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Development of the Polyamide Composite Reverse Osmosis Membrane and Reverse Osmosis Membrane System: A Case Study of Toray Industries, Inc.

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IIR Case Study CASE#12-08

2012年7月6日

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Development of the Polyamide Composite Reverse Osmosis Membrane and Reverse Osmosis Membrane System: A Case Study of Toray Industries, Inc. [1]

December 2010
(Translated in July 2012)

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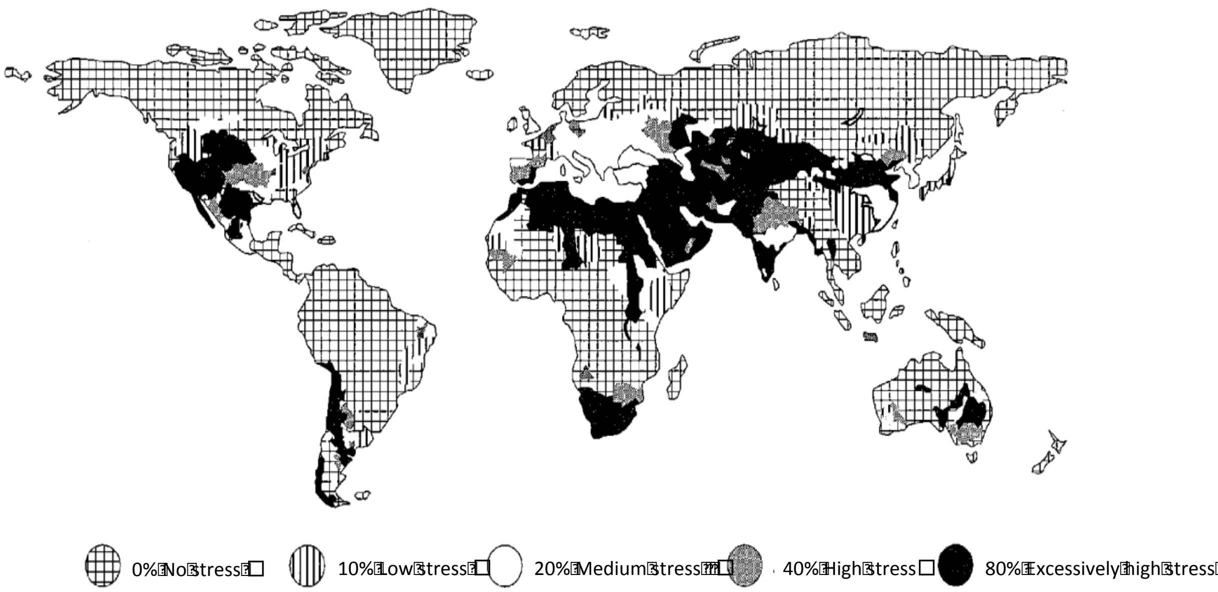
1. Introduction

The worldwide water business market is expected to be worth 100 trillion yen[2]. It has attracted serious attention and is being watched with anticipation around the world. It is because freshwater resources easily used by humans, such as rivers, lakes and marshes, account for a mere 0.01% of all the water on earth[3]. Moreover, driven by factors such as global economic development, rising populations and environmental pollution, water shortages are beginning to be a serious problem and are creating significant water demand. Fig. 1 shows the projected global distribution of water stress in 2025. The most notable aspect is that severe water shortages are projected in many parts of the world, and particularly in Africa and the Middle East. The production and supply of potable water has become a serious social need.

Fig. 1: Global water stress in 2025 (estimated)



Source: *World Water Vision* River and Water Committee (2001), p.96.

“The water business” involves a broad scope of operations. The first images that come to mind are those of water-related services – the water supply and sewer maintenance and management service, with its strong shading of public works, and the ultrapure water and groundwater supply services. Supporting these water-related services is the systems and plants construction business.

In addition there is the materials and components business, which manufactures the membranes, chemicals and membrane units utilized in systems and plants. Within this broad sector, the area in which Japanese firms are especially conspicuous is the materials and components business related to the upstream portion of the water business. To narrow the area specifically, it is the business for membranes referred to as reverse osmosis membranes.

Reverse osmosis membranes are membranes with extremely tiny pores, measuring 1nm or less in diameter, which endows them with the ability to separate ionic components such as salt from water, based on the principle of osmotic pressure. By using reverse osmosis membranes, a country without freshwater resources can produce fresh water out of seawater to meet its water resource needs. The ability of Japanese firms to maintain a strong showing in the reverse osmosis membrane market is remarkable. Looking at global market share in FY2009, the leading firms were The Dow Chemical Company (35%), Nitto Denko Corporation (28%), Toray Industries, Inc. (20%) and Toyobo Co., Ltd. (7%). When market share is divided by country, Japanese firms reign supreme.

Among these Japanese companies, the first to enter the business from its earliest stages was Toray Industries. Toray formally began reverse osmosis membrane development in 1968. Thirty-five years later, in 2003, Toray was comprehensively evaluated not only for having developed reverse osmosis membranes that demonstrate better performance than traditional membranes, but also for its superior technology for the production of such membranes and its seawater to freshwater conversion systems, and received the 49th Okochi Memorial Production Prize (FY2002) for its "development of the polyamide composite reverse osmosis membrane and the reverse osmosis membrane system"[4]. How was it possible for Toray to undertake the development of reverse osmosis membranes, over such a long span of years?

2. Technology and Industry Overview

2.1 Overview of membrane water treatment methodology

Water treatment technology is a set of methodologies that has been developed in tandem with population growth. Humans originally obtained and processed water by using nature's self-cleansing effects. Eventually, however, this approach was no longer able to keep up with rapidly expanding populations. As a consequence, during the 1800s efforts were undertaken to create and develop water treatment technology.

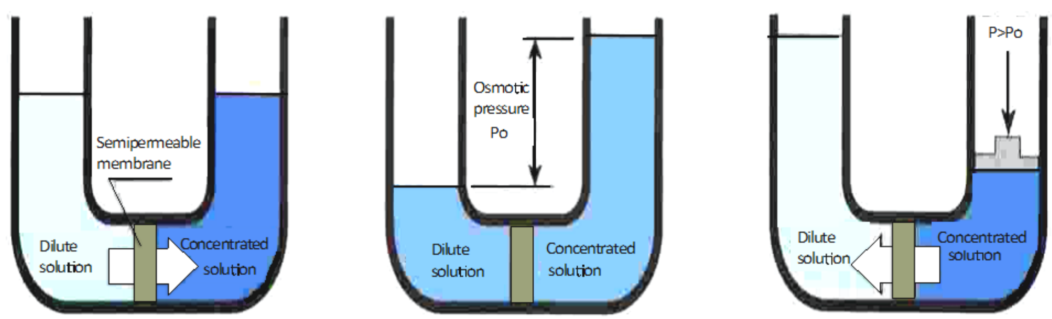
The main technologies differ depending on their application. Slow filtration systems and rapid filtration systems that utilize sand filtration were devised for clean water treatment use, while treatment systems employing microorganisms were developed for use in treating sewage and wastewater. For many years, evaporation methods were the main technology for producing fresh water from seawater[5]. As its name indicates, this is a method of evaporating seawater, then cooling the steam to obtain fresh water. Because of the large quantity of fuel required to produce evaporation, this method has been adopted mainly in the Middle East region where water resources are extremely limited and there are ample heat resources produced from oil. As part of this search for treatment techniques, water treatment technologies utilizing membranes have attracted attention in recent years. These are generally referred to as membrane treatment methods.

Membrane treatment methods are techniques for removing impurities by using separation membranes called semipermeable membranes. A semipermeable membrane is a membrane that does not allow molecules or ions larger than a certain size through the membrane's pores, yet enables molecules and ions of smaller sizes to pass. Semipermeable membranes can be divided broadly into two types, according to the method for eliminating impurities. These are membranes that eliminate impurities based on filtration principles, and reverse osmosis membranes that are based on the osmotic pressure principle. Membranes based on the filtration principle can be understood by visualizing a typical filtration process. A slightly more detailed explanation might be required, however, to grasp how reverse osmosis membranes based on the osmotic pressure principle work.

Fig. 2 is a diagram describing the osmotic pressure principle. Look at the pictures sequentially from the left. Normally, when water in a container is separated by a semipermeable membrane, and the salt concentration is high on one side of the membrane and low on the other, the water on the low salt concentration side will pass through the semipermeable membrane to the side with high salt concentration, until the salt concentration on either side of the container is the same. This is referred to as osmosis, and the differential pressure that arises when equilibrium has been reached is called osmotic pressure. However, when pressure that is higher than the osmotic pressure is placed on the water having the higher salt concentration, the water from the side with a high salt concentration will flow oppositely to the low concentration side. This phenomenon is called reverse osmosis. A semipermeable membrane that removes impurities by using this principle is called a reverse osmosis membrane.

Fig. 2: Osmotic pressure principle

Osmosis | Osmotic pressure | Reverse osmosis



The solutions with different concentrations seek to reach equilibrium through the semipermeable membrane, and the dilute solution flows to the concentrated solution side.

When osmosis reaches equilibrium, a pressure difference between the two liquids exists (= osmotic pressure)

When pressure greater than the osmotic pressure is applied, the solution is transferred from the concentrated solution side to the dilute solution side.

Source: Toray Industries, Inc. presentation materials

Besides that, the semipermeable membranes are divided into some categories depending on the size of their pores. Membranes based on the filtration principle are divided into three types, in order from the largest pores: large pore diameter filtration membranes (LP), micro filter membranes (MF) and ultra filter membranes (UF). On the other hand, semipermeable membranes based on the osmotic pressure principle are divided in order from large pore size into nanofiltration membranes (NF) and reverse osmosis membranes (RO). Naturally, the smaller the pore size, the finer the impurities that can be removed. RO membranes in particular with a pore size smaller than 1nm can eliminate ion components such as salts. RO membranes are therefore well-suited for seawater desalination.

Among these many alternatives, this case study focuses on NF and RO semipermeable membranes. Furthermore, normally the term “reverse osmosis membrane” is used in some cases to generically indicate semipermeable membranes based on the osmotic pressure principle (NF membranes and RO membranes), and in other cases to indicate those semipermeable membranes with a pore diameter less than 1nm (RO membranes only). In this study, we will refer to these separately, using the term reverse osmosis membranes when referring generically to NF membranes and RO membranes, and using the term RO membranes when indicating RO membranes only. Now, let’s turn to a description of reverse osmosis membrane structure.

2.2 Reverse osmosis membrane structure and product formats

Reverse osmosis membranes consist of two layers, a dense barrier layer and a supporting layer. The dense barrier layer, which exists on the membrane surface, is the layer that removes impurities. The supporting layer is porous and contributes very little to eliminating impurities, but accounts for most of the membrane. The reason for having a supporting layer that doesn’t contribute to eliminating impurities is that the membrane would tear if it consisted solely of the extremely thin dense barrier layer.

In some cases the materials used for the dense barrier layer and supporting layer are identical, while in other cases they differ. Membranes that use the same material are called asymmetrical membranes because their structure is asymmetrical in the direction of the membrane thickness. Cellulose acetate (CA) membranes are a typical asymmetrical membrane. This was the first material to be commercialized as a reverse osmosis membrane, and is currently still in use.

