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RESEARCH

Physicochemical Properties and Characterization of a New Product: Spray Dried Hempseed Milk

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Abstract
The objective of the present study was to investigate the physicochemical properties and powder characterization of hempseed milk powders obtained by whole hempseed and cold-pressed whole hempseed paste (de-oiled). Whole hempseed and de-oiled hempseed paste were used to produce plant based milk powder applying spray drying process. The influence of oil content on physicochemical features, emulsion and rheological properties of the powders was examined. Results showed that dry content, total protein, loose density, tapped density, viscosity, foaming capacity and foaming stability of sprayed-powders produced in milk obtained using whole and de-oiled hemp seeds were not statistically different from each other ($p > 0.05$). By using de-oiled hempseed cake in feed solution preparation, spray dryer process efficiency increased from 31 to 44% without using any carrier agents. Hempseed powder product with improved properties such as apparent density, solubility, hygroscopicity and emulsion stability index was obtained.

Keywords Hempseed milk · Powder · Spray dryer · By-product · Plant-based milk

Introduction

Hempseeds are composed of protein (20–25%), lipids (25–35%), 20–30% carbohydrate, 10–15% insoluble fibre, 4–7% ash, depending on the variety [1, 2] and by this way it can be considered as a rich source of oils, proteins [3] and total polyphenols [4]. One of the valuable compounds is hemp seed oil because of containing essential fatty acids (Linoleic acid (LA, C18:2, ω-6 and α-linolenic acid (ALA, C18:3, ω-3) with the recommended value for human nutrition of 3:1 [5]. Hempseed is a good source of nutritious protein thanks to contain of highly digestible amino acids. In addition, the amount of compounds can ensure needing for infants or children as recommended by FAO/WHO [6]. Moreover, typical terpeno-phenolic compounds as prenylated flavonoids, stilbenoid derivatives, lignanamides and flavonoids were found in hempseed cake [4].

Hempseed cake/meal is a by-products obtained after oil extraction from hempseed such as other methods; solvent extraction [7], supercritical CO2 extraction [8] and cold-pressing process [4, 9]. Hempseed cake is a valuable nutritional supplements with fatty acid composition, antioxidant properties and anti-proliferative capacity and it is a kind of food supplement with positive health effects and an interesting by-product utilizable in food [10]. Studies have shown that hempseed cake is not only used in animal nutrition [11], but also added to food formulations such as meat, bread and potato chips [12].

All the mentioned factors make hempseed oil and its by product (hemp seeds cake) important foods in terms of both market and health values. After hempseed oil production, de-oiled hempseed cake can be sold directly or integrated other food matrix for the enrichment of protein percentage. Hempseed milk can be produced both of materials as whole hempseed and de-oiled whole hempseed. Like other milk alternatives made from plant seeds (soybean, almond, etc.), hemp milk has an unstable emulsion system that tends to coalesce and flocculate due to its oil content. This leads to poor quality, shelf life and consumer acceptance. Emulsifier/stabilizers are used or some technological processes (high

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pressure homogenization, ultrasonic treatments, pulsed electric field processing etc.) are applied in order to reduce these negative effects and to obtain a stable product [13, 14]. Plant based milk substitutes can also be spray-dried [15] or drum-dried [16] to produce a stable powder, which can be reconstituted to the desired product.

In this study, physicochemical properties and characterization of hemp milk powder produced from both whole hempseed and de-oiled hempseed paste was investigated. The purpose of using different materials (whole hempseed and cold-pressed whole hempseed cake) were to seek an answer to the question of whether hempseed milk powder could be obtained without using any carrier agents. In addition, using possibility of de-oiled hempseed cake was examined the feasibility of the production of the instant hempseed powder from waste-by product. It was also aimed to contribute to increase the usage potential in food matrix of hemp seed plant-based milk, which attracts attention in the food industry.

Materials and Methods

The Materials and Methods section is presented as supplementary Online Resource 1.

Results and Discussion

Spray Drying Process and Product Yield

One of the parameters for the spray drying process is inlet temperature [19] and it was selected as 150 °C according to pre-trials for the hempseed milk drying process. Man and Minh [20] also determined 150 °C as the drying inlet temperature of coconut milk with 50% oil content. Powders obtained have shown in Fig. 1.

