



Influence of Carrying Agents on the Physicochemical Properties of Freeze-Dried Black Garlic Powder as a Functional Ingredient

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Abstract

Black garlic is widely recognized for its high antioxidant content and potential health benefits, making it a valuable functional ingredient in the food and pharmaceutical industries. This study investigated the effects of different carrier agents maltodextrin, isomaltodextrin, and fructooligosaccharide at concentrations of 20%, 25%, and 30% on the physicochemical properties of freeze-dried black garlic powder. The parameters evaluated included total soluble solids, color, moisture content, water activity, bulk density, flowability, and antioxidant activity (DPPH, ABTS, and FRAP). The results indicated that both the type and concentration of the carrier significantly influenced ($p < 0.05$) the quality of black garlic powder. Among the tested carriers, 25% maltodextrin showed the greatest potential by improving powder stability, reducing moisture content (4.53 %w.b.), enhancing flowability (33.14°), and maintaining high antioxidant activity. These findings provide valuable insights into optimizing carrier selection for the production of black garlic powder with improved quality and stability.

Keywords: Black garlic, Freeze-drying, Carrier

1 Introduction

Garlic (*Allium sativum* Linn.) has been widely recognized for its numerous health benefits, including its ability to reduce cholesterol levels, regulate blood pressure, and enhance immune function (Alvino Granados et al., 2021). However, fresh garlic has a limited shelf life and a pungent odor that may be undesirable for some consumers. To overcome these challenges, fermentation has been widely used to produce black garlic, a functional food ingredient with enhanced bioactivity and improved sensory characteristics (Kaewpiboon & Boonnak, 2020). During processing, black garlic undergoes a non-enzymatic browning reaction known as the Maillard reaction, resulting in the formation of bioactive compounds such as polyphenols, flavonoids, and S-allyl cysteine, which contribute to its strong antioxidant activity (Kim et al., 2013).

Despite its potential as a functional food, black garlic presents challenges in processing and storage due to its sticky texture and high sugar content (Orefice et al., 2022). These issues become particularly

pronounced during powder production, where hygroscopic properties can lead to clumping and caking, thereby reducing flowability and shelf stability. To address these concerns, freeze-drying has been identified as an effective drying technique because it retains bioactive compounds and minimizes thermal degradation (Shrestha et al., 2007). However, the presence of low-molecular-weight sugars in black garlic contributes to increased stickiness, particularly when the product approaches its glass transition temperature (Tg) (Alvino Granados et al., 2021).

To enhance the physical stability of freeze-dried black garlic powder, carrier agents are commonly employed. These carriers prevent caking, improve powder flow properties, and enhance the reconstitution ability of the final product (Fang et al., 2008). Moreover, some carriers may provide additional health benefits, aligning with the growing demand for functional food ingredients. However, limited studies have systematically investigated the influence of different carrier agents and their concentrations on the physicochemical properties and stability of freeze-dried black garlic powder. Therefore, further

investigation is needed to optimize carrier selection for improving powder quality and functionality.

The objective of this study is to evaluate the effects of different carrier agents, maltodextrin (MD),

2 Material and Methods

2.1 Material

Black garlic (*Allium sativum* L.) was provided by GRAZIE & AVIKA Co., Ltd., Thailand. Maltodextrin (DE 10-11, Maxway Co., Ltd., Thailand), Isomaltodextrin (Hayashibara Co., Japan), Fructooligosaccharide (COSUCRA, Belgium), 2,2-Diphenyl-1-picrylhydrazyl (DPPH), 2,2'-azino-bis (3-ethylbenzthiazoline-6-sulphonic acid) (ABTS), 2,3,5-Triphenyltetrazolium chloride (TPTZ) and 6-Hydroxy-2,5,7,8-tetramethylchroman-2-carboxylic acid (Trolox) were purchased from Thermo Fisher Scientific Co., Ltd., Japan. All other chemicals and reagents were of analytical grade.

