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ORIGINAL ARTICLE

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Clarification of jamun juice by centrifugation and microfiltration: Analysis of quality parameters, operating conditions, and resistance

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Abstract

Jamun is a subtropical minor fruit having many medicinal values. It has a distinguish flavor and color. The fruit pulp was depectinized with enzyme at low temperature and the obtained juice was used for clarification process. A comparison between physicochemical properties and operating parameters were performed between centrifuged and microfiltered clarified juice. Several operating conditions (speed and time) for centrifugation and pressure and membrane pore size for microfiltration (MF) had been chosen. Juice obtained from MF with a pressure of 137.89 kPa and membrane pore size of 0.45 μm has the best result in terms of clarity, turbidity, color, and protein concentration. Permeate flux declination was observed after a certain time period (130 min) due to macromolecules and fiber particles present in the initial juice. Fouling of the membrane was due to cake layer resistance (65%), which gave the maximum contribution to the total resistance, but a good percentage of restoration of membrane was observed after CIP treatment. The study will be helpful for scaling up the clarification process in industrial level.

Practical applications

Syzygium cumini, known as jambul, jambolan, jamblang, or jamun, is a tropical tree in the flowering plant family of Myrtaceae. A fresh food contains all the nutrients needed for good health, but because it may not always be possible to obtain fresh food, preservation becomes necessary. Clarification is necessary for making homogeneous juice. An attempt has been taken to clarify the jamun juice with microfiltration with different membrane as well as pressure. The data have been compared with traditional clarification process, that is, centrifugation. This article describes the optimization parameter for clarification process as well as the membrane resistance. The results will be helpful to clarify the juice in industrial level and for further research to clarify any other pulpy juice.

1 | INTRODUCTION

Package juice industry is the blooming sector of India with a growing trend of 12% per annum. Making juice from whole fruit needs some major postharvest operations. One of the major unit operations is the clarification process. The discoloration and haze formation in the fresh juice is due to the suspended pectin in cell wall (Pinelo, Zeuner, & Meyer, 2010). There are several types of primary clarification processes used in juice industry, such as enzymatic clarification, clarification through centrifugation, and microfiltration (MF) through membrane. Except citrus fruits all pulpy fruits need a primary clarification for settling down the macromolecular particles (protein, pectin, polyphenol, and cell wall). Other importance of clarification is to increase the acidity of the juice and to minimize the sucrose inversion level for longer shelf life (Doherty, Greenwood, Pilaski, & Wright, 2002). Initial clarification process helps for further processing of the final juice (filtration and evaporation). To obtain a homogeneous final juice, clarification process is needed. Enzymatic clarification is useful for the freshly prepare juice and to separate the pectin protein bonds (Sin, Yusof, Hamid, & Rahman, 2006), whereas centrifugation is used to separate the large macromolecules in the juice and MF even helps to remove bacteria, yeast, and mould.

Jamun is one of the evergreen subtropical minor fruits of India belongs to family Myrtaceae having a high amount of antioxidant,

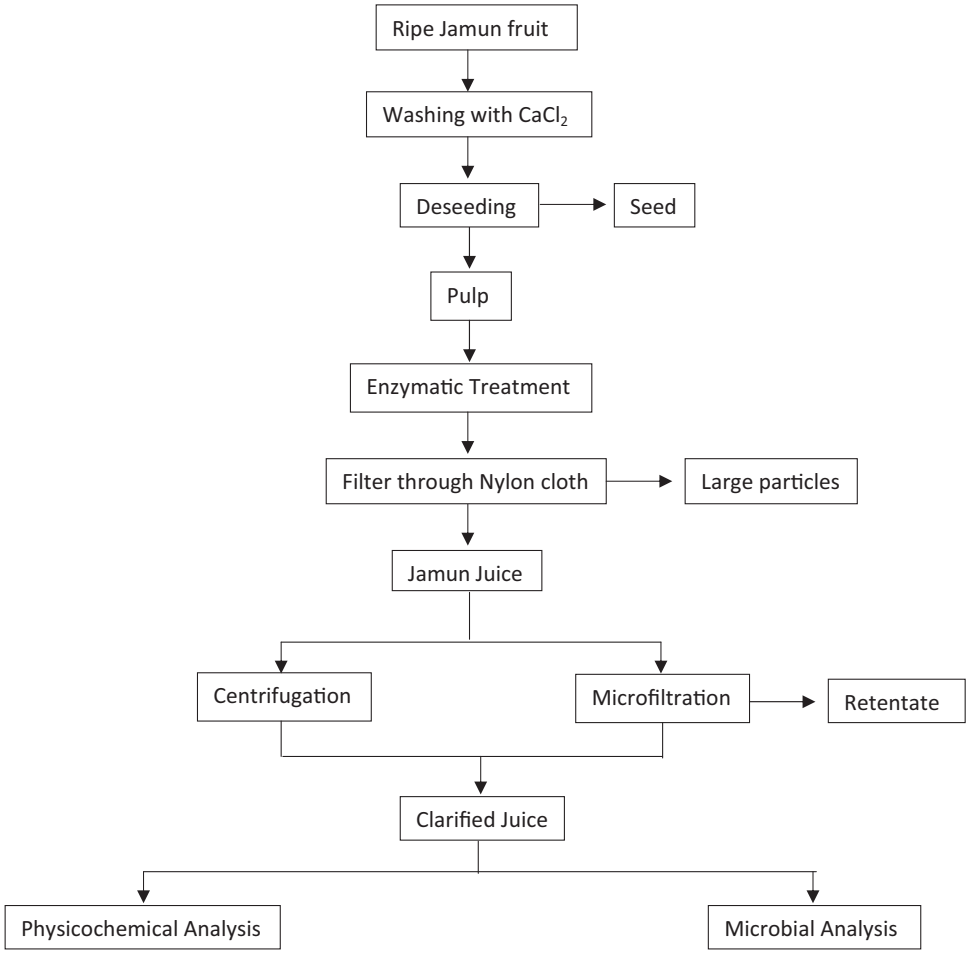

FIGURE 1 Process flow chart for clarification of jamun juice

polyphenols, and minerals. The fruit is known as wonder fruit for its medicinal value (Banerjee, Dasgupta, & De, 2005). The fruit as well as juice is beneficial for the diabetic people, but the fruit is available only for 2 months in the monsoon season. So to meet the demand of the juice throughout the year, package juice is used. The fruit is pulpy in nature, and hence for getting a high yield, enzymatic treatment is needed. Optimization of jamun fruit juice with pectinase enzyme has been already studied by Ghosh, Pradhan, and Mishra (2017).