Oil content showed a significant effect on the product yield ($p < 0.05$). While product yield was 31.34% in HSMP, it was 44.15% in D-HSMP. The reasons for this the low product yield could be the oil content and the use of no carrier/wall material. As stated in other studies [20] in which products had high oil content were tried to be sprayed at high inlet temperature level, high oil content caused adhesions in the cyclone and product loss by passing to liquid form. Particles with high particle size retain more oil in their bodies [19]. When D-HSMP and HSMP samples were compared, because of containing less oil on their (D-HSMP) outer surfaces, the adhesion to the cyclone was slightly reduced and the product yield was increased compared to the HSMP.

Proximate Analysis

Dry matter content, total ash, protein and oil content of the spray dried samples were summarized in Table 1. Dry matter and protein contents of powder samples were not statistically different from each other ($p > 0.05$) while oil and ash contents show difference ($p < 0.05$). The moisture content of powder samples was around 7% and the value is above the 3–4% limit value that powder foods should have [21], but it is in the moisture range (91–98%) determined in powders obtained from oilseed meal at other study [22]. The oil content of the HSMP was determined as 35%, which is in line with the literature [23, 24]. In addition, Kiralan, Gül [25] reported that the oil content of twenty one different hempseed samples from Turkey ranged from 29.6 to 36.5%. The oil ratio in D-HSMP produced from the hempseed cake obtained cold-press process was determined as 10.49%. Supporting this result, House, Neufeld [26] reported that the remaining seed cake or meal containing approximately 10% residual oil. It depends on the cold press process yield and extraction capability but the process can ensure reducing three times of oil content of the seed. Protein content of HMSP and D-HSMP determined as 34% and 31%, respectively ($p > 0.05$). The remaining seed meal has a high protein content of 35%, which is why it is commercially offered as a source of vegetable protein in the form of hemp protein powder, hemp flour and in shake drinks [26]. Latif and

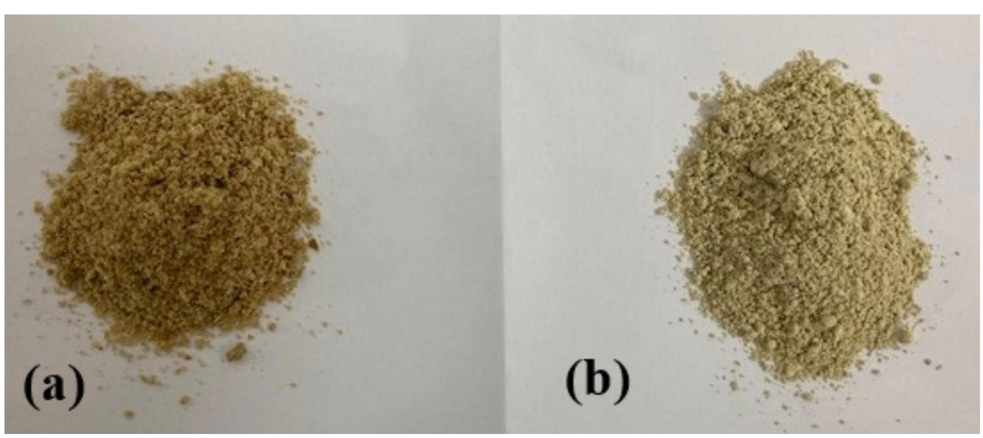


Fig. 1 Digital images of HSMP (a) and D- HSMP (b). HSMP: Hempseed milk powder, D-HSMP: De-oiled hempseed milk powder