2.2 Freeze-dried black garlic powder preparation

Black garlic, prepared at room temperature was blended using a manual industrial mincer (SW98, Kolbe Foodtec, Germany). Each of maltodextrin (MD), isomaltodextrin (IMD), and fructooligosaccharides (FOS) was prepared as a 50% (w/w) solution. The carrier solutions were then mixed with finely ground black garlic at black garlic to carrier solution ratios of 60:40, 50:50, and 40:60 (w/w), respectively, to obtain final formulations containing 20%, 25%, and 30% (w/w) carrier solids. The resulting samples were designated as MD 20, MD 25, and MD 30, depending on the carrier solid content. The black garlic samples were dehydrated using a freeze dryer (Kryo 'D' Freezer, ITC, Thailand) as shown in Fig. 2. The samples were loaded into trays and spread to a thickness of 1 cm from the tray bottom. The samples were frozen at -50 °C, and the core temperature was monitored using an RTD probe. During the primary drying stage, the system was

isomaltodextrin (IMD), and fructooligosaccharides (FOS), at varying concentrations on the physicochemical properties and stability of freeze-dried black garlic powder.

operated at a pressure below 40 Pa, while the shelf temperature was gradually increased from -20 °C to 20 °C. In the secondary drying phase, the shelf temperature was further increased to 40 °C (Fig. 3). The dried product was ground and stored in aluminum foil bags under controlled conditions at 25 °C and 30-40% relative humidity until further analysis.

2.3 Total soluble solids content (TSS)

For the TSS 1 g of sample was dissolved in 10 mL distilled water. And mixed for 1 min, the solution was measured using digital hand pocket refractometer (PAL-1, ATAGO, Japan). The TSS value was then multiplied by the number of dilutions to obtain the final TSS value.

2.4 Moisture content (M.C.)

The moisture content was determined by the hot air oven (AOAC, 2000). Samples were dried at 70 °C until a constant weight was achieved (defined by three consecutive readings) Then, the following relation was used:

$$\text{Moisture content} = \frac{(W1-W2)}{W1} \times 100 \quad (1)$$

Where: W1 = initial weight of the sample before drying (g); W2 = final weight of the sample after drying (g)

2.5 Water activity

The water activity of the black garlic powder was measured using an AquaLab 4TE (Meter Group Inc., USA). Three replications of water activity were performed for each sample.

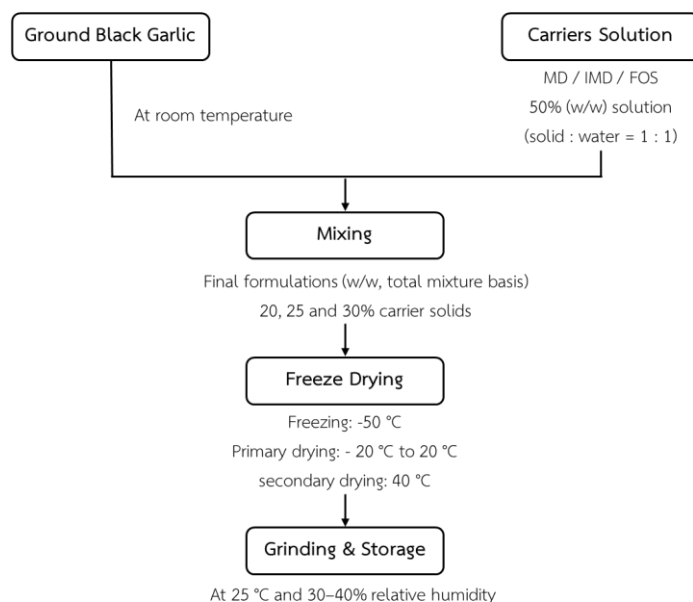


Fig. 1. Preparation of the freeze - dried black garlic powder

2.6 Bulk density

The samples were placed in a container of known volume and weighed. The bulk density was calculated using the following formula:

$$\rho = \frac{m}{v} \quad (2)$$

Where ρ is bulk density (g cm^{-3}), m is the mass of samples (g) and V is the volume (cm^3).

2.7 Color

The color of the powder was measured using spectrophotometer (ColorFlex, Hunter Lab, UK). The color difference was calculated according to Equation.

$$\Delta E = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2} \quad (3)$$

Where L^* represents the lightness (0 - 100), a^* represents the scale of greenness (-) to redness (+),

and b^* represents the scale of blueness (-) and yellowness (+).

2.8 Flowability (angle of repose)

The flowability of freeze-dried black garlic powder was determined using the angle of repose method. A fixed cone height was established, and a known quantity of powder was measured. The powder was then poured onto a horizontal surface (Camacho et al., 2022), after which the height and diameter of the formed cone were measured.

$$AoR = \arctan \times \frac{2h}{d} \quad (4)$$

where h = height from the top of the formed product cone to the horizontal surface (cm); d = maximum cone product diameter (cm).