Several studies have been reported for the clarification of juices by centrifugation. Orange juice has been homogenized and centrifuged, and effects of the treatments on color, cloudiness, and particle size have been mentioned by Sentandreu, del Carmen Gurrea, Betoret, and Navarro (2011). Beveridge (1997) mentioned in a report about various types of centrifuges and its application in the juice industry. Clarification of passion fruit juice and the comparison between centrifugation and enzymatic treatment process has been studied by Domingues, Junior, Silva, Cardoso, and Reis (2012). In that study, chitosan-treated centrifuged juice obtained the best result.

MF is a type of physical filtration process where a special pore-sized membrane is used to separate microorganisms and suspended particles from process liquid. MF is based on symmetric as well as asymmetric filters having pore size in the range of 0.05–10 μm. The separation principle is a sieving device and transport over the membrane by an operating force. Various studies have been reported for the clarification of juices through different types of microfilter for decade. Microfilter was used for orange, chicory, passionfruit, pineapple, kiwi, camu-camu, pomegranate, banana, and melon juice clarification (Barreto, Cabral, Matta, & Freitas, 2013; Cassano, Figoli, Tagarelli, Sindona, & Drioli, 2006). Cassano, Conidi, and Drioli (2010) evaluated the effect of MF on cactus pear juice quality and observed that the retention was enriched with antioxidant compounds, such as polyphenols, vit C, sugars, and amino acids. Aguiar et al. (2012) conducted an experiment on the clarification of apple juice by membrane separation processes at Brazil, to evaluate the final quality. The juice was clarified by MF, and the quality was better in terms of clarity and homogeneity.

Few studies have been reported with the comparison of MF and centrifugation as the primary clarification. Sagu, Karmakar, Nso, and De (2014) studied the comparison of banana juice clarification through centrifugation and MF and concluded that MF method is more suitable for further clarification in terms of viscosity, pectin, and clarity. Same type of result obtained by de Oliveira, Docê, and de Barros (2012) for passion fruit juice clarification.

No work has been reported till date for jamun juice clarification by centrifugation or MF method. The extraction of the juice has been optimized at low temperature with the help of pectinase enzyme (Ghosh et al., 2017). So the aim of the present study is to compare

centrifugation and MF process with different operating parameters for the initial clarification of the jamun juice and analysis of the obtained juice. Several physicochemical and nutritional parameters have been evaluated for the comparison. Also, fouling mechanism, analysis of membrane, cake layer resistance, and the effect of it to total resistance were also evaluated for membranes in case of MF process.

2 | MATERIALS AND METHODS

2.1 | Raw material

Fresh and ripe jamun (variety: Ram Jamun) was procured from local market of Rourkela, Odisha (India) and cleaned with $CaCl_2$ solution. As it is a highly perishable seasonal fruit, it was stored immediately in a deep freezer at −20 °C for further use (Ghosh, Pradhan, & Mishra, 2016).

2.2 | Preparation of jamun juice

Before starting, the experiment jamun fruits were kept outside the deep freezer and thawed for 4 hr until it reaches the room temperature. After that, the fruits were deseeded manually and the pulp was stored in a clean container. The pulp was mixed in a mixer (Bajaj Mixer, Bajaj Rex 500-Watt Mixer Grinder, Hyderabad, Telengana, India) for 5 min at its high speed. Based on the preliminary work, the grinded pulp was treated with pectinase (*Aspergillus aculeatus*) enzyme (Pectinex Ultra SPL; activity 3,800 U/mL; Sigma-Aldrich, Mumbai, India) with a concentration of 0.05% (wt/wt) for 80 min at 44 °C. Details of juice preparation have been mention by Ghosh et al. (2016). The obtained juice was used for further treatment (Figure 1).

2.3 | Clarification process

2.3.1 | Centrifugation

Centrifugation of jamun juice was performed in a lab scale refrigerated centrifuge (2–16KL; Sigma-Aldrich). The operation was done on batch mode method with 50 mL capacity tubes at 25 °C. The independent variables for centrifugation operation were rotational speed (rpm) and time (min). The rotational speed was varied from 6,000 to 8,000 rpm with a time period of 20, 40, and 60 min, according to the previous scientific results (Rai, Rai, Majumdar, Das Gupta, & De, 2006) and on the basis of preliminary trials. The supernatant was collected for further physicochemical analysis (total soluble sugar [TSS], pH, color value, turbidity, clarity, protein, and polyphenol) after centrifugation as primary clarified juice.

2.3.2 | MF

MF of jamun juice was carried out using a laboratory scale filtration system attached with hollow fiber membrane (Explorer 12, polysulfone material, effective area 0.005 m^2, inner diameter 0.013 m with a height of 0.312 m) supplied by CleanSep (Mumbai, India). Schematic diagram of MF process is given in Figure 2. A total of 500 mL of juice was used for each trial under batch concentration mode (permeate collected separately with recirculation of retentate) until the recovery was more than 80%. The filtration system consists of a feed tank (capacity 2 L) from where the feed was passed through peristaltic pump. Flow rate of

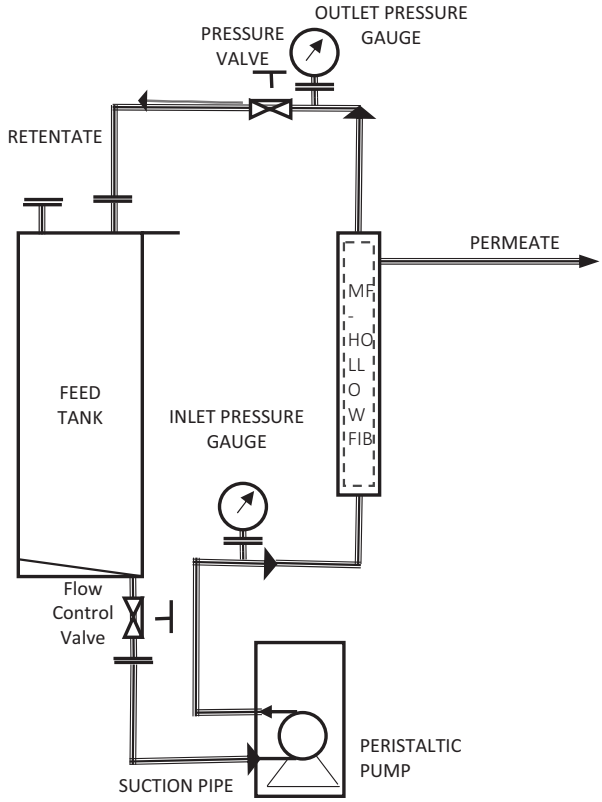

FIGURE 2 Experimental set up for microfiltration process

the feed can be controlled by the feed flow control valve. In the experiment, feed flow rate was constant. The pump passes the feed to the microfilter membrane. Inlet (P1) and outlet (P2) pressures of the membrane were visible through the attached pressure gauge. The transmembrane pressure (TMP = [P1 + P2]/2) was calculated on the basis of the average of both the pressure. After passing through membrane, filtrate was obtained on one side and, the other side, retentate was returned to the feed tank. To maintain the back pressure, pressure valve was used throughout the time of experiment. The amount of filtrate was collected in a measuring cylinder. Time to collect each 10 mL of juice was noted down, and the permeate flux (J) was calculated. Filtrate as well as retentate was collected for physicochemical analysis.

