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Combining various wall materials for encapsulation of blueberry anthocyanin extracts: Optimization by artificial neural network and genetic algorithm and a comprehensive analysis of anthocyanin powder properties

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ABSTRACT

Various wall materials, including maltodextrin (MD), β-cyclodextrin (β-CD), whey protein isolate (WPI) and gum Arabic (Gum-A) were combined together as the wall materials for encapsulation of blueberry anthocyanin extracts through freeze drying. Simplex lattice mixture design was used to make the experimental design. Artificial neural network (ANN) combined with genetic algorithm (GA) was successfully applied to model the influences of formulation composition on encapsulation productivity (EP) and encapsulation efficiency (EE), as well as obtain optimum formulations. Four optimum formulations were provided by the ANN-GA approach. In all the optimum formulations, WPI had the highest content. Using the optimum formulations, EP values were higher than 96% and EE values exceeded 82%. On the other hand, the properties of resulting anthocyanin powders were analyzed from different aspects. Although there were some differences in bulk density, particle size and glass transition temperature among encapsulated powders using different formulations, all the samples exhibited similar moisture content, water activity, color property and crystallinity. More importantly, encapsulation using resulting optimum formulations was effective to protect blueberry anthocyanins against degradation during heating.

1. Introduction

Food color is an important property of food products. Food industry requires a large amount of colorants annually. According to their origin, food colorants can be mainly divided into two categories, including natural and synthetic colorants [1]. Many synthetic colorants used in food industry are chemical compounds with potential harmful impact on human health. On the other hand, many studies have demonstrated that natural colorants have the health-promoting properties, such as antioxidant, anticancer, antimicrobial and anti-inflammatory properties [2–5]. Therefore, food industry is encouraged to use natural colorants instead of synthetic colorants.

Anthocyanins are one of the example natural colorants widely used in food industry. Specifically, anthocyanins are a group of water soluble pigments that are secondary metabolites in various plants [6]. It is well-known that blueberries are rich in anthocyanins. Due to the short shelf-life of blueberries, blueberries are sometimes used as raw materials to produce anthocyanin-based natural colorants [7,8]. The main drawback of anthocyanins including blueberry anthocyanins is that they are considerably unstable and can be easily affected by many factors, including temperature, pH, light, oxygen, enzyme and metallic ions during processing and storage [6]. Thus, to produce high-quality colorants using blueberry anthocyanins, it is of paramount importance to enhance the stability of anthocyanins.

Encapsulation is a common technique to protect anthocyanins against degradation and enhance anthocyanin stability. Encapsulation is defined as a process to entrap active substances (core materials) within other substances (wall materials) to produce particles with diameters from a few nanometers to a few micrometers [1]. During encapsulation, core and wall materials are first homogenized and then submitted to either freeze drying or spray drying. Regarding wall materials, different carrier agents can be used for anthocyanin encapsulation, such as carbohydrates, proteins, gums, fibers, etc. For example, whey protein isolate (WPI) alone was used to encapsulate blueberry extracts in the study of Flores et al. [9]. The mixture of gum Arabic and maltodextrin was used by Turan et al. [8] to encapsulate blueberry bioactive compounds. Furthermore, Wilkowska et al. [10] used β-cyclodextrin alone as carrier agent for encapsulation of blueberry juice polyphenolic compounds. Based on these results, it can be concluded that the

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composition of wall materials has a profound influence on encapsulation productivity, encapsulation efficiency and quality properties of resulting anthocyanin powders.

On the other hand, there are several studies reporting that the combinations of different wall materials may benefit the encapsulation of core materials compared with single wall material. For example, Davidov-Pardo et al. [11] found that the proper combinations of maltodextrin, mesquite gum and zein were more efficient than each single material to encapsulate grape seed extract. The study of Rajabi et al. [12] also revealed that the combination of maltodextrin, gum Arabic and gelatin at the weight ratio of 0.94:0.05:0.01 retained the highest amount of saffron bioactive components. Klein et al. [13] claimed that the interactions among various wall materials could generate complexes with interfacial and amphiphilic properties, thus improving encapsulation efficiency. Furthermore, it was found by Pérez-Alonso et al. [14] that the combination of protein, mesquite gum and maltodextrin could improve the properties of resulting microcapsules. As can be seen, the combinations of various types of wall materials can be considered during encapsulation of bioactive compounds. However, according to our best knowledge, there are few studies about encapsulation of blueberry anthocyanins using combinations of different wall materials and optimization of formulations used [8]. Thus, it is of significance to conduct researches to investigate the feasibility of combinations of various wall materials for encapsulation of blueberry anthocyanins, optimize the formulation and evaluate the quality of encapsulated powders.

Furthermore, modeling can be a useful and low-cost method to optimize the encapsulation process. Among the current simulation methodologies, artificial neural network (ANN) is a promising tool due to its strong ability to correlate any forms of non-linear relationship between processing factors and detected responses without prior specification of proper fitting functions [15]. Meanwhile, genetic algorithm (GA) is occasionally used to optimize the input space of ANN models, so as to obtain optimum processing parameters. According to our knowledge, ANN-GA method has already been used to model and optimize various bioprocesses, including extraction and fermentation [15,16]. Therefore, the ANN-GA approach can be considered when it comes to optimize the formulation composition used for encapsulation of blueberry anthocyanins.