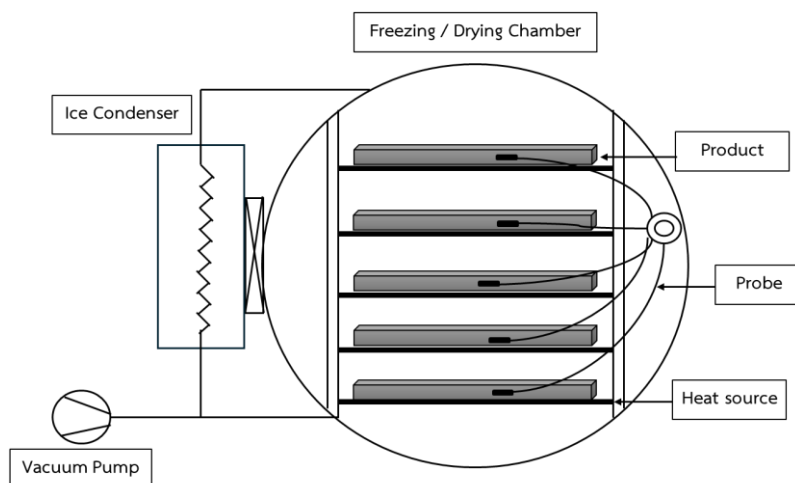


Fig. 2. Freeze dryer, Kryo 'D Fz - Itc (1993) co. ltd

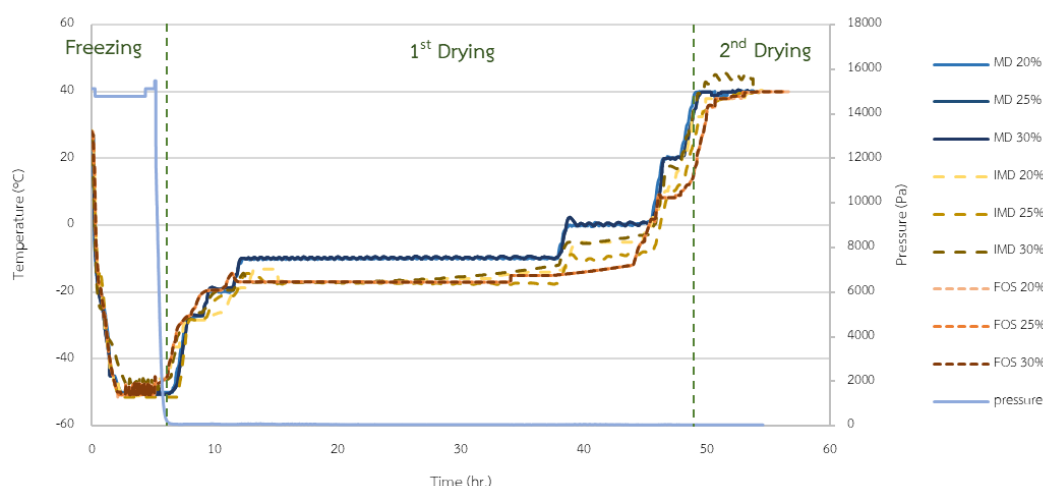


Fig. 3. The setting temperature and pressure operation of freeze dry machine.

2.9 Antioxidant activity

2.9.1 DPPH radical scavenging activity

The DPPH assay was conducted following the method of Singh et al. (2002) with slight modifications. An aliquot (10 μ L) of the extract was mixed with 2.99 mL of DPPH working solution in the dark for 60 min before measuring absorbance at 515 nm using a spectrophotometer (Libra S12, Biochrom, England). Results were expressed as mg Trolox equivalents (TE)/g dry matter.

2.9.2 ABTS Radical Scavenging activity

The ABTS assay was conducted following the method of Kriengsak et al. (2006) with slight modifications. An aliquot (10 μ L) of the extract was mixed with 2.99 mL of ABTS working solution and incubated in the dark for 10 min before measuring absorbance at 734 nm using a spectrophotometer (Libra S12, Biochrom, England). Results were expressed as mg Trolox equivalents (TE)/g dry matter.

2.9.3 Ferric reducing antioxidant power (FRAP)

The FRAP assay was conducted following the method of Benzie and Strain (1996) with slight modifications. An aliquot (10 μ L) of the extract was mixed with 2.99 mL of FRAP reagent and incubated in the dark for 30 min prior to measuring absorbance at 593 nm using a spectrophotometer (Libra S12, Biochrom, England). Results were expressed as mg Trolox equivalents (TE)/g dry matter.

