Using Nanofiltration and Ultrafiltration for the Concentration of Amla (Phyllanthus Emblica) Juice

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Abstract

This study aims to evaluate the performances of the membrane processes during the treatment of Amla juice for concentration and purification. Amla juice was concentrated and purified by membrane processes such as Ultrafiltration and Nanofiltration. Amla juice concentration and purification was done through spiral wound ultrafiltration (UF) and nanofiltration (NF) (NMWCO 15Kda) membrane module. The ash contents, ascorbic acid, clarity, electrical conductivity (\(\mu\)S), \(pH\), total soluble solids \(^{\circ}\)Brix and TDS (mg/l), of the purified and concentrated juice were determined. Experiments were performed in selected operating conditions according to the batch concentration procedure up to a final volume reduction factor (VRF) of 2. Almost all total soluble solids and ascorbic acid of the original juice were recovered in permeate. The resulting juice after filtration showed a complete removal of suspended solids with a consequent improvement of colour and clarity. Therefore, the concentrated and clarified juice retained the food value of the original juice. Experimental work carried out for purification and concentration of Amla juice using membrane technology shows that ultrafiltration followed by NF resulted in retained of valuable component present in amla juice. It is observed that maximum recovery rate achieved up to 71.4\% in nanofiltration method.

Keywords: Amla juice; Juice Clarification; Juice Concentration; Nanofiltration; Spiral Bound Membranes; Total Antioxidant Activity; Ultrafiltration.
1. INTRODUCTION
Nowadays, the demand for the natural bio products, which do not contain chemicals, is rising. These demand areas in the case of medicines and medicinal products. Amla (Phyllanthus emblica) is that type of herbs. The amla (Family: Euphorbiaceae, Latin names: Phyllanthus emblica) fruit being highly astringent in taste. It is not popular as a desert fruit [8]. However, its high vitamin C, tannin and mineral contents and medicinal properties offer tremendous scope for processing into various juice based beverages such as syrups, squashes, nectar and ready-to-serve drinks [2]. The amla can be used for several diseases. Amla juice contains polyphenols so it helps in controlling high blood pressure as it calms the mind and body Amla juice has the power to fight cancers and improve immunity [2]. The production of concentrated fruit juices like amla is of interest at an industrial level. Amla juice can be used as ingredients in many products such as ice creams, fruit syrups, jellies and fruit juice's beverages. When the concentration is carried out by evaporation, most of the aroma compounds contained in the raw juice are lost and the aroma profile undergoes an irreversible change with a consequent remarkable qualitative decline. This paper deals with the investigations of the use of nanofiltration and ultrafiltration method for the concentration of the amla juice. This study discussed the suitability of nanofiltration (NF) and ultrafiltration (UF) methods to reserve the valuable components of the amla juice. It means that the heating and cooling were neglected and the concentration was carried out by membrane processes instead of evaporation. The results of the tests indicated that membrane separation method consumed low energy as compare with the traditional methods like evaporation [7]. Our paper will discuss in detail the process and analysis of concentrated juice related to acidity, Ph, soluble solid and ascorbic acid.

2. THEORY AND EXPERIMENTAL
Amla fruit (Family: Euphorbiaceae Latin names: Phyllanthus emblica) were purchased from a local market in south east India. Fruit were manually washed in water and then peeled by hand, with a knife. Seeds were removed with a squeezer and washed with water. Amla juice is obtained by squeezing or mixer. The suspended matter was removed from juice by using masline cloth, filter paper no. 40 for primary filtration and sodium azide was added to amla juice solution(02. % w/w), in order to prevent their fermentation during the experiments. Cross-flow ultrafiltration (UF) was used in batch mode for the clarification, to remove the suspended solids from the fresh amla juice.

The main goal of our work was to examine the concentration of the amla juice with different membrane separation methods. During our researches, the applicability of different membrane separation methods was examined. The concentration was examined with the removal of the suspended solids with different methods, such as conventional filtration method and without the removal of the suspended solids. The optimal operating parameters like pressure, temperature, recirculation flow rate were determined and the concentration measurements were carried out on them. The following analytical assay was carried out: determination of pH, carotene- and total
phenol content, determination of the element composition, colour and clarity. With the membrane separation, the evaporation can be replaced; the energy costs and the heat damage can be reduced.

For experimental purpose, Perma Reverse Osmosis system is use. System is a modular ultrafiltration system designed for separation of cells, bacteria, viruses, micro molecules, colour, fibres, crystals and starch. Nanofiltration system designed for separation of dissolved solids like polysaccharides, proteins, tannins, aroma compound, salts, pigments and sugar [5]. The membrane after each run was taken out of the system for cleaning. The membrane was first flushed with tap water to remove the cake layer attaching on the membrane surface. The membrane was soaked in NaOH 2% solution for 12 hours. The membrane was then washed with tap water and soaked in HNOH 1% solution for 4 hours. The membrane subsequently washed with tap water again and was measured membrane resistance before used in the next run. It can also clean with sodium metabisulphite. Membrane characteristics are given in Table 1.

