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Production and characterization of chitosan based edible films from *Berberis crataegina's* fruit extract and seed oil

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ABSTRACT

Chitosan-based edible films were prepared by supplementing *Berberis crataegina* DC.'s seed oil and fruit extract into chitosan matrix. The produced films have characterized both physiochemically (SEM, DSC, FT-IR, UV–vis, contact angle and mechanical analysis) and biologically (anti-quorum sensing, antimicrobial and antioxidant). Chitosan-fruit extract film revealed higher thermal stability, antioxidant, antimicrobial and anti-quorum sensing activity compared to other films. Addition of *B. crataegina's* seed oil and fruit extract into the chitosan film notably decreased the UV–vis transmittance but ameliorate the tensile strength values. Hydrophobicity of the chitosan-seed oil film was observed to be higher (92.64 ± 4.17) than chitosan-control film (84.67 ± 1.50) while chitosan-fruit extract film exhibited slightly lower hydrophobicity (73.82 ± 7.42) than chitosan film. The overall high thermal stability, antioxidant and antimicrobial activity of chitosan-fruit extract film revealed that *B. crataegina's* fruit extract can be used as an effective ingredient for the production of the edible film with enhanced physicochemical and biological properties.

1. Introduction

Recently production of edible films gained growing interest due to its biodegradable and nontoxic nature. The major reason behind this increasing interest is the environmental concerns as these films were produced using natural biopolymers such as chitosan, gelatin and lipids etc. (Amadori et al., 2015; Dwivedi, Backers, Harishchandra, & Galla, 2014); Tharanathan (2003). These natural edible films can be used as a carrier of natural active substances (antioxidant and antimicrobial) which could contribute to the enhancement of shelf life of any food commodity (Atarés, Chiralt, & McElhatton, 2016). Edible films carrying active compounds could also offer a more control release of these substances into food media (Ponce, Roura, del Valle, & Moreira, 2008). Up to now many studies has been conducted on production of active food packaging films enriched with certain natural antioxidant and antimicrobial agents (Jeannine Bonilla & Sobral, 2016; Chen et al., 2016; Duran et al., 2016; Genskowsky et al., 2015; Khalifa, Barakat, El-Mansy, & Soliman, 2016; Moradi et al., 2012; Ponce et al., 2008; Talón et al., 2017; Tesfay & Magwaza, 2017; Valenzuela et al., 2015). But still there are many plant based extracts and essential oils which needs to be characterized for its antioxidant and antimicrobial properties in edible films production. In the current study for the first time chitosan based edible films enriched with *Berberis crataegina's* seed oil and fruit extract were produced.

The genus *Berberis* has about 500 species commonly distributed in Europe, North America, South America, Asia and Africa (Bhardwaj & Kaushik, 2012; Mokhber-Dezfuli, Saeidnia, Gohari, & Kurepaz-Mahmoodabadi, 2014). *Berberis* species are rich in polyphenolic constituents such as anthocyanin and have shown significant free radical-scavenging activity (Charehsaz et al., 2015). Gulsoy, Ozkan, and Ozkan (2011) showed that leaves and fruits of *B. crataegina* contain predominant phenolic compounds which includes rutin and chlorogenic acid. No study has been conducted on oil composition of seeds from *B. crataegina*. In the current study *B. crataegina* was selected due to its wide

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distribution in different regions of Turkey making it an economical alternative as natural antioxidant and antimicrobial agent which can be used in edible films.

Chitosan is a de-acetylated derivative of chitin which is the second largest biopolymer in terms of its presence on the planet followed by cellulose (Xu, Kim, Hanna, & Nag, 2005). Chemically chitosan is comprised of repeating units of D-glucosamine and *N*-acetyl-D-glucosamine linked through β-(1-4) bond (Tripathi, Mehrotra, & Dutta, 2009). The antimicrobial properties of chitosan are attributed to its cationic nature which interacts with the negatively charged microbial cell surface consequently result in membrane seepage (Hernández-Muñoz, Almenar, Del Valle, Velez, & Gavara, 2008). These properties make chitosan an ideal alternative for synthetic polymers to be used in edible film production.

The current study was aimed to produce chitosan based edible film enriched with *B. crataegina's* seed oil and fruit extract for the first time. The produced films were characterized using SEM, ATR/FT-IR, and DSC. Additionally, some important properties were determined such as transparency, thickness, hydrophobicity, contact angle analysis and mechanical characteristics. Also antioxidant, anti-quorum sensing and antimicrobial activities of the films were evaluated.