2.10 Statistical analysis

All results were presented as mean $\pm$ standard deviation (SD). The experimental data were analyzed using a 3 $\times$ 3 factorial design and subjected to two-way analysis of variance (ANOVA) to evaluate the effects of carrier type and concentration. Statistical analyses were performed using IBM SPSS Statistics version 20. Significant differences among means ($p < 0.05$) were determined using Duncan's new multiple range test.

3 Results and Discussion

3.1 Drying time and TSS of black garlic powder

Drying time ranged from 53.25 to 56.77 hr, while total soluble solids (TSS) varied from 76.4 to 83.3 %Brix depending on carrier type and concentration (Table 1). Black garlic exhibits an amorphous structure and contains a high concentration of low-molecular-weight sugars, which makes it susceptible to structural changes during drying. Powdered products may transition into a sticky, viscous state when exposed to temperatures above the glass transition temperature (T_g) or when additional moisture is absorbed. Therefore, the incorporation of carrier agents or stabilizers can increase the glass transition temperature (T_g), thereby preventing melting during the freeze-drying process and reducing the stickiness of the resulting powder (Mani et al., 2002; Shrestha et al., 2007). In addition, the inclusion of carrier agents generally increases the total soluble solids (TSS) content of the formulation. The drying time is

influenced by several factors, including moisture content, structural characteristics, solute concentration, thermal conductivity of the material, and freezing temperature (Rattanapanon, 2015). As shown in Table 1, samples containing fructooligosaccharides exhibited longer drying times than those containing maltodextrin and isomaltodextrin. This trend corresponds with the total soluble solids content, where higher soluble solid concentrations result in longer drying times. Materials with higher TSS typically exhibit increased viscosity, which can slow down moisture diffusion and consequently extend the drying duration (Ratti, 2001).

3.2 Quality of black garlic powder

The moisture content and water activity of black garlic powder are presented in Table 1., ranging from 4.07 - 5.96 %w.b. and 0.2536 - 0.2955. Both parameters decreased as the concentration of carrier agents increased. Among the samples, the powder containing maltodextrin exhibited the lowest moisture content and water activity, followed by the sample containing isomaltodextrin, while the sample containing fructooligosaccharide showed the highest moisture content. These results indicate that the type of carrier agent significantly influences the moisture content of the powder after drying due to differences in water-holding capacity (Michalska-Ciechanowska et al., 2021). In addition, all black garlic powder samples showed water activity values below 0.6 (Table 1). Water activity is an important parameter for evaluating the availability of water in food systems, as it directly affects shelf life, microbial growth, and overall food safety (Ganaie et al., 2021).

The bulk density of black garlic powder showed variations among carrier types and concentrations, with values ranging from 0.94 to 0.88 for maltodextrin, 0.95 to 0.93 for isomaltodextrin, and 1.01 to 1.00 for fructooligosaccharide, depending on carrier concentration. The bulk density decreased with increasing concentrations of carrier agents. This phenomenon can be attributed to the density difference between black garlic and carrier materials, as black garlic possesses a higher density than the carriers. Consequently, the addition of carriers reduced the proportion of black garlic solids in the formulation, resulting in increased porosity and void formation within the powder structure and, therefore, a lower bulk density (Jaya &

Das, 2004; Cano-Chauca et al., 2005). During the freeze-drying process, structural modifications may occur in the sample, particularly due to the partial dissolution of carrier materials. This effect becomes more pronounced during the secondary drying stage, where the product may undergo partial structural collapse (Shrestha et al., 2007), which subsequently influences the bulk density and overall physical properties of the powder.

A comparison among samples containing different carriers indicated that black garlic powder formulated with maltodextrin and isomaltodextrin exhibited similar bulk density values, both of which were lower than those of the samples containing fructooligosaccharide. Maltodextrin and isomaltodextrin are carbohydrates with molecular structures that facilitate polymer formation and possess relatively high-water retention capacity, promoting the development of a more porous structure during the drying process. In contrast, fructooligosaccharide exhibits different intermolecular interactions, resulting in structural variations in the final product and consequently leading to a higher bulk density.