Table 1 Physicochemical characterization of spray dried powders

Analysis	D-HSMP	HSMP	sig.*	Analysis	D-HSMP	HSMP	sig.*
Dry matter content (%)	93.48 ± 0.07	93.31 ± 0.03	0.055	D*10, µm	73.57 ± 4.27	52.58 ± 2.13	0.00
Total ash (%)	11.8 ± 0.19	7.85 ± 0.06	0.00	D*50, µm	106.21 ± 4.96	75.33 ± 1.22	0.00
Total protein (%)	31.62 ± 1.74	34.17 ± 0.29	0.282	D*90, µm	151.86 ± 5.76	106.5 ± 2.26	0.00
Total oil (%)	10.49 ± 1.09	35.3 ± 3.80	0.00	D[4,3), µm	109.71 ± 5.15	77.65 ± 0.90	0.00
Water activity (a_w)	0.300 ± 0.00	0.507 ± 0.00	0.00	D[3,2), µm	102.09 ± 5.12	72.45 ± 1.44	0.00
Loose bulk density (ρL), g/cm^3	0.340 ± 0.01	0.368 ± 0.015	0.052	Product yield, %	44.15 ± 5.59	31.34 ± 5.28	0.006
Tapped bulk density (ρT), g/cm^3	0.551 ± 0.01	0.540 ± 0.02	0.458	Hygroscopicity, %	12.26 ± 0.27	3.86 ± 0.28	0.00
Apparent density, g/cm^3	1.37 ± 0.01	1.17 ± 0.01	0.00	Solubility, %	64.12 ± 2.68	32.37 ± 4.36	0.00
Carr index (CI), %	38.29 ± 2.13	31.91 ± 2.18	0.022	Viscosity, mPas (50s^{-1})	0.841 ± 0.14	0.76 ± 0.04	0.396
Hausher ratio (HR)	1.62 ± 0.06	1.47 ± 0.04	0.023	Foaming capacity, %	23.33 ± 8.82	19.44 ± 3.47	0.516
Angle of repose (AOR), °	30.99 ± 0.35	28.03 ± 1.50	0.029	Foaming stability, %	82.22 ± 16.77	96.97 ± 5.25	0.22
Porosity (Ɛ), %	59.77 ± 0.79	54.15 ± 1.61	0.006	L	74.21 ± 0.17	60.69 ± 0.26	0.000
Emulsion stability index (ESI), %	69.87 ± 3.79	51.10 ± 2.04	0.002	a	-1.22 ± 0.04	2.43 ± 0.02	0.000
Emulsion activity index (EAI), m^2/g	34. 42 ± 5.46	47.68 ± 0.39	0.014	b	18.26 ± 0.15	21.62 ± 0.17	0.000

*Significant value of independent t test at 95% confidence level. HSMP: Hempseed milk powder, D-HSMP: De-oiled hempseed milk powder

Anwar [9] reported that enzyme-assisted extraction had no noticeable effects on the amounts of protein in hemp seeds. While the total ash content was 7.85% in the HSMP, it was 11.8% in the D-HSMP ($p < 0.05$). The increasing of ash percentage can be caused removing oil from the seed and total compound percentage.

Physical Characterization of Powder

The water activity values of the powder samples were statistically different from each other ($p < 0.05$) (Table 1). Accordingly, it can be concluded that the oil content creates a barrier to moisture transfer [17]. As the water activity of the powder samples varies between 0.3 (D-HSMP) and 0.5 (HSMP), it can be defined as a microbially safe product. It is recommended that the water activity value, in which microbial growth can not occur, should be lower than 0.6 [22]. There is no statistical difference between the loose and tapped bulk densities of HSMP and D-HSMP powder samples ($p > 0.05$) (Table 1). Apparent density of D-HSMP was higher than HSMP ($p < 0.05$). D-HSMP, which contains less oil content with lower density, was naturally more dense. As can be seen in Table 1 the oil content caused a statistically significant difference on the flowability property of powders. Carr index and Hausher ratio values were calculated in order to evaluate the flow properties of the powders. Both powder samples showed high cohesion properties (> 1.4). As noted in some study high cohesive behaviour has resulted in poor flowability [27]. Carr index values also supported the high cohesive behaviour. When the flowability property was classified considering the Carr index, sample HSMP could be classified as "fair" with the value of 31.91 ± 2.18, and sample D-HSMP represented "bad" flowability performance with the value of 38.29 ± 2.13. The poor flowability of powders could be explained by high levels of oil which obstructed to flow as in accordance with some study [21]. The porosity value of the D-HSMP samples obtained from the de-oiled hempseed cake milk was found to be higher ($p < 0.05$), having the highest mean particle size ($p < 0.05$). Particle enlargement can cause porosity [18]. SEM image also supported this result. Especially in the Fig. 2d, porous structures are observed on the particles surface. Furthermore when the water holding capacity was examined, it was concluded that this porous structure also affected the hygroscopicity value. Because, higher hygroscopicity was determined in the D-HSMP sample, which had high porosity (Table 1). Large particles leading to large porous structure could be facilitated the water penetration from the structure. In addition, as in HSMP sample, high oil content also created a barrier to water transfer, resulting in less hygroscopicity (3.86%). Statistically significant differences in the color parameters (L*, a*, b*) of powders were observed ($p < 0.05$) (Table 1). The changes in L* value are similar to the study [28] that reported de-oiling had significant effects on the color of hempseed cake samples. In that study, higher L* value, lower a* and b* values were obtained in the oil-removed hempseed cake compared to the hempseed cake. Similarly, in the present study, the yellowness (b value) of HSMP was higher than in the D-HSMP (Figs. 2 and 3). This is a result of the more oil containing being more yellow because of more yellowish color of hempseed oil [9]. The red–green (a*) color parameter of D-HSMP was in the negative region with the value of -1.22 ± 0.04, more toward green, while that of HSMP moved to positive region with the value of 2.43 ± 0.02, more toward red. Solubility is one of the most reliable criterion to evaluation of the behavior [22]. The oil content made a statistical difference on the solubility of the samples ($p < 0.05$) (Table 1). D-HSMP had almost twice as much solubility property as HSMP. This means that it is possible to obtain a food ingredient with increased