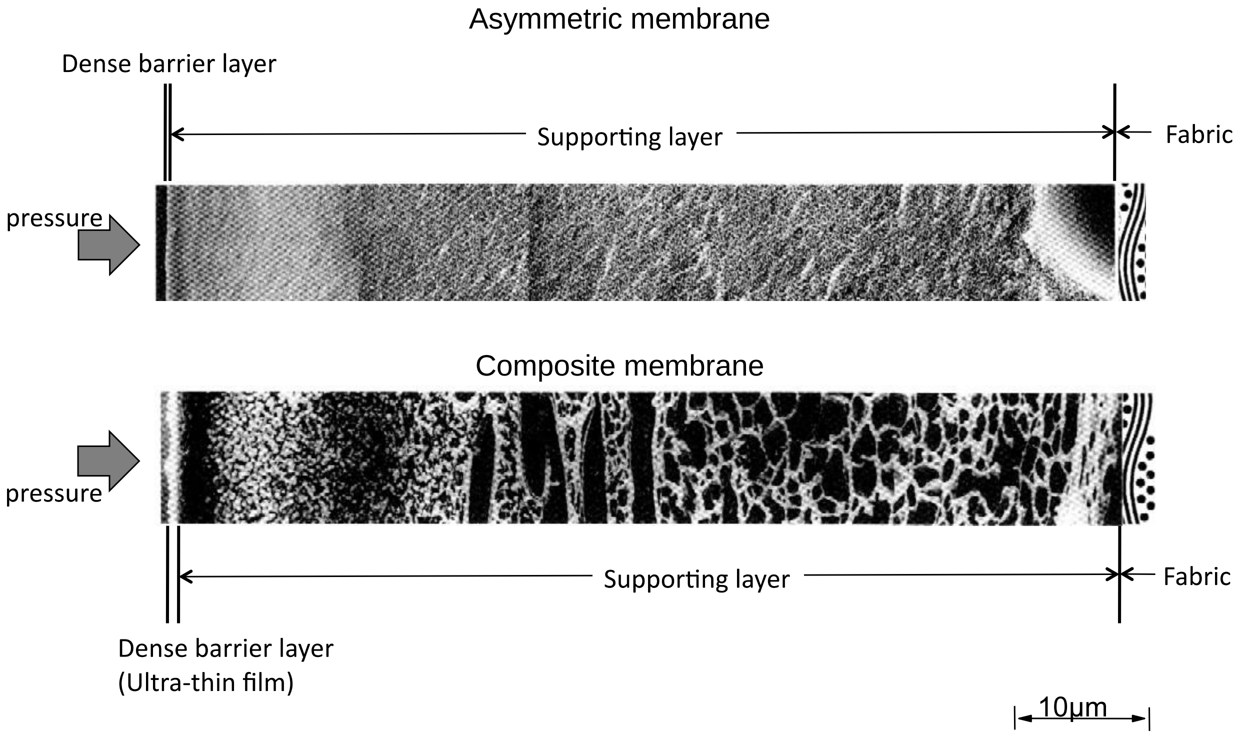
With composite membranes, on the other hand, the dense barrier layer and supporting layer can be produced utilizing different materials optimized for each function. The difference in cross section from asymmetric membranes is illustrated in Fig. 4. For a composite membrane a dense barrier layer must be produced again on the surface of the supporting layer. A variety of formation techniques have been invented, including processes to spread polymers directly on the supporting layer surface, processes to create polymers by spreading monomers, and processes to construct water soluble polymers, but interfacial polycondensation has become the most prevalent method at this time. Polycondensation is the process of creating the polymers by linking into a chain several compounds as they bind together, and in interfacial polycondensation, the polymer is formed continuously at the boundary surface (that is, the interface) of the aqueous phase and the organic phase.

The following is a more specific process for creating membranes. First, a porous membrane made of polysulfone is produced on fabric as the supporting layer. An amine solution is spread on the surface to form the aqueous phase. Interfacial polycondensation is then caused by applying an organic solvent solution of oxychloride over the aqueous phase. This produces a thin polyamide membrane with a thickness of 0.2μm or less as the dense barrier layer. This is shown in Fig. 3.

The advantage of interfacial polycondensation is the fact that even if the membrane tears during the formation process, the membrane can be re-formed because the aqueous phase comes into contact again with the organic phase. This self-restoration aspect mitigates the difficulty of membrane formation.

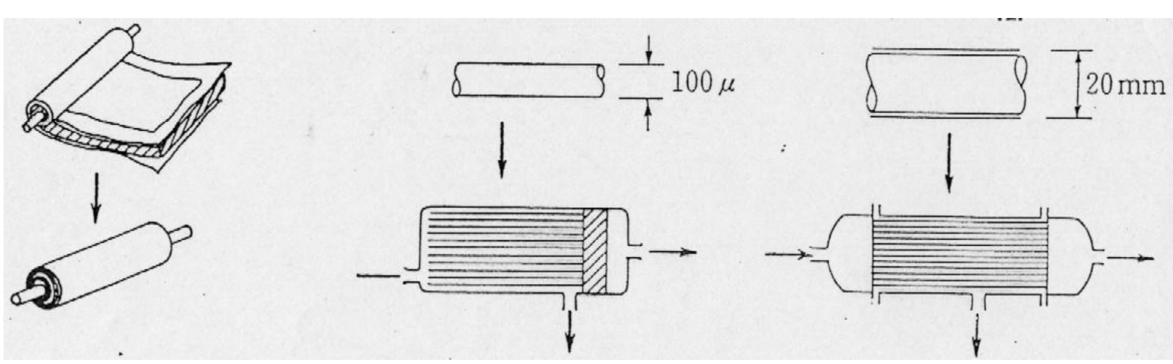
Reverse osmosis membranes are classified into three types depending on the product form they are later given; spiral wound membranes, hollow fiber membranes and tubular membranes. These forms are illustrated in Fig. 4. To form spiral wound membranes, a flat sheet membrane is first produced, then wound around a cylinder to produce the final product. The completed product is called a reverse osmosis membrane element. An element size measuring eight inches (about 20cm) in diameter and 40 inches (about 1m) in length has become the *de facto* industry standard. Hollow fiber, on the other hand, is a thin fiber with a hollow core; MF membranes and UF membranes used for medical care and dialysis treatment, and for water purification, are the main products. Tubular membranes are similar to hollow fiber membranes, but with a larger inside diameter. Of these three types, this case deals mainly with spiral wound reverse osmosis membrane elements.

Fig. 3: Cross-sectional photographs of an asymmetric membrane and composite membrane



Source: Prepared by the authors based on Toray Industries, Inc. presentation materials and *Clean Water Membranes (2nd Edition)* Editing Committee (2003), p. 65.

Fig. 4: Product forms



Spiral wound membranes | Hollow fiber membranes | Tubular membranes

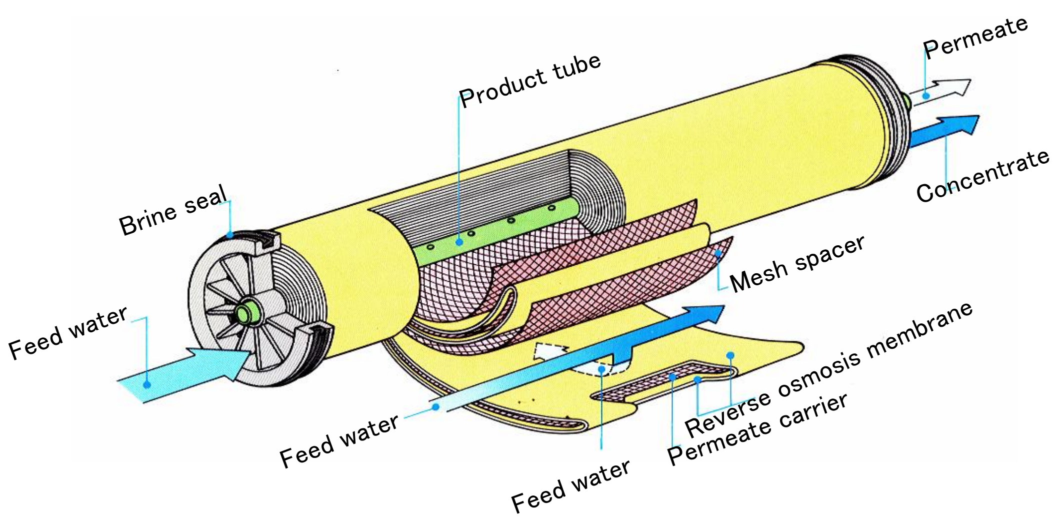
Source: Toray Industries, Inc. presentation materials

The mechanism by which RO membrane elements used for seawater desalination produce fresh water from seawater is shown in Fig. 5. The seawater (feed water) enters from the left side of the diagram, and pressure is applied to remove salts as the water penetrates the RO membrane surface. Permeate that has entered into the RO membrane is forced to pass through the mesh spacer into the center portion where outlets have been opened. Holes on the surface of the product tube collect the permeate in the tube at the center of the element. The result of this mechanism is fresh water (permeate), which flows out to the right from the product tube. On the other hand, the remaining water (concentrate) flows through the raw water permeate carrier and is separately discharged from the right side. As discussed previously, pressure must be applied for filtration and salt removal. The elements are used by placing them inside a pressure vessel for this purpose.

Advantages of spiral wound membranes are heat tolerance and strength, as well as their resistance to fouling – that is, the build-up of impurities. On the other hand, hollow fiber membranes do not require the use of a permeate carrier, and their membrane density and capacity efficiency can be increased. This has an advantage in that even if the water penetration capacity per membrane area is low, the filtration flow rate per unit capacity of the membrane module is improved[6].

The above is a summary of membrane water treatment methodology, reverse osmosis membranes and product structures. Although this discussion may have been a bit detailed, it will have served its purpose if it has helped you realize that semipermeable membranes include membranes based on the filtration principle (LP membranes, MF membranes and UF membranes) and reverse osmosis membranes based on the osmotic pressure principle (NF membranes and RO membranes), and that reverse osmosis membranes are divided into asymmetrical membranes and composite membranes, depending on their materials. Furthermore, depending on the type of product they are used to form, reverse osmosis membranes are divided into spiral wound membranes, hollow fiber membranes and tubular membranes.

Fig. 5: Structure of a RO membrane element



Source: Toray Industries, Inc. presentation materials

2.3 Market conditions

Desalination demand has expanded rapidly in recent years. According to the International Desalination Association, the total desalination capacity of installed desalination plants worldwide rose from 5.07 million tons in 1980 to 14.8 million tons in 1990, 26.7 million tons in 2000 and a record 53.32 million tons in 2008 (IDA, 2009). New demand likewise showed a similar trend, climbing from 950,000 tons (1980) to 890,000 tons in 1990, 1.6 million tons in 2000 and 5.57 million tons in 2008. Growth has been especially remarkable during the first decade of the 21st century. While this does not necessarily mean every desalination plant uses reverse osmosis membranes, it can be said that growth since 2003 is being driven by seawater desalination plants utilizing reverse osmosis membranes.

The size of the market for reverse osmosis membranes has undoubtedly increased along with the growth in desalination demand as well. The global market expanded from 69.0 billion yen (FY2007) to 72.2 billion yen (FY2008) and 54.9 billion yen (FY2009) (Fuji-Keizai Group, 2009, 2010). Although demand slumped in FY2009 because of the global financial crisis, over the long term demand is expected to follow a positive trend.

Reverse osmosis membrane use has been divided roughly into four stages. The first application to be opened was the conversion of brine into fresh water. Here, membranes are used to produce fresh water by removing dissolved salts and treating water such as river, lake and marsh water that contains low concentrations of salts. Today this is the largest application. Membranes were next applied in the production of ultrapure water for semiconductor fabrication. As the semiconductor industry developed from the 1970s through the 1980s, the manufacture of high-performance semiconductors demanded ultrapure water, and reverse osmosis membranes capable of eliminating impurities played a key role.

This was followed by the use of membranes to convert seawater to fresh water. The development of reverse osmosis membranes was originally begun in the United States for the purpose of seawater desalination. Because of the difficult challenges, this application did not take off quickly as an industry. As the result of technological advances, however, seawater desalination using reverse osmosis membranes spread rapidly in the 2000s. Finally, an application that has begun to expand in recent years is the use of membranes for recycling sewage and wastewater. This is literally their use for processing sewage and wastewater to make it reusable.

Looking at the players in this market shown in Table 1, as of 2010, there are five companies that supply NF membranes and nine companies that supply RO membranes. Each company’s market share is shown in Table 2. The companies supplying RO membranes and NF membranes are nearly the same, and these figures are thought to also approximate their shares of the RO membrane market.

As of 2010, spiral wound cross-linked aromatic polyamide composite membranes and hollow fiber (cellulose triacetate) asymmetric membranes are the only membrane types used in the RO membrane market for seawater desalination. The RO membranes and product formats that each company has adopted are summarized in Table 3. As can be seen, Toray, Dow Chemical, Nitto Denko and Koch Membrane Systems, Inc. use spiral wound compound membrane elements made with polyamide. Toyobo is the only company making hollow fiber RO asymmetric membranes.

From the standpoint of plant manufacturers who purchase membrane products, a market where several manufacturers who use identical materials have introduced polyamide spiral compound membrane elements, so that their products can be alternatively selected, mitigates the risk. Consequently the primary market demand today is for polyamide spiral compound membrane elements. According to FY2009 data, the market share for polyamide reverse osmosis membranes has reached 92%, with cellulose membranes having just 8% of the market (Fuji-Keizai Group, 2010).

How exactly were these reverse osmosis membranes developed, and how was the market established? In the next section, we'll answer these questions by first tracing the history of reverse osmosis membrane development.

