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Research article

Encapsulation of anthocyanins in black bean (*Phaseolus vulgaris*) extracts using spray-drying and their stability under various pH and temperature processing conditions

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Abstract

Importance of the work: Anthocyanins from black beans are a natural pigment providing health benefits to consumers; however, they are heat- and pH-sensitive compounds during food processing.

Objective: To stabilize anthocyanins in black beans under food processing conditions using microencapsulation techniques with a suitable, low-cost wall material.

Materials & Methods: Black bean extracts were mixed with maltodextrin of dextrose equivalent (DE) 10 and 15. The encapsulation process was conducted at various spray-drying temperatures. The stability of the bioactive compounds in the pigment powders was observed by solubilizing in a buffer solution with a pH range of 3–9 under different heat treatments.

Results: The levels of anthocyanins and phenolic compounds in the black bean powders decreased with increasing air-drying temperature (180–210°C) and DE value (from DE10 to DE15). However, the physical properties and antioxidant activities measured using the 2,2-diphenyl-1-picrylhydrazyl and 2-2'-azino-bis (3-ethyl-benzothiazoline-6-sulfonic acid) diammonium salt methods were not significantly different among the drying conditions. The bioactive compounds and antioxidant activities were more stable in the encapsulated pigment powders for a broad pH range of the buffer solution, based on pasteurization compared to sterilization. The pigment powder encapsulated with the higher DE value of maltodextrin provided a lower content of bioactive compounds; however, the antioxidant activities were stabilized under pasteurization.

Main finding: The stability was highest for the bioactive compounds and antioxidant activities in black bean powders encapsulated with low DE maltodextrin using an air-drying temperature below 200°C when solubilized in buffer solutions over a pH range of 3–7 due to the co-pigment affect. This powder could be used as a functional ingredient for the food and pharmaceutical industries for a range of pH conditions.

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Introduction

Food color is one of the first impressions to capture consumer attention. The color used as a food additive in a regular operation of the food industry should be able to retain or reconstitute itself to provide an appropriate color intensity. Although various synthetic pigments are commonly used in food processes, the application of synthetic pigments in foods has been of concern, as many studies have reported adverse health effects of chemical residues in humans (McCann et al., 2007; Aguilera et al., 2016). In addition, awareness of healthy diets and natural foods has been promoted simultaneously, resulting in natural pigments being increasingly used to improve the acceptability of food colorants. For example, black bean is a low-cost agricultural product with versatile utilization as plant-based source of antioxidants and colorants, as well as containing proteins (20–30%), dietary fiber, phytochemicals, anthocyanins and phenolic compounds (Xu and Chang, 2008;, Aguilera et al., 2016; Mojica et al., 2017; Cappa et al., 2018). Furthermore, Aguilera et al. (2016) reported that the anthocyanins content in the black bean seed coat was as high as that of berries and plums. Teixeira et al. (2021) extracted anthocyanins from black bean hulls using a pressurized ethanol-citric acid solution and produced a high content of phenolic compounds.

It is well-known that anthocyanins provide health benefits including the prevention of cardiovascular diseases, anticancer activity, anti-inflammatory activity, neuroprotective activity, anti-obesity and antidiabetic activity (Kim et al., 2008; Žilić et al., 2013). The health benefit of this natural red-to-purple color has been reported as a flavonoid group acting as an antioxidant (Xiao, 2008). However, pigment powders obtained from natural plants are sensitive to heat, light and oxygen during processing and storage (Hernandez-Herrero and Frutos, 2014). Another important factor for anthocyanin stability is the pH; in particular, alkaline conditions cause a decrease in the flavylium cation concentration (Rodríguez-Mena et al., 2022). Thus, it was suggested to stabilize the flavylium cation form of anthocyanins rather than the carbinol and chalcone forms that prevented undesirable color shifts of anthocyanin pigments during various storage conditions or in different food matrices. With a pH shift, the structural change of anthocyanins affected the redox potential of the molecules, resulting in lower radical scavenging activities at the natural pH compared to the acidic condition (Borkowski et al., 2005; Jhin and Hwang, 2014). Baublis et al. (1994) reported that anthocyanins acylated with aromatic acids and naturally bound to sugar residues resulted in color stability of plant organs. Hence, the stability of these bioactive compounds could be improved using the encapsulation technique which entraps sensitive compounds by using suitable wall materials.

Recently, the most commonly used encapsulation technique has been spray-drying due to the desirable physical properties of the products, the short drying time and the economic potential for up-scaling (Robert and Fredes, 2015). The desired physical properties of the products were obtained by applying various drying parameters during spray-drying, such as the inlet air temperature and carrier agent. Nonetheless, natural pigments and other heat sensitive compounds were degraded by the spray-drying temperature because, in general, the content of heat sensitive compounds decreased with increasing temperature due to the heat and the removal of oxygen (Shishir and Chen, 2017). Several works have reported different heat-sensitive compounds, such as betacyanin in pithaya powder (Tze et al., 2012), anthocyanins in acai powder (Tonon et al., 2008), vitamin C in guava powder (Patil et al., 2014), lycopene in pink guava puree by-product powder (Kong et al., 2010), anthocyanins in black carrot powder (Murali et al., 2015) and anthocyanins in blackberry (Weber et al., 2017). The carrier agent is one of the spray-drying aids that provides a high yield percentage and reduces the stickiness and hygroscopicity of powdered products (Shishir and Chen, 2017). Common carrier agents for improving the drying process efficiency include maltodextrin, gum arabic, gelatin, starches, pectin, methyl cellulose, alginates, tricalcium phosphate and their combinations (Kha et al., 2010; Mishra et al., 2014). Romero-González (2020) reported that using maltodextrin improved the phenolic retention and color stability in maqui powders stored for 60 d at 25°C. Similarly, Jafari et al. (2016) reported that microencapsulation of anthocyanins from saffron petals was more effective with added maltodextrin. In addition, using maltodextrin as a carrier agent in spray-drying of lychee juice successfully reduced thermal and oxidative losses of anthocyanins (Kingwatee et al., 2015). Novel and inexpensive processes, such as the use of maltodextrin, are required to improve pigment stability. The dextrose equivalent (DE) value of maltodextrin represents the amount of the reducing sugar content. The most commonly used DE of maltodextrin is in the range 3–20 due to maltodextrin having a high molecular weight, high glass transition temperature (T_g), good solubility and low viscosity (Schutyser et al., 2012). However, a higher DE value results in lower stability of the encapsulated product as more glucose units are obtained (Levine and Slade, 1986).