The flow properties of powders are important material characteristics and play a crucial role in the production process, as they influence processing efficiency, product uniformity, and shelf life. The results of the flow property analysis of freeze-dried black garlic powder are presented in Table 1. For maltodextrin, isomaltodextrin, and fructooligosaccharide, the values at 20%, 25%, and 30% carrier concentrations ranged from 36.42 to 30.09, 36.59 to 31.55, and 44.45 to 40.67, respectively. The flowability index increased from cohesive to easy flowing as the carrier concentration increased. Samples with lower carrier content exhibited poorer flowability, which can be attributed to their lower glass transition temperature (T_g) and higher hygroscopicity. A lower T_g increases molecular mobility, leading to particle stickiness and agglomeration, while higher hygroscopicity promotes moisture absorption, further reducing flow performance (Roos, 1995; Fitzpatrick et al., 2004). Among the carrier agents, maltodextrin generally exhibited better flowability due to its ability to form a stable matrix with reduced stickiness (Gharsallaoui et al., 2007). Isomaltodextrin showed

comparable but slightly lower flow performance, which may be related to its more highly branched structure affecting particle packing and matrix compactness. In contrast, fructooligosaccharide demonstrated poorer flowability, likely due to its lower molecular weight and higher hygroscopicity, which enhance moisture uptake and promote particle cohesion (Franck, 2002). Increasing carrier

concentration improved flowability across all carriers by increasing Tg and reducing moisture-related interactions. Similar findings were reported by Nurhadi and Roos (2017), who observed improved flowability in honey powder with increasing maltodextrin concentration.

Table 1 Quality characteristics of black garlic powder

Sample	Drying time (hr.)	M.C. (%w.b.)	Water activity	TSS (%Brix)	Bulk density (g cm ⁻³)	AoR (°)
MD 20	53.25	4.85 ± 0.16 ^d	0.2742 ± 0.0110 ^{abcd}	76.4 ± 0.1 ^h	0.94 ± 0.03 ^b	36.42 ± 0.73 ^c
MD 25	53.58	4.53 ± 0.04 ^e	0.2683 ± 0.0127 ^{bcd}	78.1 ± 0.1 ^f	0.92 ± 0.00 ^b	33.14 ± 0.35 ^d
MD 30	53.93	4.07 ± 0.03 ^f	0.2536 ± 0.0126 ^d	79.1 ± 0.0 ^{de}	0.88 ± 0.00 ^c	30.09 ± 2.37 ^e
IMD 20	54.08	5.19 ± 0.08 ^c	0.2799 ± 0.0089 ^{abc}	76.8 ± 0.0 ^g	0.95 ± 0.01 ^b	36.59 ± 0.26 ^c
IMD 25	54.38	4.93 ± 0.05 ^d	0.2744 ± 0.0106 ^{abcd}	78.9 ± 0.2 ^e	0.94 ± 0.01 ^b	33.21 ± 2.27 ^d
IMD 30	54.65	4.42 ± 0.17 ^e	0.2639 ± 0.0118 ^{cd}	79.3 ± 0.1 ^d	0.93 ± 0.01 ^b	31.55 ± 0.37 ^d
FOS 20	56.18	5.96 ± 0.07 ^a	0.2955 ± 0.0218 ^a	80.8 ± 0.2 ^c	1.01 ± 0.02 ^a	44.45 ± 0.25 ^a
FOS 25	56.52	5.63 ± 0.04 ^b	0.2894 ± 0.0088 ^{ab}	81.9 ± 0.1 ^b	1.00 ± 0.01 ^a	41.38 ± 0.03 ^b
FOS 30	56.77	5.25 ± 0.05 ^c	0.2817 ± 0.0095 ^{abc}	83.3 ± 0.2 ^a	1.00 ± 0.02 ^a	40.67 ± 1.69 ^b

Data are presented as mean ± standard deviation (n=3). a-d superscripts represent a significant difference within the same column (p<0.05) according to Duncan's multiple range test.

3.3 Color of black garlic powder

The color characteristics of black garlic powder are presented in Table 2. The addition of carrier agents prior to drying increased the lightness (L*) and yellowness (b*) of freeze-dried black garlic powder. This observation is consistent with previous studies by Caliskan and Dirim., 2016; Moolwong et al., 2023 who reported that higher levels of maltodextrin added during both freeze-drying and spray-drying processes resulted in powders with greater lightness. Similarly,

increasing the maltodextrin ratio has been shown to enhance the lightness of avocado powder. This effect can be attributed to maltodextrin forming a white powder upon drying, thereby increasing the overall lightness of the final product. Among the carrier agents tested, samples containing maltodextrin exhibited the highest lightness values, followed by those containing isomaltodextrin, whereas samples containing fructooligosaccharide showed the lowest lightness.