**Table 1: Specification of Nanofiltration system**

| Type                          | Perma Reverse Osmosis System 3 Module HPA 250 |
|-------------------------------|-----------------------------------------------|
| Configuration                 | Spiral Bound                                  |
| Membrane Polymer              | Polyamide                                     |
| NMWCO                         | 15 Kda                                        |
| Membrane Surface Area         | 1.75 m²                                       |
| Average Pores Diameter        | 59 Å                                          |
| Ph Operating Range            | 2-11                                          |
| Temperature Operating Range   | 25 To 40°C                                    |
| Pressure Operating Range      | 300 Psi For NF                                |
|                              | 150 Psi For UF                                |
| Suspended Solid               | Less Than 5 ppm                               |
| Turbidity                     | Less Than 1 Ntu                               |
| Feed Flow Rate                | 960 Lit/Hr                                    |
| Permeate Flow Rate            | 60 X 180 Lit/ Hr                              |
| Concentration Flow Rate       | 780 To 9000 Lit/ Hr                           |

The performance of a NF system is quantified by permeate flux. There are four important operating parameters that affect the flux: pressure, temperature, feed concentration and flow rate [4]. The pressure-flux behaviour in typical NF processes is pressure dependent at low pressures and pressure independent at high pressures; increasing pressure increases permeate flux when the pressure is low, but permeate flux becomes independent of pressure when the pressure is high [6]. The resistance model describes correctly the observed pressure-flux behaviour of the NF processes. In the resistance model, the increase in pressure increases the flux and compact the boundary layer [5]. The compaction of the boundary layer increases its hydraulic resistance and thus opposes further increases in flux. On the other hand osmotic model does not consider the hydraulic resistance of the boundary layer [5].
3. RESULTS AND DISCUSSION

Batch concentration experiments showed that the permeate flux in the selected operating conditions decreased gradually with operating times by increasing the volume reduction factor (VRF) due to concentration polarization and fouling phenomena. The volume reduction factor (VRF) was calculated by

\[ VRF = \frac{V_o}{V_R(t)} = \frac{V_o}{V_o - V_p(t)} \] ……….. 01

Where \( V_0 \) is the initial feed volume (300 cm\(^3\)), \( V_R(t) \) and \( V_P(t) \) the retentate and permeate volumes at \( t \) time, respectively.

Rejection of the membrane was obtained by the following equation:

\[ R(\%) = \left( 1 - \frac{C_P}{C_R} \right) \times 100 \] ……….. 02

Where, \( C_P \) and \( C_R \) are permeate and retentate concentration respectively.

In the initial stage the rapid flux decline, more evident for the Polymer membranes, can be attributed to the adsorption, growth of a polarized layer formed by leftover pectin, protein and high molecular weight compounds present in the juice. However, internal fouling due to pore plugging at the early stage of the process can also occur. The slower decline towards a quasi-steady state can be attributed to fouling due to pore blocking and cake build-up. Permeate flux values observed with the NF membranes process were lower than that observed with the UF membranes process. Steady-state permeates fluxes were 34 L/m\(^2\)h and 39 L/m\(^2\)h, respectively. Tables 2 show the influence of the concentration and clarification treatment with UF and NF membranes process respectively on amla juice composition. The permeate of the UF juice treated with NF membranes process contains almost all the soluble solids and acids in terms of ascorbic acid of the initial feed. Concentration, color and clarity of juice were improved after filtration because of the removal of suspended colloidal
particles and higher molecular weight soluble solids of the juice. NF and UF membranes process showed a lower rejection towards TSS, total phenolics and TAA in comparison with the conventional process. Fig. 1 shows the changing of the solid content of the retentate in case of concentration with a nanofiltration. It is encouraging that at the end of the measurements the solid content of the retentate was four times lower than the starting concentration. The solid content of permeate could not be measured with a refractometer. The measurement had to be stopped after one hour because of the fouling of the membrane.

![Graph](image1.png)

**Fig. 1 Concentration with Nanofiltration**

![Graph](image2.png)

**Fig. 2 Concentration with Ultrafiltration**

The measurements were carried out at 500 L/h recirculation flow rate, on 30° C and on 30 bar pressure. The solid content of the juice increased two times higher, which is shown in Fig. 2. It is a promising start. The solid content of permeate could not be measured with the refractometer. The measurements were carried out at 150 L/h recirculation flow rate, on 30° C and on 30 bar pressure. In the case of reverse osmosis, there was no problem with the membrane fouling, but then the juice was a clear liquid, and it did not contain suspended solids. The clarification was made with ultrafiltration. The measurement will be continued with concentration of the fresh pressed juice with and without the removal of suspended solids using nanofiltration and ultrafiltration.

Table 2: Analytical measurements on samples coming from UF and NF of Amla juice

| Sr. No. | Parameter | Filtration (Feed to UF) | Ultrafiltration | Nanofiltration |
|---------|-----------|-------------------------|----------------|---------------|
|         |           | Permeate | Concentrate | Permeate | Concentrate | Permeate | Concentrate |
| 01      | Ash %     | 0.48     | 0.16 | 0.32 | 0.08 | 0.22 |
|         |           | 0.81     | 0.18 | 0.31 | 0.08 | 0.38 |
|         |           | 1.02     | 0.20 | 0.56 | 0.16 | 0.52 |
4. CONCLUSIONS

Amla juice was concentrated and clarified by using UF and NF process. The clarification of amla juice by nanofiltration and ultrafiltration method was perfect, but because of the total fouling of the membrane another method has to be found in the separation of the suspended solids.

Experimental work carried out for purification and concentration of Amla juice using membrane technology shows that ultrafiltration followed by NF resulted in retained of valuable component present in amla juice. It is observed that maximum recovery rate achieved up to 71.4% in nanofiltration method.

Color and clarity of the juice were improved after filtration because of the removal of suspended colloidal particles and higher molecular weight soluble solids of the juice.

Acidity, pH and soluble solids did not change during the processing and presented average values of 0.76_0.80% w/w Ascorbic acid, 3.35_3.40 and 10.6_118Brix, respectively. Therefore, the concentrated and clarified juice retained the food value of the original juice.

However, in the concentrated and clarified juice obtained with NF and UF a higher recovery of antioxidant compounds, especially in terms of total phenolics compounds, was observed in comparison with the conventional processes.

Finally it can be concluded that the application of Nanofiltration and ultrafiltration for purification and concentration may be a better alternative to conventional processes.
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