Journal of Food Processing & Technology
ISSN: 2157-7110

Marella et al., J Food Process Technol 2013, 4:9
DOI: 10.4172/2157-7110.1000269

Review Article Open Access

Application of Membrane Separation Technology for Developing Novel Dairy Food Ingredients

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Abstract

In several processing industries, separation of different components from a mixture is an important unit operation. Sometimes the separated component is an important product, and in some instances it is a waste product. There is a variety of technologies available for use in separations, each operating based on physical and chemical properties of the mixture. One of the fundamental separation processes that brought about a major change in processing of dairy co-product streams is membrane separation technology. Membrane separations work on the basis of differences in size and shape of the molecules. Today dairy industry accounts for major share in the total membrane area installed in food processing, accounting for about 300,000 square meters of membrane area installed worldwide. Reverse Osmosis (RO), Nanofiltration (NF), Ultrafiltration (UF) and Microfiltration (MF) processes have been in use in the dairy industry for about 4-5 decades. Each of these processes is used for specific application. The phenomenal growth in the application of membrane separation technology in the dairy processing brought into focus the need for novel membranes and processes that enable production of new dairy based ingredients. Now-a-days wide pore UF process is used to develop α-Lactalbumin enriched protein products, loose NF process is used to recover and purify Oligosaccharides, high pressure UF process is used to replace the conventional NF process used for concentrating dairy product streams. In the present paper, new developments in the application of membrane separation in the dairy industry are presented along with the experimental data from the research conducted by the authors.

Keywords: Wide pore ultrafiltration; Milk minerals; Spiral wound; Membrane separation; Milk protein concentrate

Introduction

In several processing industries, separation technology is widely used to separate and in some cases to purify a particular component from the rest of the mixture. The target component might be the desired product or an unwanted component, separated to increase the purity of the original mixture. Separations take advantage of differences in physical or chemical properties of the mixture of components [1-3]. Of the several separation technologies available, membrane separation technology brought a significant change in dairy food processing. There are several advantages of membrane separation technology when compared to other processes. These include, separation of components at a lower temperature, separating the component in its native state, less energy use etc.

Reverse Osmosis (RO), Nanofiltration (NF), Ultrafiltration (UF) and Microfiltration (MF) are four commonly used membrane separation process in the dairy food processing. These processes have evolved from consistent research and development in the area of new membrane material development as well as applications [4]. Membrane separations have been extensively used in dairy process industry and are used for selective separation of different species. Commonly used separation processes are Microfiltration, Ultrafiltration, Nanofiltration and Reverse osmosis. These processes differ in membrane characteristics, their pore size and operating pressures to which they are exposed to. Reverse osmosis is mainly used to concentrate all the solutes present in a mixture, while removing water in the process. Nanofiltrationis used to concentrate the solutes while partially allowing the passage of some lactose and monovalent salts, thereby minimizing the effect of osmotic pressure. Conventional ultrafiltration is used to remove lactose and soluble salts from dairy mixtures. Microfiltrationis widely used to remove bacteria, somatic cells, fat and lately micellar casein from skim milk [5].

Applications in Dairy Industry

Milk is an essential constituent of many foods. It is a complex mixture of different components like fat, protein, lactose, minerals, etc. These components have specific nutritional and functional properties. Fractionation of these components will enable pure ingredients to be produced that have the advantage of constant quality [4,6]. Accordingly membrane processing is implemented in the dairy industry on a wide scale. The dairy industry accounts for the lion's share of the total membrane area installed in the food industries. It is estimated that about 500,000 m2 of membrane area is installed in dairy applications worldwide, and more than 70% of this area is in whey processing [7] especially in preparation of whey protein products. Specific applications of membrane processing in the dairy industry include fractionation of milk fat from whole milk, removal of bacteria and spores from skim milk, production of milk protein and native casein concentrates recovery and fractionation of whey proteins etc. Various applications

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Received July 31, 2013; **Accepted** September 28, 2013; **Published** October 08, 2013

Citation: Marella C, K Muthukumarappan, L E Metzger (2013) Application of Membrane Separation Technology for Developing Novel Dairy Food Ingredients. J Food Process Technol 4: 269. doi:10.4172/2157-7110.1000269

of membrane separations in the dairy processing have been extensively reviewed [8-10].

