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Effective clarification of pomegranate juice: A comparative study of pretreatment methods and their influence on ultrafiltration flux

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ABSTRACT

The aim of this study was to introduce an effective pre-clarification step in order to improve performance of subsequent ultrafiltration (UF) and to obtain a high quality pomegranate juice (PJ) with improved clarity. The effects of various pre-clarification treatments utilizing gelatin, bentonite and polyvinyl polypyrrolidone (PVPP) on UF performance were evaluated comparatively through analysis of flux behavior and membrane fouling. Quality attributes of the PJs (pH, total acidity, total phenolic content, total monomeric anthocyanins, individual phenolic acids, organic acids, total antioxidant activity and color characteristics) following various pre-clarification treatments were also investigated. On the whole, pre-clarification treatments that included PVPP exhibited a higher overall adsorption capacity, especially of low molecular weight phenolics. The best results with regard to both the fouling behavior of the UF membrane and the juice clarity were achieved by sequential application of PVPP and bentonite. Since lesser amounts of fining agents were used in the pre-clarification treatments, quality attributes of PJ were well preserved comparing conventional clarification applications.

1. Introduction

Over the past few years, there is a worldwide increasing tendency for the consumption of pomegranate juice (PJ) due to the substantial increase in the number of scientific papers concerning potential health benefits of pomegranate. Significant anti-atherosclerotic (Aviram et al., 2008), antimicrobial (Reddy et al., 2007; Su et al., 2010), anti-hypersensitive (Basu and Penugonda, 2009) and anti-inflammatory (Viladomiu et al., 2013) effects of PJ have been proposed by various researchers. These beneficial activities of PJ have been attributed to its remarkably high antioxidant capacity, which are correlated with their polyphenol content. Gil et al. (2000) have shown that PJ's antioxidant capacity is three times higher than that of red wine and green tea. Potential use of PJ as a natural antioxidant in food products has been proposed by various researchers.

Since high molecular weight tannins present in the husk are extracted into juice during pressing, PJ contains higher amount of phenolic compounds than edible parts of the fruit. The soluble polyphenol content in PJ varies within the range of 0.2–1.0%, depending on variety, including mainly hydrolyzable tannins, ellagic acid derivatives and anthocyanins (Turfan et al., 2012). The phenolic constituents of pomegranates play a large role in the acquisition of sensory properties (color, bitterness, etc.) of the juice. On the other hand, the presence of excessive amount of these compounds can also have undesirable effects for the fruit juice, for instance, formation of astringent flavor which makes it undesirable to consumers (Turkyilmaz et al., 2013). The phenolic compounds also contribute to haze and sediment formation along with color loss and browning during processing and storage through formation of polymeric complexes between polysaccharides, sugars, metal ions, and proteins. The eventual development of post bottling haze and sediment during storage is one of the greatest hindrances to the marketability of pomegranate juice, affecting the overall acceptability adversely. Therefore, a clarification step is necessary to prevent formation of sediments and turbid appearance at the bottom of the juice during storage (Onsekizoglu, 2013). The bitter taste due to high tannin content can also be avoided and the taste of the product can be improved by means of clarification. Conventional clarification typically involves addition of fining agents where gelatin and bentonite are the most common ones among them. Gelatin is positively charged in the low pH range of fruit juices and reacts with negatively charged phenolics such as tannin species. The main effect of bentonite on clarification depends on its adsorption capacity, mainly proteins. Polyvinyl polypyrrolidone (PVPP) which is cross-linked polyvinylpyrrolidone, is another well-known adsorbent, which exhibits high selectivity for phenolic

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compounds. The regenerative use of PVPP due to its insolubility in water is the most important reason for its acceptance in the beverage processing industry. The typical dosages in common practice is in the range of 1–2 g/L for gelatin (Fischer et al., 2011; Turfan et al., 2012; Vardin and Fenercioglu, 2003), 0.3–0.75 g/L for bentonite (Alper et al., 2005; Fischer et al., 2011; Vardin and Fenercioglu, 2003) and 0.2–2.5 g/L for PVPP (Alper et al., 2005; Vardin and Fenercioglu, 2003).

Ultrafiltration (UF) has been replacing conventional fining for clarifying fruit juices with the advantages of elimination of fining agents together with their associated problems. However, studies suggest that ultrafiltered juices are more susceptible to post bottling haze than traditionally clarified juices, as potential haze precursors may not be eliminated to the same extent. Moreover, the rapid reduction of permeate flux interfere the commercial utilization of UF in juice processing (Domingues et al., 2014; Gokmen and Cetinkaya, 2007; Onsekizoglu, 2013). Up to now, an efficient UF process for production of high quality pomegranate juice with improved clarity has not yet been accomplished. Therefore, the aim of this study was to evaluate the feasibility of implementation of a pre-clarification step in order to improve performance of subsequent UF and to obtain a stable product with improved clarity. The effects of pre-clarification with fining agents such as gelatin, bentonite and PVPP on UF performance were evaluated through an analysis of flux behavior. The comparative analysis of membrane, fouling and cake layer resistances, their contribution to the total resistance, was also performed through the evaluation of the hydraulic permeability of the membrane measured before and after the treatment with PJ and cleaning procedures. Furthermore, variations in quality attributes of the PJs (pH, total acidity, total phenolic content (TPC), total monomeric anthocyanins (TMAs), individual phenolic acids, organic acids, total antioxidant activity (TAA) and color characteristics) following various pre-clarification treatments were also investigated.