The operating parameter for MF was membrane pore size (0.1, 0.2, and 0.45 μm) and TMP (68.9, 103.42, and 137.89 kPa).The feed flow rate was constant (10 L/h) with the help of the pump speed and feed control valve. A total of nine experiments had been conducted. The permeate flux J (L/hm^2) was calculated according to Khemakhem, Gargouri, Dhouib, Ayadi, and Bouaziz (2017):

$$\text{Permeate flux } (J) = \frac{V_P}{t \times A} \quad (1)$$

where V_P (L) was permeate collected at a certain time period t (h) for the effective surface area A (m^2) of the membrane. The volumetric reduction ratio (VRR) was also calculated according to the following:

$$\text{VRR} = \frac{V_0}{V_R} \quad (2)$$

where V_0 and V_R are initial feed volume and retentate volume, respectively.

Cleaning of membrane and measurement of hydraulic permeability

Before starting the experiment, water flux was calculated for the individual membrane in fixed operating condition with feed flow rate (42 L/h) at 30°C. With the help of the plotted graph against water flux versus TMP, the hydraulic permeability was calculated through the slope obtained from the graph. The hydraulic permeability for the new membrane with water was referred as L_P^0, and after passing jamun juice, it is denoted as L_P^1.

After finishing each experiment, the membranes were cleaned in two steps. At first step, the membrane was rinsed with tap water for 30 min at maximum flow rate and minimum TMP (13.78 kPa) for washing out the reversible polarized layer and measured the hydraulic permeability as L_P^2. In the second step, first NaOH (0.1%) (wt/wt) (Sigma-Aldrich) solution at 50°C was passed through the module for 30 min at maximum flow rate and minimum TMP of 13.78 kPa, and permeability was measured as L_P^3. Then, again tap water was used for cleaning until neutral pH (pH 7) obtained. Next, HNO_3 (0.1%) (wt/wt) (Sigma-Aldrich) solution at 50°C was used for cleaning at same TMP and flow rate for 30 min. Finally the permeability was measured as L_P^4. Again, it was rinsed with tap water till the neutral pH obtained. The membrane fouling was expressed by the following equation:

$$\text{Fouling index} = \left[1 - \left(\frac{L_P^1}{L_P^0}\right)\right] \times 100 \tag{3}$$

Analysis of resistance

Permeate flux decline was analyzed by resistance in series model (Ennouri et al., 2015). Permeate flux can be written as the ratio of TMP difference and total resistance:

$$J_P = \frac{\text{TMP}}{(\mu \times R_t)} \tag{4}$$

where J_P is the permeate flux (L/m^2/hr), TMP is transmembrane pressure (kPa), μ is the dynamic viscosity of the solution (Pa s). R_t (1/m) is the total resistance composed of four resistance and can be calculated as

$$R_t = R_m + R_c + R_{frev} + R_{firr} \tag{5}$$

where R_m is noted as intrinsic membrane resistance of clean membrane and can be calculated as Equation 6, R_c is known as cake layer resistance due to the concentration polarization and deposition of solids. So it can be washed out at the time of cleaning with water. R_{frev} is the reversible adsorbed layer, which can be removed with chemical wash, and R_{firev} is the layer formed due to the adsorption of the material and cannot be washed after chemical wash:

$$R_m = \frac{1}{\mu_w L_P^0} \tag{6}$$

where μ_w is the viscosity of the water (Pa s) and $L_P^0 = J_w/\text{TMP}$ and J_w is the flux of distilled water

Again R_t can be calculated as by following equation:

$$R_t = \frac{1}{\mu_w L_P^1} \tag{7}$$

$L_P^1 = J_w^1/\text{TMP}$ and the permeability were calculated after jamun juice treatment.

Next, the present resistance after cleaning with water (L_P^2) can be written as

$$R_m + R_{frev} + R_{firrv} = \frac{1}{\mu_w L_P^2} \tag{8}$$

$L_P^2 = J_w^2/\text{TMP}$, and the permeability was calculated after washing with water and before starting the detergent cleaning. Similarly, after alkaline and acid wash, the hydraulic permeability can be written as

$$R_m + R_{firrv} = \frac{1}{\mu_w L_P^4} \tag{9}$$

$L_P^4 = J_w^4/\text{TMP}$, and the permeability was calculated after acid wash. With the help of the above equations, all the resistances were calculated after obtaining the experimental data.

2.4 | Physicochemical analysis of juice

Raw jamun juice, centrifuged jamun juice, filtrate, and retentate collected from MF unit were analyzed for different physicochemical analysis. pH, TSS, turbidity, total dissolve solid (TDS), color difference, chroma, hue, protein content, polyphenol content, clarity, and microbiological analysis had been done.

2.4.1 | Analysis for pH, TSS, TDS, and turbidity

Abbe-type digital Refractometer was used for TSS and expressed in degree Brix (°B). Turbidity was measured by Digital Turbidity-meter (Model 335; Deluxe Company, Delhi, India), and the unit of turbidity was nephelometric turbidity units (NTU) (Sin et al., 2006). The pH was measured with the help of a Elico pH meter. Total dissolve solid was measured by TDS meter (HM Digital, TDS-3) and expressed as parts per million (ppm).