Table 1: Global membrane manufacturers and membrane products

		RO membranes	NF membranes	UF membranes	MF membranes	MBR membranes
Overseas	Dow Chemical (US)	◎	◎		○	○
	Koch (US)	○		○	○	○
	General Electric (US)	○	○	◎		◎
	Siemens (Germany)					○
	Norit (Netherlands)			◎		○
	Woongjin Chemical (Korea)	○	○			
	MOTIMO (China)			○	○	○
	Vontron (China)	○				
Japan	Toray Industries	◎	○	○	○	○
	Nitto Denko	◎	◎	○	○	
	Mitsubishi Rayon				○	○
	Toyobo	○		○		
	Kubota Corporation					◎
	Asahi Kasei			○	◎	○
	Daicel Chemical	○		○		
	Kuraray Aqua			○	○	
	METAWATER				○	

◎: High market share products ○: Other product on the market

Source: Toray Industries, Inc. presentation materials

Note 1: MBR (Membrane Bio-Reactor) membrane refers to the membrane separation activated sludge method.

Note 2: Toray Industries is unique in that it has consistently developed the five types of membranes internally.

Table 2: Share of global reverse osmosis membrane market (Total value basis, %)

FY \ Company	Dow Chemical	Nitto Denko	Toray	Toyobo	Woongjin	Koch	Trisep	Other
2008	39.1	27.0	16.2	5.3	3.5	n.a.	n.a.	9.0
2009	34.6	28.2	20.0	7.3	2.7	2.6	1.8	2.7

Source: Fuji-Keizai Group (2009, 2010)

Table 3: RO membranes for the global seawater desalination market

Membrane manufacturer	Product	Membrane material	Membrane	Product format
Toray	TM-800	Cross-linked aromatic polyamide (PA)	Composite membrane	Spiral wound
Dow/FilmTec	SW-30			
Koch/Fluid Systems	TFCL-HP			
Nitto Denko /Hydranautics	NTR-SWC			
Toyobo	HOLLOSEP	Cellulose triacetate (CTA)	Asymmetric membrane	Hollow fiber

Source: Prepared by the authors based on Toray Industries, Inc. presentation materials (July 15, 2010 presentation).

3. History of Reverse Osmosis Membrane Development

3.1 Initial exploratory research in the United States: The 1950s[7]

During the 1950s, exploratory research on reverse osmosis membranes was conducted independently in the United States by researchers at the University of Florida and the University of California, Los Angeles (UCLA).

At the University of Florida, Professor Charles Reid undertook research on seawater desalination with the support of The Office of Saline Water. The Office of Saline Water was an organization established within the U.S. Department of the Interior after the central government, concerned about future water shortages, enacted the Saline Water Act on July 2, 1952. Professor Reid pursued his research as part of an effort by The Office of Saline Water, which conducted a seawater desalination research program at the national level.

Reid showed the effectiveness of reverse osmosis membranes in 1953, and in 1955 proposed a research study on *Osmotic Membranes for Demineralization of Saline Water*, on which he had pursued specific development. As a result of experimenting with a variety of commercially available polymer films, Reid discovered that cellulose acetate membranes demonstrated the highest salt rejection values. Then, in 1959, along with clarifying the desalination mechanism of cellulose acetate membranes, Reid and Ernest Breton announced a new desalination system (Reid and Breton, 1959). This system proved unable to reach desalination volumes more than 1 GFD (gallons per square foot per day), however, far less than the amount demanded for practical use.

On the other hand, following WWII California's population growth jumped sharply, leading to serious water shortage problems. Seawater desalination research was therefore given high priority at UCLA. UCLA's "saline water conversion" research had been kicked off by report written by Gerald Hassler in 1949, entitled *The Sea as a Source of Fresh Water*, and was further along than the research at the University of Florida. Hassler continued his work and in 1950 proposed desalination using a reverse osmosis membrane. An experimental device to verify this idea was completed in 1954. The technology proposed by Hassler was unable to ensure sufficient quantities of desalinated water, however, and in 1960 the project was canceled[8].

A separate project at UCLA was led by Samuel Yuster. Two researchers participating in this project, Srinivasa Sourirajan and Sidney Loeb, greatly advanced the efforts needed for practical application of reverse osmosis membranes[9]. These two researchers found that cellulose acetate membranes are asymmetric in the direction of the thickness, and that one side of the membranes achieves the desalination function. The two later began to create membranes, using commercially available cellulose acetate polymers, and successfully developed a cellulose acetate (CA) membrane that combined high salt rejection with practical levels of desalinated water. The desalinated water was over ten times the volume achieved by Reid. This result, announced in 1960[10], was called a Loeb-Sourirajan membrane.

3.2 Practical application of reverse osmosis membranes: The 1960s

Through patents, papers and various presentations, the Loeb-Sourirajan membrane had become well-known by 1961 and the possibility of creating low-cost desalination devices was widely understood. In the first half of the 1960s, Havens Industries put their membranes to practical use to build a desalination plant utilizing tubular reverse osmosis membranes. Another company that used Loeb-Sourirajan reverse osmosis membranes to move the technology toward commercialization was Aerojet-General Corporation[11].

Around this time, the most significant strides towards practical use were taken by General Atomic Company in San Diego. Ulrich Merten, Bob Riley, George Lonsdale and Donald Bray, young researchers at General Atomic, pursued R&D aggressively with support from The Office of Saline Water. In 1963 they developed the spiral reverse osmosis membrane element[12], which was patented in 1968 and went on to become the main product in today's market[13].

By the mid-1960s, two leading chemical companies, The Dow Chemical Company and E.I. DuPont de Nemours and Company (DuPont), had also begun full-scale development of reverse osmosis membranes aimed at commercialization. Dow developed the hollow fiber element, based on CA membrane research that Henry Mahon had conducted at the company since the latter half of the 1950s. Dow's product was unable to beat the Loeb-Sourirajan membrane in terms of performance aspects, however, and Dow's efforts were not fruitful as a business.

In contrast to Dow Chemical, which was using CA membranes, DuPont focused on the development of synthetic membranes. In 1967 its researchers synthesized an aromatic polyamide membrane, which DuPont used as the basis for development of a hollow fiber element. Although it produced a lower volume of desalinated water than CA membranes, this product offered superior salt rejection, making it suitable for seawater desalination. As a result, when DuPont commercialized the membrane and began selling it under the brand name "Permasep," the product proved to be highly popular and attained substantial commercial success at the time. This development success by DuPont would become the direct opportunity for Toray to launch its development of reverse osmosis membranes as described below.

In this way, the spiral wound element utilizing a CA membrane from General Atomic and DuPont's hollow fiber element made from a polyamide membrane drove the market during the 1960s.

3.3 Development of composite membranes and acceleration of commercialization: From the 1970s through the 1980s

As the 1970s began, there was progress in enhancing the performance and practical application of CA membranes, and advances in composite membrane development. Learning how to form a dense barrier layer on the surface of the supporting layer was critical for the development of composite membranes. Consequently various methods were sought out, and the most successful of these was interfacial polycondensation of amine and acyl chloride[14].

A key role was played by John Cadotte, who developed the composite membrane using interfacial polycondensation. Cadotte was a researcher affiliated with the North Star Division of MRI (Midwest Research Institute), a non-profit research institute. He developed the first reverse osmosis membrane using interfacial polycondensation in 1972, which successfully boosted both salt rejection and desalinated water volume.

It was Cadotte who also invented the basic structure of today's composite membranes. In 1977, Cadotte succeeded in developing a cross-linked aromatic polyamide compound membrane by using interfacial polycondensation[15]. This cross-linked aromatic polyamide compound membrane made using difunctional amine and tri-functional acid chloride later became the reverse osmosis membrane standard (Baker, 2004). In the following year (1978), Cadotte and several colleagues independently established FilmTec Corporation, and obtained a U.S. patent in the same year. This patent, generally referred to as "the '344 patent" because of the patent number – 4,277,344 – became the critical patent for reverse osmosis membrane development[16]. The reverse osmosis membrane developed by Cadotte was subsequently placed on the market by FilmTec Corporation as the "FT-30".

On the other hand, Bob Riley at General Atomic also pursued the development of composite membranes made by interfacial polycondensation. His effort preceded that of Cadotte. In 1974 General Atomic sold its business related to reverse osmosis membranes to Fluid Systems Inc., a subsidiary of UOP (Universal Oil Products), and in 1975 Fluid Systems commercialized the composite membrane developed by Riley. This product was delivered for a large-scale seawater desalination plant in Jeddah, Saudi Arabia in 1979.

In the 1980s, both CA membranes and composite membranes entered the phase of full-scale practical use. Dow Chemical, which had developed the CA membrane business, acquired FilmTec Corporation in 1985 after having sold off its own patents, making FilmTec a wholly-owned subsidiary and acquiring that company's composite membrane technology. As a result, FilmTec Corporation's patents, production facilities and Cadotte himself moved to Dow. Dow thus accelerated the commercialization of composite membranes, and leapt to the top of the global market.

The 1980s were also the years when the semiconductor industry developed. Ultrapure water with fewer impurities was required for the manufacture of semiconductors with advanced high performance, and the demand for reverse osmosis membranes for ultrapure water production increased rapidly. This was also the stage in which Japanese firms began to take an active role. Teijin Limited, Asahi Chemical Industrial Co., Ltd., Sumitomo Chemical Co. Ltd., Nitto Denko Corporation, Toyobo and other firms had conducted R&D on reverse osmosis membranes. Among these firms, the three that ultimately battled it out in the RO membrane market were Toray Industries, Nitto Denko and Toyobo.

In the 1970s Toray Industries, which formally began reverse osmosis membrane research in 1968, commercialized CA membranes and also conducted R&D on composite membranes. Despite having developed a new composite membrane that achieved salt rejection superior to the products of other companies, performance stability was a problem and Toray's membrane failed to attain commercial success. The situation changed when Toray invented a high-performance aromatic polyamide from four components in 1985, which it commercialized for ultrapure water production at semiconductor plants in 1987. This technology won Toray the Okochi Prize.

Reverse osmosis membrane development at Nitto Denko Corporation was begun in 1973. In CA membranes, the company focused on the semiconductor industry, which grew rapidly after other companies introduced the technology in 1985, and devoted itself to the ultrapure water business. In 1986 it established a membrane factory at its Shiga Plant and created an organization for supplying membrane products. In the following year, Nitto Denko purchased Hydranautics, a U.S. company headquartered in San Diego, making that company a wholly-owned subsidiary in 1987. Nitto Denko used this acquisition as a turning point to accelerate development of its composite membrane business. Toyobo Co., Ltd. began its asymmetric membrane development in 1972[17], developing a hollow fiber reverse osmosis membrane in 1978 that the company has marketed since 1979 under the brand name "Hollosep". Since then, this company has consistently undertaken the development and sale of hollow fiber asymmetric membranes.

This concludes a brief history of reverse osmosis membrane development until the 1980s. The next section examines how Toray launched its efforts as part of this development, and clarifies how the company has evolved.

4. Reverse Osmosis Membrane Development at Toray

4.1 Start of reverse osmosis membrane development

When DuPont successfully developed an aromatic polyamide hollow fiber reverse osmosis membrane in 1967, the news traveled rapidly. This news kick started Toray's reverse osmosis membrane development effort. "Reverse osmosis? What's that?" Using DuPont as a technology development benchmark, studies concerning reverse osmosis membranes were soon initiated by several research organizations within Toray.

The information on DuPont's development success was received at the Industrial Materials R&D Department in Toray's Fibers & Textiles Research Laboratories from the company's representative in New York. *Ryoichi Bairinji*, a researcher at the time, had worked on asymmetric CA membrane development. The team responsible for the development of a reverse osmosis membrane element utilizing the CA membrane developed by *Bairinji* was a development team under *Naokatsu Kanemaru* at the Engineering Research Lab. Toray's reverse osmosis membrane research sprang from the opportunity created by the news from DuPont and was formally begun in 1968, the following year.

Additionally, based on its own inquiries Toray's Central Research Laboratories had also learned about the DuPont information. Then, in 1970, development of polyamide composite membranes led by *Kojuro Ikeda* was begun in the Applied Research Department. Consequently, reverse osmosis membrane development proceeded in parallel at three locations: the Industrial Materials R&D Department at the Fibers & Textiles Research Laboratories, the Engineering Research Lab and the Applied Research Department at the Central Research Laboratories.

As reverse osmosis membrane-related R&D advanced at these three divisions, *Maseru Kurihara*, who would later lead Toray's reverse osmosis membrane development, enrolled to study at The University of Iowa in September 1970 in the United States. His initial reason for studying abroad, however, was not the development of reverse osmosis membranes.

