A synopsis report on

Clarification and concentration of Sugarcane juice using membrane process: Fouling, Cleaning studies, Modelling and Process Simulation

Submitted by

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Introduction

Rapid urbanization as well as growth of population especially in developing countries has led to the setting up of various industries. Among these, food industries play a vital role in the development of economy of a country. Sugarcane juice is one of the most essential juices from which sugar is produced in the industry. Sugarcane cultivation in India is taking place from the period of 1400 to 1000 BCE and it is extensively accepted that *Saccharum* species are originated in India [1]. India is the second largest producer of sugarcane in the world by contributing 18.4% of the world's total sugar production i.e., 348 million metric tons (Horticultural statistics at a glance 2018, Ministry of Agriculture and Farmers' Welfare, Government of India).

Generally, products such as sugar crystals, jaggery and other similar products are manufactured by concentrating the sugarcane juice. The nutritional value of sugarcane juice per serving is: 28.35 g of sugarcane juice contains 26.56 kcal energy, 27.51 g carbohydrate, 11.23 mg calcium, potassium 41.96 mg, 0.27 g protein, iron 0.37 mg and 17.01 mg sodium. The extracted sugarcane juice contains large (75-90%) quantity of water, due to which large number of challenges are faced in preservation, storage and transportation. Major challenge of the sugar industries for processing of sugarcane juice are old and inefficient methods. Unfortunately, during the processing(clarification, concentration) and storage certain common problem occurred such as is gel formation at high concentration, browning (enzymatic) of juice, loss of vitamins and the components responsible for the juice flavour, taste due to the thermal and chemical pre-treatment. Conventional method of the sugarcane juice processing involves the pre-treatment with lime, sulfur dioxide and heat treatment above 100 °C. Carbonation, sulphitation or activated carbons are also used for the clarification of sugarcane juice. Numerous literatures reported the application of MF, UF for clarification of juice. Due to high viscosity and high osmotic pressure and large volume it is very difficult to handle. Hence, mostly polymeric membranes are widely used for sugarcane juice clarifications. Ceramic membranes by virtue of their high stability (chemical, mechanical and temperature), and long life are being increasingly used for a variety of separation applications. With the presence of foulants such as protein, polysaccharide, starch, gums, suspended impurities, sediments of Ca, Mg, Al, Fe, and phenolic compounds, robust membrane cleaning technique based on chemical cleaning using a high concentration of

NaOH, NaOCl, HNO_3, enzymatic detergents and hot water was used by many researchers for sugarcane juice purification.

Based on literature the scope of the membrane clarification might be quite promising since it will reduce the use of chemicals for clarification of juice. However, improvements on the study of fouling and cleaning process are needed. In literature inline scraping during ultra filtration was not explored at all for regeneration of membranes. Hence, there is scope to go for a physical cleaning which can reduce the use of chemical for removing foulants.

Conventional methods such as multiple effect evaporators are commonly used for the concentration of sugarcane juice to 50-60% by weight and vacuum filtration is used for further concentration. To replace or complement the conventional processes emerging technologies have been investigated which include membrane technology, ohmic heating, microwave heating, gamma irradiation, pulsed electric field and ultrasound. Existing method concentration of juice include nano filtration(NF), reverse osmosis (RO), forward osmosis (FO) membrane technology. On the other hand, FO process has more privileges over the RO process in terms of operating pressure and temperature, less energy consumption in presence of effective draw solution (DS) regeneration, high product recovery, low reverse solute flux and less fouling. The viability of using FO process was studied using test cell experiments by few researchers. These studies however are limited to FO experiments to measure the flux for sugarcane NaCl/seawater system using both commercial and lab prepared FO membrane.

There are some challenges which have to be faced during the concentration of sugarcane juice by FO processes such as low water flux, concentration polarisation (CP) and selection of DS, fouling. It has been also reported that use of clarified liquid food has reduced fouling intensity and provide stable FO operation. Therefore, this study was focused on the design of FO experiments to achieve the optimal operating conditions to minimize the water flux as well as to mitigate membrane fouling for sugarcane juice concentration process.

