Transferring academic research to industry

A case study: ultrafiltration for potable water production

by Philippe Aptel

L'entrata in vigore della nuova legge 297 sul finanziamento della ricerca industriale potrebbe contribuire a significative azioni di collaborazione tra ricerca pubblica ed iniziative private. L'esperienza descritta dal Dr. P. Aptel, in Francia, può essere esemplificativa di quanto potrebbe avvenire in Italia.

During my academic membrane science career, I have had the opportunity to transfer few ideas to industry. This short paper is the story of my secondment from the Centre National de la Recherche Scientifique (CNRS) to Lyonnaise des Eaux between 1985 and 1992.

Project presentation and program definition

In early 80's, a group of researchers at the Lyonnaise des Eaux research centre (CIRSEE - Centre International de Recherche Sur l'Eau et l'Environnement) under the impulsion of François Fiessinger, have reviewed foreseeable evolutions of treatment technology for municipal potable water production. Their conclusions can be summarised as follows:

- the simple improvement of the conventional processes will not satisfy the growing demand for quality;
- a breakthrough is imperative and this break must pass by the use of finer filtration system than the traditional sand filter and the reduction or even the elimination of the addition of chemicals.

Considering the recent progresses in related separation technologies, the idea of using membrane filtration emerges as "the" solution. The main advantages of membrane operations were already known: membranes constitute an absolute barrier and the pore size can be tailored to reach complete removal of particles and micro-organisms giving a constant water quality far beyond current international standards for clarification and desinfection. However a major problem had to be overcome: the economical barrier. At this time membrane filtration began to be used for milk concentration and for fruit juice clarification i.e. for products with a far higher cost (factor of 1.000!) than potable tap water.
In the same time, at Laboratoire de Génie Chimique (LGC), I was developing spinning experiments to elaborate hollow fibres for several applications as ultrafiltration, pervaporation and air-stripping membrane contactors. One day, I had the opportunity to present our work on VOC's removal from drinking water to Lyonnaisès researchers. After my presentation, a large exchange of ideas started on the potential use of membrane operations in the water field. This was the beginning of a long collaboration between Lyonnaise des Eaux and CNRS on this subject.
During the years '84-'85, researchers from LGC and CIRSEE worked together to prepare an ambitious program which was officially launched in November 1985. To insure the success of the program a staff of specialists was formed to bring together the expertise of water scientists from CIRSEE, of process and system engineers from Degrémont and of membrane scientists from CNRS. An agreement was also signed with DSS, the well-known Danish membrane company and later with Zenon (the now well-known Canadian membrane company) in the frame of the two European programs BRITE and EUREKA. Moreover a GdR (Groupement de Recherche) was launched between Lyonnaise des Eaux and four laboratories from CNRS having complementary expertise in Chemical Engineering, Fluid Mechanics, Polymer Physical Chemistry and Textile Engineering.
In January 1986, a Membrane Laboratory was opened in Toulouse under my direction. On a six years period (1986-1991) up to 50 people (technicians, engineers, researchers and PhD students) were involved, for a total budget of around 30 Million Euro (80% was provided directly from Lyonnaise de Eaux and 20% from CNRS, French

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Ministry of Research and European Community). At my knowledge, it was the most important industrial R&D effort ever made in membrane field for the development of one specific product. The only exception might be the Reverse Osmosis program performed by Dupont in the early sixties.
A first and clear objective has been assigned: an ultrafiltration potable water plant had to be in operation before December 1998. That means that starting from a lab spinning set-up and our academic knowledge in membrane fouling, we had three years to solve:

- all the scientific problems concerning the choice of the hollow fibre (nature, dimensions etc.);
- the design of a large module and the optimisation of the UF process;
- all the technological problems associated with the scale-up of the spinning line and the module assembly.

As already mentioned, we started with two main ideas: the first one was the choice of ultrafiltration compared to microfiltration, since we decided to insure in one step the functions of clarification and desinfection; the second one was the choice of hollow-fibre modules compared to other designs: plate and frame, spiral wound and tubular.
Hollow fibres have three decisive potential advantages:

- the highest membrane surface to weight ratios;
- the highest membrane surface to volume ratios;
- the highest backwashing capability.

Step 1 - 1986: Process fundamentals and membrane screening

The main effort of the initial research work was aimed at the resolution of two problems related to improve the overall economics and system design for ultrafiltration of water:

- the choice of a commercial suitable polymer compatible with water treatment conditions and with the spinning process;
- the study and the control of fouling relating to the constraint of continuous operations (24h/24h) at high flux avoiding too frequent chemical cleaning steps.

To select the membrane - chemical nature and pore size - and to ascertain a basic set of operating parameters, different commercial and experimental UF and MF hollow fibres, including polysulfone, polyvinyldifluoride, polyacrylonitrile and cellulosic derivatives, were tested on surface and underground resources. They were tested in small modules with a shell-and-tube design, the feed flowing inside the tube. The choice of the inside-out mode of filtration was made for an obvious reason: the scaling-up of the process operating parameters, from studies based on small modules (10 or 20 hollow fibres), was reasonable in this mode while quite impossible, in a short time, in outside-in configuration.
Small-scale pilot units have been designed to test two modules in parallel on the same feed and at the same wall shear rate. After one year, the following conclusions have been drawn:

- in continuous cross-flow operation (without backwashing) all the membranes gave the same “critical” flux;
- backwashing was efficient to restore the initial permeability only with cellulosic derivatives hollow fibres;
- in analysing the flux decreases during the first 10-20 minutes of filtration, we observed only few or even no difference as a function of the cross flow velocity.