During the period before his study abroad *Kurihara*, who joined Toray in 1963, had consistently conducted research on heat-tolerant polymers, including five years at the company's Basic Research Laboratories and two years at the Central Research Laboratories. His initial objective in studying abroad was also the development of heat-tolerant polymers.

At the university where he enrolled to study, however, *Kurihara* thought he would like to pursue new research, rather than continue researching heat-tolerant polymers. *Kurihara* therefore asked his academic advisor at the University of Iowa to list up research topics. One of the themes on the list was membranes. Membranes had applications for seawater desalination, as well as in kidney dialysis machines. With his interest piqued, *Kurihara* decided to begin research on membranes. *Kurihara* talks about his demeanor at that time as follows:

> Because of the high level of enthusiasm among international students at that time, I felt quite strongly that I wanted to bring back work that was not being conducted at Toray… When I brashly asked my professor, “Please make me a list of the research topics you’re pursuing,” membranes were on the list. They seemed interesting, and were also being used for desalination and kidney dialysis. And membranes are made by processing polymers. So I said, “Please let me work on this theme.”

Despite conducting work on membranes, however, *Kurihara* did not research reverse osmosis membranes. Although *Kurihara*’s theme was desalination membranes for seawater desalination, his research was not on expelling water under the application of pressure in the manner of reverse osmosis membranes, but oppositely on technology to push out salts. *Kurihara* pursued this research with 50% of his research expenses covered by Toray and 50% covered by The Office of Saline Water at the U.S. Department of the Interior.

At that time, The Office of Saline Water was regularly issuing reports, including some on desalination technology. *Kurihara* therefore forwarded these to *Bairinji* and *Ikeda*, who were researching reverse osmosis membranes in Japan. Although he did not directly conduct research on reverse osmosis membranes themselves, during the period until he returned home in 1972 *Kurihara* played a key role as contact point for information with domestic development teams that were well versed with the status of development efforts in the United States concerning reverse osmosis membranes.

Following his return from study abroad in September 1972, *Kurihara* was assigned to the Environmental Technology Research Department in the Engineering Research Lab. During this period Toray proceeded to centralize its R&D organization. As part of this effort, in 1971 the research on reverse osmosis membranes that was proceeding concurrently in the initial three divisions was integrated into the Environmental Technology Research Department at the Engineering Research Lab, where about ten researchers were engaged in the R&D. *Kurihara* joined this group. *Kurihara* was urged at this time by the head of the lab to do research on either artificial kidneys or on reverse osmosis membranes, and he decided to pursue the development of reverse osmosis membranes.

4.2 Composite membrane research: PEC-1000

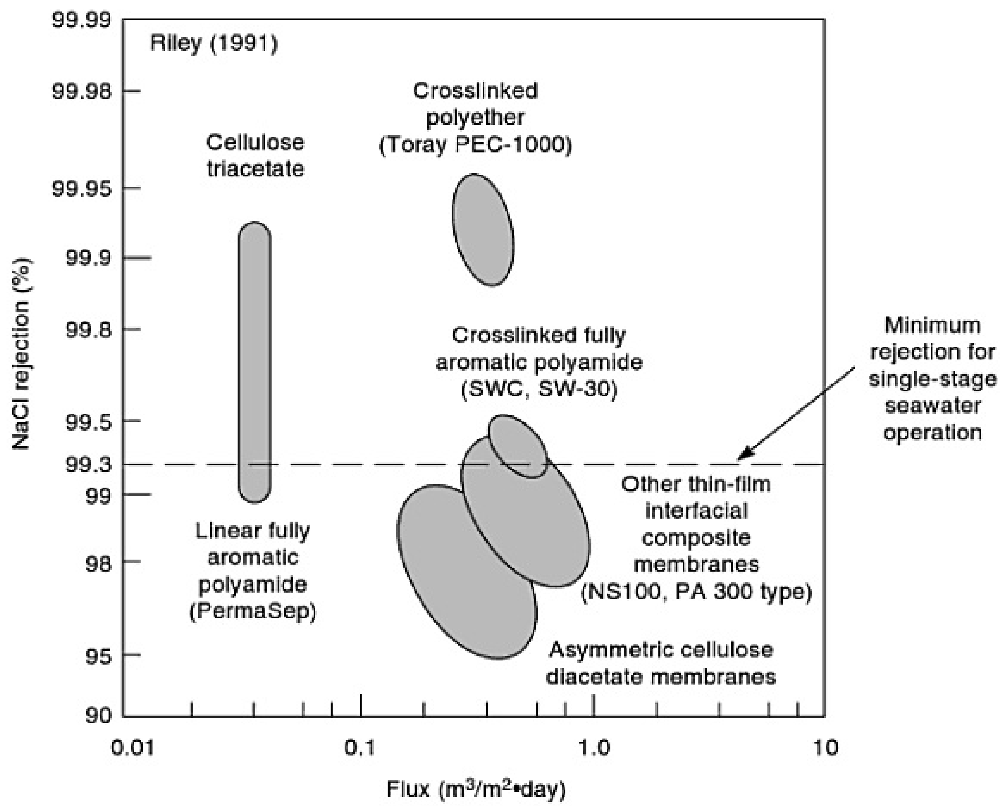
The theme *Kurihara* researched in the PEC Group within the Engineering Research Lab’s Environmental Technology Research Department was development of composite membranes as one type of reverse osmosis membrane. During this period, other companies including Japanese firms were already engaged in efforts aimed at commercial development of reverse osmosis membranes, and severe competition in the future was predicted. This included development work that had already been taken internally at Toray to commercialize CA membranes. Consequently the development of composite membranes as the next-generation technology was given to *Kurihara* as a theme. The thinking was that for Toray, which had expanded in the synthetic fibers and textiles business, similarly synthesized composite membranes would be a likely winner.

Kurihara pushed the development of a cross-linked polyether composite membrane, which was named PEC-1000. This was created by spreading a monomer on the supporting layer to form a dense barrier layer through polymerization on the surface, rather than by interfacial polycondensation. As a result PEC-1000 demonstrated very high performance. For example, in contrast to a recovery rate of 30% (proportion of fresh water to volume of feed seawater) for the reverse osmosis membrane that

DuPont and FilmTec announced at the international symposium held in Nice in 1978, Toray's PEC-1000 and CA membranes from Toyobo demonstrated a recovery rate of 40%. A high recovery rate, simply put, means high salt rejection. Even today, PEC-1000 offers a high salt rejection level and excellent performance.

Fig. 6 is a comparison with reverse osmosis membranes manufactured by other companies. The overwhelming superiority of PEC-1000 in terms of both salt rejection and desalinated water capacity ("flux") is readily apparent. PEC-1000 was a composite membrane that exceeded the trade-off between salt rejection and flux. Consequently the PEC group including *Kurihara* was transferred to the development division to undertake commercialization, and Toray decided to begin selling the product in 1980.

Fig. 6: PEC-1000 comparison



Source: Baker (2004) p.206

However, PEC-1000 had two problems. The first was its weak chlorine tolerance. This was a troublesome problem because chlorine is typically used when washing reverse osmosis membranes. The second problem was the membrane's extreme vulnerability to the dissolved oxygen in seawater. These problems were serious, and *Kurihara* finally advised Toray to halt sales of PEC-1000. *Kurihara* looks back as follows:

> The first phase was having this product PEC-1000 in the 1980s. Even though performance of the elemental technology was good,… the product never made it to (the) *Ehime* (Plant). It

> was made in *Shiga*, but before it went to *Ehime* I had recommended to the head of R&D that "we should stop." We researchers were the ones who initially made the argument that "we should stop." Maybe it made me look like a fool, but I said "I'm sorry" (and apologized to my boss)[18].

4.3 Commercialization of CA membranes

As the time required to commercialize composite membranes dragged on, the commercialization of CA membranes, which was achieved first, supported the business and enabled Toray to remain in and continue the reverse osmosis membrane business. In fact, from the beginning Toray was not considering development of the business based on just CA membranes. Because Toray was a firm that had grown based on synthetic materials, the idea was that the company could eventually develop its business with proprietary composite membranes synthesized in-house. It appeared such development would require a significant amount of time, however. The company therefore decided to develop the business with the CA membranes it had, and wait for the results of composite membrane development. *Kurihara* described the situation with a comparison to baseball:

> Imagine you go to your boss at Toray and take only cellulose, and because your boss has synthetic nylon on his mind you say, "There's no way this can be made into a business with the materials companies out there are using." So Kawabata and *Bairinji* (who were responsible for cellulose) said, "Let us do this first, because they're holding back their fast ball (pitcher)."[19]

By way of explanation, for CA membranes as well, which had been developed as a stand-in product, the applied market was exceedingly difficult to identify in the first half of the 1970s and Toray had run into a wall on commercialization. At seawater desalination plants that were envisaged as the most likely market, most plants were using evaporation methods, and from a cost perspective the membrane treatment methods at the time absolutely could not compete.

It was during this period, in 1975, that the CA membrane development team was moved from the Environmental Technology Research Department to the Development Division, to focus on element development and commercialization. In the following year (1976), Toray became the first Japanese firm to announce the commercialization of reverse osmosis membranes, and at the end of the same year Toray received its first order from IBM's *Yasu* Plant. To recycle wastewater, IBM adopted Toray's CA membrane as a purification membrane. The CA membrane spiral elements were produced at Toray's Shiga Plant, and introduced successfully at the IBM *Yasu* Plant in 1977. Nevertheless, wastewater treatment was a niche market and was still too small to be called a business.

This situation was changed completely by the rapid growth of the market for production of ultrapure water at semiconductor plants. This put the wind in the industry's sails. As Japan's semiconductor industry developed briskly from the 1970s through the 1980s, the result was rising demand for efficient production of the ultrapure water used at fabrication facilities. Until this period, ultrapure water had been produced by the ion exchange method, but obtaining purity sufficient to meet the needs of semiconductor miniaturization, and clean room fabrication was difficult with this method. It was at this point that reverse osmosis membranes, with their high separation performance, proved well suited for this market. Toray seized this new business opportunity with its spiral membranes. The improved CA membrane achieved performance sufficient for semiconductor fab plants. *Tadahiro Uemura*, who had pursued reverse osmosis membrane development together with

Kurihara, looked back on this period as follows:

> Well it really was a good tailwind, I guess, ultrapure water. Japan's semiconductor business just burst onto the world stage, and a single wafer required one ton of ultrapure water, and they just had to make ultrapure water, even if it cost around a thousand yen per ton.
> These semiconductor businesses were still able to make of go of it even though they were buying reverse osmosis membranes at prices that are dozens of times higher than today's prices. So it truly was a tailwind. Because at the start no one was thinking, "The semiconductor business is coming. Let's get into reverse osmosis membranes." So our product came to be used for ultrapure water, and grew to the point where we made enough to enable a couple of dozen researchers could eat, even though we didn't sell so much. So semiconductors were tied to our business, and to our R&D[20].

Afterwards, Toray's reverse osmosis membrane business expanded in lockstep with the growth of the semiconductor industry. Compared with today, the market certainly was not large, but Toray had stolen a march on other companies and had nearly 100% a market share, and the market was of a sufficient size to give validity to reverse osmosis membrane development.

Even though CA membranes were thus commercialized, the commercialization of composite membranes required still more time. After 1975, when the CA membrane development team was transferred to the Development Division, about ten members who had been responsible for composite membranes continued development work at the Environmental Technology Research Department as the PEC Group.

4.4 Toray throws in the towel on PEC-2000 commercialization

While *Kurihara* himself was responsible for PEC-1000 development, the development of PEC-2000 as a next-generation membrane had been put in the hands of *Uemura*. *Uemura* had been assigned to the Environmental Technology Research Department at the Engineering Research Lab soon after joining the company in 1974, and after having supported industrialization and development of CA membranes for about a year was one of the individuals who had consistently been responsible for composite membrane development. PEC-2000 was a polyamide composite membrane formed by causing polycondensation of a polymer amine and acyl chloride monomer; variations of this membrane type included UTC-20 (for NF membranes), UTC-30 (RO membrane for seawater desalination) and UTC-40 (RO membrane for brine conversion). As commercialization of PEC-1000 lagged, there were major expectations for PEC-2000, the follow-up product.

In 1982, a reshuffle moved the PEC-2000 development group to the Development Division, where it turned its attention to mass production and development. The remaining four or five researchers including *Kurihara* and *Uemura* formed an independent functional membrane group. When the Functional Membrane Laboratory was established in the Development Laboratories (formerly the Central Research Laboratories) in October of the same year, the functional membrane group was transferred to that lab, where it provided support for the mass production of PEC-2000 and took over the development of other new materials until about 1984.