2.4.2 | Color analysis

Color measurements were performed by colorimeter (ColorFlex EZ; Hunter Lab, Reston, Virginia, USA). Instrument was standardized during each sample measurement with a black and a white tile and the color values represented brightness/darkness (L^*), redness/greenness (a^*), and yellowness/blueness (b^*). Change in color (ΔE) with respect to raw juice, chroma, and hue value was also evaluated according to Fernández-Vázquez, Stinco, Meléndez-Martínez, Heredia, and Vicario (2011) to measure the qualitative attribute off color:

$$\Delta E = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2} \tag{10}$$

$$\text{Chroma} = \sqrt{(a^*)^2 + (b^*)^2} \tag{11}$$

$$\text{Hue} = \arctan\left(\frac{b^*}{a^*}\right) \tag{12}$$

TABLE 1 Analytical determination of centrifuged jamun juice with different speed and time

Parameter	Speed (rpm)	Time (min)		
		20	40	60
pH	6,000	3.52 ± 0.005^{ax}	3.53 ± 0.025^{bx}	3.57 ± 0.011^{cx}
	7,000	3.32 ± 0.01^{ay}	3.39 ± 0.01^{by}	3.5 ± 0.005^{cy}
	8,000	3.4 ± 0.01^{az}	3.4 ± 0.005^{bz}	3.5 ± 0.001^{cz}
TSS	6,000	18.17 ± 0.04^{ax}	17.86 ± 0.11^{bx}	18.27 ± 0.11^{cx}
	7,000	18.4 ± 0.01^{ay}	18.73 ± 0.11^{by}	19.07 ± 0.11^{cy}
	8,000	18.78 ± 0.02^{az}	19.16 ± 0.05^{bz}	19.73 ± 0.11^{cz}
Turbidity	6,000	81.2 ± 0.1^{ax}	58.44 ± 0.245^{bx}	34.25 ± 0.005^{cx}
	7,000	51.19 ± 0.081^{ay}	36.76 ± 0.057^{by}	14.43 ± 0.275^{cy}
	8,000	2.1 ± 0.001^{az}	1.0 ± 0.057^{bz}	0.20 ± 0.005^{cz}
ΔE	6,000	6.14 ± 0.01^{ax}	6.13 ± 0.011^{ax}	6.44 ± 0.04^{bx}
	7,000	6.12 ± 0.005^{ay}	6.17 ± 0.005^{ay}	6.46 ± 0.005^{by}
	8,000	6.14 ± 0.001^{ay}	6.15 ± 0.001^{ay}	6.34 ± 0.005^{by}
Chroma	6,000	0.97 ± 0.005^{ax}	0.82 ± 0.02^{bx}	0.21 ± 0.015^{cx}
	7,000	0.72 ± 0.005^{ay}	0.68 ± 0.005^{by}	0.14 ± 0.015^{cy}
	8,000	0.93 ± 0.005^{ay}	0.65 ± 0.001^{by}	0.42 ± 0.02^{cy}
Hue	6,000	0.27 ± 0.005^{ax}	0.35 ± 0.015^{bx}	0.23 ± 0.035^{cx}
	7,000	0.95 ± 0.01^{ay}	0.58 ± 0.01^{by}	0.24 ± 0.01^{cy}
	8,000	0.81 ± 0.01^{az}	$0.59 \pm .001^{bz}$	0.19 ± 0.005^{cz}
Protein	6,000	19.63 ± 0.057^{ax}	17.93 ± 0.036^{bx}	16.70 ± 0.02^{cx}
	7,000	17.88 ± 0.08^{ay}	16.74 ± 0.025^{by}	14.18 ± 0.08^{cy}
	8,000	11.88 ± 0.072^{az}	11.38 ± 0.08^{bz}	6.39 ± 0.09^{cz}
Polyphenol	6,000	14.95 ± 0.035^{ax}	14.28 ± 0.0032^{bx}	13.21 ± 0.104^{cx}
	7,000	12.37 ± 0.01^{ay}	12.5 ± 0.1^{by}	10.95 ± 0.01^{cy}
	8,000	9.55 ± 0.01^{az}	7.05 ± 0.05^{bz}	6.27 ± 0.02^{cz}
Clarity	6,000	30.3 ± 0.001^{ax}	63.53 ± 0.005^{bx}	73.80 ± 0.005^{cx}
	7,000	61.5 ± 0.057^{ay}	79.73 ± 0.02^{by}	81.20 ± 0.01^{cy}
	8,000	78.42 ± 0.032^{az}	$80.31 \pm .066^{bz}$	83.60 ± 0.005^{cz}

Values expressed are mean ± standard deviation. Means in the same column with different letters were significantly different at $p \leq .05$ (a, b, and c for time and x, y, and z for speed).

2.4.3 | Determination of protein and phenol content

Spectrophotometric method was used for both protein and polyphenol concentration. Protein concentration was determined with bovine serum albumin as standard, according to the dye binding method and phenol concentration was measured at 650 nm by Folin–Ciocalteu method with gallic acid standard as portrayed by Ghosh et al. (2017).

2.4.4 | Determination of clarity

Clarity of the juices was evaluated by transmittance (%T) at 660 nm utilizing the spectrophotometer (Model:AU 2701; Systronics India, Ltd., Ahmedabad, Gujarat, India) (Ghosh et al., 2017).

2.4.5 | Microbiological analysis

Bacteria and yeast and molds count were determined with slide modification of the method described by Cruz-Cansino et al. (2015). To enumerate the total aerobic microbial count, the juice samples were 10-fold serially diluted in peptone water (0.1%) and for the viable count of yeasts and molds, the juice samples were serially diluted in Millipore water. Then, those were plated out in duplicate on nutrient agar standard for total aerobic plate count as well as on potato dextrose agar for yeasts and molds. Plates were incubated at 30 °C for 48 hr and 25 °C for 72 hr for TPAC and yeast and mould count, respectively. Colonies were enumerated, and results were expressed as the logarithm of colony-forming units (cfu) per milliliter.

All the experiments were conducted in controlled environment. To prevent microbial contamination in pipes and pumps from microorganism, they were cleaned with H_2O_2 (30%) and kept it for 15 min at a temperature of 80 °C. Samples were collected in sterilized glass bottles without head space. The bottles were air tight with wax sealing. Before any microbiological analysis, the laminar flow chamber was cleaned with ethanol and UV lamp was switched on with door closed.

2.5 | Statistical analysis

All results were demonstrated as mean ± SD of three independent evaluations. Turkey's test and ANOVA were used for comparing the mean. Significance level was settled as $p < .05$ for all the differences.

SPSS Statistics 20 (SPSS, Inc., Chicago, IL, USA) was used for all the statistical analysis.

3 | RESULTS AND DISCUSSION

In India, no well-defined standard is present for clarified juice. The available jamun juice in the market has pulpy in nature where clarification has not been done. The final selection used to be done by the acceptance of the consumer with respect to its organoleptic property as well as physicochemical properties.

3.1 | Effect of centrifugation on the juice

The enzymatically treated juice was put in centrifuge at 6,000, 7,000, and 8,000 rpm for 20, 40, and 60 min. The physicochemical properties (pH, TSS, turbidity, color, protein, polyphenol, and clarity) are tabulated in Table 1.

