https://doi.org/10.48048/tis.2024.7247

Influence of Spray Drying Inlet Temperature on the Physical Properties and Antioxidant Activity of Black Garlic Extract Powder

Novita Wijayanti[1,*], Tri Dewanti Widyaningsih[1], Siti Narsito Wulan[1] and Muhaimin Rifai[2]

[1]*Department of Food Science and Biotechnology, Faculty of Agricultural Technology, Brawijaya University, Malang, Indonesia*
[2]*Department of Biology, Faculty of Mathematics and Natural Sciences, Brawijaya University, Malang, Indonesia*

([*]Corresponding author's e-mail: n_wijayanti@ub.ac.id)

Received: 25 June 2023, Revised: 20 July 2023, Accepted: 25 August 2023, Published: 25 December 2023

Abstract

Black garlic is a fermented product from garlic (*Allium sativum* L.) that is processed by heating fresh garlic at a certain temperature and humidity for several days. Black garlic has higher antioxidant activity and is rich in organosulfur compounds. Therefore, black garlic has an unpleasant taste and odor. Spray drying of black garlic extract is important for wider application, preservation and prolongation of its shelf-life. Black garlic powders were produced using SD-18A mini spray dryer. The purpose of this research was to evaluate the effect of inlet temperatures on the physical properties and antioxidant activity of aqueous black garlic powder. The inlet temperatures ranged from 165 to 225 °C, whereas other conditions were kept constant such as maltodextrin concentration (8 %) and feed flow rate (4.5 mL/min). The black garlic powders were examined for powder yield, moisture content, hygroscopicity, bulk density, wettability, antioxidant activity and total phenolic and flavonoid content. The optimal conditions for the drying process were achieved with an inlet temperature of 225 °C. An increase in the inlet temperature of the spray dryer has increased the powder yield, hygroscopicity, antioxidant activity and total phenolic and flavonoid content of the black garlic powder, but reduced the water content, bulk density and wettability of the spray-dried product.

Keywords: Antioxidant activity, Black garlic, Inlet temperature, Maltodextrin, Spray drying

Introduction

Black garlic is a fermented product made from fresh garlic that is aged for several days at a temperature between 65 to 80 °C and a relative humidity between 70 to 80 % [1]. This process will increase polyphenol and flavonoid content [2], as well as S-allyl-L-cysteine [3], in black garlic when compared to fresh garlic. The antioxidant activity in black garlic is also higher than fresh garlic [4].

Black garlic is rich in organosulfur compounds that have an unpleasant smell and odor. The aqueous extract of black garlic can be processed into a soluble powder that can be consumed as an instant drink as well as a herbal supplement. Therefore, it is necessary to process the black garlic extract by encapsulation. This method also has the benefit of protecting the bioactive compounds in black garlic.

Encapsulation is the procedure of transforming tiny droplets or liquid particles into tiny dried particles known as microcapsules [5]. During the encapsulation procedure, it is important to protect the bioactive compounds using the appropriate encapsulating agent as a coating material [6]. The mostly used coating material is maltodextrin because it is more effective, cheaper and colorless. It also has low viscosity at high solids and good water solubility. Furthermore, it is bland in flavor and available in a variety of molecular weights [7]. Spray drying using maltodextrin as a coating material has the advantage of producing powders with lower water content compared to guar gum, Arabic gum, inulin, pectin and alginate [8].

Inlet temperature has an impact on powder attributes such as moisture content, bulk density, particle size, hygroscopicity and morphology. The increased drying temperature reduces moisture content, bulk density, powder yield and hygroscopicity [9]. The input temperature for the spray-drying method is between 150 to 220 °C. Several experiments have shown the effect of spray drying inlet temperature on the encapsulation process. The inlet temperature of 150 °C for encapsulation of Jamun fruit juice powder gave a higher powder yield [10]. The inlet temperature of 190 °C in skimmed coconut milk powder produces the optimum yield [11]. Spray-dried Sapodilla processed at a 200 °C inlet temperature shows higher total phenolic content [12]. Therefore, the research aimed to evaluate the effect of different inlet temperatures (165, 180, 195, 210 and 225 °C) on the physical properties and antioxidant activity of the spray-dried black garlic powder. The results of this research would be useful in developing black garlic herbal supplements.