Many researchers have studied on hollow fibre reverse osmosis (HFRO) mathematical model with experimental validation. However, hollow fibre forward osmosis (HFFO) module performance have not reported by considering DS and Feed solution (FS) in batch mode. In literature, no mathematical model is reported for FO process by considering DS and FS in batch mode with tank model equation to estimate the performance of the batch process. There is thus a scope for the development of a mathematical model for the FO process to analyse the effect of DS flow rate in co- current and counter-current mode and validation of experimental result along with analysis of different process flow sheet simulation for finding the optimal result with respect to sugarcane juice concentration, energy consumption and reverse solute flux.

Objectives of the work

From the above research gaps, the objective of this doctoral thesis are derived as follows,

- To study the characteristic change of sugarcane juice by UF ceramic membrane.
- To study effect of conventional and unconventional physical cleaning mechanism on ceramic UF membrane system for sugarcane juice clarification.
- Experimental study of the concentration of sugarcane juice by HFFO Aquaporin membrane along with fouling and cleaning study.
- Development of a mathematical model and validation for concentration of sugarcane juice by aquaporin HFFO membrane.
- Performance evaluation of HFFO aquaporin membrane system by process simulation study for sugarcane juice concentration to optimize the membrane system parameters for minimizing the specific energy consumption with maximum yield.

Organisation of the thesis

The thesis is organised in six chapters, a brief summary of each chapters are presented in the following sections

Chapter1: Introduction

Chapter 2: Summarised literature review and objective of the thesis

Chapter 3: Sugarcane juice clarification by ultra filtration membrane with fouling and cleaning study

Chapter 4: Concentration of sugarcane juice using forward osmosis membrane with fouling and cleaning study

Chapter5: Modeling, validation and process simulation of forward osmosis membrane for the concentration of sugarcane juice

Chapter 6: Conclusion and future directions

Chapter 1: Introduction

This chapter presents a summary of fundamentals of sugarcane juice processing technologies along with the challenges in sugarcane juice processing. This chapter provides a brief introduction of membrane, ceramic membrane and forward osmosis membrane. Also the importance of clarification of sugarcane juice by membrane processes, importance and need of cost effective process for concentration of sugarcane juice by membrane processes are discussed. This chapter also include various studies which focus on mathematical model development for FO module

Chapter 2: Literature review and objective of the thesis

This chapter covers the detail literature review on (1) Application of membrane technology on sugarcane juice clarification and concentration and its problems during the application of membrane processes, (2) State of the art on the methods associated to clarification and concentration of sugarcane juice through ceramic membrane and forward osmosis membrane has been elaborately discussed, (3) It also deals with detail literature of modelling and validation of FO membrane for concentration process, (4) Review literature that focuses on the experimentation and simulation on FO membrane. Based on the state of the art, the scope for further research has been identified and the aim of the present work has been summarized. From the outcome of the literature survey, the objectives of the present research work are defined at the end of this chapter and finally, the organization of the thesis has been presented here.

Chapter 3: Sugarcane juice clarification by ultra filtration membrane with fouling and cleaning study