Therefore, the current research aimed to determine the effect of microencapsulation using a spray-drying technique at various air-drying temperatures and different DE values of maltodextrin on the stability of anthocyanins and phenolic compounds obtained from black beans. In addition, the pigment stability was investigated using different heat treatments and pH levels (3–9) to study the utilization of this natural pigment powder under circumstances similar to a food processing environment with a broad pH range applicable to many food products.

Materials and Methods

Black bean (*Phaseolus vulgaris*) was obtained from a local market in Ubon Ratchathani, Thailand. 2,2-Diphenyl-1-picrylhydrazyl (DPPH) was purchased from Sigma (Germany) and (±)-6-hydroxy-2,5,7,8-tetramethylchromane-2-carboxylic acid (Trolox) from Aldrich (Saint Louis, MO, USA). 2-2'-Azino-bis (3-ethyl-benzothiazoline-6-sulfonic acid) diammonium salt (ABTS) and Folin-Ciocalteau reagent were purchased from Fluka (Germany). Maltrodextrin DE 10 and DE 15 were obtained from Sahasinwattana (Thailand).

Anthocyanin pigment powders process

Extraction method

Black beans were washed thoroughly to remove extraneous matter. The washed beans were blended at 1,500 rounds/min for 30 s after draining using a blender (Robot coupe, model no. BLIXER 3 V.V; Australia). The extraction method followed Jitpisoot et al. (2007) with modifications. Blended samples were extracted using a buffer solution containing 0.2 M boric acid, 0.05 M citric acid and 0.1 M sodium phosphate solution at pH 2.5. The blended samples were mixed with the buffer solution (1:4 sample-to-solvent ratio) before processing using an ultrasonic sonicator (Elma; model no. T 890/H; Germany) at 60±2°C for 50 min, after which the filtrate was collected using a filter suction pump. The pigment extract was further evaporated using a vacuum evaporator (Didacta; model no. TA62/D; Italy) at 50±2°C and a pressure of 66±2 kPa to obtain 5% soluble solid content.

Spray-drying process

The final total soluble solid content of pigment extract was adjusted to 10–11% using the two types of studied maltodextrin (DE10 and DE15). The blending process was conducted using a hand-held homogenizer. All spray-drying experiments used a spray-dryer (Niro; model A/S; Denmark). The extracted pigment solution was passed through a peristaltic pump for spray-drying using an atomizer with a solution feed flow rate of 16.67 mL/min using three different inlet and outlet temperature settings (180±0.6°C, 78–79°C, respectively; 195±0.6°C, 84–85°C, respectively; 210±0.7°C, 90–94°C, respectively). The pigment powders were packed in vacuum-sealed packages and stored in a desiccator at 25–30°C until further analysis. The anthocyanin pigment powder process is summarized in Fig. 1. The physiochemical properties and bioactive compounds of the encapsulated pigment powders were determined. The encapsulation conditions of the pigment powders were conducted following a 2×3 factorial in completely randomized design with three replications. The obtained data were analyzed using analysis of variance and Duncan's multiple range test were used to determine differences among means, with $p < 0.05$ considered as significantly different.

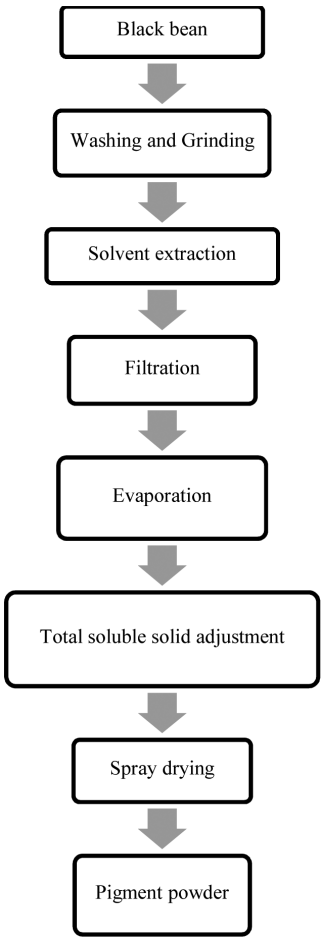

Fig. 1 Processing flow chart for anthocyanins pigment powders obtained from black beans

Stability of bioactive compounds in pigment powders

The stability was investigated of the anthocyanin powders, obtained using the different microencapsulation techniques based on spray-drying, using a buffer solution (0.2 M boric acid mixed with 0.05 M citric acid solution). The pH was adjusted by adding 0.1 M sodium phosphate buffer to obtain values in the pH range 3–9. The stability study of bioactive compounds using the various pH and heat treatment conditions to solubilize the powder in liquid form followed Sadilova et al. (2006) and Jiang et al. (2019) with modifications. Samples (1 g each) of the powder were transferred into Pyrex tubes containing 10 mL of buffer solution which was well capped to avoid evaporation. Two conventional heating treatments were used: 1) pasteurization at 70°C for 30 min using a water bath; and 2) sterilization at 121°C for 15 min using an autoclave. These treatments were compared with the control (no heating). After being heated, the pigment solution was immediately cooled to 4°C using iced water. All treatments were stabilized at 29°C by placing the tubes in tap water before analysis. The stability of the bioactive compounds in the pigment powders was monitored by determining the total phenolic compounds (TPCs), total anthocyanin content and antioxidant activities (DPPH and ABTS methods).

The experiment was designed using a 2×3 factorial in completely randomized design with three replications. Factor A was three levels of heating treatment and Factor B was four levels of pH for each selected encapsulation process. Analysis of variance and Duncan's multiple range tests were used to analyze the differences in the experimental data, with $p < 0.05$ considered as significantly different.

Analysis of moisture content and water activity

The method of Association of Official Analytical Chemists (2006) was used to determine the moisture content using a hot-air oven at 103–105°C (BINDER; Model FED240-M; USA). Water activity (a_w) was measured using a Thermoconstanter (Novasina; Model PS200 S/N 9809020; Switzerland) calibrated with a known value of the standard sample in the range 0.11–0.99. The experimental data were obtained using three replications.

Surface color measurement

The surface color of the pigment powders was measured using a colorimeter (IKA; Model Genius 3; Germany). Color was expressed in the CIE system based on values determined for L* (whiteness and blackness), a* (redness and greenness), and b* (yellowness and blueness). In addition, color intensity (chroma) and hue angle were calculated using the Equations 1 and 2, respectively:

$$\text{Chroma}(C^*) = \sqrt{a^{*2} + b^{*2}} \quad (1)$$

$$\text{Hue}(h) = \arctan\frac{b^*}{a^*} \quad (2)$$

Bulk density analysis

The bulk density of the pigment powders was determined following Habtegebriel et al. (2018) with slight modifications. A sample (2 g) was placed in a 50 mL graduated cylinder and the volume of powder was read. The bulk density was calculated by dividing the mass of the pigment powder by the volume it occupied in the cylinder.