Materials and methods

Materials and chemicals

Garlic (*Allium sativum* L.) variety Lumbu Hijau was obtained from a local farmer in Malang, East Java, Indonesia. Maltodextrin was obtained from Xingmao. Acetone, acetic acid, aluminum chloride, ethanol, $FeCl_3.6H_2O$, Folin Ciocalteau, hydrochloric acid, methanol, sodium acetate, sodium carbonate, sodium chloride, sodium hydroxide and sodium nitrite were purchased from Merck (Germany). The chemicals ABTS (2,2'-Azino-Bis-3-Ethylbenzothiazoline-6-Sulfonic Acid), DPPH (2,2-diphenyl picryl hydrazyl), gallic acid, potassium persulfate, quercetin, Trolox (6-hydroxy-2,5,7,8-tetramethylchroman-2-carboxylic acid) and TPTZ (2,4,6-tripyridyl-S-triazine) were obtained from Sigma Chemicals (St. Louis, USA).

Preparation of raw materials

The fresh garlic (*Allium sativum* L.) was cleaned with tap water. The garlic was placed in a rice warmer machine at 73 ± 2 °C for 15 days. Then, the black garlic was peeled to remove the skin, and the pulp was ground into a paste.

Water soluble extract preparation

The black garlic paste was weighed and homogenized with 10 mL/g of water to get the black garlic extracts. The mixture was extracted using the infused technique for 60 min at 100 °C. The black garlic extract was then filtered using filter paper to remove the residue. The extracted solution was maintained in a closed bottle and kept at 4 °C before was used for the further experiment.

Drying condition

Maltodextrin was dissolved in black garlic extract with a concentration of 8 % (w/v) and then filtered using a sieve (80 mesh) to remove insoluble particles that could block the nozzle of a spray dryer. Black garlic extract and maltodextrin were spray-dried at 165, 180, 195, 210 and 225 °C with the same intake air volume of 60 % and feed flow rate of 4.5 mL/min. For further investigation, the resulting powders were kept in opaque glass vials.

Product yield

The product yield of the black garlic powder was calculated by using the following Eq. (1), as shown below [13].

$$\text{Product yield} = \frac{\text{Weight of product}}{\text{Weight of ingredient}} \times 100\ \% \tag{1}$$

Moisture content

The black garlic powder moisture content was measured by the hot air oven method [14]. One g of black garlic powder was weighed on a petri dish and dried in an oven at 100 °C for constant weight. The samples were weighed after cooling in the desiccator. The product moisture content was calculated by using the following Eq. (2), as shown below.

$$\text{Moisture content} = \frac{\text{initial weight} - \text{final weight}}{\text{final weight}} \times 100\ \% \tag{2}$$

Hygroscopicity

The hygroscopicity measurement was modified from Cai and Corke [15]. Black garlic powder was weighed at approximately 0.5 g and placed in a pre-weighed 10 mL glass. The powder was then placed in a glass desiccator maintained at a relative humidity of 75 % by filling the bottom part of the desiccator with a saturated sodium chloride solution (36.5 g/100 mL) [15]. The powder was then weighed after 5 h. The hygroscopicity was calculated using the following Eq. (3), as shown below.

$$\text{Hygroscopicity} = \frac{\text{weight after storage} - \text{weight before storage}}{\text{weight before storage}} \times 100\ \% \tag{3}$$

Bulk and tapped densities

The bulk density of black garlic powder was measured by adding 1.0 g of black garlic powder into a graduated measuring cylinder of 10 mL before the initial volume was recorded. The cylinder was tapped 100 times, and the volume of the sample was recorded to determine the tapped density of black garlic powders. As shown below, the bulk and tapped densities were calculated using the following Eqs. (4) and (5).

