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Separation and Recovery of Milk Components from Dairy Effluent

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Abstract- **Dairy is considered to be one of the most important agriculture based industry in Indian scenario but despite of this fact dairy industry is also one of the most polluting industries in terms of the organic content in dairy effluent. This effluent contains a high concentration of organic matter mainly lactose, protein, whey, and mineral salts. They can be harmful to the environment, if discharged directly with the other liquid effluents from the dairy industry. It requires multistage processing before its discharge. Additionally, the milk components present in the effluent possess large applications in food, chemical and drug industry. In this study, separation and recovery of components from dairy effluent was investigated. The Polysulfone (PSF) membrane based ultrafiltration process was adopted to separate lactose and protein with high yield and purity. The quantitative analysis were done for the recovery of the milk components from the effluent and it was observed that upto 90% of the lactose recovery could be achieved using the advanced separation technology.**

Keywords: Dairy Effluent, Membrane, Ultrafiltration, Polysulfone, Recovery.

I. INTRODUCTION

Milk and Milk Products have to been considered to be an important nutritional food because they are good sources of proteins, vitamins and calcium [1]. Lactose is one of the main components of human and animal milk. Due to its physiological and functional characteristics industrially manufactured lactose is used today in a large number of food stuffs, as well as in pharmaceutical industries [2]. A large amount of effluent is generated during the processing of milk in dairy.[2] This Effluent is characterized by their relatively increased temperature, high organic content and wide pH range, which requires special purification in order to eliminate or reduce environmental damage [3]. Conventionally it is done by the distructive methods like aerobic or anaerobic digestion [4]. This leads to loss of nutritional components like proteins and lactose from the effluent.

These components can be recovered from the dairy effluent with the help of membrane separation technique. The composition of the components present in the dairy effluent is solids (7%), whey/proteins (3%), fats (1%) and lactose (3%) [5]. Due to presence of these components, the effluent is characterized by high biological oxygen demand and chemical oxygen demand concentrations [6]. It decomposed rapidly and depletes the dissolved oxygen level of the receiving streams immediately resulting in release of strong foul odor due to nuisance condition [7]. The dairy industry generates about 0.2-10 liters of effluent per liter of processed milk with an average generation of about 2.5 liters of waste water per liter of the milk processed [8]. The effluent is generated from different processes including waste water from the production line cooling water, domestic waste water, acid whey and sweet. The sweet whey forms the most polluting effluent by its biochemical composition rich in organic matter and is from 60-80 times more polluting than domestic sewage [9].

Due to these conditions it highly necessary to treat the effluent before its discharge. On the other hands the components from this effluent possess very high nutritional, food and drug value. Hence their recovery is highly advisable. Use of ultrafiltration membranes can be one of the way for the treatment of the effluent and recovery of components.

II. OBJECTIVES

Current work is targeted towards optimization of membrane properties for recovery of components from dairy effluent before its disposal. The separation and recovery would be carried out using Polysulfone (PSF) based ultrafiltration membranes. Membrane formation parameters and its transport properties would be optimized for removal of components like butter protein, lactose and lactic. These products have large market value and separation would provide large economic benefits. Along with the transport properties, optimization of membrane would be targeted towards increase in fouling with reduction in fouling.

The objectives of treating dairy wastes are

- Reduce the organic content of the waste water.
- Remove or reduce nutrients that could cause pollution of receiving surface waters or groundwater.
- Remove or inactivate potential pathogenic microorganisms.
- Dairy effluent requires a specialized treatment to prevent or minimize environmental problems, as it contains high biodegradable organic compounds and this increases the complexity of the treatment process.

III. MATERIALS AND METHOD

A. *Material:*

Polysulfone (PSF) pellets of molecular weight 36000 is used as the material for membrane obtained from otto. N, N'-demethyl acetamide (DMAc) of 98% extra pure grade was purchased from avra synthesis pvt. Ltd. Indigeneous polyester membrane backing with 120 gfm was obtained from ShivOhm Membrane Pvt. Ltd.

B. *Casting:*

The dope solutions for membrane casting were prepared using DMAc as solvent under constant stirring by using magnetic stirrer. Predefined concentration of PSF was added to DMAc. The solution was stirred continuously for more than 48 hrs at a speed of 300rpm.The stirring speed was maintained using magnetic stirrer with rpm indicator. After 48 hrs the solution was degassed for removal of entrapped air with the help of probe sonicator for 2 minutes. It is followed by centrifugation at 3000 rpm for 15 min.