The juice was acidic in nature. From the table, it can be observed that time and speed have a significant effect on pH value at $p < .05$, but overall the variation of pH value is very low ranges between 3.32 and 3.57. In case of commercial products, acidity regulator has been used and the pH of the market juice was 3.5, whereas the raw juice has a pH of 3.6. In case of TSS, the value ranges from 17.86 to 19.73°B. With an increasing speed of centrifuge, TSS contains increases in the juice significantly at $p < .05$, but with change in time, significant differences have not observed. The optimum TSS has been observed at 7,000 rpm, 40 min. Clarified juice should possess less turbidity. As the rpm of the centrifuge increases up to 8,000, reduction of turbidity observed (0.20 NTU) at significant level. The highest turbidity obtained in the case of centrifugation was 81.2 NTU at 6,000 rpm and 20 min as suspended particles still there in the juice. Colloidal and suspended solids are removed in high speed and time duration. Change in color or ΔE value has a certain range with its own significance. When the value is greater than 6, it signifies that the change in color is great (Wibowo et al., 2015). Where ΔE has a qualitative value in a three dimensional space; on the other side, chroma signifies the quantitative attribute characteristics. The minimum change in color observed at 7,000 rpm with 20 min time. With time, there is no significant effect ($p < .05$) on color change, but significance difference has been noticed in chroma. Hue is the qualitative representation of the chromatic nature of the color. The red color is denoted either by 0 or 360° (Robertson, 1978). The values are in 0°, which implies the red color of the juice. Both time and speed have a significant effect on hue value. The highest amount of protein (44.62 mg/g) and polyphenol (45.25 mg/g) contents obtained at the lowest speed and minimum time, i.e., 6,000 rpm and 20 min, respectively. As protein and polyphenols are macromolecules, with an increase in speed high molecular weight substances separated and the concentration decreases in the juice. For both the cases, significant difference has been observed at $p < .05$ with change in speed and time. The amount of protein percentage decreases up to 6.39 and polyphenol percentage was 6.27 at 8,000 rpm and 60 min. Similar type of data was observed by Sagu et al. (2014). Clarity of the juice is proportionally depends on time and centrifugal force significantly. As the speed and time increases, clarity of the juice reached from 30.3 to 83.60%T. As the macromolecular particles as well as suspended particles are removed at higher speed and time, the juice became clear. Maximum clarity 83%T has been observed at 8,000 rpm and 60 min. Same trend of clarity has been obtained by Sagu et al. (2014) and Sentandreu et al. (2011). In case of centrifugation, considering all the physicochemical and nutritional parameter, the optimized process speed and time were obtained as 7,000 rpm and 40 min, respectively.

3.2 | MF operating conditions

Temperature and flow rate of the jamun juice (30 °C and 10 L/h) was maintained throughout the experimental process. Figure 3 shows the variation of permeate flux at three different membrane pore size. With

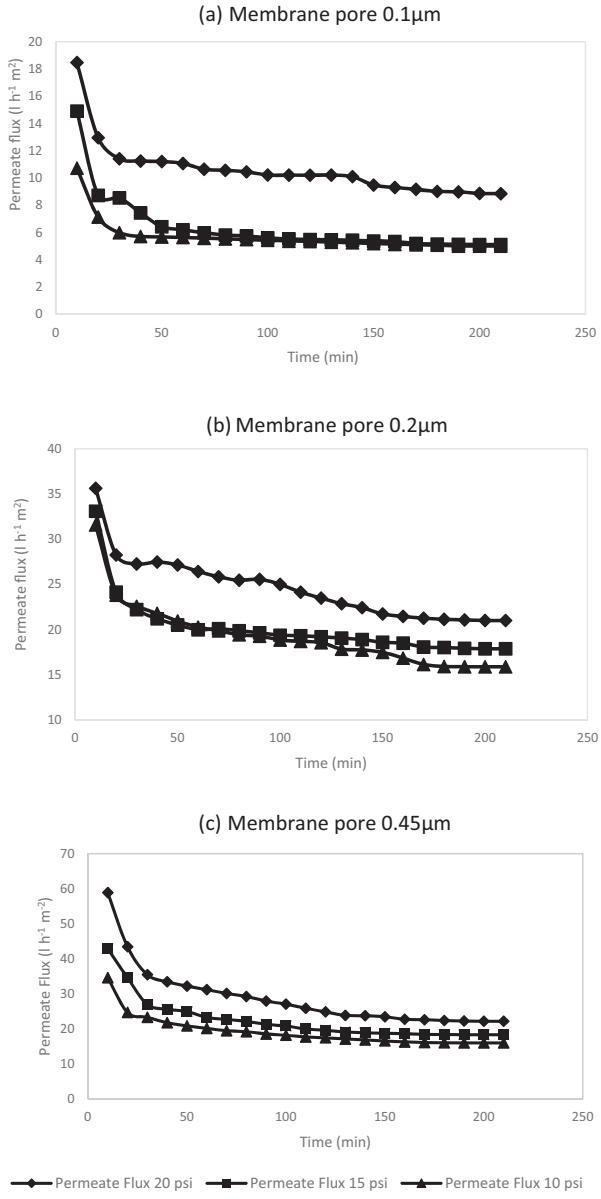

FIGURE 3 Variation of permeate flux versus time for jamun juice microfiltration at different trans membrane pressure for different membrane pore sizes (a) pore 0.1 μm, (b) pore 0.2 μm, and (c) pore 0.45 μm

TABLE 2 Analytical determination of microfiltered jamun Juice and retentate with change in pressure and membrane pore size