$$\text{Bulk density} = \frac{\text{Weight of black garlic powder (g)}}{\text{Initial volume (mL)}} \tag{4}$$

$$\text{Tapped density} = \frac{\text{Weight of black garlic powder (g)}}{\text{Initial volume after tapped (mL)}} \tag{5}$$

Cohesiveness

The cohesiveness values of the black garlic powders were measured in terms of the Hausner ratio (HR). The HR was calculated from the bulk density and tapped density of the black garlic powder by using the following Eq. (6), as shown below.

$$\text{Hausner ratio} = \frac{\text{tapped density}}{\text{bulk density}} \tag{6}$$

Different ranges for the HR in defining cohesiveness are below 1.2, low cohesiveness; 1.2 < HR < 1.4, intermediate cohesiveness; and above 1.4, high cohesiveness.

Flowability

The flowability values of the black garlic powders were measured in terms of Carr index (CI). CI was calculated from the bulk density and tapped density of the black garlic powder by using the following Eq. (7), as shown below.

$$\text{Carr Index} = \frac{\text{tapped density} - \text{bulk density}}{\text{tapped density}} \tag{7}$$

Excellent flowability can be reached if CI is below 15 %; good flowability when CI is between 15 - 20 %; fair flowability when CI is between 20 - 35 %; bad flowability when CI is between 35 - 45 %; and poor flowability when the CI is above 25 %.

Wettability

Wettability was calculated based on the solubility of black garlic powder in water. This analysis was modified from Duangchuen *et al.* [11] as follows: 1.0 g of black garlic powder was poured into a 250 mL beaker glass containing 100 mL of water at 25 °C. The time duration for black garlic powder to completely immerse in water was measured.

Total phenol content

The total phenolic content of black garlic powder was measured by the Folin-Ciocalteu method with slight modifications. Briefly, 1 mL of the sample was mixed with 2 mL of Folin-Ciocalteau 10 %, and the mixture was incubated for 2 min at room temperature. Subsequently, 2 mL of sodium carbonate (75 g/L) in water was added. The mixture was incubated at room temperature for 30 min. Finally, the absorbance was measured at 765 nm against a blank using a spectrophotometer, and the results are expressed as mg GAE/g dry weight. Gallic acid at a concentration of 10 - 50 µg/mL was used as the standard curve [16].

Total flavonoid content

The total flavonoid content of black garlic powder was measured by the $NaNO_2$-$AlCl_3$-NaOH method [25] with some modifications. Briefly, 0.5 mL of samples were diluted with 2.0 mL of distilled water. Then, it was mixed with 0.15 mL of $NaNO_2$ 5 % (w/v) solution. Five min later, 0.15 mL of $AlCl_3$ 10 % (w/v) was added, and the mixture lasted for 6 min. Then, 1.0 mL of NaOH 1 M solution was added, and the total volume was made up to 5.0 mL with distilled water. Then, the mixture was thoroughly mixed and incubated for 30 min at room temperature. Finally, the absorbance was measured immediately at 325 nm against a blank using a spectrophotometer, and the results are expressed as mg QE/g dry weight. Quercetin was used as the standard curve at a concentration of 10 - 100 µg/mL.

DPPH assay

The DPPH radical scavenging activity of black garlic powder was measured using a colorimetric method [18] with certain modifications. Firstly, 0.1 mL of the sample was mixed with 2.9 mL of DPPH at 0.03 mg/mL in methanol. Then, the mixture was incubated for 20 min at room temperature in the dark. The mixture was centrifuged for 10 min at 6,000 rpm. Finally, the absorbance of the supernatant was measured immediately at 517 nm against a blank (absolute methanol). Trolox (concentration 25 - 150 μg/mL) was used as the standard curve. The result was expressed as mg TE/g dry weight. The DPPH reagent was prepared daily and freshly.

