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Clarification of Synthetic Juice through Spiral Wound Ultrafiltration Module at Turbulent Flow Region and Cleaning Study

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***Abstract*—**Synthetic juice clarification was done through spiral wound ultrafiltration (UF) membrane module. Synthetic juice was clarified at two different operating conditions, such as, with and without permeates recycle at turbulent flow regime. The performance of spiral wound ultrafiltration membrane was analyzed during clarification of synthetic juice. Synthetic juice was the mixture of deionized water, sucrose and pectin molecule. The operating conditions are: feed flowrate of 10 lpm, pressure drop of 413.7 kPa and Reynolds no of 5000. Permeate sample was analyzed in terms of volume reduction factor (VRF), viscosity (Pa.s), ^{0}Brix, TDS (mg/l), electrical conductivity (µS) and turbidity (NTU). It was observe that the permeate flux declined with operating time for both conditions of with and without permeate recycle due to increase of concentration polarization and increase of gel layer on membrane surface. For without permeate recycle, the membrane fouling rate was faster compared to with permeate recycle. For without permeate recycle, the VRF rose up to 5 and for with recycle permeate the VRF is 1.9. The VRF is higher due to adsorption of solute (pectin) molecule on membrane surface and resulting permeateflux declined with VRF. With permeate recycle, quality was within acceptable limit. Fouled membrane was cleaned by applying different processes (e.g., deionized water, SDS and EDTA solution). Membrane cleaning was analyzed in terms of permeability recovery.

***Keywords*—**Synthetic juice, Spiral wound,ultrafiltration, Reynolds No, Volume reduction factor.

I. Introduction

CLARIFICATIONS of fruit juice are important study for storage of juice for a long duration and provide the juice for non-seasoning duration. There are several processes available to clarify the juice, but without pretreatment it is difficult to get the maximum clarified juice. Pretreatment process is applied to remove the maximum suspended materials and pectin molecule from the raw juice. Different pretreatment methods are available to clarify the juice, such as, mechanical separations (centrifugation), addition of fining agents (gelatin, bentonite and gelatin–bentonitecombination), combination of centrifugation and fining agent addition. Each pretreatment method has some merits and demerits. In centrifugation pretreatment method, power consumption is high and is time consuming; fining agent added pretreatments depend on fining agents concentration and the removal of suspended materials is low; combination of centrifugation and fining agents has improved the maximum impurities from the raw juice, but increase the cost [1].

After the pretreatment methods, some suspended impurities are left in the raw juice. Membrane separation process is applied to remove the remaining suspended materials and pectin molecule from the pretreatment juice. Membrane separation process has additional advantages, such as, the non-requirement of any chemical addition, the capability of generating permeate of acceptable quality and requiring less energy relative to conventional separation technologies (e.g. distillation, extraction and even adsorption processes) [2]. Different membrane modules are used in membrane separation process. Membrane module is arranged in a device to separate the feed streams into permeate and retentate streams. Various membrane modules, such as, tubular module; hollow fiber module; flat sheet module and spiral wound module are used [3].

The purpose of this work is to evaluate the membrane performance and observe permeate quality at two different operating conditions, such as, with and without permeate recycle. Permeate and retentate sample qualities are analyzed in terms of reduction viscosity, turbidity and electrical conductivity. Cleaning experiments for fouled UFmembranes were performed by using deionised water, sodium dodecyl sulfate (SDS) and ethylenediaminetetraacetic acid (EDTA) solution respectively.

II. Materials and Methods

A. Chemicals

The syntheticjuice was a mixture of sucrose, pectin and deionized water. Sucrose and pectin were procured from LobaChemiePvt Ltd, Mumbai, India. Cleaning agents, namely, sodium dodecyl sulphate (SDS) and ethylenediaminetetraacetic acid (EDTA) were also supplied by LobaChemiePvt Ltd, Mumbai, India.