Parameter	Samples	Pressure (kPa)	Membrane pore diameter (μm)		
			0.1	0.2	0.45
pH	Filtrate	68.9	3.53 ± 0.005^{ax}	3.6 ± 0.001^{bx}	3.503 ± 0.005^{cx}
		103.3	3.54 ± 0.005^{ax}	3.61 ± 0.001^{by}	3.52 ± 0.001^{cx}
		137.8	3.61 ± 0.005^{ay}	3.61 ± 0.005^{by}	3.507 ± 0.005^{by}
	Retentate	68.9	3.71 ± 0.005^{ax}	3.61 ± 0.001^{bx}	3.51 ± 0.001^{cx}
		103.3	3.75 ± 0.005^{ay}	3.64 ± 0.001^{by}	3.51 ± 0.001^{cx}
		137.8	3.67 ± 0.006^{az}	3.64 ± 0.005^{by}	3.56 ± 0.01^{cy}
TSS	Filtrate	68.9	15.4 ± 0.001^{ax}	16.03 ± 0.057^{bx}	16.2 ± 0.001^{cx}
		103.3	15.13 ± 0.115^{ay}	16.17 ± 0.057^{by}	16.6 ± 0.001^{cy}
		137.8	15.47 ± 0.115^{ay}	16.8 ± 0.001^{bz}	17.2 ± 0.001^{cz}
	Retentate	68.9	16.06 ± 0.057^{ax}	17.33 ± 0.115^{bx}	15.4 ± 0.001^{cx}
		103.3	15.4 ± 0.001^{ay}	17.33 ± 0.115^{bx}	15.6 ± 0.001^{cy}
		137.8	17.40 ± 0.001^{az}	17.2 ± 0.115^{bx}	15.8 ± 0.001^{cz}
Turbidity	Filtrate	68.9	0.30 ± 0.057^{ax}	0.24 ± 0.39^{ax}	11.46 ± 0.001^{bx}
		103.3	0.51 ± 0.1^{ay}	0.38 ± 0.072^{ay}	10.50 ± 0.001^{by}
		137.8	0.12 ± 0.152^{az}	0.48 ± 0.075^{by}	8.56 ± 0.001^{cz}
	Retentate	68.9	78.33 ± 0.057^{ax}	89.56 ± 0.057^{bx}	91.01 ± 0.001^{cx}
		103.3	81.51 ± 0.1^{ay}	100.10 ± 0.11^{by}	103.46 ± 0.001^{cy}
		137.8	87.40 ± 0.251^{az}	135.43 ± 0.55^{bz}	138.61 ± 0.173^{cy}
ΔE	Filtrate	68.9	6.37 ± 0.005^{ax}	6.15 ± 0.01^{bx}	6.22 ± 0.02^{cx}
		103.3	6.35 ± 0.005^{ay}	6.45 ± 0.03^{bx}	6.23 ± 0.01^{cx}
		137.8	6.26 ± 0.01^{az}	6.17 ± 0.005^{by}	6.33 ± 0.015^{cy}
	Retentate	68.9	6.09 ± 0.01^{ax}	6.39 ± 0.035^{bx}	5.43 ± 0.041^{cx}
		103.3	6.40 ± 0.037^{ay}	6.16 ± 0.026^{bx}	5.10 ± 0.015^{cy}
		137.8	6.41 ± 0.01^{ay}	6.17 ± 0.01^{by}	5.06 ± 0.03^{cy}
Chroma	Filtrate	68.9	0.29 ± 0.005^{ax}	0.83 ± 0.005^{bx}	0.99 ± 0.005^{cx}
		103.3	0.42 ± 0.01^{ay}	0.20 ± 0.005^{by}	0.72 ± 0.006^{cy}
		137.8	0.65 ± 0.005^{az}	0.84 ± 0.006^{by}	0.47 ± 0.001^{cz}
	Retentate	68.9	2.79 ± 0.01^{ax}	3.34 ± 0.01^{bx}	5.70 ± 0.01^{cx}
		103.3	1.30 ± 0.015^{ay}	2.94 ± 0.015^{by}	4.49 ± 0.032^{cy}
		137.8	1.28 ± 0.015^{az}	1.88 ± 0.02^{bz}	3.65 ± 0.04^{cz}
Hue	Filtrate	68.9	0.14 ± 0.005^{ax}	0.39 ± 0.006^{bx}	0.57 ± 0.005^{cx}
		103.3	0.27 ± 0.005^{ay}	0.25 ± 0.006^{by}	0.47 ± 0.006^{cy}
		137.8	0.32 ± 0.01^{az}	0.40 ± 0.005^{bz}	0.29 ± 0.053^{cz}
	Retentate	68.9	0.13 ± 0.005^{ax}	0.25 ± 0.02^{bx}	0.27 ± 0.005^{bx}
		103.3	0.25 ± 0.01^{ay}	0.28 ± 0.005^{ax}	0.26 ± 0.005^{bx}
		137.8	0.33 ± 0.015^{az}	0.27 ± 0.01^{bx}	0.29 ± 0.005^{by}
Protein (%)	Filtrate	68.9	34.50 ± 0.047^{ax}	71.34 ± 0.055^{bx}	74.54 ± 0.025^{cx}
		103.3	47.63 ± 0.066^{ay}	76.16 ± 0.081^{by}	91.51 ± 0.047^{cy}
		137.8	50.22 ± 0.025^{az}	80.74 ± 0.025^{bz}	92.11 ± 0.072^{cz}
	Retentate	68.9	14.57 ± 0.015^{ax}	6.84 ± 0.02^{bx}	2.62 ± 0.015^{cx}
		103.3	28.45 ± 0.095^{ay}	8.01 ± 0.026^{by}	8.38 ± 0.015^{cy}
		137.8	18.78 ± 0.02^{az}	7.77 ± 0.02^{bz}	3.25 ± 0.02^{cz}
Polyphenol (%)	Filtrate	68.9	22.35 ± 0.032^{ax}	38.28 ± 0.112^{bx}	48.62 ± 0.061^{cx}
		103.3	29.24 ± 0.023^{ay}	43.5 ± 0.081^{by}	51.47 ± 0.115^{cy}
		137.8	31.34 ± 0.015^{az}	46.76 ± 0.108^{bz}	53.49 ± 0.032^{cz}
	Retentate	68.9	58.1 ± 0.015^{ax}	47.84 ± 0.045^{bx}	50.44 ± 0.035^{cx}
		103.3	60.45 ± 0.026^{ay}	49.54 ± 0.02^{by}	47.46 ± 0.111^{cy}
		137.8	60.21 ± 0.011^{az}	40.41 ± 0.04^{bz}	44.56 ± 0.01^{cz}
Clarity	Filtrate	68.9	65.2 ± 0.001^{ax}	85.36 ± 0.06^{bx}	87.53 ± 0.115^{cx}
		103.3	72.5 ± 0.001^{ay}	86.36 ± 0.057^{by}	88.3 ± 0.001^{cy}
		137.8	80.5 ± 0.36^{az}	85.3 ± 0.1^{bz}	93.5 ± 0.001^{cz}
	Retentate	68.9	37.86 ± 0.152^{ax}	25.36 ± 0.404^{bx}	0.20 ± 0.001^{cx}
		103.3	53.3 ± 0.17^{ay}	7.46 ± 0.057^{by}	0.50 ± 0.001^{cy}
		137.8	40.5 ± 0.1^{az}	14.70 ± 0.001^{bz}	0.20 ± 0.001^{cz}

Values expressed are mean ± standard deviation. Means in the same column with different letters were significantly different at $p \leq .05$ (a, b, and c for time and x, y, and z for speed).

