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The Impact of Fermentation Broth on Concentration of Citric acid by using Nanofiltration

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Abstract:

Recent developments in membrane technology have expanded the range of applicability of these traditional processes offering the processing industry as new alternatives to the more traditional technological approaches. One such development is Nanofiltration (NF). Nanofiltration is a pressure-driven membrane process which basically separates molecules large enough to avoid Reverse Osmosis(RO).NF membranes provide high rejection of organic molecules or multivalent ions having molecular weights ranging from 150 to 1000 with a relatively high-water flux, but lower operating pressure compared to RO. The main objective of this research work was to develop thin film composite membranes that can be used mostly in NF processes to concentration.The water present in feed was removed by applying pressure, so that the concentration of citric acid in feed has been enriched from 1% to 3%. This method enables very precise and controlled rejection and flux properties of the membrane.

Key Words:

Nanofiltration, Reverse Osmosis, Citric Acid, Flux, Concentration, Fermentation Broth.

Introduction:

Citric acid (2-hydroxy-1,2,3- propane tri carboxylic acid) is the most important commercial product. Citric acid [1] is a weak organic acid found in citrus fruits (lemon). It is a good, natural preservative and is also used to add an acidic (sour) taste to foods and soft drinks. More than million tones are produced every year by fermentation. In general, membrane separation processes are often more capital intensive but energy efficient when compared with conventional separation processes. Membrane devices and systems are almost always compact and modular. In addition, membrane processes can sometimes achieve totally novel results [10]. The membrane is highly selective and controls the passage of components from one side to another side. In membrane separation processes, the feed is separated into a stream that goes through the membrane, which is known as permeate, and the fraction of feed which doesn't go through the membrane, is called as reject or concentrate. Today membrane processes are used in a wide range of applications and the number of such applications is growing. In most membrane separation processes; different species are separated when they pass through the membrane because of their difference in the charge of transport. The transport rate of species through a membrane is inversely proportional to the membrane thickness. High transport rates are desirable in membrane separation processes for economic reasons. Therefore, a membrane should be as thin as possible. Every membrane separation process is characterized using a membrane to accomplish a particular separation. The membrane could transport one component more readily than other because of differences in physical or chemical properties between the membrane and the permeating components in the feed. In many cases the permeation rate through the membrane is proportional to the driving force i.e., the flux force. The pores in a NF membrane [7] correspond to 10 A° or 1 nm. This process is extensively used in the food processing industry and for the removal of divalent and multivalent ions from a feed mixture. Nanofiltration has exhibited broad application prospects in the field of precise separation attributed to its unique sieving performance for ions and small molecules, high permeation flux and low energy consumption. However, it remains a great challenge for current nanofiltration membranes (NFMs) to improve the permeability while maintaining the high rejection for divalent (or multivalent) ions. Separation in these types of membranes is mainly a function of molecular size and pore size distribution i.e., the particles whose size is larger than the largest pore of the membrane will be completely rejected. A key component in a successful sustainable and economical implementation of NF/RO systems in wastewater polishing is to find a disposal or reuse strategy for the resulting concentrates/rejects [17, 3]. Concentrates from NF process cannot be discharged directly into water bodies due to legislation and environmental regulations. For any discharge concept, apart from dilution to meet discharge levels, it is imperative to minimize the concentrate volume [18].Nano Filtration (NF) has drawn much attention in the field of separation and purification compared to other technologies as it offers flexibility in manipulating the product yield and purity by either adjusting the process parameters or/and by changing the membrane properties. NF was also reported to have high selectivity towards divalent compared to monovalent anions. Commercial NF membranes on a pilot scale are made mostly from polymers such as aromatic polyamide, polysulfone[8], and polyether sulfone [11] etc. The influence of operating parameters such as feed pressure 0-21 kg/cm^2 and total dissolved solids (TDS), water flux and impurity rejection were determined. In addition, NF membranes have molecular-scale pores throughout their selective layer results in generating a relatively high permeate flux at low operating pressure [5]. Concentration of citric acid [9] from fermentation broth[14] by NF will influence the operating conditions such as feed concentration, permeate pressure and temperature on performance is revealed.

Materials and Methods:

Nanofiltration/Reverse Osmosis experimental procedure:

Description of Nanofiltration system:

Fig.1 (a) and (b) represent diagrams of the NF pilot plant used in the studies. A feed tank of 30 L capacity made of stainless steel 3l6 was provided for storage and supply of industrial effluent [13] to the membrane system. A polypropylene (PP) pre-filter cartridge of 5 µm pore size was installed upstream of the flat sheet membrane module to prevent the entry of suspended solid particles. A high-pressure pump (Hironisha, Japan) capable of maintaining a feed pressure up to 25 kg/cm^2 was installed for transporting the feed liquid throughout the system. The pump was run by a 2 HP single phase motor (Crompton, India).

