Contents lists available at ScienceDirect

Food and Bioproducts Processing

journal homepage: www.elsevier.com/locate/fbp

Development of a sequenced enzymatically pre-treatment and filter pre-coating process to clarify date syrup

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ARTICLE INFO

Article history:
Received 30 March 2015
Received in revised form 20 November 2016
Accepted 23 November 2016
Available online 30 November 2016

Keywords:
Date syrup
Pectinolytic enzyme
Filter aid
Clarification
Filtration
Response surface methodology

ABSTRACT

An enzymatically pre-treatment and a filter pre-coating processes were used to decrease turbidity and darkness intensity of date syrup. Response surface methodology was applied to study effects of enzymatically pre-treatment parameters, namely, pectinolytic enzyme (0.005–0.015 mg/100 ml syrup) and gelatin (0.005–0.015 mg/100 ml syrup) concentrations, and filter pre-coating concentrations including, cellulose (5–10 g/100 ml water), diatomite (5–10 g/100 ml water) and crud perlite (5–10 g/100 ml water) on dependent variables. Color, turbidity and polyphenol content of clarified date syrup were considered as response variables. Second order regression models, with high coefficient of determination values ($R^2 > 0.874$) were significantly ($p < 0.05$) fitted for predicting the response variables of clarified date syrup. Optimum concentrations of enzyme and gelatin were predicted to be 0.011 and 0.009 g/100 ml syrup, respectively. Optimum concentrations of cellulose, diatomite and crude perlite were obtained at 5.76, 8.23 and 5.91 g/100 ml water, respectively. Color, turbidity, and polyphenol content of clarified syrup at optimum conditions were obtained at 4936 ICUMSA, 38 NTU and 325 mg/l, respectively. Results obtained using FTIR spectroscopy, indicated that melanoidins, polyphenols and pectin were the main compounds caused turbidity and darkness of date syrup. The concentrations of off-color compounds were drastically decreased during filtration at obtained optimum conditions.

1. Introduction

Date palm (*Phoenix dactylifera*) is one of the oldest agriculture crops and grows largely in dry and semi dry regions like North Africa and middle-east countries (Ahmed et al., 1995; Besbes et al., 2009). Date is a good source of carbohydrates, pectin, salts, minerals (e.g. boron, calcium, cobalt, copper, fluorine, iron, magnesium, manganese, potassium, phosphorous, sodium and zinc), dietary fibers, vitamins (e.g. vitamin C, and vitamins B_1 thiamine, B_2 riboflavin, nicotinic acid (niacin) and vitamin A.), fatty acids (e.g. palmitoleic, oleic, linoleic and linolenic acids), and proteins including 23 types of amino acid (Gabsi et al., 2013; Al Juhaimi et al., 2014).

Over harvest and storage, dates lose up to 60% of their quality due to such high nutritional value and weak texture (Al-Farsi, 2003; Mrabet et al., 2008). The presence of high sugar content in low quality date fruits is made them an important natural sugar source industrially to produce many products such as, jam, jelly, vinegar, chutney, and candy. Date syrup can be considered as one of the most innovative and attractive by-products in date processing. It may also be substituted for honey or other similar sweeteners in confectionary and drinks (Al-Farsi, 2003; Alanazi, 2010).

Juice extraction, clarification, and concentration are the unit operations which industrially are applied to produce date syrup (Al-Farsi, 2003; El-Nagga and Abd El-Tawab, 2012). Raw date juice contains carbohydrates, proteins, lipids, pectin, salts and minerals. This product has also high turbidity and dark-brown color which in turn, make it unattractive for consumers. Furthermore, during other unit operations used to produce date syrup like concentration, the turbidity and darkness intensity of the product increase due to decrease its water content and increase its soluble compounds (Alanazi, 2010; Gabsi et al., 2013). Therefore, an important step in production of date syrup is clarification and discoloration of date juice (Nasehi et al., 2012; Ahdno and

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http://dx.doi.org/10.1016/j.fbp.2016.11.008

Jafarizadeh-Malmiri, 2015). The most common colored components in juices are melanins, melanoidins, phenolic compounds, pectin, and caramels that cause a darkening of the date syrup. Melanoidins are the dark pigments which are formed during the Maillard reaction, a non-enzymatic browning reaction (Simaratanamongkol and Thiravetyan, 2010). Caramel is produced during heat treatment of sugar at high temperature, usually at temperature above 80 °C (Matmaroh et al., 2006; Jamshidi Mokhber et al., 2008). Phenolic substances are other main colored compounds which naturally present in date fruits. These compounds contain at least one aromatic ring with one or more attached hydroxyl groups in their chemical structure (Simaratanamongkol and Thiravetyan, 2010). Pectin is one of the main components of date fruit which is caused lack of clarity in date juice due to its high molecular weight and low solubility in water (Dharmadhikari, 1994). Depectinisation is essential for most of the juice clarification process. To remove off-color compounds, in particular polyphenols, activated carbon is used for its high adsorptive capacity, and sufficient pore size distribution (Arslanoğlu et al., 2005; Karnib et al., 2014; Ozdemir et al., 2014). The proper concentration of activated carbon depends on the type and amount of off color compounds and the type of activated carbon, for example, powder or granule (Nasehi et al., 2012). During filtration process, different types of filter aid are used to remove very fine particles and to avoid blockage of the filter (Coenen et al., 2007; Zandleven et al., 2007).