Centrifuged solution is spread on the polyester backing with the help of doctor knife with variable clearance 0.1 – 0.2 µm, on automated casing system. It allowed to pass through gelation tank maintained are 8 ˚C. It followed by rinsing at room temperature and final coagulation tank at 44 ºC [Fig 2]

Figure 1. Polysulfone Pellets

Figure 2. Pilot Scale Casting Machine

The formed membranes were analyzed visually for back penetration, and delamination of solution from backing. The membrane is cut and kept in the coagulation bath for 24hrs. Different concentration of membrane was casted using this machine (21%, and 25%).

Figure 3. Prepared Membrane of 25% Concentration

C. *Analysis of Membrane Properties*

1) *Bubble Point*

In order to determine the largest pore size of a membrane, the pressure at which bubble is reached is required. In order to reach bubble point, sufficient gas pressure must be applied to overcome the capillary forces of the pores [10]. The bubble point was calculated as defined by [11]. Obtained bubble point was calculated by using bubble point method [13, 15]. The smallest pore size can also be calculated by increasing the gas pressure till all pores has been emptied and gas flow through the membrane is that of a dry membrane profile. At pressures below the Bubble point, gas can only pass through the membrane through diffusion. We can calculate bubble point by the formula,

$$r_{p_i} = \frac{2\sigma * \cos\theta}{p_i}$$

Where, θ = angle of air and water contact = 70.5

σ = surface tension = 42.1 J/m^2

Pi = Pressure (bar)

2) *No. of Pores*

The bubble point was further used in calculation of pore size as described earlier [11, 13, 15].

$$\mathbf{ni} = \left(\mathbf{Ji} - \frac{\mathbf{J(i-1)} - \mathbf{Pi}}{\mathbf{P(i-1)}}\right) * \frac{\mathbf{8} * \boldsymbol{\eta} * \mathbf{l}}{\boldsymbol{\pi} * \mathbf{Pi} * \mathbf{rpi^4}}$$

Where, η = viscosity of water

r_{pi} = radius of membrane (m)

ni = number of pores (nm)

Ji = Water Flux (LMH)

Pi = Pressure (bar)

l = thickness of membrane (micrometer)

3) *Water Flux*

Water flux is expressed as amount of water passed through a membrane per unit area per unit time. It is used to express the rate at which water permeates the membrane. Its units are measured in GSFD and LMH. It is calculated using Amicon type cell with the membranes of 5 cm diameter.

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The water flux was measured using distilled water at pressure varying in the range of 0.4 to 1 bar.

Flux = V/A
V = v/t

Where, V = Volumetric Flow Rate (lit/hr)
v = volume (liter)
t = time (hrs)
A = area of membrane (m^2)

4) Molecular Weight Cut-Off

Molecular Weight Cut-Off (MWCO) is a method of characterization used in filtration to describe pore size distribution and retention capabilities of membranes. It is defined as the lowest molecular weight (in Daltons) at which greater than 90% of a solute with a known molecular weight is retained by the membrane.
MWCO is calculated using the PEG analysis of different molecular weight for the concentration of the membrane. The different molecular weight PEG solution was passed through the 21% and 25% PSF Membrane using the UF cell and rejection is collected and diluted in the ratio of 1:20 and is reacted with the reagent i.e. iodine solution which is diluted in 1:10 ratio. From the diluted rejection 0.6ml is reacted with 6ml iodine diluted solution and is kept for 30minutes for the reaction to occur at room temperature. After 30minutes analysis of the rejection is done at a wavelength of 535nm using UV Spectrophotometer.

IV. RESULTS AND DISCUSSION

A. PSF

PSF is used due to its chemical stability and thermal stability, it also has high strength and stiffness. It is water purified to remove all the suspended particle and dirt [16]. PSF were dissolved in N,N- Dimethylacetamide.

- Chemical Stability and Thermal Stability.
- They are characterized by high strength and stiffness, retaining these properties between 150°C to -100°C.
- It allows easy manufacturing of membranes, with reproducible properties and controllable sizes of pores down to 40nm.
- They have a high dimensional stability.
- It is highly resistant to mineral acids and electrolytes.
- It is resistant to oxidizing agents therefore it can be cleaned by bleaches.
- It is also resistant to surfactants and hydrocarbon oils.
- They are hydrophobic in nature.

B. N,N'-Dimethyl acetamide (DMAC)

- DMAC is a good solvent for a wide range of organic and inorganic compounds and it is miscible with water, ether, ketones and other compounds.
- The polar nature of DMAC enables it to act as a combine solvent and reaction catalyst in many reactions producing high yields and pure product in short time period.
- DMAC is a versatile solvent due to its high boiling point and good thermal and chemical stability.

C. Polyester Backing

- Polyester backing had the different properties i.e. it had high tear resistant, its durability is very high and polyester backing is highly flexible.
- Polyester backing is mainly recommended for heavier and very specific technical applications like removal of components from different effluents in industry.
- Also the most effective to use of polyester backing is it resist the heavier loads during process.

