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## **Freezing–melting process and desalination: review of present status and future prospects**

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**Abstract:** The main factors affecting the use of freezing-melting (FM) process are the capital cost and the process complexity. The FM technology was successful when these two factors were compensated by other advantages. The success in food industry was mainly due to its ability of producing high-quality products compared to the available thermal technology in the market. In chemical industry it is generally adopted when there are no other alternatives. It would be difficult to utilise the above advantages to adopt FM process for desalination. Furthermore, misconceptions and negative attitudes also affected the progress of FM process. In desalination, a number of existing technologies are available. The pilot studies in several countries indicated that the hybrid techniques of combining FM process and other desalination methods have high potential for development. The strategies for the commercial success of the FM process in desalination industry are identified in this paper.

**Keywords:** desalination; freezing–melting process; direct contact freezing; indirect contact freezing; vacuum freezing; eutectic freezing.

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## 1 Introduction

Desalination for water supply has grown steadily since the 1960s. In fact, seawater desalination is currently meeting most if not all requirements of domestic freshwater supply for some countries (*e.g.*, Persian Gulf countries) and is a supplemental source for others. Desalination refers to a water treatment process that separates water from salt solution. Based on the process, desalination plants can be categorised into two types. The first involves plants that employ a phase-change process. In such plants, desalination takes place while there is a change of phase (*i.e.*, evaporation or freezing). Plants that follow such a process include the following:

- Multi-Stage Flash (MSF)
- Multi-Effect Distillation (MED)
- Vapor Compression Distillation (VC)
- Solar Humidification/Dehumidification Desalination (SHD)
- Freezing–Melting (FM).

The second type of desalination plants are those that involve no phase change. In such plants, the extraction of water or salt takes place while the salts or pure water remains in the solution phase. These include:

- Reverse Osmosis (RO)
- Electrodialysis (ED).

Buros (2001) has categorised the desalting processes as major and minor processes based on its uses. Further details of the desalination processes are given by Ettouney *et al.* (1999), Fernandez *et al.* (1968), Hassan *et al.* (1998), UNEP (1996), Shwartz and Probstein (1969), Singh and Tembrock (1999), Thampy *et al.* (1999), and Wangnick (2003).

Although the Freezing–Melting (FM) process is not widely used commercially, the process has some advantages. Perhaps, the greatest potential advantage of desalination by freezing is the very low energy requirement compared to that of distillation processes (Brian, 1971; Johnson, 1979). The reduction in energy costs results as the latent heat of fusion of ice is only one-seventh the latent heat of vapourisation of water. FM separation could achieve 75% to 90% reduction of the energy required by conventional thermal process (Heist, 1979). The FM processes have the advantage of a low operating temperature, which minimises scaling and corrosion problems (Brian, 1971; Agnew and Anderson, 1992; Hartel, 1992). Inexpensive plastics or low cost materials can be used at low temperature (Johnson, 1976; 1979; Agnew and Anderson, 1992; Maguire, 1987). A very high surface area and high heat transfer coefficient can be achieved with a direct contact between the brine and refrigerant. FM process does not need pretreatment step, thus chemicals required for pretreatment could be avoided. In addition, it is not subject to fouling, similar to membrane separation (Johnson, 1976; Schroeder *et al.*, 1977), and low ecological impact (Maguire, 1987).

Traditionally, the disadvantages of freeze concentration compared to evaporation and reverse osmosis have included higher capital costs and higher operating costs during the ice separation (Muller, 1967). Other disadvantages are (Wiegandt and Von Berg, 1980):

- Retention of undesirable flavours and aromas (initially present in the feed saline water) that may come into the produced fresh water (Braddock and Marcy, 1987).
- Freezing process needs to include the growing, handling, and washing of ice crystals steps, and the need for mechanical vapour compressors.
- Compressors represent an expensive method of furnishing the energy requirements of the system (Brian, 1971).
- Probably the greatest deterrent to general acceptance of the freezing process is the fact that large plants cannot be designed and optimised with confidence, owing to the complexity of the unit operations in the freezing unit, melting unit, and wash-separation column.
- Trapping of salt solution in the ice during crystallisation needs crushing and recrystallisation of ice.
- A progressive increase in the concentrations of the dissolved substance, and non-condensable gases.
- High quality energy is required for crystallisation compared to low quality energy used in many evaporation processes.
- Certain amount of fresh water is required to wash ice for reducing salt content in the product water.
- Limited knowledge of ice crystallisation and growth in a slurry system, practicalities of handling ice slurries, good methods for complete separation of ice from brine, and hydration behaviour.