B. Experimental Setup

As shown in “Fig. 1”, the Perma pilot plant, a spiral wound configuration with UF membrane module was used for clarification of synthetic juice. The UF membrane module specification is as: (materials: polyamide), cross sectional area (A_m: 1 m^2), module length (L: 0.51 m) and module diameter (d: 0.073 m). Membrane module was kept in SS cylindrical cell**.** Spiral wound pilot plant consisted of a feed tank with a capacity of 10L, cooling tank capacity of 10L; retentate line; permeate collection line; pressure gauge and rotameter. High pressure (up to 6894 kPa) triplex plunger pump was attached

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in pilot plant to circulate the feed (synthetic juice) in UF membrane module. Flow rate was maintained with constant flow rate (up to 10 lpm) using two rotameters and a control valve (fully open or fully closed) which were attached with module. Before experiment, UF membrane module was compacted with deionised water at 758 kPa for one hour.

The operating conduction for clarification of synthetic juice through spiral wound UF membrane module was shown in Table I. Temperature has great impact to effects the permeate flux. Cooling tank was provided to maintain the temperature of feed tank.

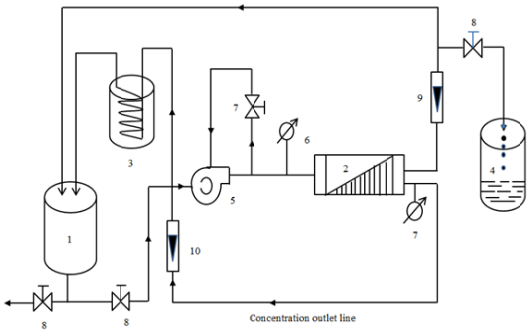

Fig.1 Systematic diagram of spiral wound UF membrane: (1) Feed tank, (2) UF membrane module, (3) Cooling tank, (4) Permate collection, (5) Plunger pump, (6) Inlet pressure gauge, (7) Outlet pressure gauge, (8) Control valve, (9) Permeate flow rate, (10) Concentrate flow rate

TABLE I
OPERATING CONDITIONS FOR CLARIFICATION OF SYNTHETIC JUICE THROUGH UF SPIRAL WOUND MEMBRANE

Parameters	value
Transmembrane pressure (kPa)	413.7
Flow rate (LPM)	10
Reynolds No	3638
pH	3.5
Temperature(ºC)	28±2

C. Sample Analysis

Different physical properties such as sucrose contents in terms of ºBrix analysis, clarity, turbidity, TDS, electrical conductivity, pH, viscosity, and pectin content in terms of alcohol insoluble solids (AIS) were analyzed for synthetic and clarified juice obtained from spiral wound UF membrane module. All the properties were measured thrice. Sucrose content was measured in terms of refractive index value using an ABBE -3L BenchtopRefractometer (Thermospectonic, USA). After that refractive index data was converted into ^{0}Brix [4]. Clarity was measured by percentage transmittance at 660 nm frequency using a UV spectrophotometer (Perkin-Elmer Precisel, Lamda-35, Canada). Turbidity, an optical characteristic or property of a liquid, which in general terms describes the clarity, or haziness of the liquid was measured by turbidity meter provided by VSI Electronics Pvt. Ltd., Punjab (India). Total dissolved solids (TDS (mg/l), electrical conductivity (m.mho/cm) and pH of feed, permeate and retentate streams at each operating conditions were measured by digital portable water/soil analysis kit provided by VSI Electronics Pvt. Ltd., Punjab (India). Viscosity was determined using a rheometer at a constant water bath temperature of 28±1ºC (HAAKE rheostress, Thermo Scientific, USA) [5]. Pectin material content was measured in terms of alcohol insoluble solids (AIS). AIS was determined by boiling 20 ml of sample mixed with 300 ml of methanol solution for 30 min. After simmering, the filtered residue was again washed with methanol solution. The residue was dried at 100ºC for two hours and was expressed in terms of g/l.

