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# Clarification and storage study of bottle gourd (*Lagenaria siceraria*) juice by hollow fiber ultrafiltration

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ABSTRACT

Ultrafiltration of bottle gourd juice was undertaken in this study using hollow fibers with the objective to have juice with long shelf life without heat treatment, preservatives and additives maintaining the natural taste, flavor and nutritional quality intact. Five blend hollow fiber membranes (M-1 to M-5) with 15 wt% polyacrylonitrile (PAN) and 5 wt% cellulose acetate phthalate (CAP) were prepared. No additive was used in M-1 membrane. For M-2 and M-3 membranes polyethylene glycol (PEG) of molecular weight 200 Da was used as an additive with concentration 1.5 and 3 wt%, respectively. 3 wt% PEG of molecular weight 1500 Da was used in M-4 and same concentration of PEG of molecular weight 4000 Da was used in M-5 membrane. The membranes were characterized in terms of permeability, molecular weight cut off, permeate flux, permeate quality and anti-fouling characteristics. M-3 membrane was selected based on performance. Experiments were conducted both in total recycle and batch concentration mode. In case of total recycle mode, transmembrane pressure drop (TMP) was varied from 35 to 104 kPa and that of cross flow rate (CFR) was from 10 to 20 l/h. Quality of the filtered juice was analyzed in terms of color, clarity, total soluble solid, polyphenol, concentration of protein, sodium and potassium. TMP 104 kPa and CFR 20 l/h were found to be suitable operating conditions for filtration. The filtered juice was stored for 8 weeks under normal refrigeration. All the nutritional parameters (total protein, polyphenol, sodium, potassium and total soluble solids) including taste were monitored and it was found that the quality of the juice was maintained with a positive purchase intention.



## 1. Introduction

Vegetables and fruits contain a wide variety of essential nutrients and they provide a natural protection to human health (Kubde et al., 2010; Shah et al., 2010). Bottle gourd (*Lagenaria siceraria)* is one such widely available vegetable that has many nutritious and medicinally important components. Bottle gourd contains significant amount of proteins, various vitamins, minerals, carbohydrate, fiber, flavonoids, other antioxidants, vitamin C, sodium, and potassium (Shah et al., 2010). It is cardioprotective, cardiotonic (Fard et al., 2008), diuretic (Duke, 1992), and anticancerous (Sharma et al., 2006). Thus, clarified bottle gourd juice with high shelf life is a desirable product and the juice form is easy to assimilate having high nutritional value.

High shelf life of the juice can be achieved by removing suspended materials, cell debris and high molecular weight protein (Sagu et al., 2014). These haze forming substances are potential source of microbial growth (Fang et al., 2007), thereby requiring unit operations, like, clarification. Typical clarification steps of any juice processing system involves centrifugation, use of fining agents like gelatin and bentonite, filtration by diatomaceous earth, polished filtration, and finally sterilization for removal of microorganisms (Amerine et al., 1976; Fang et al., 2007; Yang et al., 2015). Adsorption is the major mechanism for

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these traditional methods but they are inherently slow and time consuming. Moreover, these methods need addition of chemicals, thereby, risking contamination. Additionally, the vitamins, aroma and other nutritious substances like polyphenols, etc., are degenerated during sterilization. Membrane based processes can be a promising alternative in this regard. The major advantages of these processes are, (i) they are operated at room temperature; (ii) no external chemicals are added; (iii) low energy intensive; (iv) modular in nature and hence easily scalable; (v) they are rate governed processes and hence can be operated at high rate of productivity. Various membrane based operations, like, microfiltration (MF) and ultrafiltration (UF) are extensively used for clarification of fruit juice. MF of average pore diameter 0.2 micron is sufficient for removal of microorganism (Porter, 2005). However, there are reports where the microorganism is not completely removed by 0.2 micron filter (Li et al., 2003). Therefore, one can select a suitable primary clarification step followed by an appropriate UF to get a finally clarified juice devoid of suspended materials, high molecular weight solutes and other haze forming substances. If the resultant juice is packaged aseptically, then one can obtain a clarified juice which is free of microorganism, having high shelf life, without any additives and preservatives maintaining the entire natural flavor, aroma and inherent nutritional substances. Thus, the membrane based processes are known as "cold sterilization" and they have become an integral part of any modern juice treatment plant. In this regard, selection of the appropriate molecular weight cut off (MWCO) of the membrane is essential. Application of MF and UF for treatment of fruit and vegetable juice is numerous. Rai et al. (2006a) and Chhaya et al. (2013) used MF as a pretreatment step for clarification of depectinized mosambi juice and treatment of Stevia extract for recovery of Steviol glycosides, respectively. Cross flow microfiltration was used to clarify pineapple (Carneiro et al., 2002), melon (Vaillant et al., 2005), watermelon (Chhaya et al., 2008), passion fruit juice (Oliveira et al., 2012), apple (Girard and Fukumoto, 1999; Riedl et al., 1998; Youn et al., 2004), pomegranate (Onsekizoglu, 2013; Mirsaeedghazi et al., 2010), bergamot (Conidi et al., 2011), chicory (Luo et al., 2013; Zhu et al., 2013), acerola (Matta et al., 2004), chicory juice and pineapple juice (Laorko et al., 2011), cactus pear juice (Cassano et al., 2010; Mondal et al., 2014). Similarly, UF has been a final polished filtration step for many fruit juice. Both Rai et al. (2006b) and Chhaya et al. (2012) used 30 kDa UF for treatment of mosambi juice and Stevia extract, respectively. UF has been used for clarification of shamouti orange juice (Merin and Shomer, 1999); pomegranate (Baklouti et al., 2012); West Indian cherry and pineapple (Barros et al., 2004); orange and lemon (Capannelli et al., 1992); tangerine juice (Chamchong and Noomhorm, 1991); passion juice (Jiraratananon and Chanachai, 1996); grape (Bailey et al., 2000); sugar beet (Loginov et al., 2011); sugar cane (Saha et al., 2007); apple (Vladisavljević et al., 2003; Bruijn et al., 2003) kiwi fruit (Tasselli et al., 2007); pineapple (Barros et al., 2003); Clementine mandarine (Cassano et al., 2009).