On April 2, 1984, as it began moving toward the mass production of PEC-2000 in addition to CA membranes, Toray set up a Membrane Products Department with the goal of making reverse osmosis membranes a core product. Toray declared its policy at this time would be to increase division sales to 10.0 billion yen in three years[21]. Considering the fact that annual sales for this business in FY 1983 were less than 2.0 billion yen, this was an ambitious plan.

Based on this plan, Toray established a new mass production facility for reverse osmosis membranes within its *Ehime* Plant in 1985, and also prepared a line for mass production of PEC-2000. The *Ehime* Plant General Manager at the time who pulled the reverse osmosis membrane production to *Ehime* was *Katsunosuke Maeda*, who later served as Toray's president. *Maeda* appreciated the potential of the reverse osmosis membrane business, and strove vigorously to locate the mass production system at the *Ehime* Plant, rather than in *Shiga*. As a result, little by little resources within the company were redirected to mass production and sales.

Even during these events, however, *Kurihara* was still recommending that Toray cancel efforts to commercialize PEC-2000. The reason was that simultaneously with PEC-1000, Toray was unable to solve the chlorine tolerance problem. This time, however, Toray had already set up a mass production plant. For a developer to call a halt to commercialization under such conditions required considerable determination. *Kurihara* was convinced, however, that because of the fundamental weaknesses of the PEC-2000 molecular configuration, Toray could not compete in the seawater water conversion business. The Development Division and plant pushed back strongly, asking "Why give up now, before we even try?" *Kurihara* commented on the conditions at that time:

> If I had to say what was my most painful moment, that was it. Because here I was, saying "you really should stop making that material," when the company felt it had to go with this (PEC-1000), and 2000 was also at *Ehime* and being gradually ramped up, and the Development Division general manager was in too deep to call everything off, and I was always fighting with the Development Division general manager and director of the Development Laboratories. Chemical engineers and mechanical engineers, you see, want to show that even their most hapless son is a winner. (So) they say, "Why do you want to declare defeat when no one is saying the product is weak."

Abandonment of the effort to commercialize PEC-2000 after PEC-1000 put a question mark on the commercialization of reverse osmosis membranes for seawater desalination. The seawater desalination market was extremely small and sales opportunities only came every few years, and it was necessary to build a large-scale facility when introducing a product. For an independent organization established as a division, equipping a mass production plant to prepare for a large wave that might or might not come once every several years could hardly be deemed reasonable. Indeed, the alternative of undertaking development with an awareness of the market for ultrapure water or brine conversion developed using CA membranes also seemed more assured commercially. *Uemura* looked back on this situation as follows:

> At the time, seawater desalination was not steady year in and year out like it is today, and the situation was that you'd get orders for large facilities to desalinate several thousand tons just once every couple of years, which would be built in no time flat. People would ask, "If we build a production facility just for that, what will we do with it when we don't have any orders and don't make anything on the line?" PEC was developed originally for desalination, and if we tried there would only be a project once every several years. (That's why people said,) "Desalination is no good as a target. Rather than that business, make something that we can sell continually every year at a constant pace, to a lot of customers, because the product is good, even if it's small, for markets like ultrapure water or brine conversion[22].

Of course, the development group had not thrown away seawater desalination, one of its largest objectives. As a practical judgment, however, the group was told to develop, for ultrapure water and brine conversion, the polyamide membranes on which it had now begun working. Thus UTC-60 was newly developed based on UTC-20, and commercialized as a NF membrane[23].

5. Development of a New Polyamide Composite Membranes

5.1 The search for new membrane materials and a breakthrough

PEC-1000 was produced using commercially available monomers as a material. For PEC-2000, the polymer amines were synthesized by modifying some of the chemical structures. However, the chlorine tolerance problem that plagued PEC-1000 and PEC-2000 had remained. Although a membrane "for brine" had been set as the realistic development objective, this problem had to be fundamentally solved when focusing on seawater desalination. Consequently, *Kurihara* and his team members considered it necessary to begin by synthesizing new monomers to achieve this goal.

In 1984, *Kurihara* assigned the "synthesis of an ideal membrane" to one of his new employees as a research theme. *Yoshio Himeshima*, who had just been assigned to the Functional Membrane Laboratory, was the researcher responsible for this theme. *Himeshima* was perfectly suited to the task, having studied organic synthesis in college and graduate school. He would forge ahead with the search for a material that realized excellent chlorine tolerance while offering separation performance identical to conventional materials.

From various past development experience, including work conducted at other companies, the basic orientation for forming polyamide membranes by producing interfacial polycondensation between an amine solution and an organic solvent solution of acyl chloride had been clarified. The point, specifically, was what type of structure was it best for a material to have? As a benchmark, *Himeshima* mainly used the performance of the cross-linked polyamide membrane developed by Cadotte from the interfacial polycondensation of a difunctional aromatic amine and tri-functional aromatic acyl chloride. The objective was development of an ideal membrane that surpassed this performance. *Himeshima* described this as follows:

> When you're talking about aromatic-type monomers, that's Cadotte. So I searched for information by eagerly devouring the pertinent patents or… symposium papers. This was because in those days, Cadotte was like a god. …My attitude was that I wanted to catch up while following such people, and I wanted to find something, even if just one thing, that they hadn't noticed, and make something great.

As he continued working to solve the various performance trade-offs, *Himeshima* had narrowed down the materials while searching through trial and error for the best parameters. He synthesized several hundred monomers, passing them to veteran researchers who turned them into membranes and evaluated their performance. One monomer he found through these repetitions was triaminobenzene. For Toray's later reverse osmosis membrane business, it was a key discovery. In May 1985, the development group began to study RO membranes that used triaminobenzene. Roughly one year had passed since *Himeshima* had begun his research.

In and of itself, triaminobenzene was not a new monomer. It was not being marketed as a commercial product, however. Therefore *Himeshima* had continued and developed a proprietary method to produce triaminobenzene at a high purity. In parallel with *Himeshima*'s discovery, a group at the functional membrane laboratory centered on *Uemura* proceeded with the work on identifying the specific membrane structure. They finally arrived at the new polyamide composite membrane UTC-70. Toray created an ultra-thin film that greatly increased flux while achieving an exact reticulation structure offering high selectivity and durability.

It was later understood that when the composite membrane was formed by interfacial polycondensation, the membrane surface took on a pleated condition that widened the surface area. As a result, it became easy for the feed water to penetrate the membrane because the amount of surface area broadened, even though pore size became much smaller. It was this phenomenon that produced both high salt rejection and high flux.

In 1985, the same year in which it pushed UTC-70 development, Toray established an RO Production Department and began focusing its attention on the creation of a mass production organization for RO membranes. The UTC-70 technology was transferred to this RO Production Department in February 1987. Toray commercialized UTC-70 as "SU-700," and delivered the product to *Toshiba Corporation's Oita Plant*, in the same year. This was the 1MB DRAM plant where *Toshiba* had gathered its state-of-the-art technology. Toray also pushed forward with commercialization for brine water conversion. As a result, Toray's reverse osmosis membrane business rapidly shifted its main focus from CA membranes to composite membranes.

These processes of the development of reverse osmosis membranes are shown in Supplemental Fig. 2.

5.2 Development for seawater desalination

Two points about the development success of UTC-70 throw light on the future of Toray's reverse osmosis membrane business.

First, UTC-70 not only replaced CA membranes in the markets for ultrapure water and brine water conversion, it also began to produce major commercial results. Consequently, as the next development issue the development team sought to lower the operating pressure of the reverse osmosis membrane elements and reduce the cost of desalted water. As it achieved improvements in factors such the membrane production conditions, reactive catalysts and control of the nano structure, the team made progress in lowering the pressure. Table 4 shows the performance of the RO membranes Toray introduced for brine water conversion. From the table it is clear the membrane elements put on to the market achieved high salt rejection and flux even as the required pressure was reduced.

Second, UTC-70 again opened the way to commercialization of RO membranes for seawater desalination. Because top management also knew the developers aspired to create a membrane for seawater desalination, they grabbed the opportunity from the development success of UTC-70 to once again begin pushing commercialization. In contrast to the effort to reduce the pressure in the case of RO membranes for brine water conversion, the critical point in the development of RO membranes for seawater desalination was improving the pressure tolerance of the membrane.

As described at the beginning of this case, operating pressure greater than the osmotic pressure must be place on water with a high concentration of salts to cause the reverse osmosis phenomenon. Because the osmotic pressure rises as the concentration of salts increases, removing fresh water from seawater demanded operating pressure greater than pressures used in the past, making it necessary to develop RO membranes capable of withstanding the operating pressure. The pressure tolerance of the polyamide membrane DuPont was developing in 1969 was about 5.5MPa, and the product did not achieve a 20% fresh water recovery rate. Although a membrane that endured 6.9MPa appeared in the 1980s, the recovery rate was merely 25%.

Table 4: Toray's development of RO membranes for brine water conversion

		Low pressure		Ultralow pressure	Super-ultralow pressure
Membrane element name (Year introduced)		SU-720 (1987)	SU-720L (1988)	SUL-G20 (1996)	SUL-H20 (1999)
Performance	Salt rejection (%)	99.4	99.0	99.4	99.4
	Flux (m^3/day)	26.0	22.0	26.0	26.0
Test condition	Operating pressure (Mpa)	1.5	1.0	0.75	0.5
	Temperature (°C)	25	25.0	25	25
	Feed condition (mg/L)	1500	1500	1500	1500
	Brine flow rate (L/minute)	80	80.0	80	80

Source: Uemura and Henmi (2008)

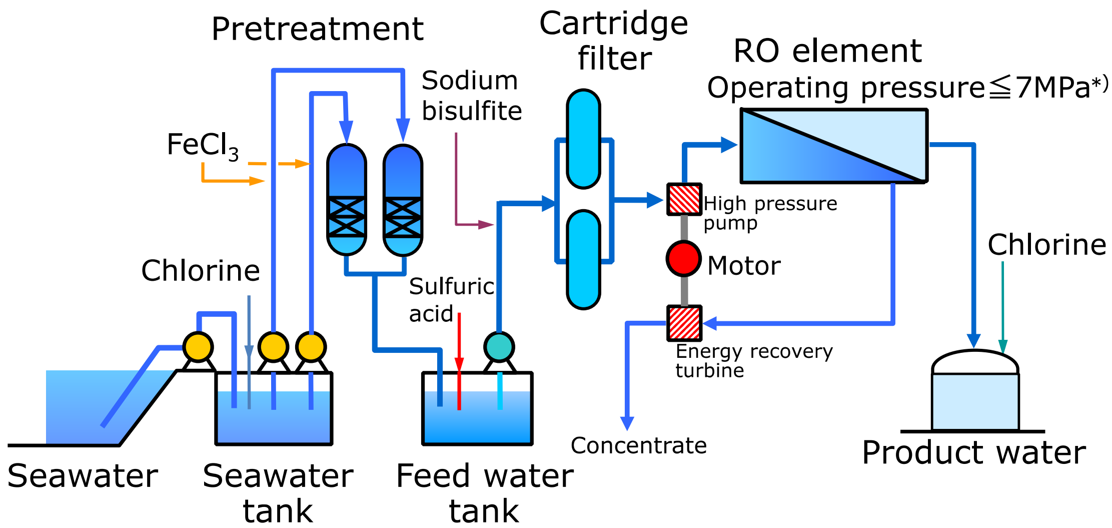
On the other hand, the UTC-70 developed by Toray could also be developed sufficiently for seawater desalination. Therefore the development group developed UTC-80, a RO membrane for seawater desalination, by increasing the membrane's salt rejection and improving the structure of the element while using triaminobenzene, the chemical utilized for UTC-70. UTC-80 was commercialized as "SU-800" in 1991 and delivered for the first time to *Kitadaito-jima* and *Minamidaito-jima* in *Okinawa*. It was also commercialized as "SU-820" (eight inches), which was introduced in 1996 for the desalination plant in *Chatan*, *Okinawa* (40,000m^3/day). The product's performance characteristics were an operating pressure of 6.5MPa, a desalination rate of 99.75% and flux of 16.0m^3/day[24].

Yet even though the business of RO membranes for seawater desalination had been established, the size of the business remained too small to be gratifying. The reason was the existing evaporation method was superior by far because the cost of attaining desalted water using RO membranes remained high and the results were also insufficient. So the hard struggle continued. *Katsunosuke Maeda*, who by this time had already become Toray's president, finally grew exasperated and in 1994 strongly ordered the developers to achieve a drastic improvement in Toray's desalination systems.