The feed tank had a provision for recycling of the concentrate after it passed through a heat exchanger that was installed for maintaining a constant feed temperature (27 ± 1°C). The heat exchanger consisted of a lengthy glass shell, which was circulated with ice-cooled water while the effluent flowed in the tube side of concentric glass coils placed inside the shell, which provided a large heat transfer area. The reject coming out of the heat exchanger was then recycled to the feed tank as concentrate. A restricting needle valve was provided in the concentrate outlet line at an appropriate position after the membrane module to pressurize the feed to a desired value as indicated by a pressure gauge installed upstream of the needle valve in the reject line. Permeate and concentrate flow rates were measured using calibrated rotameters.NF membrane of effective area 0.015 m^2 was fixed in the indigenously fabricated flat sheet test cell unit. Initially, the experiments were carried out using deionized water to study the effect of feed pressure on pure water flux. Deionized water was allowed to pass from side to side the membrane module using the high stress pump. To maintain constant feed concentration[4], the concentrate line was allowed to flow into the drain instead of recycling it to the feed tank.

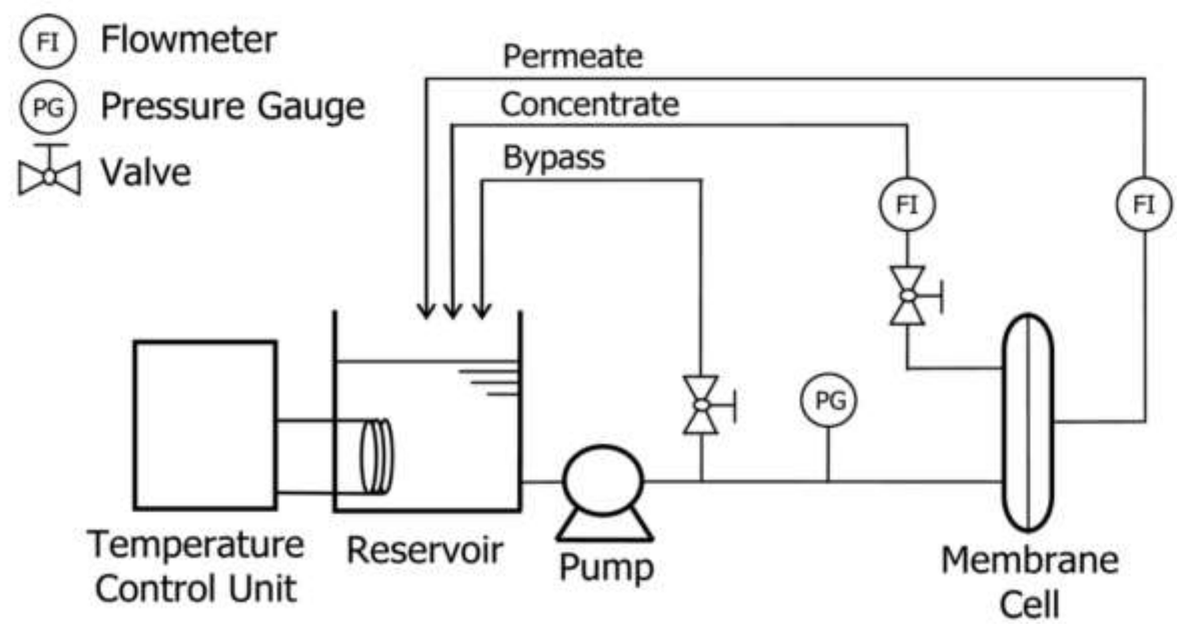

Fig.1: (a)Schematic diagram of the NF/RO system experimental setup.