This chapter presents the potential application of novel low cost Lanthanum phosphate ($LaPO_4$) coated tubular ceramic membrane for the clarification of sugarcane juice. The main aim of this work is to prove potential suitability of sugarcane juice clarification by attainting quality of permeate without using chemicals which can directly be used for further concentration process. In this chapter design of the experimental setup (Fig. 1) and experimental procedure is illustrated. Here the application of $LaPO_4$ ceramic membrane for the clarification of sugarcane juice is explored without the application of lime by measuring colour removal efficiency, turbidity, polyphenol oxidase (PPO) enzyme, bacteria removal efficiency, permeate flux decline profile and cleaning efficiency. This chapter includes the various methodologies and techniques (FESEM, EDX, and FTIR) used for characterisation of membrane, sugarcane juice and fouling. A hybrid cleaning strategy involving an innovative brushing action on the membrane surface for physical cleaning combined with chemical cleaning was implemented in this study. A comparison of permeate flux decline profile of without physical cleaning for coated membrane, without physical cleaning for uncoated membrane and with physical cleaning for coated membrane and another comparison of cleaning efficiency of coated membrane for only chemical cleaning and for physico-chemical cleaning is studied. The experimental results conclude that raw sugarcane juice clarification using $LaPO_4$coated UF membrane was viable with the use of innovative physical cleaning method as proposed in this study.

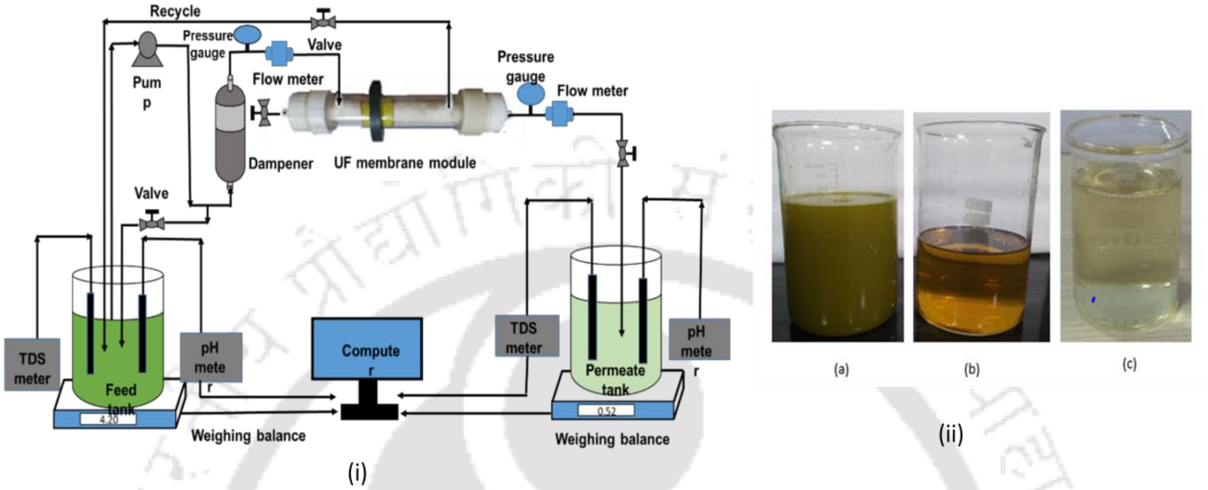

Figure 1. (i) Experimental setup with physical cleaning mechanism for sugarcane juice clarification, (ii) colour of feed, permeate of uncoated membrane, permeate of $LaPO_4$ coated membrane

Chapter 4: Concentration of sugarcane juice using forward osmosis membrane with fouling and cleaning study