Analysis of solubility of pigment powders

Solubility was determined using the method of Takashi and Seibi (1988) with some modifications. Briefly, 1 g of powder was dissolved in 10 mL distilled water at 30°C in a centrifuge tube. The suspension was stirred constantly for 30 min before centrifugation using a laboratory centrifuge (Hettich; Model MIKRO 200/200R220R, USA) at 3,000 rpm for 10 min. The supernatant was collected and dried at 105°C until a constant weight was recorded. The weight of the dried mass was used to calculate the water solubility (%).

Analysis of total anthocyanins

The total anthocyanins were determined using the pH differential method (Association of Official Analytical Chemists, 2006). The dilution was performed in a solution at pH 1 and pH 4.5, with quantification of absorbance at wavelengths of 510 and 700 nm, respectively, using an ultraviolet/visible spectrophotometer (TG Instruments; model no. T60, Germany) according to Wrolstad et al. (2005). The results were expressed as milligrams of cyanidine-3-o-glucoside per 100 g on a dried basis (db.).

Analysis of total phenolic compounds

The TPCs were determined using Folin-Ciocalteau reagent with gallic acid as a standard (Slinkard and Singleton, 1977). The sample solution was obtained using 0.2 g of dried pigment powder mixed with 80% methanol using a shaking bath (GFL; Model 1083; Germany) at 200 rpm for 2 hr at room temperature. Then, the solution was centrifuged at 1,500 rpm for 20 min at 25°C using a refrigerated centrifuge (Hettich Universal; Model 32R; the Netherlands). After that, 2 mL of

the supernatant was mixed with 10 mL of the Folin-Ciocalteau reagent before incubation in a dark cabinet for 1–8 min. After adding 8 mL of 7.5% Na_2CO_3, the mixture was left at room temperature for 2 hr before the absorbance was measured at 765 nm. The results were expressed as milligrams of gallic acid per 100 g dried pigment powder.

2,2-Diphenyl-1-picrylhydrazyl scavenging activity analysis

The antioxidant activity was determined according to the DPPH scavenging activity method (Kitts et al., 2000) with some modifications. A sample of 1.95 mL of 0.1 mM DPPH in an 80% ethanol solution was mixed with 0.05 mL of 0.1 g/mL anthocyanin aqueous solution. Absorbance was measured at 517 nm after 30 min of incubation at room temperature. Antioxidant activity was reported as the percentage of scavenging using Equation 3:

$$\text{DPPH scavenging activity (\%)} = \frac{\text{Abs.control - Abs.sample}}{\text{Abs.control}} \times 100 \quad (3)$$

where Abs.control is the absorbance of control and Abs. sample is the absorbance of reaction mixture.

2-2'-Azino-bis (3-ethyl-benzothiazoline-6-sulfonic acid) diammonium salt scavenging activity analysis

The ABTS scavenging activity analysis was determined according to Arnao et al. (2001) with some modifications. $ABTS^+$ stock solution was prepared by mixing 7.4 mM ABTS solution and 2.6 mM potassium persulfate solution, in 10 mM phosphate buffer (pH 7.4), and keeping in the dark for 12 hr. The $ABTS^+$ working solution was prepared by mixing the stock solution in 10 mM phosphate buffer (pH 7.4) to obtain an absorbance of 0.7 ± 0.02 at 734 nm. Then 20 µL of anthocyanin solution was mixed with 1.98 mL of $ABTS^+$ working solution and the mixture was kept in the dark for 5 min before the absorbance was measured at 734 nm. The antioxidant activity of ABTS was reported the percentage of scavenging using Equation 4:

$$\text{ABTS scavenging activity (\%)} = \frac{\text{Abs.control - Abs.sample}}{\text{Abs.control}} \times 100 \quad (4)$$

where Abs.control is the absorbance of control and Abs. sample is the absorbance of reaction mixture.

Results and Discussion

Physico-chemical properties of anthocyanin pigment powders

Dried pigment powder samples were obtained using the various air-drying temperatures and maltodextrin at the two different DE values. The flow rate of pigment extract was set at 16.67 mL/min. Even though the amount of maltodextrin added was the same as the amount of the initial solid content after evaporation, there were effects of maltodextrin on some particular characteristics, such as moisture content, solubility, color and anthocyanin content. The moisture content values of the powders obtained from the different conditions were in the range 3.85–5.31% db. ($p \leq 0.05$). As shown in Table 1, the results indicated that the moisture content of the pigment powders dried at inlet air temperatures of 180°C and 195°C for maltodextrin DE10 and DE15 were not significantly different. The increase in the air-drying temperature caused by adding maltodextrin as a drying aid or wall material for pigment powder encapsulation decreased the water activity (Ekpong et al., 2016). The faster drying rate could be explained by the higher drying temperatures which supplied higher heat or energy to water molecules, causing accelerated moisture removal during drying. This result agreed with Kha et al. (2010) and Niamnuy et al. (2014). In addition, the water activity is one of the most important factors for food stability during storage. A higher level of water activity led to a shorter shelf-life due to more free water being available for biochemical degradation, while the deterioration of dried powder caused by microorganism activity and biochemical reactions could be retarded at a water activity lower than 0.6

Table 1 Physicochemical properties of anthocyanin pigment powders obtained from black beans

Inlet air temp. (°C)	Outlet air temp. (°C)	Maltodextrin (DE)	Moisture content (% db)	Water activity	Bulk density (g/cm³)	Solubility (%)	L*	a*	b*	Chroma (C*)	Hue angle (h)
180	78	10	4.65±0.12^{abc}	0.23±0.06^{b}	0.50±0.01^{a}	59.36±7.07^{ab}	61.78^{a}	21.69^{d}	-7.44^{c}	22.93^{d}	0.33^{ab}
180	79	15	5.09±0.38^{a}	0.25±0.07^{ab}	0.49±0.03^{a}	68.38±9.26^{a}	59.32^{c}	20.34^{e}	-2.62^{a}	20.48^{e}	0.12^{d}
195	84	10	5.31±0.42^{a}	0.28±0.05^{a}	0.51±0.00^{a}	54.39±2.32^{b}	58.47^{d}	23.33^{b}	-8.17^{d}	24.73^{b}	0.34^{a}
195	85	15	4.75±0.79^{ab}	0.22±0.03^{b}	0.52±0.06^{a}	58.99±4.08^{ab}	60.50^{b}	22.58^{c}	-7.40^{c}	23.76^{c}	0.32^{b}
210	90	10	3.85±0.19^{c}	0.18±0.02^{c}	0.50±0.00^{a}	51.90±6.13^{b}	59.32^{c}	19.43^{f}	-2.46^{a}	19.59^{f}	0.12^{d}
210	94	15	3.95±0.34^{bc}	0.26±0.08^{a}	0.51±0.00^{a}	53.62±4.29^{b}	54.64^{e}	26.14^{a}	-6.50^{b}	29.64^{a}	0.24^{c}

DE = dextrose equivalent; db = dry basis;

Means (± SD) superscripted with different lowercase letters are significantly ($p < 0.05$) different.