ABTS assay

The ABTS radical scavenging activity of black garlic powder was measured using the colorimetric method [18], with minor modifications. Firstly, 0.5 mL of ABTS solution (7.4 mM) was reacted with 0.5 mL of potassium persulfate solution (2.6 mM). The mixture was incubated for 12 h in the dark. Then, the mixture was diluted by mixing 1 mL of ABTS with 50 mL of methanol in a volumetric flask. A fresh ABTS reagent was prepared and used for each assay. Furthermore, 0.1 mL of the sample was mixed with 2.9 mL of ABTS reagent in a dark room for 20 min, and the mixture was later centrifuged for 10 min at 6,000 rpm. Finally, the absorbance of the supernatant was measured immediately at 746 nm using a spectrophotometer against a blank. Trolox (concentration 25 - 150 μg/mL) was used as the standard curve. The result was expressed as μg TE/g dry weight.

FRAP assay

The FRAP assay was determined by a colorimetric method [18] with some modifications. A fresh FRAP reagent was prepared by mixing 1 mL of $FeCl_3.6H_2O$ 20 mM solution with 1 mL of TPTZ solution 10 mM (HCl 40 mM solution as the solvent) and 100 mL of acetate buffer 300 mM (pH = 3.6). Briefly, 0.1 mL of samples were mixed with 2.9 mL of freshly prepared FRAP reagent. Then the reaction mixture was incubated at 37 °C for 15 min in the dark. Lastly, the absorbance of the solution was measured immediately at 597 nm against a reagent blank. Trolox (concentration 25 - 150 μg/mL) was used as the standard curve.

Statistical analysis

All the experiments were carried out in 3 replications and results were expressed as mean ± standard deviations. The analysis of variance (ANOVA) was performed using Minitab version 18. The differences between mean values were compared using the Fisher LSD test with a significance level of $p < 0.05$.

Results and discussion

Black garlic extract was spray-dried using maltodextrin of 8 % (w/v) as coating material. The physicochemical properties of spray-dried black garlic powder are shown in **Table 1**. The spray dryer inlet temperature was optimized, while other parameters such as intake air volume (60 %) and feed flow rate (4.5 mL/min) were kept constant. The range of inlet temperatures varied from 165 to 225 °C.

Table 1 Powder yield and physicochemical properties of spray-dried black garlic powder.

Parameters	**Inlet Temperature (°C)**				
	165	**180**	**195**	**210**	**225**
Product Yield (%)	3.151 ± 0.221^d	4.909 ± 0.256^c	6.145 ± 0.039^b	6.402 ± 0.175^{ab}	6.566 ± 0.107^{ab}
Moisture content (%)	4.022 ± 0.339^a	3.819 ± 0.525^a	2.663 ± 0.176^b	2.582 ± 0.281^b	2.580 ± 0.107^b
Hygroscopicity (%)	2.270 ± 0.175^b	2.303 ± 0.093^b	2.628 ± 0.176^a	2.719 ± 0.092^a	2.655 ± 0.085^a
Bulk density (g/mL)	0.334 ± 0.001^a	0.334 ± 0.001^a	0.313 ± 0.012^b	0.292 ± 0.011^c	0.268 ± 0.015^d
Tapped density (g/mL)	0.423 ± 0.010^a	0.423 ± 0.001^a	0.390 ± 0.026^b	0.376 ± 0.008^b	0.345 ± 0.019^c
Cohesiveness	1.268 ± 0.031^a	1.268 ± 0.031^a	1.247 ± 0.028^a	1.288 ± 0.025^a	1.287 ± 0.012^a
Flowability (%)	21.111 ± 1.924^a	21.111 ± 1.925^a	19.792 ± 1.804^a	22.325 ± 1.541^a	22.307 ± 0.731^a
Wettability (s)	856 ± 25^a	726 ± 13^b	655 ± 70^c	599 ± 20^c	405 ± 30^d