Table 2 Color properties of black garlic powder

Sample	Before Freeze drying			After Freeze drying			ΔE
	L*	a*	b*	L*	a*	b*	
MD 20	5.61 $\pm$ 0.18 ^b	7.48 $\pm$ 0.23 ^a	4.78 $\pm$ 0.39 ^a	38.62 $\pm$ 0.24 ^{cd}	7.89 $\pm$ 0.25 ^b	22.25 $\pm$ 0.43 ^c	37.36 $\pm$ 0.33 ^d
MD 25	5.84 $\pm$ 0.27 ^{ab}	7.50 $\pm$ 0.25 ^a	4.64 $\pm$ 0.47 ^a	41.47 $\pm$ 0.92 ^b	8.78 $\pm$ 0.19 ^a	21.24 $\pm$ 0.09 ^d	39.33 $\pm$ 0.35 ^c
MD 30	5.52 $\pm$ 0.46 ^b	8.01 $\pm$ 0.81 ^a	4.89 $\pm$ 0.29 ^a	45.21 $\pm$ 0.43 ^a	8.92 $\pm$ 0.49 ^a	22.17 $\pm$ 0.12 ^c	43.30 $\pm$ 0.32 ^b
IMD 20	5.41 $\pm$ 0.73 ^b	7.52 $\pm$ 0.65 ^a	4.66 $\pm$ 0.59 ^a	38.08 $\pm$ 0.38 ^d	7.80 $\pm$ 0.38 ^b	20.09 $\pm$ 0.58 ^e	36.14 $\pm$ 0.81 ^e
IMD 25	5.63 $\pm$ 0.31 ^b	7.45 $\pm$ 0.13 ^a	4.53 $\pm$ 0.51 ^a	39.47 $\pm$ 0.11 ^c	7.91 $\pm$ 0.57 ^b	23.18 $\pm$ 0.17 ^b	38.65 $\pm$ 0.48 ^c
IMD 30	6.45 $\pm$ 0.56 ^a	7.33 $\pm$ 0.77 ^a	4.71 $\pm$ 0.33 ^a	46.36 $\pm$ 0.44 ^a	7.78 $\pm$ 0.17 ^b	23.75 $\pm$ 0.07 ^a	44.23 $\pm$ 0.28 ^a
FOS 20	5.59 $\pm$ 0.27 ^b	7.67 $\pm$ 0.38 ^a	4.81 $\pm$ 0.72 ^a	15.91 $\pm$ 0.97 ^f	5.44 $\pm$ 0.67 ^c	9.34 $\pm$ 0.58 ^g	11.52 $\pm$ 0.72 ^h
FOS 25	5.38 $\pm$ 0.14 ^b	7.31 $\pm$ 0.12 ^a	4.42 $\pm$ 0.39 ^a	17.05 $\pm$ 1.31 ^f	5.23 $\pm$ 0.85 ^c	11.21 $\pm$ 0.23 ^f	13.70 $\pm$ 0.72 ^g
FOS 30	5.45 $\pm$ 0.36 ^b	7.22 $\pm$ 0.56 ^a	4.67 $\pm$ 0.83 ^a	21.48 $\pm$ 0.63 ^e	5.61 $\pm$ 0.43 ^c	10.97 $\pm$ 0.14 ^f	17.30 $\pm$ 0.01 ^f

Data are presented as mean $\pm$ standard deviation (n=3). a-d superscripts represent a significant difference within the same column (p<0.05) according to Duncan's multiple range test. L*, lightness; a*, greenness to redness; b*, blueness to yellowness.