(Fennema, 1996). In the current study, the water activity levels of the anthocyanin pigment powders were well below 0.6 (0.18–0.28). Furthermore, the solubility of the powders was in the range 51.90–68.38% and the highest solubility of 68.38% was observed using 180°C drying temperature with maltodextrin DE 15 ($p \leq 0.05$) which was similar to that reported for litchi powder using maltodextrin as a drying aid (Kalita et al., 2018). However, there were significantly lower levels of solubility for the conditions of air-drying at 195°C with maltodextrin DE 10 and at 210°C with maltodextrin DE 10 and DE 15 compared to at 180°C with maltodextrin DE10 and DE 15 (Table 1). The high maltodextrin content which was used as the initial solid content of the black bean extract, could provide good solubility by increasing surface water absorption and reducing stickiness and agglomeration phenomena (Ekpong et al., 2016). In addition, the added maltodextrin could be beneficial as a pigment enhancer for food matrices.

The bulk density of the spray-dried product is generally expected to be lower at higher temperatures (Shishir and Chen, 2017). However, there was no significant difference in the bulk density (Table 1) between the different drying temperatures, which could have been due to the narrow range of temperature in the study that was not sufficient to cause any differences in the bulk density.

Fig. 2 shows the surface color of the anthocyanin pigment powders under various conditions. It was found that the color of the spray-dried powders was significantly affected by drying temperature and the DE value of the maltodextrin. A significant decrease in lightness (L*) was observed with increasing drying temperature and degree of DE, except for the drying temperature at 190°C with maltodextrin DE15. The high air-drying temperatures and the large number of glucose molecules in the maltodextrin initiated a Maillard reaction that produced browning pigment compounds in the powders (Quek et al., 2007). This result was compatible with Ersus and Yurdagel (2007), who reported that an increase in DE led to lower lightness. It was clearly observed that an increase in drying temperature decreased the values of a*, chroma and hue angle of the powders when maltodextrin DE10 was used. In general, higher a* and lower hue angle values indicated a bright-purple shade of red (Fig. 2). Overall, increases in the drying temperature and degree of DE produced a lighter color of the pigment powders. As mentioned earlier, the maltodextrin content was used in the same amount as the initial solid content of black bean extract, which produced a bright color due to the dilution effect.

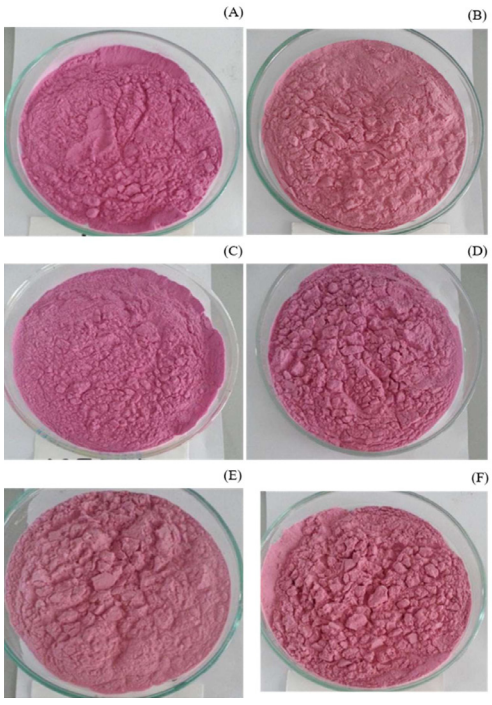

Fig. 2 Visual appearances of pigment powders obtained from black bean dried at (A) 180°C, maltodextrin at dextrose equivalent (DE)10; (B) 180°C, maltodextrin DE15; (C) 195°C, maltodextrin DE10; (D) 195°C, maltodextrin DE15; (E) 210°C, maltodextrin DE10; (F) 210°C, maltodextrin DE15

Effect of air-drying temperature and dextrin equivalent on stability of bioactive compounds and antioxidant activity

The initial total anthocyanin content determined using an anthocyanin pigment solution before spray drying, was 3,193.93 mg/100 g of dried beans. With the same DE value of maltodextrin, increasing the temperature from 180°C to 210°C reduced the anthocyanin contents from 636.79 ± 8.67 mg/100 g dried powder to 344.11 ± 6.25 mg/100 g dried powder (Fig. 3). This result was consistent with Tonon et al.

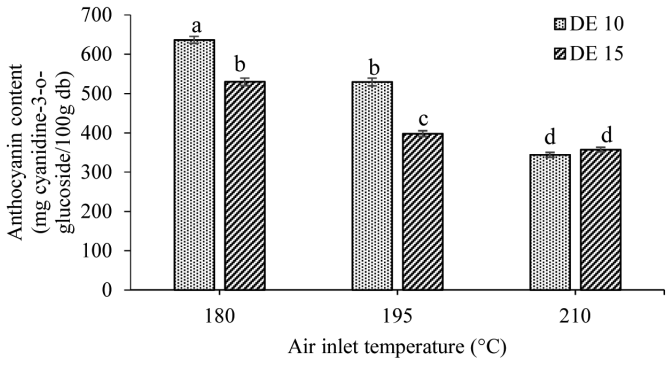

Fig. 3 Effect of dying temperature and maltodextrin at dextrose equivalent (DE)10 and DE15 on anthocyanin content of pigment powders, where different lowercase letters above columns indicate significant ($p < 0.05$) differences, error bars indicate ± SD and db = dry basis.