Product yield

The product yield of the black garlic powder ranged between 3.151 and 6.566 %. The inlet temperature showed significant ($p < 0.05$) influences on the product yield of the black garlic powder. The product yield of the black garlic powder increased by 52 % when the inlet air temperature was raised from 165 to 225 °C. Similar results like influence of the inlet temperature on product yield were previously observed in the case of spray-dried black mulberry juice [19] and Cagaita powder [20]. Powder yield increased with increasing inlet air temperature, which is likely due to improved water removal at higher temperatures, improved heat and mass transfer efficiency, and the avoidance of material sticking to the dry chamber wall [19].

Moisture content

According to **Table 1**, the moisture content of black garlic powder ranged from 2.580 to 4.022 %. Black garlic powder moisture contents were generally lower than 5 %, similar to those reported for spray-dried pink guava (2.22 - 4.46 %) [21], watermelon (1.49 - 2.78 %) [22] and guava powder (2.15 - 3.87 %) [23]. Black garlic powder spray-dried at 165 °C had the highest moisture content (4.022 %), whereas the powder heated at 225 °C had the lowest value (2.580 %). The result showed that the moisture content of black garlic powders decreased rapidly as the air inlet temperature rose from 165 to 225 °C (**Table 1**). Similar trends were also reported in spray-dried lemon grass [24], noni fruit [25], pink guava [26] and blackberry [27] powders. Elevation of the temperature gradient between the drying air and the atomized feed caused a higher rate of heat transfer into particles, increasing the driving force behind water evaporation [25], which resulted in faster water vaporization and a higher loss of water in spray-dried powder [28]. Consequently, it leads to the generation of powdered products with reduced moisture content.

Hygroscopicity

Hygroscopicity refers to the ability of a particle to absorb humidity from the surrounding environment, and an essential characteristic to be concerned about in food processing due to its impact on food stability [20]. The hygroscopicity of black garlic powder values ranged from 2.270 to 2.719 % (**Table 1**). Similar findings were reported by Bhusari *et al.* [29], who worked with the spray drying of tamarind pulp. Increasing the spray drying temperature led to a decrease in moisture content, and the encapsulated powder had more hygroscopicity. The inlet air temperature favorably impacted hygroscopicity. The maximum hygroscopicity values for black garlic powder were attained with higher drying temperatures. Lower powder moisture concentrations indicated higher hygroscopicity. Moisture content and hygroscopicity values have an inverse correlation [27]. Powders with a lower moisture content have a higher potential to absorb ambient moisture due to the higher water concentration differential between the powder particles [30].

Bulk and tapped densities

Bulk density is a crucial characteristic of powder materials because it impacts the size of the packaging, which in turn affects handling and shipping costs [31]. According to our result in **Table 1**, the bulk density of black garlic powder varied from 0.268 to 0.334 g/mL. The inlet temperature indicated a significant effect on the bulk density of the black garlic powder. According to the experiment of Nadeem *et al*. [32], the bulk density of sage powder was between 0.324 - 0.352 g/mL. The bulk density of the black garlic powder decreased when the inlet temperature of the spray dryer was increased. Similar observations also occurred in the drying process of guava powder [21], acai juice powder [30], sweet orange juice [33], amaranthus betacyanin [15], san sakng leaf [34], instant herbal tea [35] and amla juice powder [36]. On the other hand, this result followed a different trend with spray-dried kuini powder [13]. The bulk density of kuini powder increased when the inlet temperature increased from 140 to 160 °C, but then dropped when the inlet air temperature was further increased to more than 170 °C.