Novel Membrane Processes and Applications

Pervaporation

In the pervaporation process, feed liquid flows on one side of the membrane, and the permeateis removed as vapor from the other side of the membrane. Pervaporation is the only membrane process where a phase transition occurs with the feed being liquid and permeate being vapor. This is made possible by maintaining partial vacuum on the permeate side of the membrane. The components to be separated from the mixture need to be absorbed by the membrane, should diffuse through it and is expected to easily go into the gaseous phase on the other side of the membrane [11]. The required vapor pressure difference across the membrane can be maintained by a vacuum pump or by condensing the vapor produced which spontaneously creates a partial vacuum.

Pervaporation process can be effectively used for removal of water from liquid organics, water purification and organic/organic separations. Novel application of pervaporation is in purification/separation of ethanol from fermentation broths. As ethanol forms azeotrope with water at 95% concentration, pervaporation process appears promising because simple distillation will not work under these conditions. Pervaporation process is successfully used in production of pure water. A variety of membranes has been tried in these applications [5].

Electrodialysis (ED)

Electrodialysis is a membrane based demineralization process and uses ion exchange membranes. It is widely used in demineralization of liquid foods such as milk and whey and is used extensively in desalination of sea water. ED is known since 1890 but the first successful installation of ED plant was in 1952. The principle of ED process is based on the fact that when an aqueous solution containing ions of different mobilitiesis subjected to an electric field, the ionic species migrate to the respective opposite polarities of the field [11]. The ionic mobility is directly proportional to the specific electrical conductivity of the solution and is inversely proportional to the ionic concentration.

In an ED system, anionic and cationic membranes are arranged in a plate and frame configuration (just like the classic plate heat exchanger) and are placed alternately. The feed solution is pumped to the cells of the system, and electrical potential is applied. The positively charged ions migrate towards the cathode and negatively charged ions move towards the anode. Cations easily pass through the negatively charged cationic exchange membranes but are retained by positively charged anionic exchange membranes. Similarly, anions pass through anion exchange membranes but are retained by the cation exchange membranes. The net result is that one cell (pair of anionic and cationic membrane) becomes enriched / concentrated in ionic species while the adjacent cell becomes depleted of ionic species. The presence of impurities and precipitated materials, as in the case of biological material causes severe concentration polarization of the membranes. The problem is more severe with anionic membranes which are clogged by large organic anions (such as amino acids), precipitated calcium phosphate and denatured proteins [11]. This anionic membrane specific problem can be partially overcome by using neutral membranes in the place of anionic membranes. The advantages of using neutral membranes are that concentration polarization is reduced, easier cleaning cycles and extended process runs. However, the disadvantage includes low degree of separation because only one set of membranes is selective.

Membrane distillation

Membrane distillation is an evaporation process for separating volatile solvent from one side of a non-wetted microporous membrane. The evaporated solvent is condensed or moved on the permeate side of the membrane. When a hot solution and a cold aqueous solution are separated by a non-wetting membrane, water vapor will diffuse from the hot solution/membrane interface to the cold solution/membrane interface and condense there. So long as the membrane pores are not wetted by both solutions, the pressures on both sides can be different. The microporous membrane in this case acts as liquid phase barrier as water evaporation continues. This arrangement is called the direct contact membrane distillation. The main advantages associated with membrane distillation are: no possibility of entrainment, possibilities of horizontal configurations, low temperature energy sources can be used, reduced the problem of fouling due to the use of hydrophobic membranes, possibility of highly compact designs such as hollow fiber configuration [5].

Separations using liquid membranes

In separation processes using liquid membranes, the solutes diffuse through liquid contained in a porous support. These separations can be either gas or liquid separations. The solute molecules undergo dissolution in the membrane at the feed/ membrane interface. The dissolved solutes diffuse through the membrane and are desorbed at the other membrane surface. Applications using liquid membranes include waste water treatment: removal of phenol [12], removal of thiomersol from vaccine production effluents [13], trace metal treatment from natural waters. Other applications include removal of citric acid, acetic acid from fermentation broths, separation of gas mixtures, toxic heavy metal ions, separation of sugars etc.

Novel Applications of Membrane Separations in Production of Value Added Dairy Ingredients

Spiral wound microfiltration in production of Micellular casein concentrate

In recent years, there has been increased interest in use of microfiltration in production of micellar casein concentrate. Micellar casein concentrate is obtained from microfiltration of skim milk during which most of serum protein and non protein nitrogen components are removed in to permeate thereby increasing the ratio of casein to total protein and casein to true protein. The retentate obtained from this process is a concentrated colloidal suspension containing casein in micellar form, lactose, minerals and some serum proteins. Micellar casein concentrate has potential uses in cheese making, process cheese (as rennet casein replacer), nutritional meal replacements, whipped toppings, coffee whiteners etc [14-16].