The first paper in this series presented the state of the art of the FM desalination process (Rahman *et al.*, 2005). The objective of this paper is to provide a critical evaluation of its present status and future prospects for desalination.

## 2 Present and future of freezing–melting process

### 2.1 Food industry applications of FM process

The use of FM is more common (instead of evaporation) to concentrate liquid foods due to the reduced loss of volatiles, aromas and thermal degradation of product (Braddock and Marcy, 1987; Liu *et al.*, 1998). The juice and dairy industries have used the technology successfully. It has been utilised commercially for the concentration of citrus fruit juices, for vinegar concentration, and for concentration of beer and wines. This technology has also been used for concentrating coffee and tea extracts, sugar syrups, maple sugar syrups, dairy products such as milk and whey, and aroma extracts (Wagner, 1983). More common to the food industry are the indirect contact crystallisers, where the refrigeration energy must pass from the aqueous food liquid through the walls of an appropriate heat exchanger. The most common ones are static layer growth system, layer crystallisation on rotating drum, dynamic layer growth system, and suspension crystallisation unit. Grenco (1991) reported about 50 plants in commercial operation using the FM process in food industry. Some commercial applications of FM processes in the food industry are listed in Table 1. The commercial successful applications of this technology in food industry have been identified. The food industry has tailored the technology in their areas of applications and has taken the advantages of the process.

**Table 1** Applications of the commercial FM systems for food liquids

<i>Food liquid</i>	<i>Product concentration</i>	<i>System</i>	<i>References</i>
Fruit juice	40 ~ 55wt%	Grenco in USA, Japan, Italy	Grenco, 1990; Muller, 1967; Deshpande <i>et al.</i> , 1984
Vinegar	12.8 ~ 40wt% acid content	Girder in USA	Staff, 1961
	up to 400-g	Votator	Votator, 1965a; 1965b
	48wt%	Grenco in USA	Wagner, 1983
Beer and wine	12.5wt%	Phillips	Deshpande <i>et al.</i> , 1984
	32% by volume	Grenco	Wagner, 1983
	Fourfold	Grenco in UK	Grenco, 1990
Coffee extract	35 ~ 48wt%	Grenco in Brazil, Japan, UK, Switzerland	Grenco, 1990, Wagner, 1983
Sugar solution	up to 50wt%	Grenco	Wagner, 1983
Whey	up to 40wt%	CSI	Saal, 1980; Davis, 1983
Skim milk	up to 36wt%	Grenco	Deshpande <i>et al.</i> , 1984; Wagner, 1983; Basta and Fouhy, 1993
Whole milk	up to 38wt%	Grenco	van Mil and Bouman, 1990; Basta and Fouhy, 1993
Tea extract	up to 35wt%	Grenco	Wagner, 1983

## *2.2 Chemical process industry*

The biggest potential for new FM applications is in those industries that consume large amounts of energy for separation process (Rosen, 1990). Potential applications also abound in the pulp and paper, pharmaceutical, chemical, and petroleum industries. It has been said that industry would not recommend FM in situations where distillation can be applied. FM is most applicable when distillation is impossible (azeotropic mixtures, or for isomers with close boiling points) or when distillation becomes extremely energy intensive, for example attaining from 99.9% to 99.99% purity. It will find acceptance only in selected niche markets, such as treating hazardous wastes. FM has strong potential in concentrating caustic soda, salts, acids, black liquor (from pulp and paper mills), benzene, toluene, xylenes, ethanol and isopropanol (Chowdhury, 1988). One of the major problems is the high concentration of the products (Rosen, 1990). The main problem with FM in the chemical industry is that it is a new technology that people are not familiar with and they already have processes that work to some degree. In the petroleum industry, FM is used along with fractional distillation to separate aromatic isomer mixtures. Direct-clathrate systems under development may further reduce energy costs and increase product yield. FM may also be used in naphtha fractionation and sour water treatment.

