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Recovery, chemical and rheological characterization of gum from *Assyrian pulm*

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ABSTRACT

The extraction of gum from *Assyrian pulm* fruit (APH) was performed by microwave-assisted extraction technique. A Box–Behnken design (BBD) was applied to evaluate the effects of three independent variables (power of microwave (X_1: 50–350 W), extraction time (X_2: 20–90 min), and extraction temperature (X_3: 50–90 °C)) on the extraction yield of APH. The effect of temperature and concentration on flow behavior of gum solution was studied. The viscosity of 2% APH solution was 0.55 Pa s at a shear rate of 10 s^{-1} at 20 °C. The viscosity of fully hydrated gum solutions decreased as temperature increase. It was found that increasing the temperature from 10 to 60 °C causing the viscosity to decrease about 87% at shear rate 0.1 s^{-1} and 98% at shear rate 500 s^{-1}. The correlation analysis of the mathematical-regression model indicated that quadratic polynomial model could be employed to optimize the microwave extraction of APH. The optimal conditions to obtain the highest extraction of APH (18.19 ± 0.26%) were as follows: microwave power, 330 W; extraction time, 80.67 min and extraction temperature, 82 °C.

1. Introduction

Assyrian pulm or cordial myxa is a small deciduous tree belonging to Family Boreginaceae, which grows nearly all over the Indo-Pak subcontinent [1]. People in the Indo-Pak subcontinent traditionally eat the ripe fruits of the plant, while the raw fruits are used as vegetable and for making pickles [2]. Medicinal properties have been reported in every part of the tree. For example, anti-inflammatory constituents have been reported in the seeds [3,4] and phenolic antioxidants have been reported in the fruit extracts [5,6]. The mucilaginous extract of the fruit contains an anionic polysaccharide rich in uronic acid [7]. The ripe fruits contain gum composed of an anionic polysaccharide which is covalently bound with protein [8]. The polysaccharide is an arabinoglucan with the backbone of (1 → 6)-linked D-glucopyranosyl and (1/2)-linked L-arabinofuranosyl residues [9]. The gum has been reported to be an excellent emulsifier and tablet binder [10]. Prolonged-release nanoparticles for drug delivery have been prepared from this gum [11]. The mucilaginous extract is used for pasting sheets of paper and cardboard etc. Due to its better emulsifying and binding properties, it could be used as an effective carrier of additives through edible coating. It is reported that gum can be used as natural edible coating to increase the shelf-life of pine nuts. This natural coating could be used in other products to increase the shelf-life and improve the stability of food containing a high lipid proportion [12].

In recent years, the use of microwave-assisted extraction (MAE) of constituents from plant material has shown tremendous research interest and potential [13,14]. The principle of heating during MAE is based on the direct effect of the microwaves on molecules by ionic conduction and dipole rotation within the processed materials. Ionic conduction caused by the electrophoretic migration and the dipole rotation of realignment of ions under an applied electromagnetic field results in the rise of temperature within the extraction solution. This heating mode obviously enhances the transfer progress of objective solute within material matrix toward solution [15].

Response surface methodology (RSM) is a collection of mathematical and empirical techniques useful for establishing models, and for optimizing processes even in the presence of complex interactions. It not only determines the interaction between parameters, but also reduces the number of experimental trials, development time and overall cost [16]. Employing RSM could lead to simplifying the complexity of experimental trials needed to evaluate multiple variables and their interactions. Therefore, it is labor and time-saving than other methods required to optimize a process [17,18]. RSM has been successfully applied for optimizing conditions in food and pharmaceutical research [14,19–23].

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The objective of this research were: (1) to investigate the significant variables (Power of microwave, extraction time and extraction temperature) and further to optimize the process for extraction of gum from *Cordia myxa* leaf (APH) and its rheological properties using RSM and (2) to evaluate the antioxidant activity of extracted polysaccharide (APH).

2. Materials and methods

2.1. Materials

The fresh fruits of *Assyrian pulm* were purchased from local herb market (Fig. 1) (Mollasani, Iran), washed with tap water, rinsed with deionized water, and then air-dried at ambient temperature (30 °C). To inactivate the enzymes naturally present, the fruits were heated in a hot air oven at the 90 °C for 90 min. All other chemicals and solvents used were of analytical grade.

2.2. Methods

2.2.1. Extraction of hdrocolloid from Assyrian pulm (APH)

2.2.1.1. Pre-treatment of Assyrian pulm. The *Assyrian pulm* fruits (2000 g) were ground in a blender to obtain a fine powder and then were extracted for three times with 80% EtOH at 60 °C and 2 h each time to defat and remove some colored materials, oligosaccharides, and some small molecule materials under reflux in the apparatus, Soxhlet's.

2.2.1.2. Microwave extraction of APH. Pre-treated *Assyrian pulm* fruit powders (20 g) were immersed with distilled water in the microwave equipment to be extracted under different microwave power, extraction time and extraction temperature. The volume of treatment solution in every experiment was constant.