D. Process Evaluation

Spiral wound membrane performance during clarification of synthetic juice for two experiment conditions for with and without permeate recycle were analyzed in terms of volume reduction factor (VRF) and permeate flux. The VRF could be calculated by using the equation.

$$VRF = \frac{V_0}{V_R(t)} = \frac{V_0}{V_0 - V_P(t)} \quad (1)$$

where V_0 is the initial volume (ml) of feed, $V_R(t)$ is the final volume (ml) of retenate at particular time and $V_P(t)$ is the volume collected on permeate side at particular time for both experiment conditions. Permeate flux (J_w, $m^3/m^2.s$) and membrane permeability L_p for UF membrane module with time was analysed using the Darcy's law [6].

$$J_w = \frac{V_p}{A_m\ dT} = \frac{\Delta P}{\mu\ R_T} \quad (2)$$

$$J_w = L_p(\Delta P - \Delta\pi) \quad (3)$$

where A_m is the membrane cross sectional area (m^2), V_p is filtrate volume (ml) collected on permeate side at particular time interval dT (sec), L_p is the membrane permeability, ΔP is transmembrane pressure drop (kPa), $\Delta\pi$is the osmatic pressure difference between permeate and retenate, μ is viscosity (Pa.s) of permeate sample and R_T is the total membrane resistance (m^{-1}). In this study R_T is the combination of three resistances, namely, cake resistance, fouling resistance and membrane hydraulic resistance, (R_C, R_F and R_M).

$$R_T = R_C + R_F + R_M \quad (4)$$

III. RESULTS AND DISCUSSIONS

A. Effects of Operating Time on Permeate Flux and VRF with and without Permeate Recycle

Variation of VRF and permeate flux with operating time at transmembrane pressure 413 kPa for with and without permeate recycle were shown in Figs. 2 and 3, respectively. With permeate recycle, the VRF value increased from 1 to 1.9,

and initial feed volume was reduced to 7L. For without permeate recycle, the VRF increased up to 2.5 due to fouling of membrane in which an initial feed volume was reduced to 7L. For without permeate recycle, the VRF was higher compared to permeate recycle due to absorption of pectin particles on membrane surface. Increase of VRF has a significant impact on fouling behavior. The VRF value for with permeate recycle was less compared to without permeate recycle due to less deposition of solute (pectin) particle on membrane surface [7].

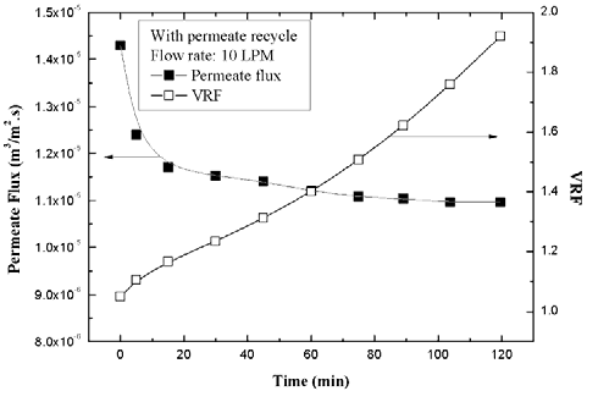

Fig. 2 Variation of permeate flux and VRF with operating time for with permeate recycle

For without permeate recycle, flux declined sharply due to deposition of pectin particles on membrane surface. Permeate flux decline sharply due to increase of concentration polarization. For without permeate recycle, flux declined sharply up to 2 min after that it started to decrease gradually due to pore blockage on membrane surface. Permeate flux decreased around 36% from 1.57×10^{-5} to 1.01×10^{-5}($m^3/m^2.s$) due to fouling of membrane surface and the pectin concentration in feed tank was reached up to 10g/l. With permeate recycle, the permeate flux decreased from original flux of 1.43×10^{-5} to 1.14×10^{-5} ($m^3/m^2.s$) within 2hrs. Higher flux decline around 36% was observed due to higher solute concentration at the membrane surface which causes a higher rate of cake formation [8]. Maximum operating times to conduct the experiment for with and without permeate recycle were120 and 8min, respectively. The steady state permeate flux at 413kPa was 1.14×10^{-5}($m^3/m^2.s$).With permeate recycle, the decline in permeate flux was small compared to without permeate recycle. Thus operating cost of cleaning the fouled UFmodule membrane was reduced compared for without permeate recycle.