To date most of the research on microfiltration of skim milk for production of micellar casein concentrate used ceramic microfiltration membranes. Ceramic membrane systems are capital intensive and membrane replacements are expensive. When compared to these systems, membrane separation systems using polymeric membranes requires less foot print, inexpensive and are familiar with most of the US dairy processors. In recent years, there has been increased interest in assessing the suitability and efficiency of polymeric membranes for production of micellar casein concentrate. It has been shown that using ceramic membranes, more than 95% of serum protein could be

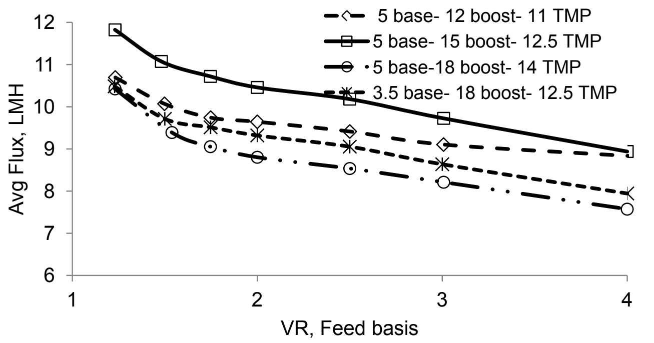

Figure 1: Effect of operating pressure on performance of spiral wound microfiltration process during production of micellular casein concentrate from skim milk. Flux is L per m2 h, TMP is transmembrane pressure and VR is volume reduction. Experiments were conducted at 65 F temperature using 0.5 μ polyvinyledene membrane.

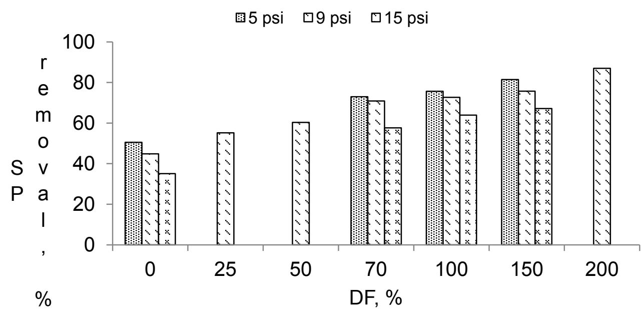

Figure 2: Effect of operating pressure on serum protein removal efficiency during spiral wound microfiltration of skim milk. SP is serum protein, DF is the amount of diafiltration water added during the process.

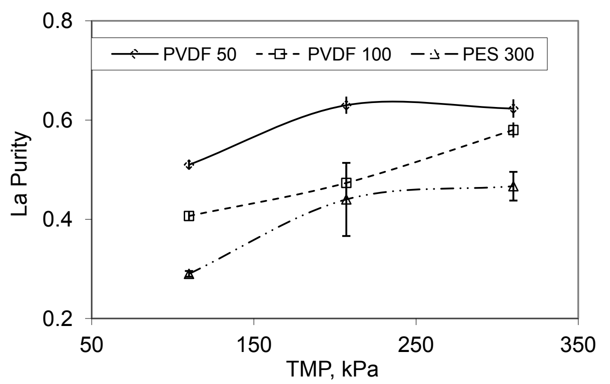

Figure 3: Purity of α-Lactalbumin obtained from wide pore ultrafiltration experiments conducted using cheddar cheese whey as feed material. La is αLactalbumin, PVDF 50 and 100 are polyvinyledene fluoride membranes with 50 and 100 kDa molecular weight cut off. PES 300 is Polyehtersulfone membrane with 300 kDa molecular weight cut off. TMP is transmembrane pressure.

removed in a 3 stage process in which diafiltration to a level of 200% (on feed volume basis) was used. Diafiltration is a process in which water is added to the retentate during microfiltration and further concentration is carried out. This step is intended to improve the serum protein removal and to control the membrane polarization phenomenon. A few studies conducted on the use of polymeric membranes for production of micellar casein concentrate showed that serum protein removal of the order of 40% was possible without diafiltration and with the use of diafiltration to the extent of 200% of feed volume, serum protein removal to the extent of 70% could be achieved [16]. However, these processes were carried out at elevated temperatures with the associated problems with energy consumption, bacterial quality etc. Marella et al. and Metzger et al. [14,17] carried out extensive research with the use of polymeric membranes for production of Micellular casein concentrate from skim milk. In this work, operating parameters such as operation pressure, level of diafiltration etc. were optimized for maximizing the serum protein removal from spiral wound microfiltration process. From this research, it was shown (Figure 1) that operating microfiltration process at a base and differential pressures of 5 and 15 psi resulted in to better flux rates. This research further showed that microfiltration process is extremely sensitive to pressure and operating the process at lower pressure results in maximum serum protein removal (Figure 2).

