale. However, for the emerging solvent filtration market, this is not the case. In this market, SMEs can be competitive by offering innovative products with excellent performance. OSN has since its emergence at the end of the nineties been a typically European research and technology focus, with all major membrane suppliers being from Europe.

The multidisciplinary consortium joining efforts and fostering co-operation between different partners representing a variety of research fields and coming from different parts of Europe helped to create the critical mass that will ensure a wide dissemination and exploitation of the R&D developments. More often than not, pockets of knowledge reside with individuals that are separated by the national borders and unknown to each other. Thus, a transnational FP-7 approach was the way to gather these people, tap on their various experiences and commercialise this knowledge to bring societal and economic benefits. The project provided the opportunities for interactions amongst SMEs and between SMEs and research organizations in Europe. Without their participation in this project, it would have been difficult for the
Organizations in Europe. Without their participation in this project, it would have been difficult for the participating SME solvent manufacturers/recyclers to enter the membrane market. Further, the intangible benefit of interaction between the various SMEs gave them the opportunity to be exposed to the challenges in their respective industries and identify niches where they can enter the market and collaborate in the future.

In addition to the direct benefits for the involved SMEs, SOLVER could indirectly influence other SMEs as well, encouraging them to become more involved in technology acquisition via partnering with international teams that include other SMEs, research organizations and larger companies. Furthermore, the project results can be transferred and developed further in other sectors for the benefit of other European SMEs helping them to gain an economic benefit and improve their competitiveness.

The fundamental scientific insights into the behaviour of membranes during PV/OSN processes acquired in SOLVER by using advanced and innovative in situ real timing monitoring techniques will not only stir interest in the scientific community, but they are also expected to enhance technology uptake in industries dealing with solvent intensive processes where OSN/PV could make a difference. For the implementation of membrane processes, it is essential to be able to guarantee stable and long-term membrane performance, high process efficiency and product quality. In situ, non-invasive and real time information of the process status can significantly contribute to achieving high process performance. FFCT-UNL has shown in this project that on-line MS monitoring is indeed a powerful tool, providing insight in the short and longer term effects of permeating compounds on the behaviour of OSN/PV membranes under different operating conditions, which in turn contributes to a more realistic understanding of solute permeation through membranes as function of length and time. This information can be used to develop a predictive model that mathematically describes transport through OSN/PV membranes. This innovative approach opens new perspectives in understanding transient mass transport phenomena in molecularly selective membranes which are at present still poorly understood.

1.4.2. Main dissemination activities

1.4.2.1. Website

The SOLVER project website (www.solver-fp7.eu) the welcome page of which is shown in Figure 15, presents the projects towards the public. The websites serves as information centre, not only for project partners but also for external researchers, institutes, enterprises, as well as the wider public. The website provides a condensed overview of the project objectives, the partners involved and the structure of the work. Project partners have been able to upload and download relevant reports, presentations and announcements. By promoting the website through their respective networks and events, the partners will continue to raise awareness of potential end-users and other target groups. Various opportunities will be used to make the website further known to target groups and to the general public. The website will be maintained and updated for at least three years after the end of the project.

Figure 15: Screen shot of the welcome page of the SOLVER project website.

1.4.2.1. Presentations and publications

Within the SOLVER project, the intellectual property rights were carefully regarded. Due to the generation
Within the SOLVER project, the intellectual property rights were carefully regarded. Due to the generation of IP sensitive results for the different industrial partners, dissemination under the form of publications in peer-reviewed journals was difficult during the project, but is currently being considered. The project was also presented at several international conferences and workshop. More detailed information is provided in Chapter 2 – Use and dissemination of foreground.

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