Tapped density is a necessary parameter of powder material because it has a relationship with the packaging, transportation and trading of powders. This value can be beneficial regarding the weight and number of products that will fit into a storage box. A high-density material can be stored in a smaller box than a low-density product. This characteristic was influenced by the spray drying temperature, showing a negative linear effect [37]. Based on **Table 1**, the tapped density of black garlic powder varied from 0.345 to 0.423 g/mL. The inlet temperature indicated a significant effect on the tapped density of the black garlic powder. This value was similar to the tapped density of sweet orange juice powder from 0.312 to 0.474 g/mL [33]. Moreover, the tapped density of black garlic powder was 0.423 g/mL when using an inlet temperature of 165 °C, while it was 0.345 g/mL when using an inlet temperature of 225 °C. The tapped density of the black garlic powder decreased when the inlet temperature of the spray dryer was increased.

This trend was also like the spray drying process of *Murraya koenigii* L. leaf [38] and jamun fruit [10]. On the contrary, the tapped density of this experiment showed an opposite trend with spray-dried Gypsophila extract powder [39].

The heavier the product, the easier it is to accommodate into the space between the powder particles, thus occupying less space and producing a greater bulk density [30]. It was found that higher input air temperatures accelerated evaporation rates, which led feed mass to dry more quickly and produced porous, fragmented products that had a lower bulk density [33]. Additionally, increasing the drying temperature results in particle inflation (puffing or ballooning), which results in larger expansion of the droplets, increases particle size and therefore reduces powder density [40].

Cohesiveness and flowability

The HRs of black garlic powder were observed in the range of 1.247 to 1.288. The HR was considered fair powders or intermediate cohesiveness as per classification. A lower the HR means that the powder has more ability to flow freely and is less cohesive [37]. CI of black garlic powder was evaluated from 19.792 to 22.235 %. CI was considered fair and had good flowability as per classification. The bulk density and tapped density would be similar in value to gain a lower CI, so the powder category was free-flowing powder with excellent flowability. Whereas, the greater the difference between bulk density and tapped density, the higher CI. Therefore, such powder was categorized as a poor-flowing powder [33].

Wettability

Wettability refers to the ability of the powder to break through the surface tension between the dry powder and water. Large pore gaps for large particles or high-porosity particles exhibit strong wettability [41]. Wettability was measured by using the solubility of black garlic powder in water. This method measures the wettability of the powder by the duration of time required to achieve total wetting of a certain amount of the powder. It is being tested starting from when the powder is dropped gently on the surface of the water until it gets submerged in water without agitation [11].

The water could penetrate 1 g of black garlic powder in 405 - 856 s. The wettability of spray-dried black garlic powder showed a significant difference ($p < 0.05$) between samples. Black garlic powders with an inlet temperature of 165 °C showed the highest wettability values (856 s), whereas the highest inlet temperature (225 °C) showed the lowest value (405 s). The results confirm that wettability decreased with a higher inlet temperature. A similar trend was reported in spray-dried jamun fruit juice (82 - 116 s) [10] and skimmed coconut milk powder (489 - 1,201 s) [11].

Wettability is inversely linked to particle size. Larger particles of powder have more space between them. The spaces between large particles in powder allow for greater interactions with the surrounding water molecules. On the other hand, smaller particles have reduced porosity, which makes it more difficult for water to enter the food matrix and leads to poor reconstitution qualities [10]. This result followed the reverse trend with spray-dried kuini powder [13]. The wettability time increased with increasing inlet temperature, indicating that it takes more time for water to penetrate the particles. The spray-dried product's quality depends on improved powder wettability, a property that may be optimized with the right parameter selection and drying method. This characteristic is influenced by several variables, such as air-drying temperature, particle size [10], the product's chemical composition, reconstitution water temperature and analytical physical parameters [30].

Table 2 Antioxidant activity, total phenol and flavonoid content of spray-dried black garlic powder.