After extraction, the vessel was allowed to cool at room temperature. The suspension was centrifuged (3000 × g, 15 min) and the insoluble residue was treated again for 2 times as mentioned above. The supernatant was incorporated and concentrated to one-fifth of the initial volume using a rotary evaporator at 50 °C under vacuum. The supernatant was precipitated by the addition of ethanol to a final concentration of 80% (v/v) and the precipitates as crude extract were collected by centrifugation (3000 × g, 15 min) (Fig. 1). After being washed three times with ethanol, the precipitate was dried 50 °C under vacuum to obtain crude polysaccharide from *Assyrian pulm* fruit (APH). The polysaccharides yield (%) is calculated as follows:

$$\text{APH extraction yield \% (w/w)} = \frac{\text{Dried crude extraction weight (g)}}{\text{Powder weight (20 g)}} \quad (1)$$

2.2.2. Experimental design

Response surface methodology (RSM) was used to estimate the effect of independent variables (Power of microwave (W), X_1; extraction time (min), X_2 and extraction temperature (°C), X_3) on the extraction yield of APH (%). A Box–Behnken design (BBD) was employed for designing the experimental data.

After determining the preliminary range of the extraction variables through the single-factor test, the relationships between the response and the three selected variables (X_1: power of microwave, X_2: extraction time and X_3: extraction temperature) were approximated by the following second order polynomial (Eq. (2)) function:

$$\text{Extraction yield (\%)} = \beta_i + \sum_{i=1}^{3}\beta_i X_i + \sum_{i=1}^{3}\beta_{ii} X_i^2 + \sum_{i<j=2}^{3}\beta_{ij} X_i X_j \quad (2)$$

where X_i is the corresponding actual value of variable. β_0 is the estimated regression coefficient of the fitted response at the center point of the design; β_i is the regression coefficient for liner effect terms; β_{ij} is interaction effects; and β_{ii} is quadratic effects.

2.2.3. Preparation of gum solution

Gum solutions at different concentrations (0.20, 0.80, 1.40 and 2.0%) were prepared by adding the powdered or extracted gum to vigorously stirred (8000 rpm) deionized water at 10–60 °C. The solutions were stirred for 2–3 h to ensure complete solubilization. Volume adjustment was made after the solutions were cooled to room temperature.

2.2.4. Viscosity measurements

Measurements were made using a Physica MCR 301 rheometer (Parr Physica, Denmark) with the courtesy of Metrohm Siam Ltd (Bangkok, Thailand). The Physica concentric cylinder measuring system was used in all experiments. The measuring system consisted of a fixed cup and a rotating bob attached to a torque bar. The samples were sheared from 0.1 to 500 1/s. Flow behavior was described by the power law model:

$$\sigma = k\gamma^n \quad (3)$$

where σ shear stress (Pa), γ shear rate (1/s), n = flow index, and k = consistency index.

2.2.5. Effect of concentration on rheological properties

Gum solutions were prepared at 0.2, 0.8, 1.4, and 2% concentrations. The viscosity was measured at 20 °C.

2.2.6. Effect of temperature on rheological properties

To study the effect of temperature, gum solutions were prepared at 2% concentration. Viscosity measurements were made at 10, 20, 30, 40, 50 and 60 °C.

2.2.7. Assay of hydroxyl radicals scavenging activity of APH

The hydroxyl radicals scavenging activity of APH was measured according to method by Sun et al. [24]. In brief, the reaction mixture contained 1 ml of brilliant green (0.435 mM), 1.0 ml of EDTA–ferrous ion solution (9 mM), 1.0 ml of H_2O_2 (8.8 mM) and different volumes of the APH solution (1 mg/ml). The final reaction volume was made up to 4 ml with distilled water. After incubation at room temperature for 20 min, the absorbance of the mixture was measured at 624 nm against a blank (distilled water instead of the APH solution). The same procedure was repeated with vitamin C, as a positive control. The hydroxyl radical-scavenging activity was expressed as follows:

$$\text{Scavenging ability (\%)} = \frac{A_0 - A_1}{A_0} \times 100\% \quad (4)$$

A_0 and A_1 are the absorbance of control (without sample) and sample, respectively.

3. Results and discussion

3.1. Effect of single factor on the extraction yield of APH

3.1.1. Effect of microwave power on the extraction yield of APH

Different microwave power was set at 50, 150, 250, 350, 450 and 550 W to investigate the influence of microwave power on the extraction yield of APH when the other extraction conditions were set as follows: extraction time 55 min, extraction temperature 70 °C (Fig. 2a). Fig. 1a shows that the extraction yield of polysaccharide was positively correlated with the increase of irradiation power, and it reached 10.8 ± 0.3% when the irradiation power of this microwave extraction equipment was 350 W. When