2. Materials and methods

2.1. Materials

The sweet pomegranates were supplied from a local market in Edirne, Turkey. Gelatin (80 Bloom), bentonite, PVPP, Folin–Ciocalteu phenol reagent, gallic acid monohydrate, phenolic standards (gallic, protocatechuic, chlorogenic, o and p-coumaric, ferulic acid, catechin, epicatechin and phloridzin), organic acid standards (citric acid, galacturonic acid, quinic acid, malic acid, methylmalonic acid, fumaric acid and succinic acid), sucrose, glucose, fructose, 2,2′-azinobis (3-ethylbenzothiazoline-6-sulfonic acid) (ABTS) were purchased from Sigma (St. Louis, MO, USA). All the other reagents and solvents used were obtained from Merck (Darmstadt, Germany) and were of HPLC or analytical grade. Oasis HLB (1 mL, 30 mg) solid phase extraction cartridges were supplied by Waters (Milford, MA, USA).

2.2. Production of clarified pomegranate juice

2.2.1. Juice extraction

Pomegranates were washed with cold tap water and drained to reduce surface dirt and microbial flora. Then fruits were cut from the middle into two pieces, pressed with a laboratory type press with a yield of 40–45%. The extracted raw juice was pre-filtered for separation of sedimented coarse particles and stored at −18 °C before use. Each set of pre-clarification treatments was done with the same batch of extracted juice in order to minimize the seasonal effects.

2.2.2. Pre-clarification of raw pomegranate juice with fining agents

The raw pomegranate juice (RJ) samples were divided into five lots. One lot of RJ was directly ultrafiltered and considered as control sample (URJ). Other samples were pre-clarified with different combinations of fining agents as shown in Fig. 1. As pomegranate juice contains only trace amounts of pectin (Magerramov et al., 2007), no depectinization was applied. The pre-clarification conditions were determined by preliminary experiments taking into account the turbidity of the resulting clarified juices. The details of these treatments are given below.

Process I. The raw juice was treated with 0.1 g/L of gelatin at 5 °C for 1 h (cold clarification). Aqueous solution of 1% (w/v) gelatin was used for pretreatment. The flocs were removed by filtering through double layer of cheesecloth.

Process II. The RJ was sequentially pre-flocculated with 0.1 g/L of gelatin and 0.5 g/L of bentonite at 50 °C for 1 h. Aqueous solutions of 1% (w/v) gelatin and 5% (w/v) bentonite were used for pretreatment. The flocs were removed by filtering through double layer of cheesecloth.

Process III. The RJ was treated with 0.4 g/L of PVPP at 50 °C for 1 h. The PVPP was removed by coarse filtration.

Process IV. The RJ was sequentially treated with 0.4 g/L of PVPP and 0.5 g/L of bentonite at 50 °C for 1 h for each treatment. The pre-clarified juice was obtained following filtration through double layer of cheesecloth.

2.2.3. Ultrafiltration system

UF experiments were performed in a pilot cross-flow filtration unit equipped with a Sepa® CF II Membrane Cell System (GE Osmonics, Minnetonka, MN, USA). A schematic diagram of the UF system is shown in Fig. 2. The juice was ultrafiltered through a 30 kDa cut-off PVDF membrane having an effective membrane area of 0.0155 m^2 (JW, GE Osmonics). UF was carried out according to the batch concentration mode in which permeate was continuously collected and the retentate stream were recirculated back to the feed tank up to a volume reduction factor (VRF) of about 5. The temperature of feed was kept constant at 25 ± 1 °C using a heat exchanger. The UF system was operated at a transmembrane pressure (TMP) of 3 bar, at a feed flow rate of 700 L/h. A digital

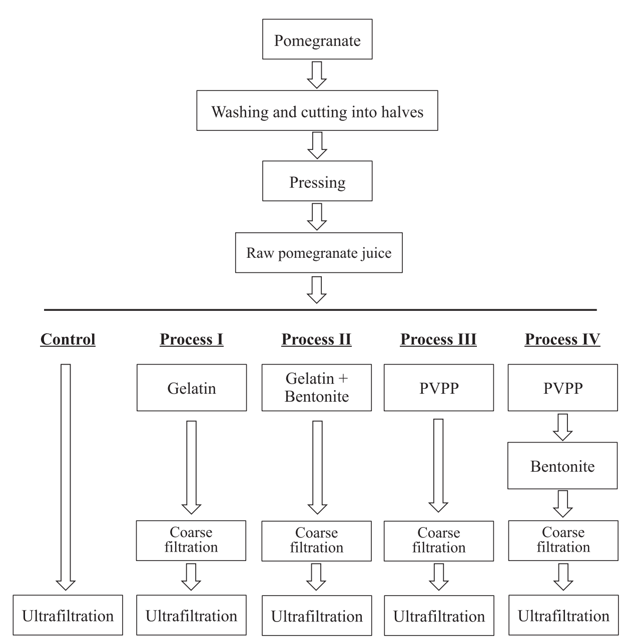

Fig. 1. Laboratory scale clarified pomegranate juice processing scheme following different pre-clarification techniques.