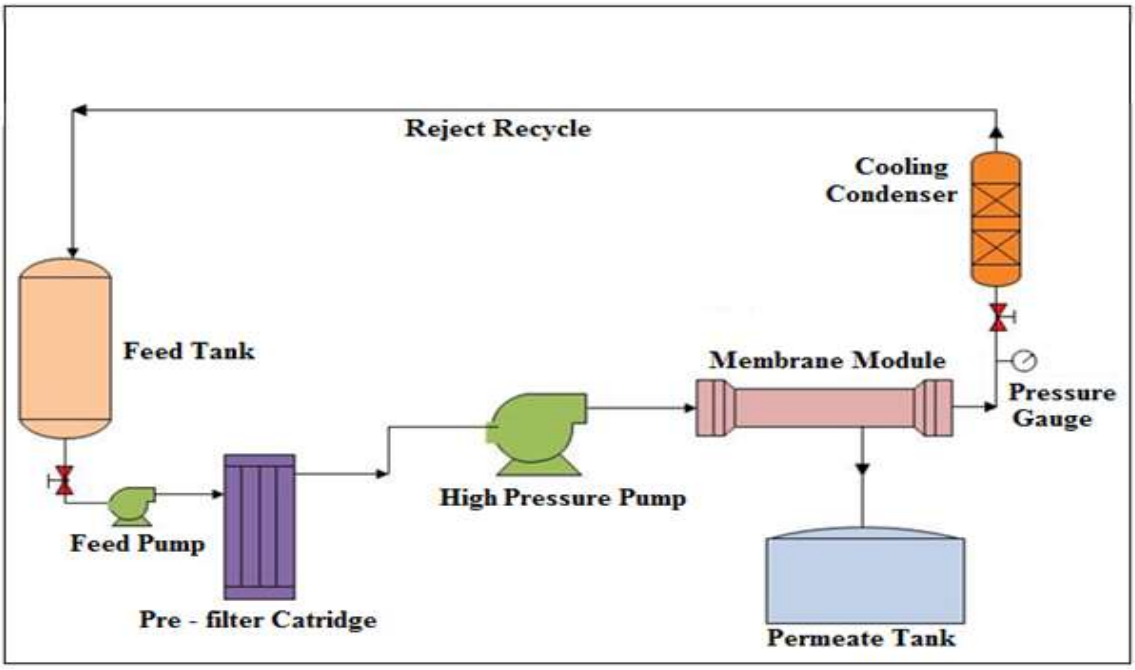

Fig.1: (b) Process flow diagram of nanofiltration system

The system was pressurized by throttling the needle valve installed in the concentrate line. The flow rates of permeate and concentrate were recorded at regular intervals of time at each pressure using rotameters.

Feed (effluent) of 30 L capacity was introduced into the feed tank and the pump was run to displace the distilled water accumulated in the system as dead volume. Initial feed sample was collected for analysis. Experiments were carried out to study the effect of feed concentration on flux and % rejection by varying the pressure from 3 to 21 kg/cm^2. This time the concentrate or reject was recycled until 65% water recovery was achieved. Permeate flow rate was measured at predetermined time intervals to observe any decline in flux. After attaining the stipulated water recovery, the samples of initial feed, final concentrate and average permeate were analyzed to determine TDS, COD, conductivity, BOD, and turbidity values.

Finally, the system was washed using cleaning chemicals such as citric acid, NaOH, EDTA chelating reagent and sodium lauryl sulfate (SLS) to remove metal precipitates, mineral and organic scales and to polish the membrane surface as per the cleaning protocols [6].

Results and Discussion:

Most Nanofiltration membranes are composite materials supported by a polymer substrate and manufactured in a spiral design compared to a flat sheet or tube geometry. The predominant model used today for industrial applications is the spiral configuration.

Characterization HPA-150 membrane with deionized water:

The effect of feed pressure on flux was studied with deionized water. The feed tank was filled with deionized water and the pressure applied across the membrane was varied from 7 kg/cm^2 to 35 kg/cm^2 and their corresponding fluxes were noted down.

Table 1: Effect of feed pressure on permeate flux

S. No.	Pressure (kg/cm^2)	Permeate Flux (L/m^{2}h)	Conductivity (µS/cm)	
			Permeate	Reject
0	0	0	42.5	42.5
1	7	19.2	32	60.9
2	14	48.0	10.4	63.4
3	21	69.6	8.5	66.0
4	28	79.2	8	67.6
5	35	93.6	7.6	71.5

Table 2: Feed pressure on permeate flux

S. No.	Pressure (kg/cm^2)	Permeate Flux (L/m^{2}h)	Conductivity (µS/cm)	
			Permeate	Reject
0	0	0	54.9	54.9
1	7	19.68	49.6	66.9
2	14	51.6	18	76.2
3	21	69.6	14.1	85
4	28	87.36	10.2	92
5	35	98.4	9	98

It is observed from the data that the flux of permeate is proportional to the applied pressure. As the pressure increased the flux of permeate also increased (Table 1 and 2).