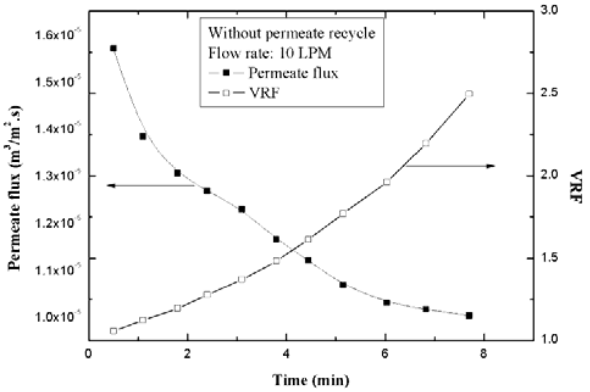

Fig. 3 Variation of permeate flux and VRF with operating time for without permeate recycle

B. Effects on Permeate Viscosity with Operating Time for with and without Permeate Recycle

In both the cases with and without permeate recycle; permeate viscosity decreased with increased operating time and was shown in "Figs. 4 and 5". With permeate recycle; sample was collected at different time intervals. It was observed that initially reduced viscosity, defined by Eq.5, decreased from 1 to 0.5 sharply within 15 min due to removal of pectin molecules. Reduction in permeate viscosity during 15 to 90 min interval was small due blockage of membrane pores. The reduction in permeate viscosity was around 50% for 413 kPatransmembrane pressure. Reduced viscosity is defined as:

$$\eta_r = \frac{\eta(t)}{\eta(0)} \tag{5}$$

where, $\eta(t)$ is the viscosity at particular time and $\eta(0)$ is the viscosity of feed.

For without permeate recycle, initially feed viscosity was higher due to higher concentration of pectin and sucrose molecules. Permeate viscosity reduced from 1 to 0.6 sharply within 1.5 min, but, between 1.5 to 8 min, the change in viscosity is marginal due to removal of pectin molecule in the permeate streams and after 3 min change in viscosity was negligible (shown in Fig. 5). After 3 min, pectin concentration on membrane surface was increased hence change in viscosity was small. The reduction in permeate viscosity was 35% at 413 kPa. Reduction in permeate viscosity for with recycle permeate was higher compared to without permeate recycle due to maximum retention of pectin molecule.

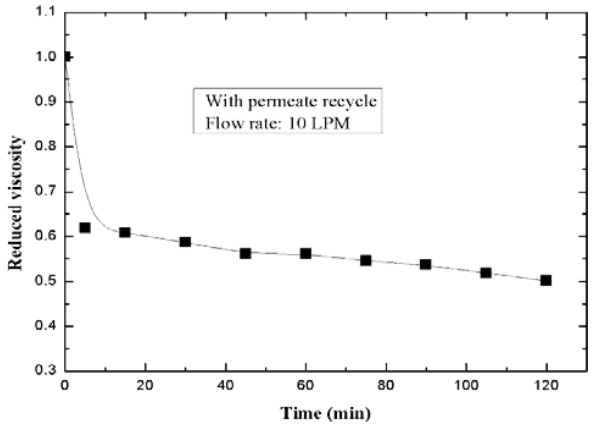

Fig. 4 Variation of reduced viscosity with operating time for with permeate recycle

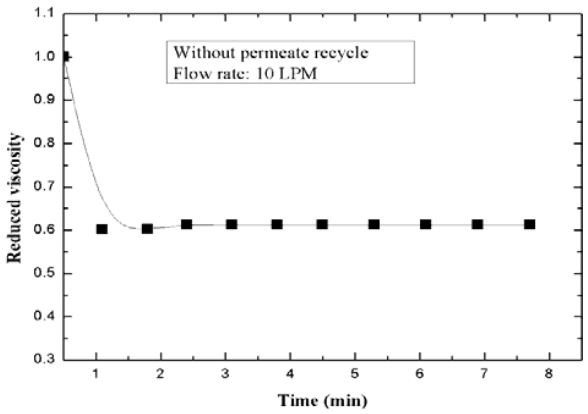

Fig. 5 Variation of reduced viscosity with operating time for without permeate recycle

IV. Cleaning of Fouled Membrane

Cleaning of fouled membrane is to remove all pectin particles on membrane surface by applying either physical or chemical methods. Fouled UF spiral wound membrane module was cleaned with different techniques, such as, using deiionized water in the module, using different chemicals, such as, SDS (sodium dodecyl sulphate) and EDTA (Ethylenediaminetetraacetic acid). Maximum operating time to perform the cleaning experiments was 20 min. During the cleaning experiments, low transmembrane pressure (69 kPa) was maintained with a flow rate of 9 lpm and temperature of 28±2°C.