Several studies have been done based on different types of filter aid such as cellulose (fibrous light weight and ashless paper), diatomite (siliceous skeletal remains of aquatic unicellular plants), bentonite, and perlite (glassy crushed and heat-expanded volcanic rock) in filtration process (Boittelle et al., 2008; Ediz et al., 2010; Lozano-Sánchez et al., 2011; Heerd et al., 2012; Lassoued et al., 2014). Pectinolytic enzymes, such as, pectinase can degrade the pectin which leads to viscosity reduction and promote the clarity. Pectinase eliminates the possible action of pectin molecules agglomerating with proteins, starch and thus, eliminates haze formation which caused turbidity in syrups and juices (Díaz et al., 2013). Gelatin as fining agent works either by sticking to the particles, or by using charged ions to cause particles to stick to each other, causing them to become heavy enough to sink to the bottom (Singh and Gupta, 2004; Silva et al., 2014). Several studies have investigated the suitable potential of using pectinolytic enzymes and gelatin to clarify and remove turbidity of juices (Sreenath and Santhanam, 1992; Sorrivas et al., 2006; Benitez and Lozano, 2007; Liew Abdullah et al., 2007; Pinelo et al., 2010; Khalil, 2013; Ajayi et al., 2014; Maktouf et al., 2014). Many researches have been done to off-color compounds removal from juices based on activated carbon as absorbent (Arslanoğlu et al., 2005; Caqueret et al., 2008; Mudoga et al., 2008; Soto et al., 2008; Devesa-Rey et al., 2011).

However, clarification of juices using activated carbon alone does not remove active haze precursors, allowing haze formation during storage. Therefore, the main objectives of this study were to determine the optimum concentrations of enzymatic pretreatment parameters and to find optimum concentrations of filter aids, in filtration process of the raw syrup using response surface methodology, to yield date syrup with minimum turbidity, color intensity and polyphenol content.

2. Material and methods

2.1. Materials

Low quality dates were purchased from Shahd Babe Pars (Tabriz, Iran). Diatomite (Celite 545, Beaver Chemicals, Ontario, Canada), Cellulose (CelluFluxx P50, Erbslöh, Geisenheim, Germany) crude and expanded Perlite (Pennsylvania Perlite, Bethlehem, USA) were prepared as filter aids. Powdered Activated carbon (PAC) was obtained from Donau Chemie Group (Vienna, Austria). Pectinolytic enzyme (Fructozyme MA-LG) and gelatin were purchased from Erbslöh and Faravari Darooi Gelatin Halal (Qazvin, Iran), respectively.

2.2. Raw date syrup preparation

Pits and stems of date fruits were removed and then sliced to small cuboid ($10 \times 10 \times 2\,mm^3$). 100 ml of distilled water was added into 25 g of prepared cubes and the mixture was stirred for 120 min at 45–50 °C to obtain soft and mushy cubes. Raw date syrup was obtained by filtration of the mixture using filter paper (Whatman, No. 1). Aliquots of this syrup were pasteurized for 5 min at 90 °C and immediately cooled to 50 °C.

2.3. Raw date syrup pre-treatment process

Raw date syrup was enzymatically treated by addition of pectinolytic enzyme (0.005–0.015 g) in 100 ml of raw date syrup, to eliminate soluble pectin, using hot plate magnetic stirrer (ARE F20520162, Velp Scientifica, Usmate, Italy) for 60 min at 40–50 °C. Gelatin (0.005–0.015 g) was then added into the syrup and mixing was continued for 60 min again at the 40–50 °C. Finally, powdered activated carbon (0.1 g in 100 ml of mixture) was added in the syrup mixture and mixing process was prolonged for 30 min at the same temperature.

2.4. Filter pre-coating process

Before the filtration process, a four-layer filter cake was formed on the surface of filter paper which acts as filter medium. Filter cake including different amounts of cellulose (5–10 g), diatomite (5–10 g) and crude perlite (5–10 g), and constant amount of expanded Perlite (5 g) in 100 ml distilled water, respectively, was built up on the surface of filter paper (Whatman No.1), which was established on a Bucher Erlenmeyer vacuum. Particle size distribution (Dp) for all the filter aids are shown in Table 1.

2.5. Clarification process of date syrup by filtration

In order to remove off-color and active haze precursors of raw date syrup, 20 ml of enzymatically pre-treated date syrup was filtered through out the prepared pre-coated filter using a Bucher Erlenmeyer vacuum connected to a vacuum pump (VP6D CPS, USA) which generates pressure of −0.4 atmospheres.

2.6. Filter aid characteristics assay

Specific surface area and bulk density of the filter aids were evaluated using Brunauer–Emmett–Teller (BET) (Chembet-3000, Quantachrome, Florida, USA) where nitrogen acts as adsorbent.

Table 1 – Particle size distribution (Dp) of the different filter aids.

Filter aid	Dp < 38 μm (%)	38 < Dp < 43 μm (%)	43 < Dp < 74 μm (%)	74 < Dp < 106 μm (%)	106 < Dp (%)
Cellulose	20	14	35	16	15
Diatomate	14	39	37	8	2
Crude perlite	16	13	41	20	10
Expanded perlite	39	18	29	10	4