These conclusions led us to the choice of cellulose ester for developing the spinning procedure and to optimise the process in dead-end filtration mode with backwashing cycles.

Step 2 - 1987-1988: Membrane development and module design

Cellulose ester hollow-fibres spinning parameters were then adjusted to provide the membrane with acceptable mechanical strength. This was not a trivial task and 15 months were necessary to get satisfactory results. During their life on the plant, hollow fibres are submitted alternately to inside (filtration) and outside (backwashing) pressures. To quantify this phenomena a backwashing simulator has been developed. The ageing is accelerated as the backwash frequency and the pressures are higher than the operating values used on plants. The number of cycles at break is higher than 100.000 for the final “standard” hollow fibre.
In the same time a first large module (100 mm in diameter and 1.300 mm in length) and a first spinning line was designed and built.
During the years 1987-1988, long term testing of the first module on a groundwater resource at Amoncourt (North-east of France) were successfully performed and the operating parameters were optimised. Then the plant was designed and built. This first UF plant has two lines with 10 modules each, a nominal throughput of 10 m^3/h and it was put in operation in November 88.
It has to be pointed out that this plant is still running today with original modules, demonstrating that membrane life-time can exceed 12 years. Moreover, the plant did never need any chemical cleaning, demonstrating the exceptional non-fouling properties of cellulosic material and the efficiency of the hydraulic backwash cycles.

Step 3 - 1988-1992: Large modules for large application and industrial plant for hollow fibres and modules manufacturing

This first success led to the decision to launch the second phase of the program with the objective of designing a larger module and a complete manufacturing plant for the industrial production of hollow-fibres and modules.
The design of a larger module has needed to overcome severe technological problems concerning mainly the mechanical resistance of the potting at the two ends of the envelope. Finally, we have used an assembly of seven single bundles of 100 mm in diameter each bundle being enclosed in a rigid perforated sleeve. In this way, the two tube plates are able to withstand the compression or tensile forces to which they are subjected during the filtration and backwash cycles. Moreover, the overall head loss on the permeate side is limited to the head loss which exists inside one single bundle. When equipped with “standard” hollow fibres, the module has a surface area of 50 m^2.
The development of the industrial spinning line has been a long task. This line consists in a succession of a tenth of unit operation starting from the dope solution preparation and ending to the hollow fibres bundling. Each step has been optimised as a function of several criteria as productivity and quality. The line is fully automated and a set of a tenth of operating parameters is precisely controlled. The industrial spinning line was put on operation in summer ’92.
In the same period, six UF plants equipped with modules of 50 m^2 were

built in France (the first one in Douchy in August '89) and two others in Macao and in UK.
Before the end of the R&D program, the decision of creating a speciality company for ultrafiltration was taken by Lyonnaise des Eaux and Dégremont. Aquasource started to operate in January '91.

Step 4 - 1991-2000: Ultrafiltration for water treatment: a growing market

During the period 1991-1996, Aquasource has been able to demonstrate the new technology in a set of reference plants. At the end of '96, Aquasource had 35 plants commissionned in different countries, delivering 120.000 m^3 of ultrafiltered water per day. Main progresses have concerned the development of hybrid ultrafiltration systems to extend the use of UF to all kind of water resources. In several cases membrane alone are not 100% effective or economical. In particular, it is the case when raw water contents micropollutants as pesticides or nitrate. Combining UF with adsorption on powdered active carbon (PAC) and/or activated sludge is now well recognised to be effective and economical. The first largest hybrid plant using PAC (Vigneux/Seine) has a capacity of 55.000 m^3/day. Aquasource has also developed emergency units which have been used world-wide for delivering potable water to population after natural catastrophic events or for refugees in time of war.
As usual with a new technology, other research and industrial groups in the world have carefully observed the success of the pioneer work performed by Lyonnaise and CNRS.
In 1997, several competitors began to commercialise UF membrane plants.
In 1999, there were already 5 main actors in this field (Memcor - with MF-, Aquasource, Zenon, Pall-Asahi, Koch and X-Flow) and the overall capacity was around 1.000.000 m^3/day.
This year the market is exploding: the overall capacity has doubled (2.000.000 m^3/day) in one year and several new actors are entering the UF water treatment arena (Ionics-Mitsubishi-Toray, Stork-Akzo, Hydranautics-Nitto, Hoechst-Daicel etc). It should also be pointed out that many of them are venture between 2 or 3 companies.

Concluding remarks

Now, back to research at CNRS, I am observing the rapid growing of the UF technology. I have the satisfaction to see that the initial ideas to promote dead-end backwashable hollow-fibres systems have been also adopted by all the tenth of competitors present today on the market.
I would like to conclude by stating that I believe that technically-driven science companies, as Lyonnaise des Eaux in this case-study, offer the opportunity to academic researchers to transfer their knowledge from the lab to the commercial products and processes. I would like to encourage colleagues to jump at such opportunity and also to encourage public and private organisations to enhance technology transfer by people exchange.