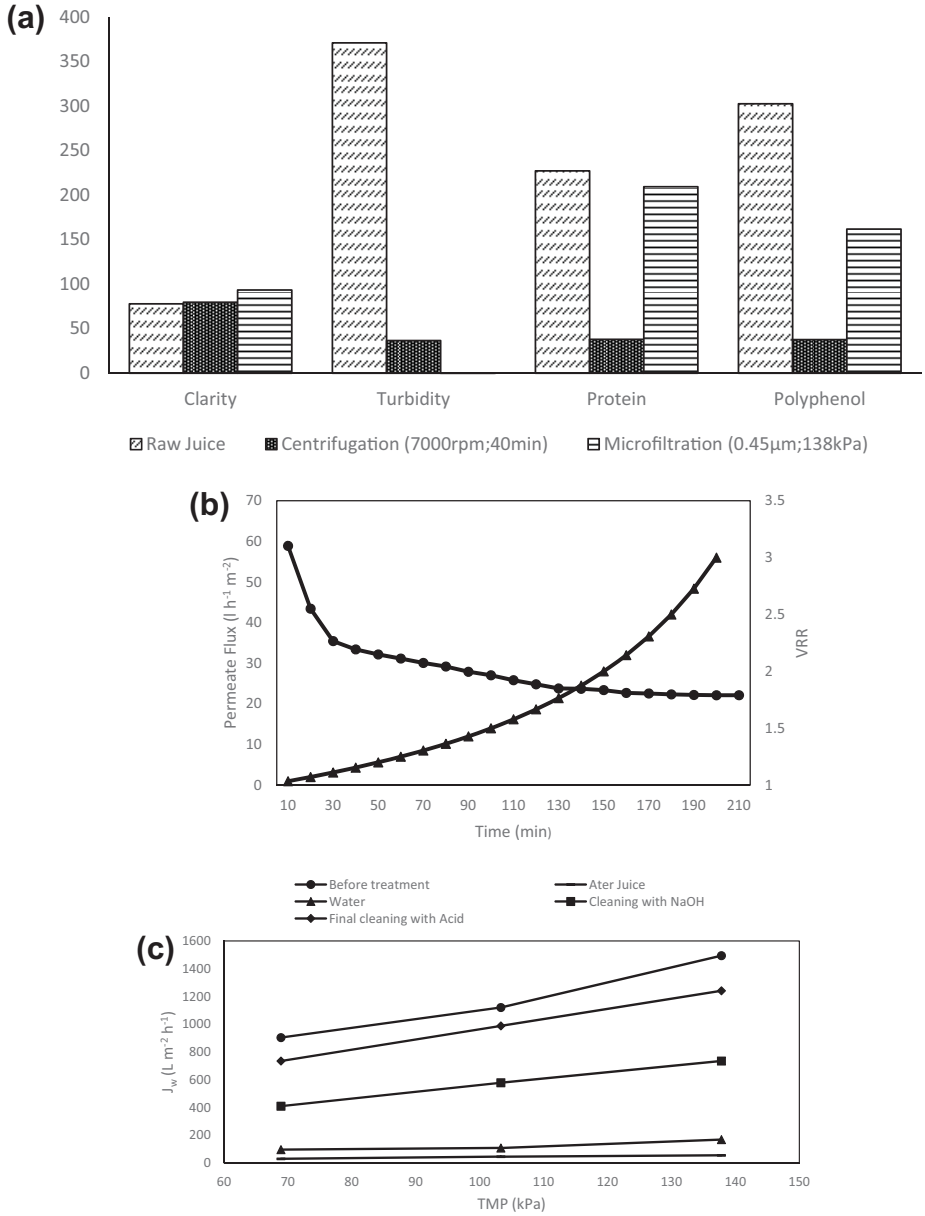

FIGURE 4 (a) Comparison of different parameters with raw juice and best treatment of centrifugation and microfiltration. (b) Microfiltration of jamun juice, Effect of time on VRR (black triangle) and permeate flux (black circle) during batch concentration mode (operating condition: membrane pore size: 0.45 μm; TMP: 20 psi). (c) Pure water flux before and after treatment through MF (0.45 μm)

the change in pressure permeate flux decreases and with an increase in the pore size, permeate flux increases from 10 to 35 L/hm^2 ($p < .05$). Sudden decreasing trend of the permeate flux observed in the first 20 min of the processing of the jamun juice. Then, the flux shows a decreasing trend up to 80 min. Finally, it reaches the steady state flow. In case of 0.1 μm pore size membrane, significant difference has not observed. The steady state permeate was same for both 68.9 and 103.42 kPa TMP, whereas for 0.45 μm pore size, membrane steady state flow has observed significantly in each TMP. This trend can be explained well by the polarization effect present in the cake layer formation. The rapid declination of the juice and decreasing trend up to a certain time indicate fouling due to cake layer formation in the membrane. Due to fibrous nature and high pectin amount in the jamun juice, deposition of the macromolecules in the membrane surface reduces its effect. The rate of deposition was less when the membrane pore size increases with an increase in permeate flux. Several reports have been reported supporting the cake layer formation (Campos et al., 2016; Sagu et al., 2014).

3.3 | Effect of MF on the juice (filtrate and retentate)

In case of MF, both filtrate and retentate were collected for analysis purpose. In Table 2, different physicochemical parameters have been measured for both the filtrate and the retentate of the juice. Primarily clarified jamun juice or filtrate was more acidic in nature than the retentate part. The pH of the filter varies within the range between

TABLE 3 Mass balance for centrifuged juice and micro filtered juice

	Treatment	Feed	Total permeate		Final retentate		Balance
Volume (mL)	Centrifugation	500	473.7	94.7%	26.3	5.3%	100%
	Microfiltration		378	75.6%	120	24%	99.6%
Protein (mg/g)	Centrifugation	227.34	38.05	16.74%	186.89	82.21%	98.95%
	Microfiltration		209.4	92.10%	7.77	3.41%	95.51%
Polyphenol (mg/g)	Centrifugation	302.69	37.8	12.5%	264.43	86.7%	99.2%
	Microfiltration		161.9	53.4%	134.87	44.56%	97.96%
Total dissolve solids (ppm)	Centrifugation	360	34.79	9.66%	325.20	90.33%	99.99%
	Microfiltration		1.63	0.45%	358.37	99.5%	99.95%

3.5 and 3.61, where the retentate ranges between 3.51 and 3.75. Significance difference ($p < .05$) has been observed in pH for changing the membrane pore size. Similar trend has been followed by Campos et al. (2016) for grape juice clarification.

For TSS content when the pore size is small, the amount of sugar retention was more in the retentate than the filtrate irrespective of pressure. For a particular pore size, membrane pressure has a positive effect on the TSS content. It can be concluded that pressure as well as membrane pore size has a significant effect on TSS. The maximum TSS obtained at maximum pressure (137.89 kPa) and highest membrane pore size (0.45 μm).

One of the major parameters for proper clarification is turbidity. The less the turbidity signifies the proper clarification. From the data in Table 2, it represents that 0.45 μm pore size has the less turbidity at maximum pressure than the other two. Reverse results had been observed in the retentate part with maximum turbidity. Membrane surface was acted as dynamic surface, which helps to reject particle according to its size. Similar trends had been noticed by Campos et al. (2016).