5.3 Creation of the highly effective brine conversion 2-stages seawater desalination system

A conventional seawater desalination system using RO membranes was like the system in Fig. 7. Under this mechanism, seawater is first taken in and pretreated by chlorination, the addition of coagulants, sand filtration and other processes. Next, the water is supplied to RO membrane elements using a high pressure pump and desalinated. The permeate is then taken and chlorine is added to make the product water. The remaining concentrate is discharged into the sea.

Fig. 7: Reverse osmosis method seawater desalination system flow



Source: Toray Industries, Inc. presentation materials

The recovery rate with this system, however, remained at about 40%. The reason was that the operating pressure applied to the RO membrane for this step had to be increased in order to improve the recovery rate, and the higher operating pressure would tear the RO membrane. Because of the RO membrane performance at the time, an operating pressure of about 5.5-7.0 Mpa was appropriate, and as a result the recovery rate hovered around a level of 40%.

Given this performance, Toray attempted to improve the recovery rate. The mechanism adopted was to pass the concentrate discharged by operation of the first stage through separate RO membranes again, thereby producing fresh water in two stages. To create this system it was necessary to raise the operating pressure on the RO membrane for the second stage to about 9.0MPa. The developers therefore created a new RO membrane named "SU-820BCM" (operating pressure of 9.0MPa, desalination rate 99.70%, flux of 16.0m^3/day), which was made with the same basic materials and a structure identical to conventional membranes but could tolerate higher operating pressure, even when applied over long periods[25].

Kurihara and the other team members dubbed this a highly effective brine conversion 2-stages seawater desalination system (Brine Conversion System: BCS), by putting the concentrated water that would simply have been discharged in the past through the separation process again using RO membrane elements for the second stage and collecting an additional 20% of fresh water. This system achieved a total fresh water recovery rate of 60% when combined with the first stage. They accomplished the basic invention during 1994, when *Maeda* handed down their orders, and successfully achieved operation of a pilot plant in *Ehime* in 1997[26]. Toray commercialized this system in 1999, delivering it to desalination plants in Maspalomas, Spain and on the Caribbean island of Curacao. Because the space required for plant installation, construction cost and running expenses can be reduced as a result, compared with conventional approaches this mechanism reduced the total desalinated water cost by about 20%.

6. Business Results and Future Development

6.1 Later efforts and business results

In 2002 Toray's BCS was adopted for the world's largest seawater desalination plant (136,000m^3/day), located in Trinidad and Tobago. Because the scale of seawater desalination plants had also increased considerably by this time, the number of membrane elements required to deliver the plant rose significantly as well. Consequently the construction of a system for stable mass production became a key problem at the *Ehime* Plant.

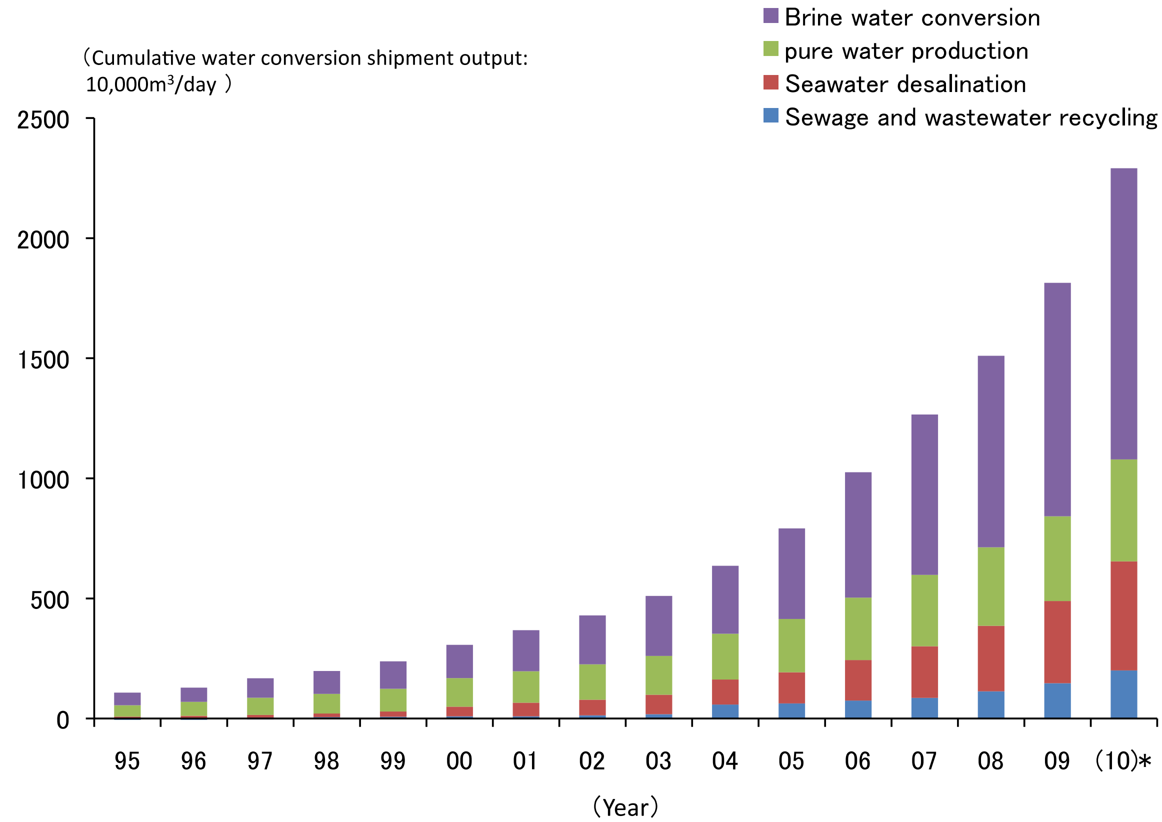
Until then, membrane element production at the *Ehime* Plant had been performed by hand. In particular the process of winding membranes to manufacture the membrane elements, which relied entirely on skill, was a critical bottleneck in the production schedule. Productivity, however, failed to improve. The members at the *Ehime* Plant therefore undertook an effort to automate the membrane rolling operation and raise productivity by introducing automatic winders. Production capacity jumped sharply as a result, which also resulted in a mass production effect, and the cost to produce reverse osmosis membranes gradually declined.

In addition, the reverse osmosis membrane development issues began to change from pressure tolerance and salt rejection to capacity to eliminate boron and low-fouling[27]. The reasons were several. First, boron's toxicity began to be raised as the cause of reproductive defects. Moreover, in order to maintain membrane element performance, users began to demand improved resistance to fouling. In response to these development issues, Toray put its UTC-70ULB RO membrane, which offered low-fouling and improved performance, on the market as "TML-720."

Because of the price reduction resulting from the series of performance gains and lower production cost, membrane treatment methods gradually became less expensive. The ratio of water treated at seawater desalination plants using the membrane treatment method, which stood at 36.7% in 1996, is believed to have exceeded the volume treated by the evaporation method in 2004[28]. In trial calculations in FY2007, the desalted water cost per ton, which had been cited as a bottleneck, was about 90-140 yen for the evaporation method and in the range of 60-80 yen for the membrane treatment method[29]. As a result, reverse osmosis membranes begin to account for the main demand in the market, and Toray's reverse osmosis membrane business began simultaneously to expand as well.

The development of Toray's reverse osmosis membrane businesses in conjunction with these changes is shown in Fig. 8. This diagram shows the cumulative flux produced since 1995 at plants that have adopted Toray's reverse osmosis membranes. From the graph we can verify that initially, the relative weighting of reverse osmosis membranes for ultrapure water production was very large, but that over time reverse osmosis membranes for brine water conversion exhibited the most remarkable growth. Today reverse osmosis membranes for brine water conversion remain the most important segment, and account for the largest weighting. From the graph we can also verify the demand for RO membranes for seawater desalination has also begun to expand since the turn of the century.

Fig. 8: Cumulative Flux at Plants that have Adopted Toray RO Membranes



Source: Toray Industries, Inc. presentation materials
Note: Data for 2010 are estimated.

6.2 Competition intensifies: Modularization and new entrants

The growth of Toray's reverse osmosis membrane business was supported by the company's development of superior reverse osmosis membranes as well as the expansion of global demand. As the world entered the 2000s, and water shortages began to be viewed as a serious problem in many regions, desalination plants began to increase in number and grow in size, and new demand for reverse osmosis membranes climbed. Because the performance of reverse osmosis membranes also deteriorated from fouling, replacement demand also emerged. For example, as mentioned at the beginning of this case, the reverse osmosis membrane market in FY2009 was 54.9 billion yen, divided between new demand of 25.0 billion yen and replacement demand totaling 29.9 billion yen[30].

Although the growth of both forms of demand provided a healthy boost to the expansion of Toray's business, it also stimulated competition among existing firms. Because a supplier could expect to monopolize the ongoing replacement demand if it was able to deliver its own reverse osmosis membrane elements once, the competition to receive orders in conjunction with the construction of new plants intensified.

Obtaining new demand, however, does not necessarily mean a manufacturer will also lock in replacement demand. The reason is that an 8-inch diameter and 40-inch length have become the *de facto* industry-wide standard module dimensions for membrane elements. Considered from the side introducing a plant, there is a possibility of selecting a new membrane manufacturer when the time comes to replace the membrane elements. The replacement at the seawater desalination plant built in

Chatan, *Okinawa* actually provides a perfect example; replaced according to the membrane specifications in 1999 and 2001, but purchased by competitive bidding since 2002[31]. Until now there were almost no major changes in delivery manufacturers when elements were replaced, but modularization has meant that the acquisition of new demand no longer brings with it the promise of securing future replacement demand.

If replacement demand means making a fresh start when selecting a manufacturer, it undercuts the dominance of the early market players and provides room for potential new entrants to break into the market. Market expansion has therefore encouraged new market entrants and vigorous M&A aimed at bolstering competitiveness. General Electric Company acquired Osmonics Inc. in 2003, acquiring that company's membrane technology. In Korea, Woongjin Chemical Co., Ltd. acquired membrane technology by purchasing Saehan Industries, while in China, Tianjin MOTIMO Membrane Technology Ltd. and Vontron Technology Co., Ltd. have plunged into the RO membrane market. Despite their small market share, these new players have become a factor driving greater competition. In addition, the appearance of automated equipment to handle production has accelerated the pace at which newly established ventures are catching up. The reason is that purchasing such equipment enables a firm to produce reverse osmosis membranes to a certain level of expertise.

To address these conditions, Toray is broadly pursuing three efforts. The first is performance differentiation through improvements to its reverse osmosis membranes as described earlier. Low-fouling and improvements in the ability to remove boron are two examples of this approach. By differentiating its product performance, Toray is working to ensure its products do not become commodities. Second, the company is not focusing only on selling reverse osmosis membranes and has also launched engineering services in pursuit of downstream opportunities. Along with its long cooperative relationship with Toray Engineering Co., Ltd., Toray strengthened its ties to downstream operations recently by making *Suido Kiko Kaisha, Ltd.* a subsidiary[32].

The third effort is global development. While the reverse osmosis membrane business was not totally removed from the global market in its early years, this business has achieved remarkable worldwide growth since 2005 in particular, after *Akihiro Nikkaku* (currently Toray's President and COO) took up his post as General Manager of the Water Treatment Division. *Nikkaku* aimed at global development, and strove to expand R&D and production capacity. Toray has subsidiaries in Switzerland and the United States, both positioned as overseas branches to produce and sell membranes. Since becoming president, *Nikkaku* has continued the overseas development of this business; in August, 2010 the company established a fully-integrated production facility in Beijing at a cost of 7.5 billion yen, thus rapidly expanding RO membrane production capacity by a factor of 1.5X[33].

6.3 Conclusion

Over 35 years, from the time Toray Industries, Inc. formally began development in 1968 until the company received the 49th *Okochi Memorial Prize* 2003, many of the firms that had propelled development were absorbed by other companies or withdrew from the market.

The RO membrane business of General Atomic, which played a critical role during the early stage of RO development, was absorbed by Fluid Systems, a subsidiary of UOP. Fluid Systems was transferred in 1994 from UOP to Anglian Water Group, and then transferred again in 1998 to Koch Membrane Systems. FilmTec Corporation, where Cadotte was employed, was acquired by Dow Chemical in 1985. Hydranautics was taken over by Nitto Denko Corporation in 1987. DuPont, the company that gave Toray its first major break in reverse osmosis membrane development, withdrew from the market in March 2001[34]. Toray is the only firm that has survived in the market while consistently maintaining in-house development. This fact alone demonstrates how difficult it is to turn development results into a viable business. Moreover, Toray has established a solid position in the market.