First, fouled spiral wound UF membrane module was cleaned with deionized water. Then, the cleaning efficiency (%) and membrane permeability was measured. Cleaning efficiency with deionized water was 61% as some of the irreversible fouling layers of pectin were largely available on membrane surface. Other chemicals, namely, SDS (0.1 to 1 mM: pH=11) and EDTA (0.1 to 2 N: pH=11) were used to clean the fouled membrane agents were used to increase the cleaning efficiency and to recover the original membrane permeability. Recover membrane permeability with SDS were 75%.Still some relative amount of fouling layer remains on membrane surface. SDS was not enough to recover the initial permeability. With EDTA, membrane cleaning efficiency was increased up to 92%. The optimized dose of SDS and EDTA were 0.5 (mM) and 1 (N), respectively, to recover maximum membrane permeability.

V. Permeate Quality

Treated synthetic juice permeate quality after clarification from UF membrane module in spiral wound membrane for permeate recycle and without permeate recycle was shown in Table II With permeate recycle, the sucrose content decreased from 9 to 8.5°Brix and it was from 9 to 8.2°Brix for without permeate recycle. The alcohol insoluble solid (AIS) in permeate for with and without permeate recycle was almost nil. The clarity of permeate had increased from 81% to 95% and from 81% to 92%, for with and without permeate recycle, respectively. Density was slightly decreased from 1.10 to 1.01g/cm^3 in both cases for with and without permeatesrecycle due to removal of pectin particle. With permeate recycle, the viscosity reduction was62% and electrical conductivity decreased from 0.23 to 0.11 µS. For without permeate recycle, viscosity reduced from 2.03 mPa.s to 1.3 mPa.s and electrical conductivity decreased to 0.15 µS from 0.23 µS. The total dissolved solid (TDS) content decreased from 180 to 75 ppm for with permeate recycle and for without permeate recycle, the TDS decrease was 50%. On the basis of viscosity, TDS, clarity and electrical conductivity reduction results, permeate quality was better in for permeate recycle compared to without permeate recycle.

TABLE II
Comparative Clarified Juice Quality for With and Without Recycle

Sample	Conductivity (mhos)	TDS (mg/l)	°Brix	Viscosity (mPa.s)	Turbuidity (NTU)	VRF	Clarity T_{660}	AIS (gm/l)
Feed	0.23	180	9	2.03	76	1.0	81.3	2
Retenate	0.25	190	10	2.50	84	--	89.3	10
With recycle permeate	0.11	75	8.5	1.25	29	1.9	95.0	0
Without recycle permeate	0.15	90	8.2	1.30	38	2.5	92.3	0

VI. Conclusion

Synthetic juice was clarified through spiral wound UF module membrane at two different operating conditions, such as, with and without permeate recycle. Membrane performances were analyzed in terms of VRF and permeate flux. It was observed that the VRF rose to 1.9 for with permeate recycle and 2.5 for without permeate recycle. For with and without permeate recycle, the flux declined up to 20% and 36%. The reduction in permeate viscosity for with and without recycle permeate were 50% and 40%, respectively. °Brix content decrease was marginal (5.5 to 9%)

for both cases. Maximum turbidity reduced for with permeate recycle. For without permeate recycle, membrane had more fouled compared to with permeate recycle. EDTA had maximum efficiency (92%) to recover original flux as well as membrane permeability recovery. With permeate recycle is the best process in terms of maximum removal of pectin from raw juice, reduction of viscosity , improvement of clarity and total dissolved solids (TDS) decrease.

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