The common polymers used to prepare the UF membranes are polysulfone, polyethersulfone, polyacrylonitrile, etc. These polymers are inherently hydrophobic in nature resulting to severe membrane fouling during processing of juice by protein, pectin and high molecular weight solutes. Blending with hydrophilic polymers, like, cellulose acetate and its derivatives is one of the less expensive and easier methods imparting hydrophilicity to the prepared membrane making it less prone to fouling. Thus, development of blend hollow fiber UF membranes having antifouling properties is an area of active research for juice clarification. Cellulose acetate phthalate (CAP), being a hydrophilic polymer compared to polyacrylonitrile (PAN) is, therefore, used to spin a CAP-PAN blend membrane for this typical application. Introduction of polyethylene glycol (PEG) as an additive enhances the hydrophilicity further.

In the current work, a detailed UF based clarification process for treatment of bottle gourd juice is presented. A suitable UF membrane was identified and the effects of the operating conditions were investigated on the clarification performance in terms of permeate flux and permeate quality as well as understanding of membrane fouling mechanism. Both total recycle and batch concentration modes were investigated. The ultrafiltered juice was stored for two months to check the shelf life and the analysis of the storage study was presented. To the knowledge of the authors, this kind of study is undertaken for the first time with bottle gourd juice.

**Table 1 – Specification of hollow fiber spinning unit.**

| | |
|---|---|
| Inner diameter of smaller needle, m | 0.0005 |
| Outer diameter of smaller needle, m | 0.012 |
| Air gap between extrusion point and gelation bath, m | 0.25 |
| Casting temperature, K | 300 |
| Pressure in polymer-melt tank, kPa | 35 |
| Water flow rate, $m^3/s$ | $3.3 \times 10^{-7}$ |
| Flow rate of polymer solution, kg/s | $5 \times 10^{-5}$ |
| Inner diameter of hollow fiber, m | 0.0008 |
| Outer diameter of hollow fiber, m | 0.0011 |
| Inner diameter of cartridge, m | 0.0118 |
| Length of cartridge, m | 0.16 |
| Number of hollow fibers | 70 |
| Total membrane area, $m^2$ | 0.028 |

## 2. Methodology

### 2.1. *Materials*

Bottle gourd was purchased from the local market. Folin–Ciocalteu's reagent (purity ≥ 99%), anhydrous sodium carbonate (purity ≥ 99%), copper (II) sulphate pentahydrate (purity 99%) and sodium hydroxide (purity ≥ 97%) were purchased from Merck Specialities Private Limited, Mumbai, India. Potassium sodium tartrate (purity 99%) and Gallic acid (purity ≥ 99%) were obtained from Loba Chemie, Mumbai, India. Bovine serum albumin (BSA) (purity ≥ 98%) was supplied by SRL Pvt. Ltd., Mumbai, India. CAP was purchased from M/s, G.M. Chemie Pvt. Ltd, Mumbai, India. PAN (homopolymer average molecular weight of 150 kDa) was procured from M/s, Technorbital Advanced Materials Pvt. Ltd., Kanpur, India. Solvent, N, N-dimethylformamide (DMF) (purity ≥ 99.8%) was purchased from M/s, Merck (India) Ltd., Mumbai, India. Polyethylene glycol of (PEG) average molecular weight 0.2, 1.5, 4, 10, 20, 35 and 100 kDa was, supplied by M/s, S. R. Ltd., Mumbai, India. Dextran of molecular weight 70 kDa was supplied by Sigma Chemicals, USA.

### 2.2. *Hollow fiber (HF) membrane and module preparation*

CAP-PAN blend hollow fiber membranes were prepared by dry-wet method. The hollow fibers were spun using two coaxial needle assembly (Thakur and De, 2012). Spinning conditions and various important parameters of hollow fibers are presented in Table 1. Solid CAP and PAN were heated to 60 °C for 2 h to evaporate the moisture completely. Similar procedure was carried out by Jung et al. (2004) in their work. Fixed amount of PEG of different molecular weight was added in premixed 15 wt% PAN and 5 wt% CAP in DMF solution and dissolved at 60 °C. The solution was prepared under constant mechanical stirring (stirring speed: 700 rpm) for at least 6 h at 60 °C to mix completely. The molecular weight of PEG was varied as 200, 1500 and 4000 Da at 3 wt% in order to get membranes of different MWCO. Concentration of PEG of molecular weight 200 Da was varied as 1.5 and 3 wt% to observe the effect of concentration. Thus, 5 membranes were prepared and the detailed composition of different membranes is presented in Table 2.

After spinning, hollow fibers were kept in distilled water for 24 h to allow the phase separation to be completed. To ensure completion of phase inversion, samples from gelation bath