In this study, four wall materials, including maltodextrin, β-cyclodextrin, whey protein isolate and gum Arabic were used as carrier agents for encapsulation of blueberry anthocyanin extracts. One of the aims was to optimize the composition of formulation by means of ANN-GA approach according to encapsulation productivity and encapsulation efficiency. Furthermore, the physicochemical properties of encapsulated anthocyanin powders using resulting optimum formulations were analyzed thoroughly. All the results can provide guidance for producing high-quality blueberry anthocyanin colorants in food industry.

2. Materials and methods

2.1. Blueberries

Blueberries (*Vaccinium ashei*) named garden-blue were purchased directly from a plantation in Lishui, Nanjing, China in the end of July, 2015. The fruits were washed, drained and stored at −18 °C in dark until use.

2.2. Extraction and purification of blueberry anthocyanins

Blueberry anthocynins were extracted and purified following the procedures reported by Cui et al. [17] with some modifications. Specifically, blueberries were thawed at room temperature for 12 h before extraction. Next, 250 g of samples were immersed in 1000 mL of solvent containing hydrochloric acid and methanol (1:99, v:v) and mashed. Anthocyanins were extracted under strong agitation at room temperature in darkness for 48 h. After that, the supernatants were obtained through centrifugation at 8000 rpm for 15 min. The acidic methanol extracts were concentrated at 40 °C by a rotary evaporator (RE-52AA, Yarong Instrument Factory, Shanghai, China) to remove organic solvents.

After extraction, the crude anthocyanin extracts were purified by partition against chloroform and ethyl acetate for several times to remove non-polar compounds. Then, the aqueous phase was subjected to an AB-8 resin column (1.6×40 cm), so as to remove aliphatic acids, sugars and other water-soluble compounds. The volume of extracts added to the column was 10 mL each time. After washing the column for several times using distilled water, adsorbed anthocyanins were eluted by 60% aqueous methanol containing 0.1% trifluoroacetic acid. Methanol in the resulting eluted solution was removed by the aforementioned rotary evaporator at 40 °C. Finally, the solution was lyophilized to obtain blueberry anthocyanin extracts.

According to the chemical analysis described later, the total anthocyanin content in the extracts was 18.06 ± 0.31 mg/g. Meanwhile, the contents of some individual anthocyanins in blueberries used in this study were measured using the HPLC method of Cui et al. [17]. The contents of cyanidin-3-*O*-glucoside, malvidin-3-*O*-glucoside and peonidin-3-*O*-glucoside in the extracts were 170.56 ± 4.32, 3841.13 ± 12.38 and 310.72 ± 7.72 μg/g, respectively.

2.3. Anthocyanin encapsulation

Anthocyanin encapsulation was performed following the procedures of Khazaei et al. [1] with some modifications. Specifically, wall materials at different ratios were mixed and dissolved in distilled water at room temperature (approximately 25 °C) to get 40% total solid concentration. After that, the resulting solution was stored at 4 °C for 24 h to reach complete hydration. Next, the aforementioned blueberry anthocyanin extracts were added slowly to the solution containing wall materials in a weight ratio of 4:1 (wall material: extract, w:w). The mixture was mixed under strong magnetic stirring and the pH value was adjusted to 2.0. To ensure complete homogenization, samples were further ultra-sonicated at 63.0 W/L and 25.0 °C for 5 min. Sonication was performed in a 20-kHz ultrasound probe system (VCX130, Sonics and Materials Inc., Newtown, USA) with a diameter of 3 mm. The dispersion was dried in a freeze drier (Freezezone 4.5, Labconoco, USA) under a pressure lower than 0.003 mBar at −45 °C for 48 h. Dried samples were ground using a mortar and pestle. Powders were packed in metallized bags, sealed and stored in desiccators at 4 °C until analysis.

2.4. Experimental design

Four carrier agents, including maltodextrin (MD with DE 10), β-cyclodextrin (β-CD), whey protein isolate (WPI) and gum Arabic (Gum-A) were used as wall materials for anthocyanin encapsulation. Two criteria were considered to optimize the formulation, being the retention of core materials inside microcapsules and the amount of core materials contained in microcapsules. Therefore, encapsulation efficiency (EE) and encapsulation productivity (EP) were used for evaluation [11]. The purpose of optimization was to find the formulations with high EE and EP values. Simplex lattice design was then employed to investigate the influences of MD (X_1), β-CD (X_2), WPI (X_3) and Gum-A (X_4) on EE and EP of encapsulated samples. Component proportions were defined as fractions of each carrier agent with a sum ($X_1 + X_2 + X_3 + X_4$) of one. Twenty combinations were generated in Design Expert 8.0 (Stat-Ease, Inc., Minneapolis, USA), which are presented in Table 1. The 20 points included 8 single-ingredient treatments, 7 two-ingredient treatments, 5 four-ingredient mixtures. All the experimental trials were carried out in duplicate.