Parameters	Inlet Temperature (°C)				
	165	180	195	210	225
Total phenol content (mg GAE/g DW)	5.077 ± 0.105^{d}	5.624 ± 0.111^{c}	5.854 ± 0.147^{c}	6.119 ± 0.130^{d}	6.566 ± 0.143^{a}
Total flavonoid content (mg QE/g DW)	1.502 ± 0.035^{b}	1.625 ± 0.013^{a}	1.628 ± 0.022^{a}	1.628 ± 0.046^{a}	1.658 ± 0.011^{a}
DPPH assay (mg TE/g DW)	0.295 ± 0.011^{c}	$0.313 \pm 0,002^{b}$	0.319 ± 0.004^{b}	0.334 ± 0.010^{a}	0.338 ± 0.008^{a}
ABTS assay (mg TE/g DW)	1.155 ± 0.026^{c}	1.225 ± 0.012^{b}	1.286 ± 0.038^{a}	1.293 ± 0.030^{a}	1.328 ± 0.036^{a}
FRAP assay (mg TE/g DW)	0.419 ± 0.007^{c}	0.472 ± 0.017^{b}	0.490 ± 0.021^{ab}	0.493 ± 0.009^{ab}	0.504 ± 0.022^{a}

Total phenol content

The total phenol content of black garlic extract powders is presented in **Table 2**. In general, the total phenol content of the black garlic powder was increased by increasing the spray-drying temperature. Total phenol content increased from 5.077 to 6.566 mg GAE/g DW when temperature increased from 165 to 225 °C. The highest and lowest total phenol contents were measured at 165 and 225 °C, respectively. A similar observation in spray-drying Sapodilla powder was reported [12]. Indeed, when the temperature increased from 140 to 220 °C, the total phenol content of spray-dried sapodilla powder increased from 1.071.22 to 1.266.78 mg GAE/100 g. The different trends that occurred in the experiment with spray-dried rapeseed honey powder [42], amla juice powder [36], Asian pear juice powder [43], noni extract powder [25] and lemon grass leaf extract powder [24], the total phenol content decreased at higher temperatures.

The increasing spray dryer inlet temperature increased the total phenol content of black garlic extract. The reason was due to the faster drying process at higher temperatures, which caused shorter exposure times and thus lesser degradation of the heat-sensitive phenolic compounds [44]. In addition, it may be caused by the polymerization as well as the synthesis of polyphenols at higher drying temperatures, which increase the total phenol content of the powder [36].

Total flavonoid content

Based on **Table 2**, the total flavonoid content of black garlic extract powder was between 1.502 to 1.658 mg QE/g. Increasing spray-drying temperatures led to an increase in the total flavonoid content of black garlic extract powder. When the temperature was elevated from 165 to 225 °C, total flavonoid content increased from 1.502 to 1.658 mg QE/g. The highest total flavonoid content was achieved at 225 °C, and the lowest one was collected at 165 °C. The total flavonoid content of black garlic powder increased with increasing temperatures of hot air dryer [45]. This trend could be explained by the fact that the flavonoid compound in black garlic extract has stable conditions at high temperatures.

A different result was found in the research of Tran and Nguyen [24], in which the total flavonoid content of Lemongrass leaf extract powder decreased from 541.82 to 258.2 mg CE/100 g dry weight when the spray-drying inlet temperature increased from 110 to 150 °C.

DPPH assay

DPPH is a free radical reagent that is frequently used to measure the radical-scavenging capacity of antioxidant components. This method is based on the presence of H^+ ion donors in bioactive compounds that lead to the reduction and production of DPPH-H (a non-radical form). The color of protonated DPPH molecules is changed to 1,1-diphenyl-2-picrylhydrazine due to the presence of a proton donor from an antioxidant compound [46].

Table 2 showed that the DPPH scavenging activity of the black garlic powders increased with increasing spray-drying temperatures. Indeed, the powders produced at 210 and 225 °C had the highest antioxidant activity, whereas the ones dried at 165 °C had the lowest values. Nadeem *et al*. [32] reported that the antioxidant capacity of sage powder significantly increased from 130 µmol TE/g dry weight to 145 µmol TE/g dry weight as the inlet temperature raised from 145 to 165 °C [38]. Tonon *et al*. [30] also recorded the same trend in spray-dried acai juice powder.