2. Material and methods

2.1. Materials

In this study, commercially available chitosan (medium molecular weight) was purchased from SIGMA ALDRICH, USA (Code number: 448877). *Berberis crataegina's* fruits were collected from Camlidere, Elmali, Turkey. Then the fruits and seeds were separated. The chitosan solution was prepared by dissolving 1 g of chitosan in 100 ml of acetic acid solution (1% v:v). The mixture was stirred continuously via magnetic stirrer for 10 h. Petroleum ether (from MERCK) was used for extraction of *B. crataegina's* seed oil. Water extract of *B. crataegina's* fruit was prepared. All the other chemicals were purchased from SIGMA ALDRICH, USA. Distilled water was used during the experiment.

2.2. Extract preparation

Fresh *B. crataegina's* fruits were dried at room temperature. After that, the dried pulps were separated from their seeds. Additionally, water extract of *B. crataegina's* fruit was prepared by maceration in boiling water. The seeds were ground by using laboratory mill. The extract of *B. crataegina's* seed oil was obtained by stirring with petroleum ether (from MERCK) for 6 h. After extraction, water and petroleum ether were completely evaporated under vacuum (Hei-VAP Advantage, Heidolph) at about 50 °C.

2.3. Preparation of the films

Three different types of films were produced. For the first film sample (chitosan-control) 60 ml of chitosan solution was used. For the second film sample, 60 ml of chitosan was mixed with 1 ml of the seed oil in a beaker. For the third film sample, 60 ml of chitosan was added with 1 g of the dried fruit extract in a beaker. 100 µl of glycerol was added as a plasticizer to each solution. Chitosan solution without seed oil and fruit extract was used as a control. Following the addition of glycerol into the chitosan solutions, the samples were continuously homogenized for 10 min at 26.000 rpm with a homogenizer (Heidolph, SilentCrusher M). The homogenized samples were poured into plastic petri dishes and allowed to dry for 48 h at 30 °C. The thickness of the films was recorded with a digital micrometer (Mitutoyo, China).

2.4. Attenuated total reflectance infrared spectroscopy (ATR/FT-IR)

ATR/FT-IR of chitosan-control, chitosan-fruit extract, and chitosan-seed oil films were recorded using Perkin-Elmer ATR FT-IR spectrometer at a resolution of 8 cm^{-1} and range of 600–4000 cm^{-1}.

2.5. Scanning electronic microscopy (SEM)

The surface morphology and cross-section of chitosan-control, chitosan-fruit extract and chitosan-seed oil films were determined using SEM (Zeiss, Evo 40, Germany). Gold- palladium coating was conducted before taking the pictures.

2.6. Differential scanning microscopy (DSC)

Mettler Toledo DSC822e (Schwerzenbach, Switzerland) was used for determining the thermal properties of the film samples. Under N_2 atmosphere and temperature range of −50 to 420 °C, 50 mg of each film sample was placed in a hermetic aluminium pan. The heating scan was performed at 5 °C/min.

2.7. Mechanical properties

Under controlled temperature (25 °C), the tensile strength of chitosan-control, chitosan-fruit extract and chitosan-seed oil films was determined. The used film samples were 0.5 cm in width, 0.05–0.1 cm thick and 8 cm long. Material Testing Systems (MTS Insight 10) device was used with a load cell of 250 N and deformation rate of 3 mm/min. MTS Test Works 4 software is used for estimating Tensile strength (TS), Young modulus (YM), and elongation at break (EB).

2.8. Optical transmittance

Optical transmittance measurements of chitosan-control, chitosan-fruit extract and chitosan-seed oil films were carried out at a wavelength range of 400–700 nm using ultraviolet-visible spectrophotometry (a Shimadzu UV-3600 UV-VIS-NIR).

2.9. Contact angle measurements

The sessile drop contact angle was carried out using a Data Physics video-based contact angle measurement system OCA20. Precise drop volume was measured using software controlled dosing volume weight-drop. Water is used for finding surface energy. Overall ten measurements were conducted for each film sample.

2.10. Film solubility in water

Film solubility tests were determined gravimetrically. Approximately 10 mg of film sample was cut into small pieces of 2 × 3 cm^2 size and placed in 20 ml of water. Experiments were performed at room temperature (25 ± 1 °C). After immersion of samples for 24 h; they were dried for 24 h at 60 °C. The procedure was repeated thrice. The solubility of the films was presented as weight loss in percentage by the following equation.

$$WL\ (\%) = \text{Weight loss/Initial weight} \times 100 \quad (1)$$

2.11. Fatty Acid Composition Analysis

The fatty acid methyl esters (FAME) were prepared according to (Paquot, 1979). The analyses were analyzed by HP 6890 N gas chromatograph (Agilent Technologies, USA) equipped with HP-88 (100 m length, 0.25 mm id, and 0.2 mM film thickness) capillary column. The identification of fatty acids was conducted by comparing retention times of the peaks derived from Altech and Accu standards.