This chapter elaborated the performance of a commercially available aquaporin HFFO membrane for concentration of sugarcane juice by adopting appropriate UF pre-treatment technique. This includes preparation of draw solution and study the effect of draw, fed solution flow rate, draw concentration and its direction of flow such as co- or counter-current on water reverse solute flux in batch mode. Figure 2 presents the experimental setup. The effect of DS flow rate on FO process performance in co and counter-current mode is studied. Counter current flow configuration between FS and DS was able to provide 13% high water flux than co current flow configuration. In case of counter current mode, the final concentration by aquaporin membrane in 12 min of batch FO operation with the initial DS concentration 100 $gL^{-}1$ was up to 53% - 60% (with negligible draw solute back diffusion) in comparison to the initial concentration. This is very close to the concentration achieved by multiple-effect evaporators in industrial operations before the feed enters the final evaporator where crystallization is done.The maximum average water flux of 5.226 ± 0.08 L h^{-1} m^{-2} was observed for higher DS flow rate (45 L h^{-1}). Both water flux and reverse specific salt flux(SRSF) improved by 15% and 39% for counter-current mode in comparison to the co-current mode at 45 L h^{-1} DS flow rate. The effect of UF clarified and raw sugarcane juice on FO membrane fouling was studied and it was observed that pure water permeability of membrane fouled by unclarified juice was reduced by 53.3% and that for UF clarified juice 11.2%. It was found that raw sugarcane juice led to severe membrane fouling that reduced water permeability after 60 min of operation. The fouled membrane was regenerated with only DI water wash for 30 min for UF clarified juice, however regeneration of membrane for the case of raw juice was not possible with DI water wash alone, and it required 0.1 M NaOH wash. From this study, it can be concluded that aquaporin HFFO membrane can be applied at industrial level since the repeatability; controllability can be done for this system and can provide an alternate solution to the evaporation and RO process where higher energy is required to concentrate the sugarcane juice.

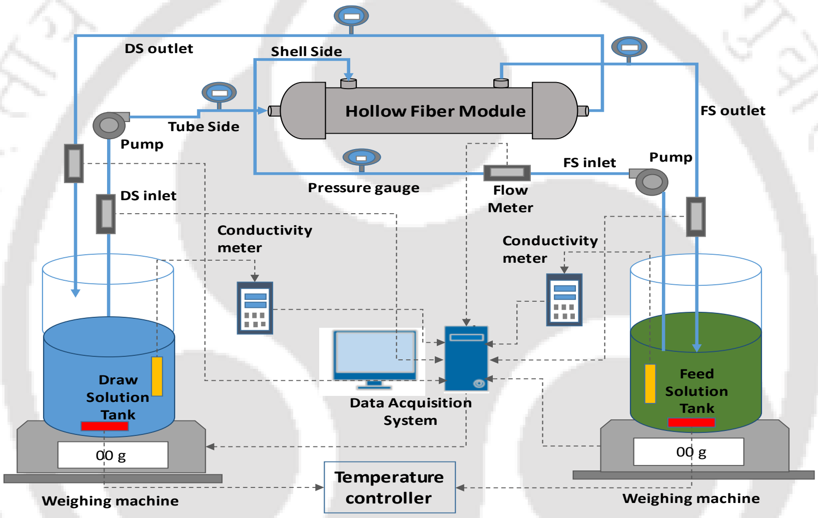

Figure 2. Experimental setup for sugarcane juice concentration

Chapter 5: Modelling, validation and process simulation of forward osmosis membrane for the concentration of sugarcane juice

In this chapter an aquaporin hollow fibre forward osmosis membrane is used in experiments. This chapter targets the modelling, validation and process simulation of forward osmosis membrane for the concentration of sugarcane juice. It discusses the mathematical model which is developed based on the tank mass balance and concentration polarization. By minimizing the error between experimental and model data, the new model is developed using Dymola software. The model equation mentioned in the theoretical section is validated with the experimental results with Modelica language in Dymola tool.

The experimental results of concentration of sugarcane juice in counter-current mode are found to be 154.4, 170.07, 173.891 g L^{-1} for DS flow rates 25, 35 and 45 L h^{-1} respectively and which is close to the model outputs with less than 10% errors. Similar results (less than 10% errors) are observed for co-current mode. The parameter such as pure water permeability (A), solute permeability (B), structural parameter (S), α_f and α_d are approximated by reducing the error between the experimental data and model output as we consider the ideal case, that the volume change in feed tank should be equal to the volume change in draw tank. The estimated parameter values are found to match the literature reported values.The reported values of parameters for aquaporin HFFO membrane are, L_p = 0.43 LMH/bar, B = 0.05 LMH and S = 210 µm.