In the pulp and paper industry, for example, the chemical separation of wood fiber in pulping operations produces a spent liquid that is then concentrated. Much of the energy used in the pulp and paper industry goes into the evaporators for concentration of the pulp. FM system is now being developed for several applications for acid recovery in pulp and paper mill liquor. Another important application of FM is the concentration of waste liquids for disposal. Environmental restrictions on industrial waste disposal have led to expensive treatments before discharge. In a number of cases, conventional treatments are ineffective for waste streams and lagoons containing highly toxic chemicals, petrochemicals, and hazardous materials used in metal plating. In other cases, FM can purify a waste stream in one step that otherwise requires several conventional processes working in series (Rosen, 1990; Slade and Dare, 1993). Organic solutes exist in natural and waste water and exert effects at concentrations below the detection and quantification capability of most analytical devices. The role of micro-chemical contaminants in water has merited increased attention (Baker, 1965). Trace organic contaminant concentration in polluted waste could be achieved by FM process (Baker, 1967; 1969; 1970).

## *2.3 Potential technology transfer of FM process to desalination*

Desalination trial using FM process is mainly limited to the direct contact refrigeration system due to its processing efficiency and economics. However it has a number of drawbacks, such as residue of refrigerant in the water, formation of hydrates, and complexity of separating refrigerants from the ice. Technologically all the FM methods used in the food industry could be used for desalination since the limitation of FM process in food industry is the high viscosity of the liquid foods due to high solids content. All the methods could go up to 100 ppm level with multiple effects. The only point to be considered is the economic analysis of the FM process for desalination since water is a low value product compared to the foods. In many cases the food product, such as dairy items, may have an extremely high price where it is easy to justify more

processing cost when the methods produce high quality. However, detailed economic analysis needs to be done for desalination before reaching a conclusion. In the food industry FM was successful mainly due to its ability to produce high quality products compared to the other available technology in the market. It would be expected to face more challenge for applying FM process in case of desalination since numbers of viable existing technology are available at present although it offers energy savings. Johnson (1979) identified the main points that need to be considered for commercial potential of the FM process are: simplicity (as compared to other freezing processes and no refrigerant contamination, containment or removal problems), a totally closed cycle refrigeration system (more integrated and compact), and all element would be at the state of the art. In addition, reduction of capital cost and use of hybrid techniques should also be considered.

#### *2.4 Practical limitations for commercial success*

The choice of a technology is usually based on the product quality, operating economy, energy cost, initial investment, and complexity of the process (Moore and Hesler, 1963). At present no commercial FM plant is available for desalination of sea water (Wangnick, 2003). Since the process of FM is almost a century old, this question may fairly be asked: why is it, then that FM is not today in wider use. The reasons of this state of affairs are manifold, such as technical, political, and financial (Johnson, 1976). The applications of FM process in other industries have been explored in order to know how they feel about this technology and their learning curves. Three applications seem to be winning favour of FM process are: treating hazardous wastes, concentrating fruit juices, and purifying organic chemicals. The main reasons of these successes are due to the development of more efficient and high capacity process, and high purity or quality products. For example, boosting the appeal of the technology today is the use of new direct-contact refrigeration cycles that are 50% more efficient than traditional ones, new commercially available crystallising-units and crystal-washers that enable production of ultra pure chemicals (99.99%) and continuous processes that permit throughputs as high as 75 million kg/yr (about five times higher than previously) (Chowdhury, 1988; Rosen, 1990).

Based on the success in the other industry, especially food and chemical process, it is evident that the principal attraction of applying FM is its capability for concentrating heat-sensitive mixtures without damaging them, and separating hazardous and flammable chemicals, and azeotropic fluids. This technology is mainly adopted when there is no alternatives. This may be the main cause why it has not been used widely in the desalination industry, where numbers of alternative technology exist.

Traditionally, the FM process has been limited by high capital costs – two to three times those of distillation or evaporation systems and production problems caused by a greater degree of mechanical complexity (Chowdhury, 1988). There is a need to justify the capital costs of FM process if it is going to replace thermal processes (Rosen, 1990). In case of chemical processes, FM system manufacturers see this as a relatively hard sell, so they are going for markets where either the existing technologies cannot fill the need or the conventional processes cannot do it in one step. The practical difficulties and developmental stage reduce its wide commercial success (Rice and Chau, 1997).