D. Membrane Property Analysis

1) Water Flux

The water flux of the membrane is one of the important characteristics defining transport properties. Hence water flux was measured using Amicon type dead end cell at 0.4 bar pressure [Table 1].

Table 1. Transport property analysis of formed membranes

Concentration of Membrane	Water Flux (LMH)	Bubble Point (bar)	Pore Size (nm)	Rejection
21%	362.52	0.2	1370	87.92
25%	84.14	2.4	114	96.51

It can be seen that the water flux was decreased from 362.52 to 84.14 lmh with an increase in PSF content in dope solution from 21 to 25 %, respectively. This can be attributed to reduction in pore size of due to increase in PSF content in dope solution. Similar reduction in water flux with increase in dope solution concentration is reported [11, 13].

2) Bubble Point

It can be seen from Table 1, that the bubble point of the formed membranes was increased from 0.2 bar to 2.4 bar with the increase in PSF concentration in dope solution from 21 to 25 %. This can be attributed to smaller pore formation. The formation of smaller size pores would require higher transmembrane pressure for transport of air by overcoming the resistance. Similar increase in bubble point with increase in PSF concentration is reported [11, 13, 14]. This reduction in pore size was supported by reduction in water flux [Table 1].

3) Pore size

An increase in dope solution PSF concentration from 21 to 25 % resulted in reduction in pore size from 1370 to 114 nm. A tenfold reduction in pore size was observed with the increase in concentration. This can be due to variation in gelation kinetics and rearrangements in PSF molecules during gelation. On exposure of dope solution to non-solvent the leaching solvent from solution starts. This triggers agglomeration of PSF molecules and formation of pores. An increase in PSF content in dope solution leaves lower amount of solvent in solution. This triggers smaller pores formation and a denser surface layer. Similar decrease in pore size with increase in dope solution concentration is reported [11, 13, 14]. This decrease in pore size can be duly supported by observed reduction in flux and increase in bubble point. The decrease in pore size would offer higher resistance for transport of water across the membrane. This resulted in observed flux reduction [Table 1]. Similarly the reduction in pore size resulted in higher resistance for transport of air, thus required higher pressure for transport of air across the membrane. This resulted in higher bubble point [Table 1].

4) PEG rejection analysis

The membrane were further analyzed for possible molecular weight cut off (MWCO) analysis using PEG rejection methodology. PEG was selected due to availability of polymer with range of molecular weight, easy analysis and non interactive nature. The solution of different MW PEG (1500, 6000, 9000 and 20000 Da) were prepared and passed through the membranes. Based upon rejection analysis, its projected MWCO was calculated [Table 2].

Table 2. PEG rejection analysis for the formed membranes

Concentration of Membrane	**Rejection of PEG MW (Da)**				**Projected MWCO (Da)**
	1500	**6000**	**9000**	**20000**	
21%	1.73	24.22	39.2	43	24000
25%	3.8	9.36	32.16	61.48	16300

It can be seen from Table 2, that the rejection for each of the PEG was increased for the membranes prepared from dope solution containing higher amount of PSF. This can be attributed to smaller pore formation with the increase in PSF content in dope solution. This reduction in pore size would increase resistance for transport and enhances the rejection properties. This resulted in increase in the projected MWCO for the membranes. Similar increase in MWCO with increase in dope solution concentration is reported [11].

E. Effluent component recovery analysis

The formed membranes were further analyzed for removal of milk components from local dairy effluent. The component removal was increased from 87.92 to 96.51 % with the increase in dope solution concentration from 21 to 25 % [Table 3].

Table 3. Milk component removal efficiency of formed membrane

Concentration of Membrane	**Recovery (%)**
21%	87.92
25%	96.51

This enhanced recovery of components can be attributed to the reduction in pore size. The milk effluent majorly contains whey, proteins and lactose contents. The 21 % membranes would be able to recover the larger active components like whey and proteins. While with the reduction in pore size the 25 % PSF based membranes could be able to recover the smaller components like lactose and lactin. This resulted in an increase in recovery from 87 to 96 %.

This show all the major components can be recovered from milk effluent, which can be used in further processing using membrane processes. These components possess large applicability in food, pharmaceutical and chemical industry. This would have a large impact on dairy economy.

V. CONCLUSION

Water reclamation is the new challenge in the new millennium. Membrane processes are proved to be convenient to treat dairy waste water for recovering of milk components present in dairy waste water and producing reusable water. The significant improvements in reliability and cost effectiveness of membrane technology have increased the recycling extent of dairy waste water. It can be seen all the major components can be recovered from dairy effluent to the extent of 96 %. The process carries out separation by physical methods thus the material is recovered in pure form and can be utilized in further processing. This would provide large economical benefits to dairy industry. Additionally recovery of the components would reduce the load on treatment unit and help to avoid pollution.

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