A simulation study of the different flow sheet was doneby fixing operating parameters Q_{FS} and Q_{DS} toestimate the optimal process design in terms of juice concentration, energy consumption, reverse solute flux and processed volume for counter-current mode. Therefore, all flow sheet simulations were done at DS flow rate 45 L h^{-1} and FS flow rate 25 L h^{-1}. Three case study was done viz. case I: FS and DS both are recycle, case II: FS recycle and DS in continuous mode and case III: both FS and DS are in continuous mode. From these three cases of simulation study, it was observed that case 3 where both FS and DS are in continuous mode was more effective in terms of concentration, reverse solute flux and specific power consumption. The specific energy consumption is 78.85 W L^{-1} for the case of both feed and draw solution in continuous mode while, specific energy consumption is 217.94 W L^{-1} in case II (FS in recycle mode and DS in continuous mode) with single membrane module and 217.74 W L^{-1} with two-module system. By varying the draw solution concentration, simulations were also performed and it was noticed that at a concentration of 100 g L^{-1} draw solution, the specific energy consumption for batch process was 312.51 W $L^{-1}$and for continuous process with two stage module the specific power consumption was 78.85 W L^{-1}.Again, for two module system, to find the optimal DS flow rate ratio, a simulation study was carried out.

Chapter 6: Conclusion and future directions

This chapter summarizes the inferences drawn from various works presented in this thesis. It includes some suggestions towers the scope for future research.

Research Output

Peer-reviewed Journal Publications

- A. Akhtar, S. Senthilmurugan, K. Mohanty, R Sundar, R. Unnikrishnan, U.S. Hareesh, Sugarcane juice clarification by lanthanum phosphate nanofibril coated ceramic ultrafiltration membrane: PPO removal in absence of lime pre-treatment, fouling and cleaning studies, ***Separation and Purification Technology***, 249, **2020**, 117157.
- A. Akhtar, M. Singh, S. Senthilmurugan, K. Mohanty,Sugarcane juice concentration using a novel aquaporin hollow fibre forward osmosis membraneFBP-Food and Bioproducts Processing.*(Under review)*
- A. Akhtar, M. Singh, S Senthilmurugan, K. Mohanty,Modelling, experimental validation and process design of forward osmosis process for sugarcane juice concentration, LWT - Food Science and Technology.*(Under review)*

Proceedings of the International Conferences

- A. Akhtar, M. Singh, S. Senthilmurugan, K. Mohanty, Energy efficient forward osmosis membrane process for the sugarcane juice concentration,International Conference on Engineering Sciences and Technologies for Environmental Care (ESTEC-2020), page 99.
- A. Akhtar, K. Mohanty, S. Senthilmurugan, A Solution to Sugar Industry by Potential Use of UF Ceramic Membrane for Clarification of Sugarcane Juice and Concentration of Juice by FO Membrane", 3rd International Conference on Materials Science and Research November 28-29, 2019, Kuala Lumpur, Malaysia. DOI: http://dx.doi.org/10.18689/2638-1559.a3.003

Proceedings of the National Conferences

- A. Akhtar, M. Singh, S. Senthilmurugan, K. Mohanty,Concentration of sugarcane juice by Hollow Fiber Forward Osmosis (HFFO) Aquaporin membrane and analysis of juice properties post-concentration, 6th Bio Processing India conference, IIT Delhi, 2018.
- A. Akhtar, M. Singh, S. Senthilmurugan, K. Mohanty,Concentration of sugarcane juice by aquaporin forward osmosis membrane: Experimental results and process design,7th Bio Processing India conference, CSIR-CFTRI, Mysuru 2019, FP-001, page 57.
- A. Akhtar,S. Senthilmurugan, K. Mohanty, Study on Clarification of sugarcane juice by Ultra Filtration and Concentration using Aquaporin membrane, Research Conclave March 14-17, 2019, IIT Guwahati.