(2010), who reported a decrease in the anthocyanins in acai at high air-drying temperatures. Laokuldilok and Kanha (2015) studied the spray-drying process of black glutinous rice bran encapsulated with partially hydrolyzed black glutinous rice maltodextrin DE10, DE20 and DE30 compared with bran samples encapsulated with the commercial maltodextrin DE10. They also reported a decrease in the anthocyanin content (from 71.96% to 47.67%) with increased air-drying temperature increased. The results in Fig. 3 indicate that higher drying temperatures caused a significant reduction in the anthocyanin content due to thermal degradation. In addition, the DE value of the maltodextrin affected the anthocyanin content. For the same drying temperature, the anthocyanin content of the sample with maltodextrin DE 15 was less than that with DE10. This result was compatible with the results of Tonon et al. (2010) and Rodríguez-Hernandez et al. (2005) but contradicted Laokuldilok and Kanha (2015), who hydrolyzed maltodextrin from black rice bran. This effect could be explained by the poorer binding properties of maltodextrin with a higher DE. Compared to maltodextrin with lower DE, the higher DE used as a coating agent in the encapsulation process could not properly bind with the other active compounds, such as anthocyanins, resulting in a lower anthocyanin content. Furthermore, a higher DE resulted in increased glucose oxidation at higher drying temperatures, resulting in less stability of the encapsulated powders (Levine and Slade, 1986; Ersus and Yurdagel, 2007).

The total phenolic contents of the encapsulated pigment powders are shown in Fig. 4. While a higher inlet air temperature significantly lowered the total phenolic contents of the pigment powders, the different DE values did not. The total phenolic contents were significantly decreased from 64.62±2.10 g gallic acid/100 g dried powder to 50.74±1.17 g gallic acid/100 g dried powder when the inlet temperatures increased from 180°C to 210°C. It was concluded that the phenolic compounds in the powders were heat-sensitive and could be degraded at high temperatures. In addition, the degradation of phenolic compounds during spray drying could have been due to changes in their molecular structure, which reduced their reactivity and extractability (Mishra et al., 2014).

The effects of drying air conditions on antioxidant activities, determined using DPPH and ABTS radical scavenging activity methods, are shown in Figs. 5 and 6, respectively. Fig. 5 indicates that the antioxidant activity was significantly

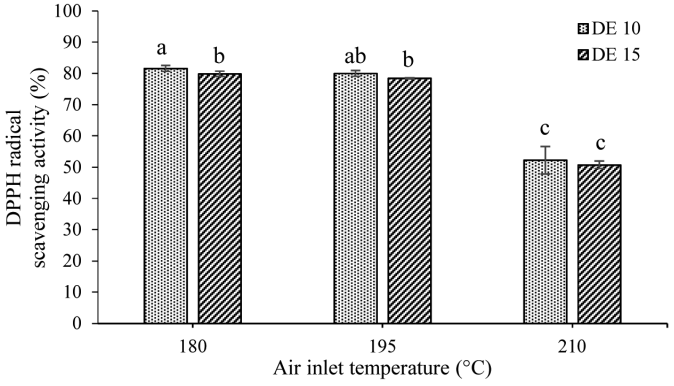

Fig. 5 Effect of drying temperature and maltodextrin at dextrose equivalent (DE)10 and DE15 on 2,2-diphenyl-1-picrylhydrazyl (DPPH) radical scavenging activity of pigment powders, where different lowercase letters above columns indicate significant ($p < 0.05$) differences and error bars indicate ± SD.

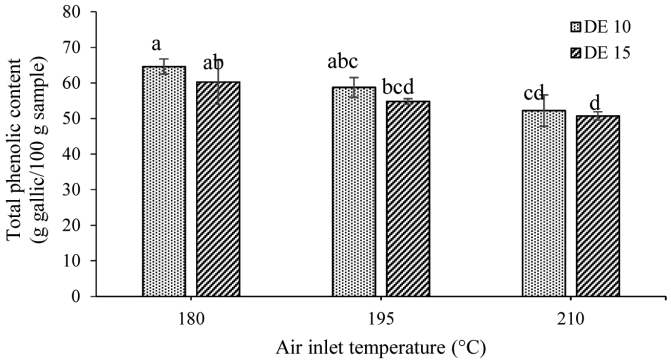

Fig. 4 Effect of drying temperature and maltodextrin at dextrose equivalent (DE)10 and DE15 on total phenolic content of pigment powders, where different lowercase letters above columns indicate significant ($p < 0.05$) differences and error bars indicate ± SD.

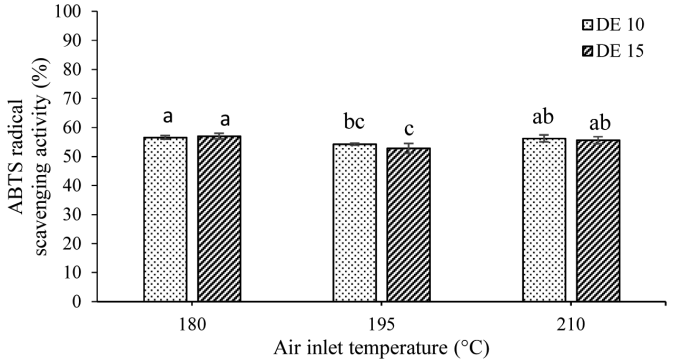

Fig. 6 Effect of drying temperature and maltodextrin at dextrose equivalent (DE)10 and DE15 on 2-2'-azino-bis (3-ethyl-benzothiazoline-6-sulfonic acid) diammonium salt (ABTS) radical scavenging activity of pigment powders, where different lowercase letters above columns indicate significant ($p < 0.05$) differences and error bars indicate ± SD.

decreased by increase inlet air temperature. The powder obtained from the lowest drying temperature (180°C) had the highest antioxidant activity (81.58%) compared to the highest drying temperature (210°C) which had the lowest antioxidant activity (77.66%). This could have been related to the high temperatures that negatively affected the structures of the anthocyanins and phenolic compounds, causing their molecular structures to break down into carboxylic acid and carboxyl aldehyde. Furthermore, the possible loss of hydrogen atoms could result in changes in the two compounds from antioxidants to free radicals during the hydrogen atom transfer mechanism (Galano et al., 2016). This reduction in the antioxidant activity was well correlated with the reduction in the total anthocyanins (Fig. 3) and TPCs (Fig. 4). In addition, the antioxidant activity was higher in the powder encapsulated with DE10 maltodextrin compared with DE15, which could have been due to the higher binding capacity of the DE 10 maltodextrin that provided a higher content of anthocyanin after drying, thus resulting in a high level of antioxidants. It was possible that the lower DE value of maltodextrin, with the longer polymeric chains, contained more hydroxyl group, that were available to interact with other food components, such as water or bioactive compounds (Castro et al., 2016).