FM is one of the most complex processes for desalination. It involves six steps plus the difficulty of handling liquid/solid slurry at its freezing point (Johnson, 1976). FM for desalination is an old standby, but today new process designs may open up applications. Continued areas of the development for FM systems include improved crystal growth, more efficient refrigeration, better design methods, and better heat recovery. FM must also demonstrate that it can match the level of concentration available with other technologies (Rosen, 1990). Several other drawbacks encumber the industrial use of FM systems. These include limited capacity, relatively high production costs, and limited maximum concentration of saline water (one of the major drawbacks) (Rosen, 1990). In several countries pilot FM processes, such as large solar-powered water desalination application in Saudi Arabia were built applying hybrid techniques (Rosen, 1990).

There has always been natural reluctance to accept a fundamentally new technique, especially when acceptable results are being obtained from an old and proven technique (Muller, 1967). There should be a need to change the attitude of the industry towards the potential technology in applying for desalination. The support of FM process from industry is very low compared to the RO or MSF processes. This has created an atmosphere conducive to easy explanations and sweeping statements.

Persistent misconceptions or myths proved damaging to the commercial development of freezing as a competitive desalination process (Wiegandt and Von Berg, 1980). They explored ten of these myths with respect to their engineering validity and a comparison is made between FM and other important desalination processes. This could reduce further damage of the FM technology since all represent incomplete, fuzzy or totally incorrect thinking. In 1952, the Organization for European Economic Cooperation convened a working party and promoted a negative impact on the development of sea-water desalting (Wiegandt and Von Berg, 1980). This team, with delegates from Belgium, Denmark, England, France, Germany, The Netherlands, and Sweden, and observers from Australia, South Africa, the USA, and the Rockefeller Foundation, made its report a year later. For atmospheric distillation and the FM method it recommended that no further work be considered; distillation because there was already sufficient knowledge of the process, and FM because the method appeared to be impossibly difficult and economically impractical. The engineering problems that needed solving to develop an FM process are more in number than that for RO or MSF distillation and far more complex than the later. Fortunately, there are a determined group of dedicated people in the universities, in industry, and in the US Office of Saline Water who realised that there would someday be an FM process for which the concepts would be sound and the engineering goals achievable (Wiegandt and Von Berg, 1980). This is evident from the literature that huge numbers of alternatives in the FM process components are being developed.

### *2.5 Hybrid techniques for use in desalination industry*

There is a high potential of combining the FM process with other desalination techniques. This hybrid approach could provide a synergy to the desalination process. One of the most promising one is the combination of reverse osmosis and freezing melting process. A zero-discharge direct-contact freezing/solar evaporator desalination complex is proposed as an efficient system to reduce the environmental impact of concentrated reject-brine from seawater desalination plants. The proposed method produced fresh water, Na, Mg and K salts and bromide (Madani, 1992). Hanafi (1994) identified

different possible desalination techniques in association with wind, tidal, and solar energy sources. An economic analysis showed that FM might be competitive with solar distillation in suitable locations. Ice collection methods, washing by natural drainage and the coupling of FM with solar distillation should be further studied (Wankat, 1973). El-Nashar (1984) presented a design analysis and economic evaluation of solar assisted Vacuum Freezing Ejector Absorption (VFEA) desalination plant with a capacity of 1 mgd and located in Abu Dhabi. The parameters that affect the design and plant costs are: seawater salinity and temperature, and solar collector outlet temperature. The collector outlet temperature was set at 90°C or 120°C using flat plate and evacuated tube collectors. The absorber loop of the VFEA system uses a sodium hydroxide solution with concentration ranging from 0.5% (dilute stream) to 0.6% (concentrated stream). The capital cost of the system increases with increasing seawater salinity and temperature, whereas cost decreases with higher collector outlet temperature (120°C). The thermal load on the concentrator increased with the seawater salinity, whereas it dropped substantially with the collector outlet temperature of 120°C as compared to 90°C. Life cycle savings in fuel costs of the solar-assisted VFEA plant were also estimated using a set of economic ground rules with the objective of specifying the optimum collector area, which yields the maximum life cycle savings. It was observed that the optimum area increases with increasing seawater salinity. Abdul-Fattah (1987) evaluated the alternative solar power systems compatible with freezing process considering the special case of Saudi Arabia. He pointed that FM can be a viable water system since freezing units of small scale are proven technology. Four designs of solar freezing are considered to select the most promising option. The decision is made on the basis of fuzzy set analysis of the criteria surrounding the choice. Taking the case of Saudi Arabia as an example, photovoltaic driven indirect freezing seems to be the most promising technology. Second in ranking is the dual vapour absorption freezing using thermal collectors.