The different gradients of DPPH scavenging activity occurred with spray-dried amla powder [36] and Asian pear juice powder [43]. There was a significant decrease in the DPPH radical scavenging activity of amla powder when the inlet temperature increased from 125 to 200 °C. The possible explanation for the high free radical scavenging activity may be because of the exposure to higher temperatures, which adversely affected the structure of phenols and flavonoids, causing their polymerization as well as their synthesis into different forms. It increases the DPPH scavenging activity of the black garlic powder [36].

ABTS assay

The principle of the ABTS assay is the reaction of hydrogen peroxide and metmyoglobin to generate the ferry 1 myoglobin radical, which changes the phenothiazine complex ABTS (2,2'-azinobis-(3-ethylben-thiazoline-6-sulphonic acid) to a radical cation ($ABTS^{\bullet+}$). Antioxidant activity determines the degree and timing of the radical cation's suppression in the presence of antioxidants [47]. Elevated inlet temperatures increased the ABTS scavenging activity of black garlic extract powder (**Table 2**). When the sprayed dried temperature increased from 165 to 225 °C, ABTS scavenging activity increased from 1.155 to 1.328 mg TE/g. Similar findings were reported by Lee *et al*. [43], who worked with the spray drying of Asian pear juice. Increasing the spray drying temperature from 130 to 170 °C increased ABTS scavenging activity.

The results might relate to the phenolic and flavonoid content as described previously. Radical scavenging activity depends on the kinds of phenol and flavonoid compounds present in the product.

Different products have different kinds of phenolic and flavonoid compounds, so high-temperature treatment can reduce or increase antioxidant activity depending on these compounds. The ABTS scavenging activity of blackberry powder decreased when the inlet temperature increased from 140 to 160 °C [48]. Similar results are observed in Gac fruit powder [28].

FRAP assay

The principle of antioxidant activity using FRAP assay of bioactive compounds in black garlic powder was the reduction of the Fe^{3+} (ferricyanide) complex into Fe^{2+} (ferrous), which acts as a potential indicator for antioxidant activity [49]. The FRAP antioxidant activity of black garlic extract powder is shown in **Table 2**. Black garlic powders with an inlet temperature of 165 °C showed the lowest FRAP antioxidant activity (0.419 mg TE/g), whereas those with an inlet temperature of 225 °C showed the highest value (0.504 mg TE/g). In general, the FRAP antioxidant activity of the black garlic powder was increased by increasing the inlet temperature of the spray dryer. However, this study's results were similar to drying process of New Zealand Tamarillo extract powder. Increasing the inlet temperature from 110 to 160 °C significantly increased the FRAP antioxidant activity of New Zealand Tamarillo powder from 80.37 to 91.05 mg TE/g [50]. In contrast, the FRAP antioxidant activity of this experiment showed an opposite trend in spray-dried bitter melon extract powder [51] and camelina extract powder [52]. Based on the experimental values, it was found a correlation between the concentration of phenolic compounds and antioxidant activity. This correlation could be explained by the fact that high spray-drying temperatures may be associated with the activation of some phenolic compounds, resulting in an increase in the antioxidant capacity of the powder [30].

Conclusions

The results of this study showed that black garlic powder could be produced using the spray drying technique. The optimum inlet temperature for spray-dried black garlic extract powder was 225 °C had the highest yield (6.57 %), the lowest moisture content (2.58 %), the lowest bulk density (0.27 g/mL), and the lowest wettability level (405 s). According to the HR and CI, black garlic powder in this research was categorized as having intermediate cohesiveness and good flowability. The hygroscopicity value was 2.65 respectively. The antioxidant activity through DPPH, ABTS and FRAP was determined as 0.339, 1.328 and 0.504 mg TE/g, respectively.

Acknowledgements

This research was supported by a research grant from Brawijaya University, Malang, Indonesia.

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