The ABTS radical scavenging activity, based on a single electron transfer mechanism, was also determined, as shown in Fig. 6. The ABTS method is expected to provide better detection of antioxidant activity than the DPPH method; for example, Floegel et al. (2011) reported that high-pigmented and hydrophilic antioxidants were reflected better by the ABTS method compared to the DPPH. However, there was no observed effect of the drying conditions on the antioxidant activity using the ABTS method in the current study. The results based on the ABTS method were in the range 52–57% for the different drying conditions. This suggested that not only those hydrophilic antioxidants, but the other bioactive lipophilic compounds, such as carotenoids, might play an important role in the radical scavenging activity detected using the ABTS method. Consequently, the antioxidant activity of the pigment powders determined using the ABTS method was not affected by the various air-drying temperatures.

Stability of bioactive compounds in pigment powers under different pH values and heating treatments

The stability was studied of bioactive compounds in encapsulated pigment powders obtained from the tested spray-drying techniques under various conditions as some disadvantages from using natural pigment have been reported under various food processing and storage conditions. Based on the values for the TPCs and antioxidant activities, the pigment powders of the highest quality were obtained using 180°C air-drying temperature with maltodextrin DE10 encapsulation and those of the lowest quality using 210°C air-drying temperature with maltodextrin DE15 encapsulation. These were both solubilized in a buffer solution of pH in the range 3–9 for stability testing. As mentioned earlier, this study aimed to determine the stability of bioactive compounds in pigment powder under circumstances similar to the food processing environment using a broad pH range for food products in liquid form. The most common food processing method uses high temperatures to extend the shelf-life of a food product, with the ingredients being stabilized, in particular food colorants or bioactive ingredients. Therefore, the stability of the bioactive compounds in the powders were investigated using heat treatments (pasteurization and sterilization) and compared with non-heated treatment samples. However, the current study did not use the extract without further processing as a control for thermal and pH stability studies which have been reported to be sensitive to processing and storage conditions (Hernandez-Herrero and Frutos, 2014). The statistical analysis indicated that heat treatment and pH significantly affected the anthocyanin contents of the pigment powders (Figs. 7A and 7B) used in the current investigation, with both these parameters causing an interaction effect with the total anthocyanin content rather than the total phenolic content (Figs. 7A–7D). An increase in temperature and pH reduced the anthocyanins, whereas a stable level of anthocyanins was observed for the untreated condition at various pH levels ($p \leq 0.05$). As expected, the anthocyanin contents of the pigment powders were significantly the highest for the treatment involving drying at 180°C with maltodextrin DE10 encapsulation and without any heat treatment at pH 3. The stability of anthocyanins tended to decrease with increases in the pH and temperature used during the pasteurization and sterilization conditions ($p \leq 0.05$), as shown in Figs. 7A and 7B. Even though there was a significant decrease in the anthocyanin content with an increase in the pH from 3 to 9 for both the sterilization and pasteurization processes ($p \leq 0.05$), the anthocyanin content at pH 5 was still relatively high. This could have been due to the benefits of the encapsulation process on the pigment powders.

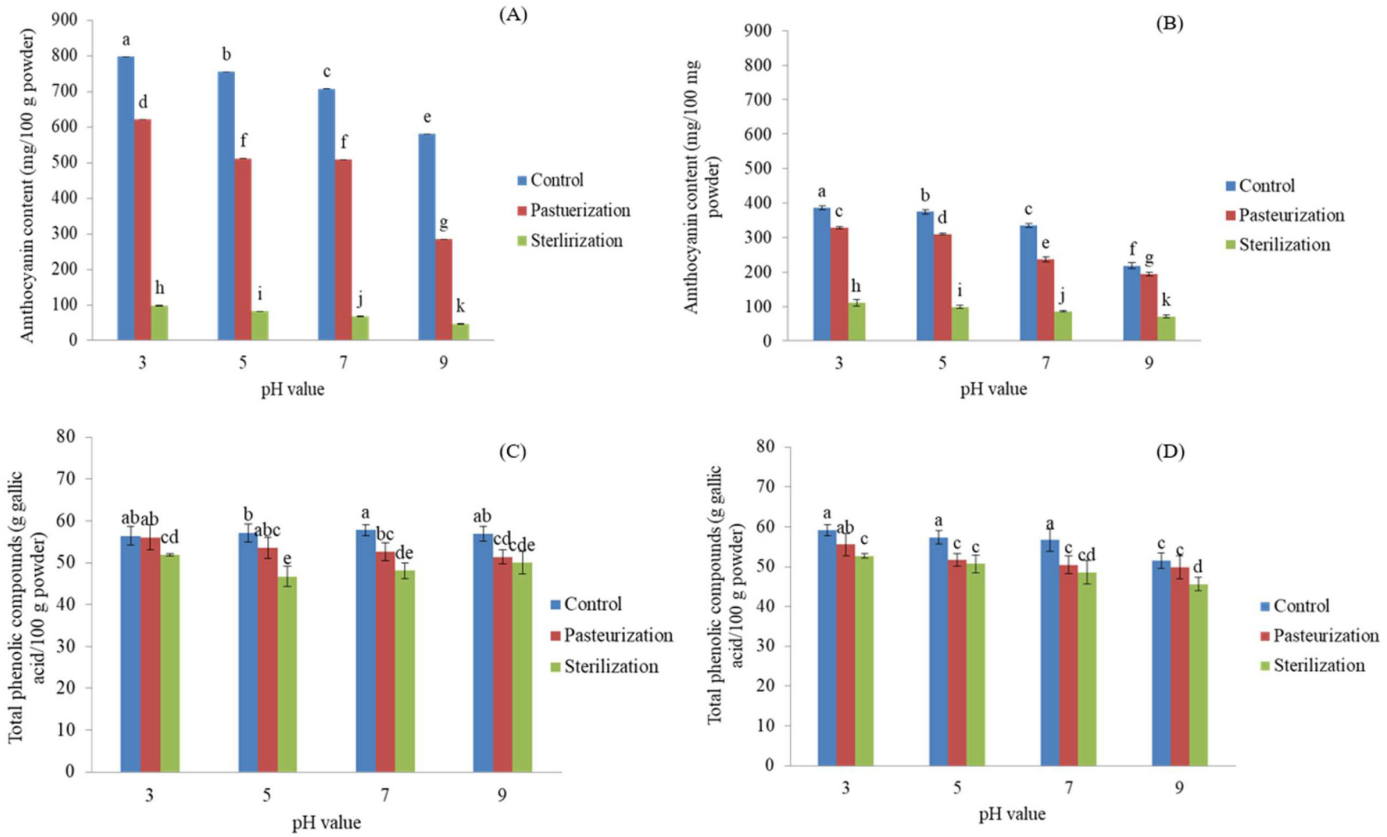

Fig. 7 Stability of bioactive compounds of pigment powders using different heat treatments for pH range 3–9: (A) anthocyanin stability at 180°C and maltodextrin at dextrose equivalent (DE)10; (B) anthocyanin stability at 210°C and maltodextrin DE15; (C) total phenolic compound stability at 180°C and maltodextrin DE10; (D) total phenolic compound stability at 210°C and maltodextrin DE15), where different lowercase letters above columns indicate significant ($p < 0.05$) differences and error bars indicate ± SD.