Wide pore ultrafiltration process for production of value added dairy ingredients

α-Lactalbumin enriched whey protein concentrate: Traditionally ultrafiltration used in dairy applications utilizes Polyether sulfone membrane with a molecular weight cut off of 10 kD. As these membranes have extremely tight pores, the ultrafiltration process using these membranes concentrates all the proteins present in either cheese whey or skim milk that is processed. When cheese whey is processed using the conventional ultrafiltration process, whey protein concentrates and whey protein isolates are obtained. These protein products are mixtures of individual and valuable protein fractions. In order to realize the true value of individual protein fractions, it is essential to fractionate these mixtures into products of individual components. One such high value protein present in cheese whey is α Lactalbumin. Previous research sah used polymeric membranes in hollow fiber configuration [18,19], combination of ceramic and polymeric membranes [20-22] and spiral wound polymeric membranes [23,24]. Using cheese whey as feed material, this research has demonstrated that α-Lactalbumin enriched whey protein concentrate can be produced with purity of 62% can be produce (Figure 3). When skim microfiltration permeate (serum whey) is used as feed material, α-Lactalbumin purity of as high as more than 80% can be obtained with proper selection of membranes and operating conditions (Figure 4).

Milk mineral from dairy process streams

Milk contains a variety of essential minerals and trace elements. The concentration of these minerals ranges from 8 to 9 g/l. Calcium, Magnesium, Sodium, and Potassium are the main cations present in the milk. Phosphate, Citrate, and Chloride are the main anions. Some of these minerals are present in dispersed form in milk serum while some of these are partially associated with milk components such as proteins (Casein, α-Lactalbumin etc.). This partial association with milk proteins gives structure and stability to milk and milk components. During manufacture of milk products, milk is subjected to various technological treatments such as filtration, acidification etc. These treatments partition the minerals present in the milk between different streams. For example, in cheese making Calcium, zinc, magnesium and phosphorus go with whey and end up in whey powders. Mineral content is higher in acid whey than in sweet whey [25].

Harvesting of milk minerals from dairy byproduct streams not only help overcome the fouling problems but also help the dairy processors to realize the true value of milk minerals. At present, milk minerals are harvested from dairy byproduct streams using some publicly known and some proprietary processes. For example, US Patent 5,639,501 describes

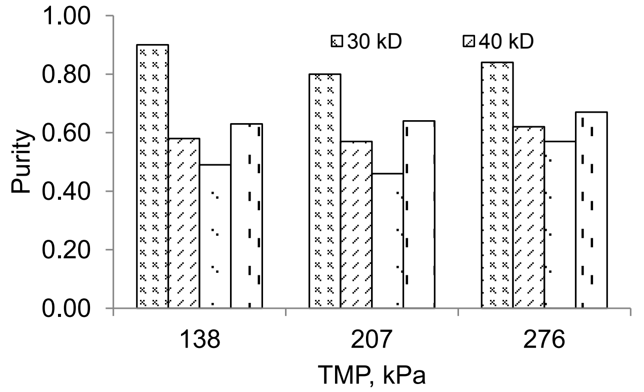

Figure 4: Purity of α-Lactalbumin obtained from wide pore ultrafiltration experiments conducted using skim milk microfiltrations permeate as feed material. 30, 40 and 100 kD are polyvinyledene fluoride membranes with 30, 40 and 00 kDa molecular weight cut off. 300 kD is Polyehtersulfone membrane with 300 kDa molecular weight cut off. TMP is transmembrane pressure. Bars with same letter are not statistically different (P <0.05).