Why was Toray able to continue development, commercialize its R&D results and build a solid position in the market in the face of competition that has developed so severely? *Kurihara*, who tenaciously pushed reverse osmosis development, highlighted two factors. The first was the fact Toray competed on performance in polyamide spiral compound membranes, the same technological arena as other firms. Technological areas where there is no competition with other companies might be proprietary, but the principle of competition isn't at work. The logic is that staking out a position in the same technological area brings market mechanisms into play and advances development. The assumption, of course, is that a company's technology has the wherewithal to compete.

The second factor was the vast capabilities of the top management people who understood and supported the development. During the early stage of development *Yoshikazu Ito*, who later became Toray's president (1981-1987), supported the development group as head of the development laboratory. *Katsunosuke Maeda*, who succeeded Ito as president and is known as the "father of Toray's rejuvenation," concentrated on creating a reverse osmosis membrane mass production system at the *Ehime* Plant while he was General Manager, and remained involved in development of the reverse osmosis membrane business even after assuming the position of president. Looking back, *Kurihara* says he never heard strong opinions calling for an end to reverse osmosis membrane development. *Akihiro Nikkaku*, Toray's current president, has consistently and vigorously pushed the global launch of this business from the time he took over as General Manager of the Water Treatment Division. Although development of the reverse osmosis compound membrane business at Toray unfolded over many long years, top management's involvement during the commercialization phase in particular contributed remarkably to improving the management results of this development.

This case is a good example of the development and commercialization gears meshing smoothly. Of course, the competition continues. As the global demand for desalted water expands, the future course of Toray's reverse osmosis membrane business will remain deeply interesting.

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<Seminars and Interviews: Honorifics omitted; alphabetical order; titles are titles at the time of the interview>

Fumiaki Fukui, Membrane Production Dept. Manager, Membrane Second Production Section August 26, 2010, 13:00-17:40, Ehime/Ehime Plant

Yoshinari Fusaoka, Advisor Water Treatment and Environment Division General Manager, Water Treatment Division, July 15, 2010, 14:00-15:45 (Seminar) , Kunitachi, July 15, 2010, 15:50-16:40, Kunitachi, and August 30, 2010, 15:30-17:45, Shiga/Shiga Plant

Yoshio Himeshima, General Manager, Research & Development Division, August 19, 2010, 15:00-16:30, Tokyo/Toray Industries, Inc. Head Office

Takeharu Inoue, Water Treatment & Membrane Technical Dept. Section Chief, Membrane Technical Section, August 26, 2010, 15:30-17:40, Ehime/Ehime Plant

Maseru Kurihara, Fellow, Doctor of Engineering, Funding Program for World-Leading Innovative R&D on Science and Technology (FIRST Program): “Mega-ton Water System” Chief Researcher, Chairman, Asia-Pacific Desalination Association (APDA), Chairman, Japan Desalination Association (JDA), August 30, 2010, 15:30-17:45, Shiga/Shiga Plant

Minyu Seiji, Manager, Administration Dept., August 26, 2010, 13:00-17:40, Ehime/Ehime Plant

Eiji Nishioka, Member, Membrane Production Dept., August 26, 2010, 13:00-17:40, Ehime/Ehime Plant

Motoyuki Tomiyama, Plant Manager, August 26, 2010, 13:00-14:00, Ehime/Ehime Plant

Fujio Ueda, General Manager, Membrane Production Dept., August 26, 2010, 13:00-17:40 Ehime/Ehime Plant

Tadahiro Uemura, Director (Technology) (Toray) Water Treatment Division , Toray Singapore Water Research Center , Doctor of Engineering, July 15, 2010, 14:00-15:45 (Seminar) Kunitachi; July 15, 2010, 15:50-16:40, Kunitachi, and; August 30, 2010, 15:30-17:45 Shiga/Shiga Plant

Kenji Ueno, Advisor Water Treatment and Environment Division Manager, Water Treatment Division (Technology & Production), July 15, 2010, 14:00-15:45 (Seminar), Kunitachi; July 15, 2010, 15:50-16:40, Kunitachi, and ;August 26, 2010, 13:00-17:40 , Ehime/Ehime Plant

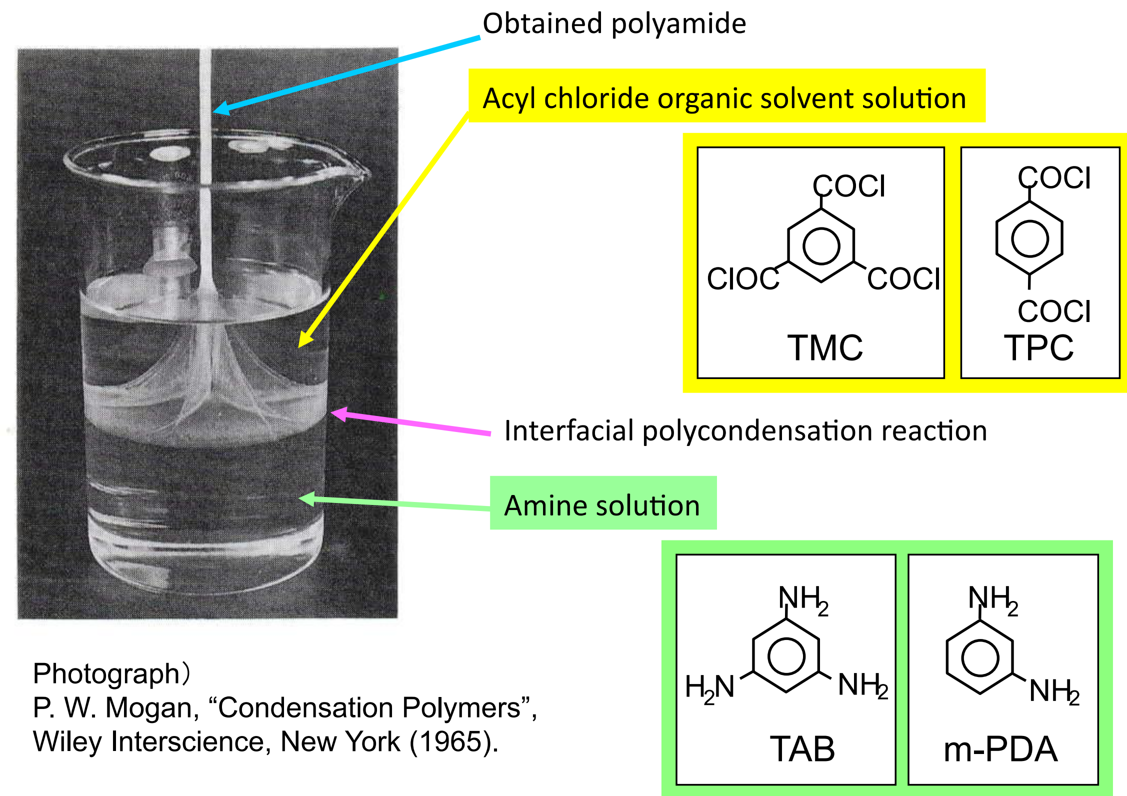
Masanori Yamamoto, Membrane Production Dept. Section Chief, First Membrane Production Section, August 26, 2010, 14:00-17:40, Ehime/Ehime Plant

< Supplemental Figures >

Supplemental Table 1: History of initial reverse osmosis membrane development period

Year	Event
1940s-1950s: Exploratory research (UCLA and The University of Florida)	
1949	Hassler submits report titled "The Sea as a Fresh Water". Continues work and in 1950 proposes desalination using reverse osmosis membranes.
1952	The Office of Saline Water is established within the U.S. Department of the Interior, and begins a national seawater desalination project.
1955	Charles Reid at The University of Florida proposes the Osmotic Membranes for Demineralisation of Saline Water research project.
1959	Breton and Reid clarify the desalination mechanism of cellulose acetate membranes.
1960s: Development for practical use (General Atomics (Fluid Systems), DuPont)	
1960	Loeb and Sorirajan at UCLA succeed in developing the asymmetric CA membrane, opening the way to practical use.
1963	General Atomics develops the CA membrane spiral wound element. Obtains patent in 1967.
1967	"Parmsep" desalination system utilizing aromatic polyamide membrane hollow fiber elements marketed by DuPont. Achieves first commercial success.
1968	Toray Industries, Inc. formally begins reverse osmosis membrane development.
1969	DuPont introduces reverse osmosis elements for seawater desalination.
1970s: Development of synthetic compound membranes (FilmTec, Fluid Systems (UOP), Toray, others)	
1972	Cadotte develops composite membranes by means of interfacial polycondensation at North Research
1975	Manufacture of composite membranes by interfacial polycondensation by Fluid Systems
1977	Under a government supported project at MRI, Cadotte develops a system of creating cross-linked aromatic polyamide composite membranes from difunctional amine and tri-functional acid chloride by interfacial polycondensation
1978	RO membranes manufactured by Fluid Systems delivered to seawater desalination plant in Jeddah, Saudi Arabia
1980s: Interfacial polycondensation polyamide membranes	
1980	Toray begins production of cross-lined polyether membrane (PEC-1000)
1985	Toray invents basic materials for four-component aromatic polyamide composite membranes. Begins production for ultrapure water production in 1987.
1985	The Dow Chemical Company acquires Filmtec
1987	Nitto Denko Corporation acquires Hydranautics Co.
1989	CA membrane hollow fiber elements manufactured by Toyobo Co., Ltd. introduced to the plant in Jeddah, Saudi Arabia
1990s	
1998	Koch Membrane Systems acquires Fluid Systems

Supplemental Fig. 1: Aspects of polyamide formation by interfacial polycondensation

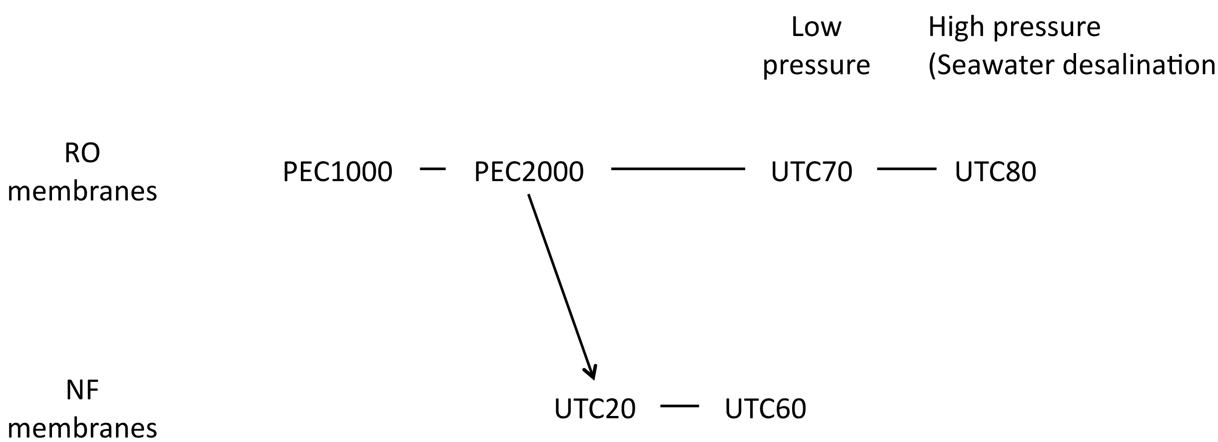


Photograph）
P. W. Mogan, “Condensation Polymers”,
Wiley Interscience, New York (1965).