Similarly, Dini et al. (2020) reported that the anthocyanin stability obtained from aphi skins decreased when the pH increased, which led to a prevalence of pseudobase structures and resulted in a colorless tone pigment. Both heat treatment and high pH caused structural changes in the anthocyanins, particularly in the 3-glycoside of the benzene ring to chalcone, which resulted in less stability during the food processing conditions (Laleh et al., 2006), which could have been due to the thermal gradation of anthocyanin being activated by high temperature through deglycosylation and scission. Sadilova et al. (2006) noted that under thermal degradation, anthocyanin glycosides were cleaved by successive losses of sugar moieties and pentoses (that were more readily split off than hexoses), while anthocyanin aglycones were further degraded by scission into phloroglucinaldehyde (cyanidin, pelargonidin), 4-hydroxybenzoic acid (pelargonidin), and protocatechuic acis (cyanidin). Similar to the pigment powders obtained by spray-drying at 180°C with maltodextrin DE10 encapsulation, anthocyanin degradation in the powder samples dried at 210°C with maltodextrin DE15 encapsulation was also observed as the pH was increased from 3 to 9 (Fig. 7B). However, the use of maltodextrin DE15 as a wall material at a drying temperature of 210°C provided high anthocyanin stability after being exposed to the pasteurization process (reduction only 11–29% of the control for the pH range 3–9). Compared to the anthocyanin degradation of the pigment powder encapsulated with maltodextrin DE10 under pasteurization, there was 22–50% reduction of the control for the pH range 3–9. This contradicted the former result on the stability of anthocyanins during the spray-drying process in which direct heating at a higher air temperature of more than 180°C was used. In this case, the higher stability of the pigment powders might have been caused by solubilizing the encapsulated powders in the pH buffer solution and treating with indirect heating, including pasteurization and sterilization. The higher stability provided by maltodextrin DE15 could be explained by the additional binding effect of the glucose units in the short-chain polysaccharides. The binding effect between glucose

and anthocyanins combined with the effect between glucose and co-pigment could result in higher thermal stability of the anthocyanins encapsulated with maltodextrin DE15 compared to maltodextrin DE10, especially at pH 3. This result agreed with Ertan et al. (2020) who reported that the anthocyanin content in strawberry nectar was stabilized during 42 d storage by using a combination of co-pigment sources (such as gallic acid) and sweeteners (such as maltose sugar) at pH 3.37–3.47. Notably, in the current study, the stability of the anthocyanins in the pigment powders was investigated over a wider pH range with the use of an encapsulation process.

The stability of the TPCs, from the co-pigment in the black bean powders, is shown in Figs. 7C and 7D. After pasteurization (Fig. 7C), the stability of the TPCs in the black bean powders was significantly different for the pH range 3–9 following spray drying at 180°C with maltodextrin DE10 encapsulation. However, there were no significant differences in the TPCs among those powders at pH values of 5, 7 and 9 after pasteurization. In Fig. 7D, the TPC in black bean powders using 210°C drying temperature encapsulated with maltodextrin DE 15 was significantly different after pasteurization at pH 5–9. In addition, the TPC of this pigment powder was not significantly different from that of the control at pH 3 after pasteurization. This was consistent with the results of the pigment powders obtained by spray drying at 180°C with maltodextrin DE10 encapsulation. Furthermore, the TPC at pH 9 was significantly less stable in the control and after sterilization but not significantly different from the powders solubilized in the buffer solution at pH 7. This result was partly consistent with the finding of da Silva et al. (2022) that the encapsulated TPC was stabilized only at pH 7, during accelerated conditions for 8 h. In contrast, the stability of the TPC in the black bean powders in the current study was stable for the whole pH range (3–9) except for those powders with maltodextrin DE15 encapsulation dried at 210°C. Instead of the maltodextrin used in the current study, chitosan, which is easily solubilized in acidic conditions, was used as a wall material by da Silva et al. (2022). For the highest stability of TPC under treatments in the pH range 3–9, the optimum conditions for black bean powders are encapsulation with maltodextrin DE10 at a drying temperature of 180°C. In summary, an increase in the temperature during food processing to above 100°C resulted in the thermal degradation of phenolic compounds and anthocyanins.

The antioxidant activity of the black bean powders obtained using by drying at 180°C with maltodextrin DE10 encapsulation was relatively high after pasteurization and sterilization compared to the control (Fig. 8A) within the pH range 3–9. Similarly, antioxidant activity was stable after pasteurization for the pigment powders encapsulated with maltodextrin DE 15 at 210°C (Fig. 8B). These results were consistent with the stability of anthocyanins in the pigment powders under the same conditions as the reduction was only 11–29% of the control. This could be explained by the binding effect between the glucose units in the short chain polysaccharides of maltodextrin DE15 and anthocyanins, which resulted in the thermal stability of the pigment and stabilized the radical scavenging activity. Furthermore, the decrease in the antioxidant activity after sterilization as measured using the DPPH method of the powders dried at 210°C with maltodextrin DE15 encapsulation (Fig. 8B) was compatible with the decrease in anthocyanins (Fig. 7B) and TPC (Fig. 7D).