Concentration of 1% Citric acid at 7 kg/cm^2 pressure by Nanofiltration:

Table 3: Concentration of TDS, conductivity, Flux and % Rejection at 7kg/cm^2 pressure

P= 7kg/cm^2	TDS (ppm)	Conductivity (mS/cm)	Volume (L)	Concentration of Citric acid (wt %)
FEED	1070	1.650	20	1.01
PERMEATE	250	0.160	12.5	0.05
REJECT	1520	1.972	7.5	2.23

% Recovery = 62.5 %
Volume of Permeate = 12.5L
Time taken for 62.5% recovery = 8.42 min
Area of membrane = 2.5 m^2
Flow rate = 89 L/h
Flux = 35.6 L/m^2h
% Loss of citric acid = 16.9 %
% Rejection of citric acid = 94.76 %

Permeate recovered = 12.5 L

Concentration of 1% citric acid by nanofiltration at 14 kg/cm^2 pressure:

Table 4: Concentration of TDS, conductivity, Flux and % Rejection at 14kg/cm^2pressure

P=14kg/cm^2	TDS (ppm)	Conductivity (mS/cm)	Volume (Liters)	Concentration of Citric acid (wt %.)
FEED	1080	1.620	20	1.06
PERMEATE	137	0.195	14.8	0.003
REJECT	1880	2.800	5.2	2.97

% Recovery = 74 %
Volume of Permeate = 14.8L
Flow rate = 178 L/h
Flux = 71.2 L/m^2h
% Loss in citric acid = 26.132 %
% Rejection of citric acid = 96.88 %
Permeate recovered = 14.8 L

Effect of feed pressure on flux and % rejection:

Table 5: Effect of feed pressure on flux and % rejection

Pressure (kg/cm^2)	Flux (L/m^2h)	% Rejection
7	35.6	94.76
14	71.2	96.88

The more the pressure applied the more will be the flux of permeate which results in less conductivity of permeate (Table 3). The more the applied pressure, the more will be the flux and hence % rejection will also be high due to solution-diffusion mechanism. The water flux increases whereas the solute flux remains the same since the hydrophilic polyamide membrane has affinity for water (Table 4 and 5).

Conclusion:

The dominant purpose of this research was to evaluate the performance of NF membrane techniques for separation, concentration and recovery of compounds and treatment of industrial effluents. Highly selective new membranes were synthesized in the form of thin film composites and studied for the performance of membranes and recovery and treatment of industrial effluents as well as aqueous solutions.

The goal in preparing these membranes is to combine high rejection together with high flux by limiting the trade-off factor between these two vital parameters which contribute directly to membrane separations. Their performance has been compared to that of commercial membranes to present NF as a more economical process through development of indigenous membranes, which otherwise becomes a highly capital-intensive technology owing to the high cost of imported membranes. The effect of operating parameters on separation performance was studied by different operating parameters such as time, feed composition, permeate pressure, flow rate and temperature to optimize conditions for achieving utmost output by concentration of citric acid from fermentation broth by NF [15] technique. The NF membrane exhibited considerably high-water permeability and substantial anti-fouling ability which clearly offers enormous scope for treatment of effluents coming from dairy, biochemical, food and bulk drug industries. The aim of this study is to highlight the capability of the NF membrane to efficiently reject larger organic molecules in the form of COD and BOD to facilitate economical treatment of industrial effluents [20] at lower pressures.

The citric acid solution also processed with NF to enrich the concentration. The water present in feed was removed by applying pressure, so that the concentration of citric acid in feed has been enriched from 1% to 3%. The optimum pressure was 14 kg/cm^2 and the flux was 71.2 L/m^2h with the solute rejection of 96%.

The effect of operating parameters [19] on separation performance was studied by different operating parameters such as time, feed composition, permeate pressure, flow rate and temperature to optimize conditions for achieving outmost output. Citric acid aqueous solution was also processed by NF toenrich its concentration. The water present in feed was removed by applyingpressure such that the concentration of citric acid in feed was enhanced from 1% to 3%. With an optimum pressure of 7 kg/cm^2, the flux was noticed to be 35.6 L/m^2h with a solute rejection of 94%. With an optimum pressure of 14 kg/cm^2, the flux was noticed to be 71.2 L/m^2h with a solute rejection of 96%. Based on the study, NF was found to be more economical than RO apart from sharing the same advantages such as economy, ecofriendly nature, process safety, ease of operation and maintenance besides with slight limitations in separation efficiency. Finally, it can be concluded that the membrane processes are eco-friendly, safe, low expensive and efficient processes, hence it can be employed in various biochemical, chemical, food, dairy, pharmaceutical industries for separation of desired products, bi products, and impurities.

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