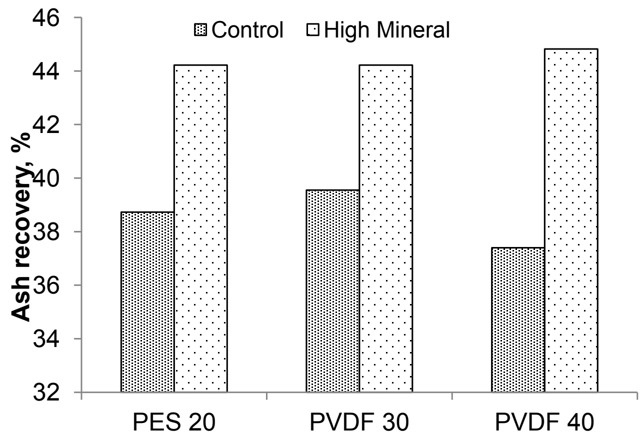

Figure 5: Mineral harvest data from wide pore ultrafiltration experiments. Ultrafiltration permeates obtained from milk protein concentrate manufacturing process were concentrated to 11% solids in reverse osmosis unit. Control is the feed that has normal level of minerals, High mineral is the feed that has higher mineral content. PES 20 is Polyether sulfone membrane with 20 kDa molecular weight cut off. PVDF 30 and 40 are Polyvinyledene membranes with 30 and 40 kDa molecular weight cut off.

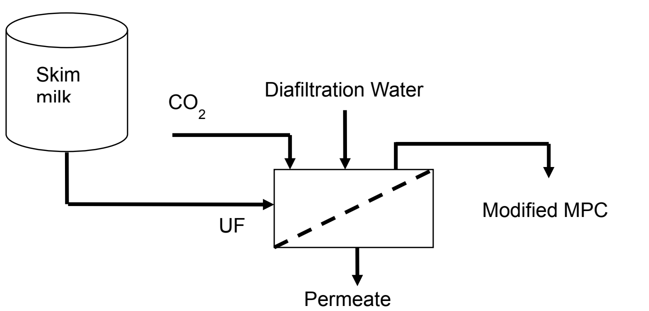

Figure 6: Process for production of milk protein concentrate with a modified mineral content. UF is ultrafiltration and MPC is milk protein concentrate.

a process wherein the pH of whey permeate stream containing about 15-24% solids is adjusted to 7.2 using a phosphate compound, heated to 155 F, and held at this temperature for 20-35 minutes in order to allow calcium phosphate to flocculate and precipitate out. Vyas and Tong [26] developed a process for recovering milk minerals from permeate stream using a combination of pH adjustment and heat treatment and reported a calcium recovery of 70%. In this research, conventional ultrafiltration membranes with a molecular weight cut off of 10 kDwas used. With the purpose recovering minerals and to develop a wide pore ultrafiltration process that has high permeation rates, Mealy et al. [27] conducted mineral harvest research using wide pore ultrafiltration membranes and reported ash recovery of 44% (Figure 5). This process using 40 kD PVDF membrane has exceptionally high flux rates of more than 100 LMH.

Filtration technology to produce mineral modified milk protein concentrates

Milk protein concentrate (MPC) is produced by ultrafiltration (UF) of skim milk to produce a product that is partially or completely delactosed and high in protein. During UF, water, lactose, NPN and some soluble salts are removed in to permeate stream. Higher molecular weight constituents such as caseins, whey proteins and some minerals are concentrated into retentate stream. In the production of MPCs, UF membranes with a molecular weight cut off of 5 and 10 kDare used to concentrate higher molecular weight components such as fat, protein and some salts. UF membranes allow passage of water, lactose, non protein nitrogen and some dissolved salts [28,29]. In some applications, diafiltration step is used in order to wash out more lactose and thereby increase the protein content. Depending on the volume reduction (VR) and extent of diafiltration (DF) applied, a variety of products are produced that range in protein content from 56 to 85%. MPCs with higher protein levels suffer from loss in solubility during storage of the product after production. Several researchers studied the reasons for loss in solubility and mineral mediated aggregation of proteins in one of the primary reasons for loss in solubility of high protein MPCs [30-32]. In-order to improve the solubility of MPCs, Baskhar et al. [33] developed an ion exchange process and showed that depletion of calcium from MPCs prevented loss in solubility of MPCs during storage. Mao et al. [34] used filtration technology where in diafiltration was conducted with the addition of sodium chloride at 50, 100 and 150 ppm and showed that this process produced MPC with a modified mineral content. Marella et al. [32] developed a process (Figure 6) for production of mineral modified MPC 80 with injection of carbon dioxide and showed that these MPCs retained its solubility when stored at room as well as elevated temperatures for up to 180 days. The mineral modified MPCs developed from this process showed superior functional properties [35].

Conclusions

Application of membrane separation technology in the dairy processing industry has brought into sea change in availability of a wide variety of dairy ingredients. Dairy applications account for major share in total membrane surface area installed in food processing industries. As more and more demand for novel dairy ingredients is growing, research is focusing on development of new processing technologies that help production of value added dairy ingredients. Membrane separation technology continues to hold a key role in selective fraction and development of novel dairy ingredients. In this paper, several new applications of membrane separation technology were discussed, and research results were presented.

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