There was no significant difference in ABTS radical scavenging activity of the black bean powders obtained using drying at 180°C with maltodextrin DE10 encapsulation, after pasteurization and sterilization for pH 3–9 compared to the control (Fig. 8C). However, the black bean powders obtained using spray drying at 210°C with maltodextrin DE15 encapsulation had the significantly lowest antioxidant activity using the ABTS method after sterilization for pH 3–9 compared with the powders after pasteurization (Fig. 8D). In addition, the radical scavenging activities of the pigment powders encapsulated with maltodextrin DE 15 at 210°C, at pH of 9 after sterilization were significantly lower than those for pH 3–7. This could be explained by the structural change of anthocyanins affecting the redox potential of the molecules and resulting in lower radical scavenging activities with the pH shift (Borkowski et al., 2005; Jhin and Hwang, 2014). This result agreed with the radical scavenging activity measured using the DPPH method, with the assumption that the antioxidant activity of the black bean powders depended on both the TPC and anthocyanins. Nevertheless, the pigment powders encapsulated with maltodextrin DE10 at 180°C provided high stability for a broad pH range (3–9). These results suggested that the pigment powders encapsulated with a single wall material, such as maltodextrin, could be used to adequately protect the bioactive compounds present in the black bean. Romero-González et al. (2020) also used maltodextrin (DE15) as a single wall material to enhance the availability of the TPC and anthocyanins in maqui powders and found that these bioactive compounds were retained after 60 d storage at 25°C compared with the powders encapsulated using maltodextrin combined with other polysaccharides.

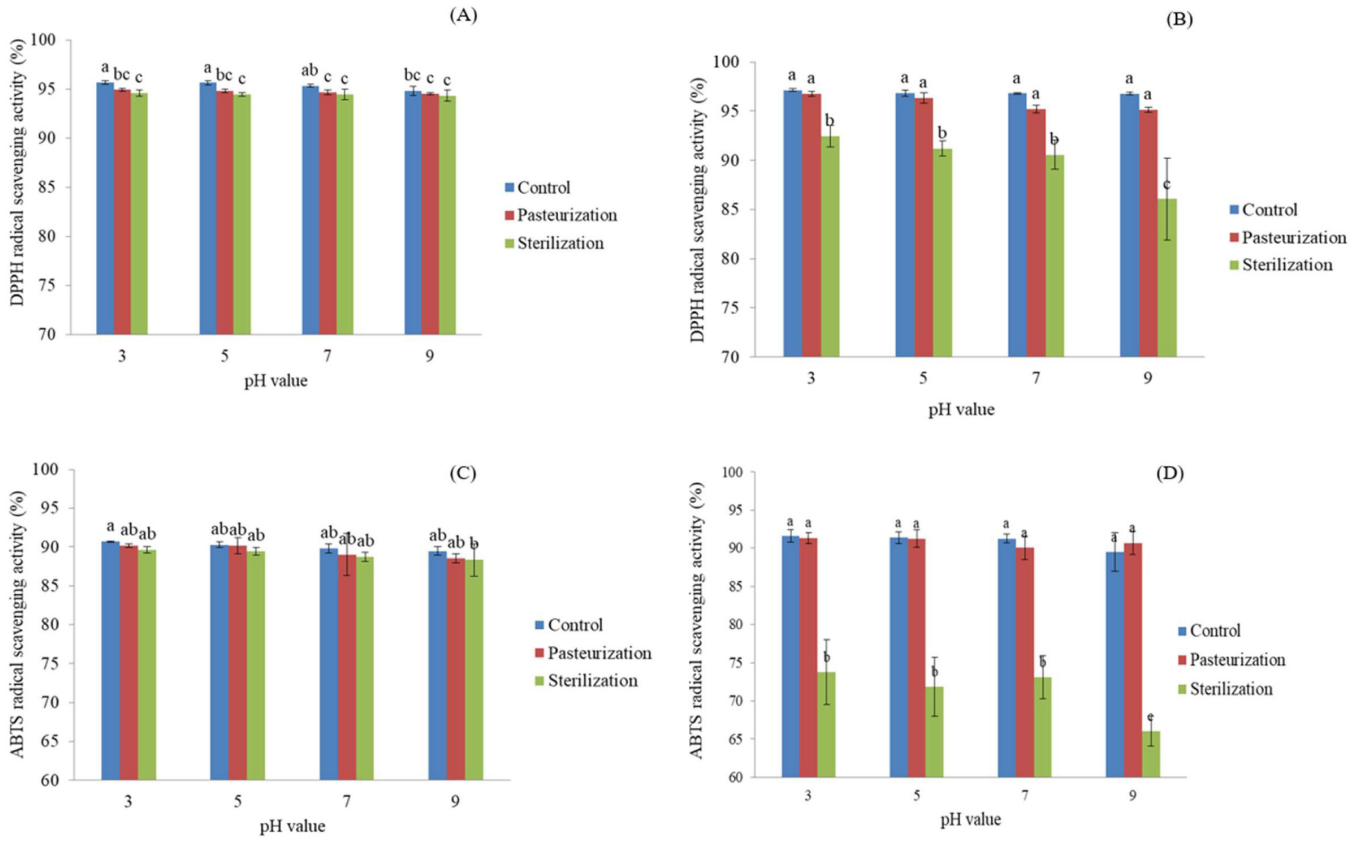

Fig. 8 Radical scavenging activities of pigment powders under different heat treatments at pH ranging of 3–9: (A) 2,2-diphenyl-1-picrylhydrazyl (DPPH) inhibition at 180°C and maltodextrin at dextrose equivalent (DE)10; (B) DPPH inhibition at 210°C and maltodextrin DE15; (C); 2-2'-azino-bis (3-ethyl-benzothiazoline-6-sulfonic acid) diammonium salt (ABTS) inhibition at 180°C and maltodextrin DE10); (D) ABTS inhibition at 210°C and maltodextrin DE15), where different lowercase letters above columns indicate significant ($p < 0.05$) differences and error bars indicate ± SD.

Thus, the pigment powders obtained from the lowest inlet air temperature (180°C) combined with the maltodextrin DE 10 encapsulation contributed to desirable physical properties, specifically that the red and green value (a*) and color intensity (C*) were higher than for the other drying conditions, whereas lightness (L*) tended to decrease. Furthermore, solubility and bulk density were not significantly different from the other drying conditions. In addition, these encapsulation conditions provided the highest contents of anthocyanins and phenolic compounds, although a decrease in both compounds was observed due to thermal degradation, which resulted in a decrease in the antioxidant activity using the DPPH method. However, there was stability of the encapsulated black bean powders as described by the co-pigment effect among anthocyanins, maltodextrin and phenolic compounds, particularly using DE10 maltodextrin at 180°C air-drying were used as the encapsulation conditions. This effect resulted in the retention of the TPC and radical scavenging antioxidant activities based on both the DPPH and ABTS methods under various conditions. Therefore, it could be concluded that encapsulation techniques with a low DE value of maltodextrin and an inlet air temperature below 200°C provided high stability of bioactive compounds in the pigment powders from black beans, which, subsequently, could be used in food products in a broad pH range (3–9) in high-temperature processes, such as pasteurization and sterilization.

Conflict of Interest

The authors declare that there are no conflicts of interest.

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