Combined wind or tidal power-desalination systems include vapour compression, reverse osmosis, electro-dialysis and freezing melting techniques. Solar energy in direct thermal form or through its conversion to electric power has the potential of usages with almost all desalination technologies. Photovoltaic solar power can be used with FM process (Hanafi, 1994).

Schwartzberg (1990) suggested that the combination of reverse osmosis and a cheaper FM could provide economical alternative for concentrating liquids. Disposal of the rejected concentration brine from reverse osmosis plants may cause serious environmental impacts. Different ways for disposal handling are adopted, including pumping into lined evaporation ponds, injection into underground rock formation, or spreading on unusable arid land. All of these are short-term solutions due to the large amount of rejected brine to be disposed of (Al-Mutaz, 1987). The amount of rejected brine from reverse osmosis plants could be minimised by a further desalination of the rejected brine. The high concentration of the RO rejected brine limits the choice of the second stage desalination unit. The energy efficiency of FM makes it a promising choice since the process is independent of fouling, and low corrosion due to the operation at low temperature. Madani and Aly (1989) conducted economical and energy comparisons between the combined system and separate RO and direct FM units of 200 m<sup>3</sup>/h. The combined system can reduce the energy consumption by about 13% and 17% compared to separate RO and direct FM plants, respectively. The combined system can reduce the rejected brine by over 90% of that of separate RO plant at the same water production.



The use of electric field and ultrasound aided process could be used in the freezing-unit in order to enhance the performance. However all these additions will make the process more complicated.

### **3 Conclusions and recommendations**

A thorough literature survey was done on the different desalination methods in order to evaluate the present status and future prospects of the FM process. Desalination refers to a water treatment process that separates water from salt solution and its use has grown steadily since the 1960s. However, the technology of FM process for desalination is still lagging behind from the commercial success point. The choice of a technology is usually based on the product quality, operating economy, energy cost, initial investment, and complexity of the process. The main factors affecting the use of FM process is the capital cost and complexity of the process. This is clearly evident from the wide varieties of alternatives available in the state of the art. The technology was successful only when above mentioned two factors were compensated by other advantages. First of all we need to identify which industry is now using FM process successfully and to explore the possible reasons of success. Some sectors of food and chemical industries have used the technology successfully. In food industry it has been utilised commercially for the concentration of citrus fruit juices, for vinegar, coffee, and tea extracts, sugar syrup, maple syrups, milk use, and whey concentration, and for concentration of beer, wines and aroma extracts. In this case only indirect FM process is used to avoid refrigerant contamination. The success was mainly due to its ability of producing high quality products compared to the available thermal technology in the market. In addition, food is high value product compared to water. In case of chemical industry, it is mainly adopted when there are no other alternatives. In this situation, the principal attraction of applying FM process is its capability for concentrating heat-sensitive mixtures without damaging them, and separating hazardous and flammable chemicals, and azeotropic fluid. It would be difficult to utilise the above advantages to progress the FM for desalination. In addition, misconceptions or negative attitude also affected the progress of FM process. In case of desalination, a numbers of existing technology are available. The practical difficulties of FM process limiting its commercial success are complexity of the process, and high capital costs. In conclusion, the reasons for failure are manifold, such as technical, political, and financial. The pilot studies in several countries indicated that the hybrid techniques of combining FM process and other desalination methods have high potential for the future development. In this case, solar assisted systems may have high potential. The following strategies could be considered for commercial success of the FM process in desalination industry:

- Development of simpler and integrated FM process for using in desalination process. A number of attempts already made in case of vacuum FM process. Commercially available FM process used in food and chemical industries should be tested for desalination.
- Complete economic analysis of FM process to identify clear economic benefits to the desalination industry, and conduct campaigns to the desalination industry for developing positive attitude towards the FM technology.
- Development of hybrid techniques by securing the synergy of the processes.

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