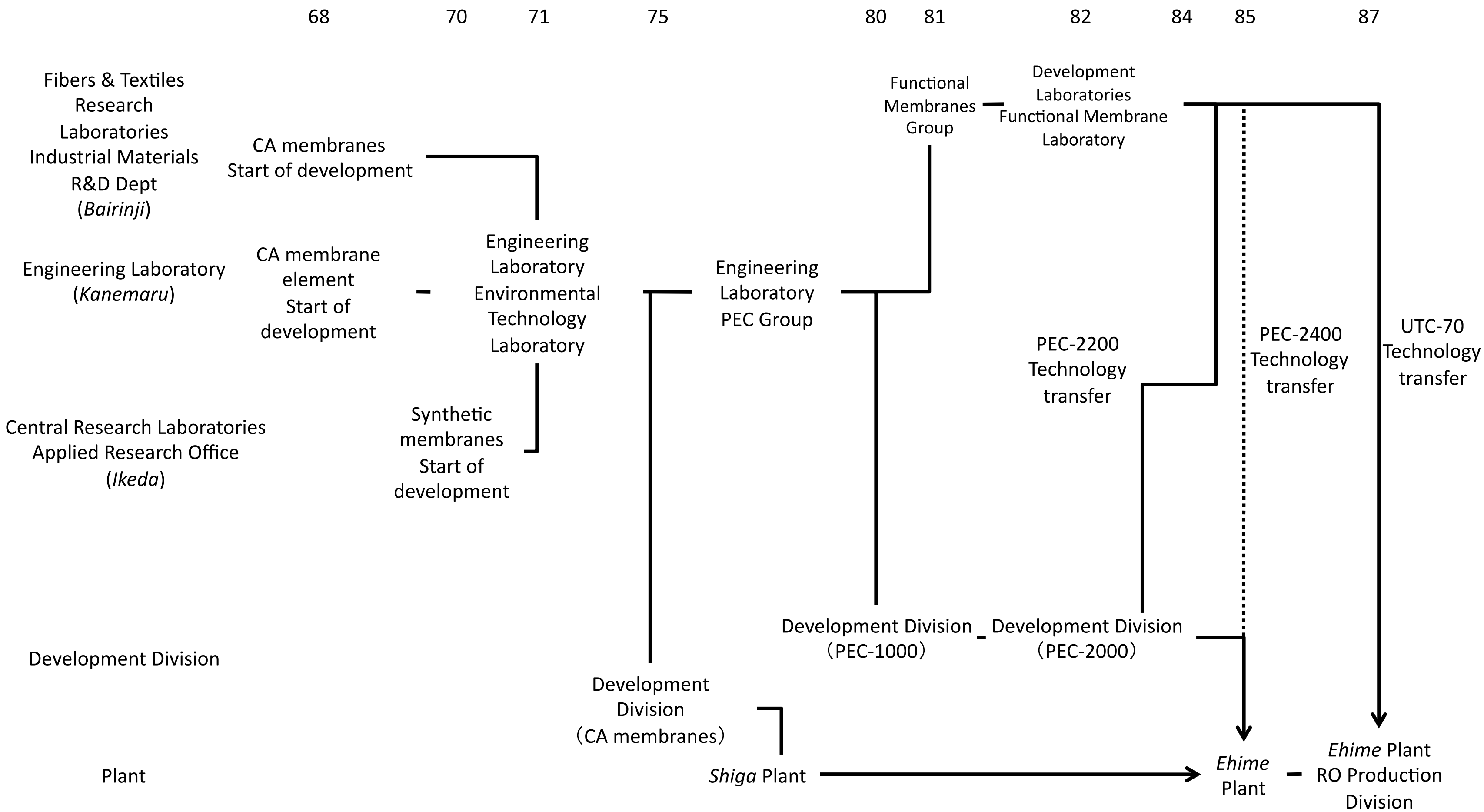
Source: Toray Industries, Inc. presentation materials

Supplemental Fig. 2: Development path

< Development of composite membranes >

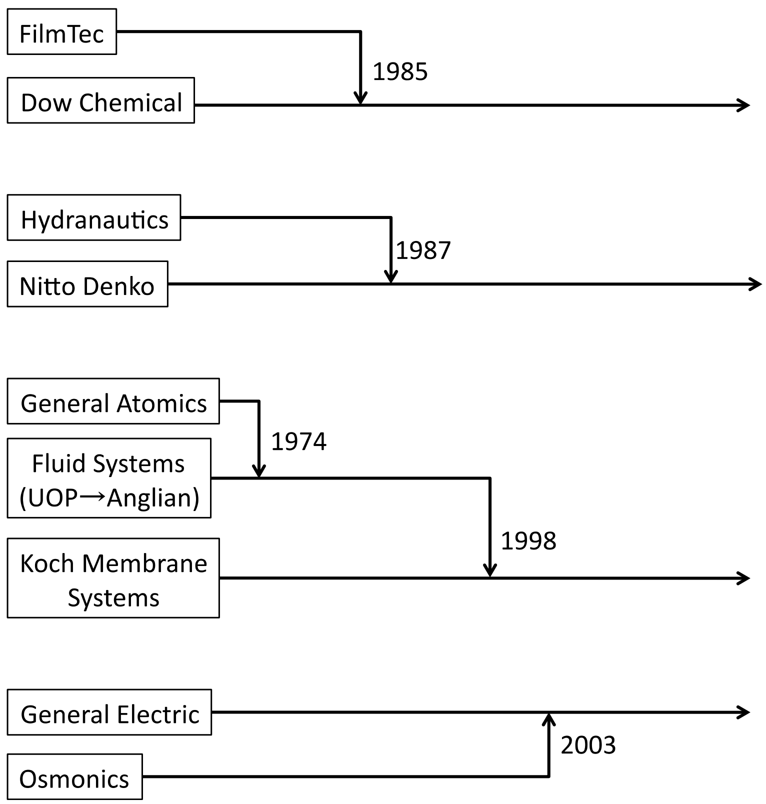


< Organizational flow >



Source: Prepared by the authors based on interviews.

Supplemental Fig. 3: Main M&A trends related to RO membrane technology



Source: Prepared by the authors based on Fuji-Keizai Group (2009) pp. 49-50.

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[1] This case is based on Fujiwara, Aoshima and Miki (2010) which is a result of research for the "Research Project on Okochi Prize Cases" project. This project is financially supported by the "Innovation in the Japanese Corporation – Education and Research Center for Empirical Management Studies" program established under the Hitotsubashi University Global COE Program. This project, which researches cases of actual results that have been awarded the Okochi Prize, analyzes topics such as summaries of technical innovations and the details of development processes, paths to commercialization and the results from commercialization, with the cooperation of the Okochi Memorial Foundation and corporations that have received the Okochi Prize. Together with compiling a library of case studies and accumulating case data on leading innovations in Japan, the project seeks to perform cross-case comparative analyses and identify the characteristics and issues affecting the innovation activities of Japanese firms. The authors wish to express their sincere appreciation for the extensive support and cooperation they received from the Okochi Memorial Foundation in the course of undertaking this project.
(Please visit the project website at http://www.iir.hit-u.ac.jp/iir-w3/reserch/GCOEokochiprize(A).html for details of the project's activities).
The authors especially wish to acknowledge the extensive support received from the following individuals for preparing the case: Toray Industries, Inc. (in alphabetic order, with titles omitted; position when interviewed shown in parentheses)
Fumiaki Fukui (Membrane Production Dept. Manager, Membrane Second Production Section)/*Yoshinari Fusaoka* (Sanji, Water Treatment & Environment Division, General Manager, Water Treatment Division)/*Yoshio Himeshima* (Director, Research and Development Division)/*Takeharu Inoue* (Water Treatment and Membrane Technical Dept. Manager, Membrane Technology Section)/*Maseru Kurihara* (Fellow, Doctor of Engineering/ Senior Scientific Director, *Funding Program for World-Leading Innovative R&D on Science and Technology (FIRST Program)*: "Mega-ton Water System"/Chairman, Asia-Pacific Desalination Association (APDA)/Chairman, Japan Desalination Association (JDA)/Seiji Minyu(Corporate Secretary)/*Eiji Nishioka* (Staff, Membrane Production Dept.)/*Motoyuki Tomiyama* (General Manager, Ehime Plant)/*Fujio Ueda* (General Manager, Membrane Production Dept.)/*Tadahiro Uemura* (Director (Technology) Water Treatment Division, Toray Singapore Water Research Center; Doctor of Engineering)/*Kenji Ueno*

(Sanji/ Water Treatment & Environment Division Manager, Water Treatment Division (Technology and Production)/*Masanori Yamamoto* (Membrane Production Dept. Manager, Membrane First Production Section)

[2] Ministry of Economy, Trade and Industry (2008), p.351.

[3] *Clean Water Membranes (2nd Edition)* Editing Committee (2003), p.15.

[4] During the same period, Toray also received the Chemical Society of Japan Chemistry Technology Award (1992) for "development of cross-linked aromatic polyamide composite reverse osmosis membranes" and the Society of Chemical Engineers Technology Award(1994) for "development of cross-linked aromatic polyamide composite reverse osmosis membranes for use in ultrapure water production".

[5] Another method for the desalination of seawater is electrodialysis. However, the percentage of fresh water produced is extremely low at just 5.5% (1996).

[6] *Clean Water Membranes (2nd Edition)* Editing Committee (2003), p. 87.

[7] This section on the initial history is based mainly on Glater (1998).

[8] Hassler and Joseph McCutchan published the results of the research from this period in 1960 (Hassler and McCutchan, 1960).

[9] Loeb joined the project in 1958. Both are assumed to have had Yuster as an instructor.

[10] Loeb, S. and S. Sourirajan (1960) "Sea Water Demineralization by Means of a Semi-permeable Membrane," UCLA-SEAS Report No. UCLA-ENG-60-60.

[11] Aerojet-General was unable to make its approach economically efficient, however, and eventually withdrew from the business.

[12] Global Water Intelligence (2008)

[13] US Pat. 3,367,504 (Inventor: Julius C. Westmoreland) and 3,417,870 (Inventor: Donald T. Bray). This element was put to practical use in 1971 in Japan as the world's largest brine water desalination plant.

[14] This method was first reported by Morgan in 1965 (Uemura and Henmi, 2008).

[15] Cadotte had studied not only interfacial polycondensation but also various methods. He made the final leap to interfacial polycondensation after finding a hint in a patent (US Pat.: 3,744,642) granted to Luciano Scalla.

[16] Dow Chemical, which acquired FilmTec Corporation and obtained the '344 patent, sued Koch Membrane Systems, Inc. and Hydranautics, which Nitto Denko Corporation had acquired, in 1991 for patent infringement. As a result of the court ruling, however, inventions related to the '334 patent were judged to be products that Cadotte had created while working under government contract during his time at MRI, and the '344 patent was ruled to be the property of the United States. Based on the court ruling, all firms that use the '344 patent are required to pay a patent fee to the US government.

[17] Sawada (2010)

[18] Interview with *Kurihara* (August 30, 2010). Clarification in parentheses added by the authors. Sales of PEC-1000 actually ended around 1993, following development of the UTC80 membrane as described below.

[19] Interview with *Kurihara* (August 30, 2010). The *Kawabata* referred to here is *Tatsuo Kawabata*, a member of the Diet from the Democratic Party of Japan and former Minister of Education, Culture, Sports, Science and Technology). At the time, he was employed at Toray, where he did research on RO membranes at the Engineering Research Lab from around 1970.

[20] Interview with *Tadahiro Uemura* (July 15, 2010)

[21] *Nikkei Sangyo Shimbun*, July 29, 1983, p. 18; March 3, 1984, p. 9; April 12, 1984, p. 12.

[22] Interview with *Tadahiro Uemura* (July 15, 2010)

[23] In contrast to UTC-20, a polymer, the newly developed UTC-60 is a monomer. The difference depends on whether the amine is a polymer or a monomer.

[24] Kurihara et al. (2001)

[25] Toray was able to develop this pressure-tolerant RO membrane ahead of other companies because it was able to take advantage of the fiber and textile technology and film technology the company had accumulated to innovate a new membrane element structure.

[26] Because *Katsunosuke Maeda*'s name is also on the patent for the BCS (Patent Publication Hei 8-108048; US Pat. 6,187,200), this implies *Maeda* was strongly involved in the reverse osmosis membrane business.

[27] One reason cited was the fact that even though a product water recovery rate as high as that available from the BCS was not necessarily required, the recovery rate improved as the result of the introduction of an energy recovery device. Fouling refers to the accumulation of impurities on the surface of the membranes.

[28] *Shukan Diamond*, January 27, 2007 issue, p. 82.

[29] *Nikkei Business*, July 24, 2000 issue, p. 83; March 19, 2007 issue, p. 134.

[30] Fuji-Keizai Group (2010). In FY2009, replacement demand exceeded new demand, which slumped because of the financial crisis. In FY2008, when new demand was larger than replacement demand, the situation was the reverse. In any event, what should be noted is that the scale of replacement demand cannot be disregarded.

[31] Kuniyoshi (2007)

[32] *Foresight*, February 2009 issue, p. 71. Other companies that have adopted strategies identical to Toray's are Dow, Koch, GE and Siemens. Nitto Denko and Toyobo supply only specific types of membranes, and are pursuing a

specialization strategy by concentrating only on the development and sale of membrane elements.

[33] *Nihon Keizai Shimbun*, July 31, 2010, p. 12.

[34] DuPont's focus on its proprietary aliphatic composite membranes, rather than on the aromatic composite membranes that became the industry-wide standard, was the main reason the company withdrew from the market.