In case of color, there is no significant effect between the juice and the filtrate, but a certain change is noticeable between filtrate and the retentate ($p < .05$). The changes in the color value of the retentate have less observed than the filtrate. The quantitative attribute or the Chroma value responsible for the intensity of the color had a huge difference. Intensity of the retentate had more effect than the filtrate as the pigment get blocked in the membrane surface, which reduces the intensity of the filtrate and makes it lighter in color. Membrane pore size and pressure have a positive significant value on chroma.

Protein and polyphenols were considered as macromolecules. A total of 0.1 μm pore size of membrane acts as a barrier for macromolecules, whereas for 0.45 μm pore size, macromolecules got more easy access to mix with the filter. Maximum percentage of protein and polyphenols used to be mix in the filtrate at 0.45 μm membrane pore size with high pressure. With an increase in pressure, the amount of macromolecules forms a layer on the membrane surface, which results to cake layer formation in further process (Sagu et al., 2014).

Clarity of the filtrate depends on the pressure. Pressure has a significant positive effect on the clarity. Due to the cake filtration on the membrane, surface and self-rejecting phenomena with the increase in pressure clarity increases. Maximum clarity observed at TMP 137.89 kPa. Similar trend has been reported by Sagu et al. (2014). There is a significant effect within the pore size of 0.1 and 0.2 μm but has no significant effect observed with the last one.

So according to the operating condition and effects on the physicochemical property, MF with the membrane pore size of 0.45 μm with a pressure range of 137.89 kPa had been chosen for further treatment.

3.4 | Comparison and optimization between centrifugation and MF for operating condition

The primary aim of the work was to optimize a particular method for clarification of the jamun juice with a better organoleptic and nutritional property. Among all the treatments in centrifugation with a speed of 7,000 rpm and 40 min processing time gives the best result and in case of MF, pore size of 0.45 μm with a TMP of 137.89 kPa gives the best result. Now comparing these best treatments with the

TABLE 4 Hydraulic permeability of the membranes before and after cleaning process

	Hydraulic permeability (M/s/kPa)		
Process	0.1 μm	0.2 μm	0.45 μm
Before treatment with juice (L_p^0)	1.36×10^{-6}	1.5×10^{-6}	2.38×10^{-6}
After treatment with juice(L_p^1)	1.46×10^{-8}	3.54×10^{-8}	1.02×10^{-7}
After cleaning with water(L_p^2)	1.94×10^{-7}	9.71×10^{-8}	2.92×10^{-7}
After cleaning with NAOH and water (L_p^3)	1.17×10^{-6}	1.36×10^{-6}	1.31×10^{-6}
After cleaning with acid and water (L_p^4)	1.31×10^{-6}	1.45×10^{-6}	2.04×10^{-6}

TABLE 5 Determination of resistances during microfiltration of jamun juice (0.45 μm)

	R_t ($\times 10^{12}$) (1/m)	R_m ($\times 10^{12}$) (1/m)	R_c ($\times 10^{12}$) (1/m)	R_{frev} ($\times 10^{12}$) (1/m)	R_{firr} ($\times 10^{12}$) (1/m)	R_m/R_t (%)	R_c/R_t (%)	R_f/R_t (%)
MF-treated Juice (0.45 μm)	11	0.47	7.16	3.30	0.07	4.28	65.01	30.70

raw juice, Figure 4a shows the comparison between all the three. It is clearly observed that microfiltered juice has the best quality and nutritional property in case of clarity, turbidity, protein, and polyphenols.

In Table 3, mass balance for both centrifugation and MF for protein, polyphenol, and dissolve solid was tabulated. Initially, 500 mL of juice was used for both the case. For centrifugation, the recovery of supernatant (juice) was 94.7% with suspended solid of 5.3%, but in case of MF, the collected permeate was 75.6% with a retentate of 24%. Volume of filtrate in case of centrifuge was more, but the quantity of protein and polyphenol was 16.7 and 12.5%, respectively, but MF permeate has a huge retention of protein and polyphenol. The overall mass balance was justified for each properties of the juice.

Total bacterial count has also enumerated. The total plate count was 35×10^3 and 21×10^2 CFU/mL for raw juice and centrifuged juice, respectively. For microfiltered juice, there was no growth of bacteria. The yeast and mould count in raw juice was 7×10^5 CFU/mL. For centrifuged juice and microfiltered juice, the value of yeast and mould were 6×10^4 and 3 CFU/mL, respectively. The best operating condition for MF is given in Figure 4b. The results shows that with an increase in time, permeate flux decreases whereas VRR increases. The maximum VRR obtained was 3 at the highest flux of 61 L/hm^2. The VRR can be divided into three subparts. Initial part was the rapid decrease of permeate flux, the second phase was up to VRR 2 where smaller decrease in the permeate flux, and the last one was changes in permeate flux in negligible up to steady state condition. Same trend of data was observed by Cassano et al. (2006) for apple juice and kiwi fruit juice respectively.

3.5 | Analysis of resistance

Three different membranes had been used for primary clarification. Table 4 describes the hydraulic permeability for all the membrane before and after cleaning. Hydraulic permeability reduces up to 98.92% after juice treatment, but recovery of the membrane was 96% after CIP. So membrane chocking was mainly due to macromolecular and suspended particles present in the juice. Among three membranes, 0.45 μm membrane gives the best result. Figure 4c represents the permeate flux of juice and water flux of membrane (0.45 μm) before and after individual treatment, whereas Table 5 shows the resistance of the membrane. Resistance in series model has been used for the measurement of individual resistance. From the table, it can be observed that membrane resistance was 4.28%, but maximum resistance was due to cake layer formation (65.01%). Fouling resistance was 30.70% with irreversible fouling of 0.63%. These data indicate that fibers and tannins present in jamun juice cause the membrane fouling, which initially blocks the membrane, but it is not responsible for the chocking of membrane. Using detergent cleaning (CIP) was helpful for the recovery of the membrane.

4 | CONCLUSION

Clarification of jamun was carried out using both centrifugation and MF. Comparison of physicochemical, nutritional, and microbiological properties was evaluated for both the treatments. It was concluded that best results were obtained in terms of clarity (93.5%T), turbidity (8.56 NTU), protein (92.11%), and polyphenol content (53.49%) for microfiltered juice. Significant difference has been observed in case of microbiological properties. The optimum values for the operating condition of micro filtration were 137.89 kPa pressure with 0.45 μm pore size. Cake layer formation gives the maximum resistance for the membrane fouling. Good restoration of the membrane was achieved by cleaning the membrane with acid and alkali. Fouling was mainly due to the presence of fibers and macromolecules. Considering quality and quantity, MF is found to be the most appropriate method for the clarification of jamun juice.

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