3.4 antioxidant capacities

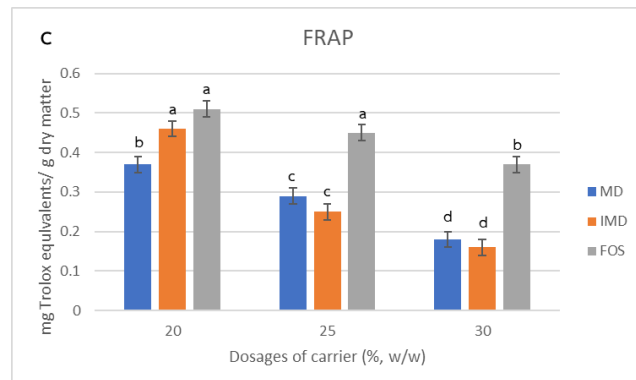
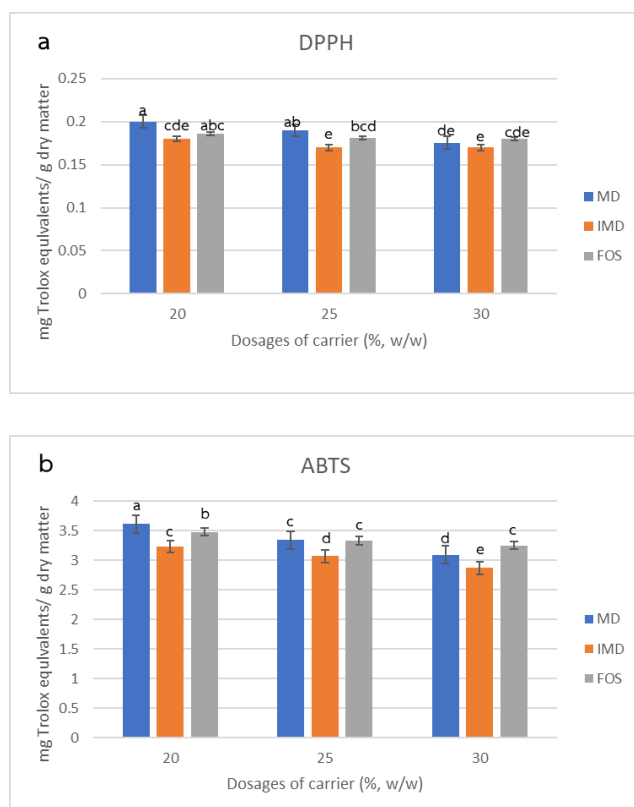


Fig. 4. DPPH (a), ABTS (b), and FRAP (c) assay of different types and quantity of black garlic samples with carriers. Values are expressed as mean $\pm$ SD (n=3) with significantly different at p < 0.05

The antioxidant capacities of black garlic powder, determined by DPPH, ABTS, and FRAP assays, are presented in Fig. 4. The results showed a decreasing trend with increasing carrier ratio. This reduction can be attributed to a dilution effect, as fresh black garlic contains high concentrations of bioactive compounds with strong antioxidant properties. The incorporation of carrier agents reduces the relative proportion of these active compounds per unit mass, leading to

lower measured antioxidant activity. Similar observations were reported by Chuaychan and Benjakul (2016), who investigated the effect of maltodextrin as a carrier agent on the antioxidative properties of spray-dried gelatin powder. Their results demonstrated that increasing the concentration of maltodextrin resulted in decreased antioxidant activity due to the reduced availability of bioactive compounds in the final powder. Likewise, Lachowicz et al. (2020) reported that increasing maltodextrin content significantly reduced the antioxidant capacity of vacuum-dried Saskatoon berry powder as evaluated by ABTS and FRAP assays. In addition, Maltodextrin showed relatively better antioxidant retention, likely due to its effective film-forming ability and stable encapsulating matrix (Vargas et al., 2024), while isomaltodextrin exhibited similar but slightly lower performance. In contrast, fructooligosaccharide provided weaker physical protection due to its hygroscopic nature and lower molecular weight. However, it showed the highest FRAP values, which may be attributed to its intrinsic reducing capacity, as the FRAP assay measures electron-donating ability rather than radical scavenging (Benzie & Strain, 1996). Additionally, thermal degradation during drying may further reduce antioxidant activity (Coskun et al., 2024). Overall, antioxidant capacity is governed by the combined effects of dilution, carrier-dependent encapsulation, thermal degradation, and assay-specific mechanisms.

4 Conclusion

This study investigated the effects of different carrier agents (maltodextrin, isomaltodextrin, and fructooligosaccharide) and carrier ratios (20%, 25%, and 30%) on the physical and chemical properties of black garlic powder. The results demonstrated that both the type and concentration of carrier agents significantly influenced the properties of the resulting powders. Among the tested conditions, 25% maltodextrin provided the optimal balance, demonstrating improved physical properties while maintaining relatively high antioxidant activity

compared to other carrier agents. These findings provide useful information for the selection of appropriate carrier agents in the production of black garlic powder. Further studies are recommended to evaluate the shelf life and long-term stability of the developed powders.

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