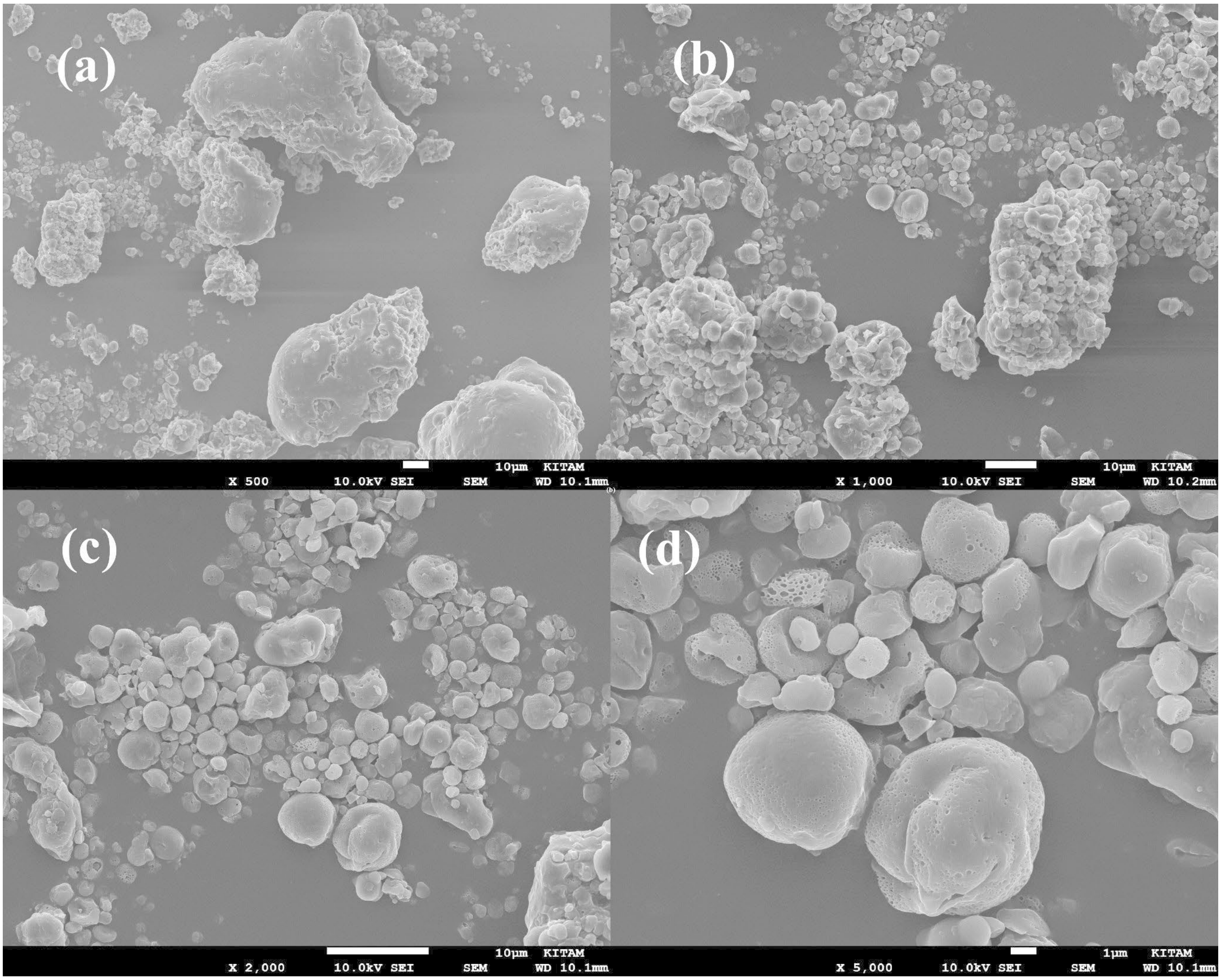


Fig. 2 SEM images of D-HSMP: 500 kV (a), 1000 kV (b), 2000 kV (c), 5000 kV (d). D-HSMP: De-oiled hempseed milk powder

solubility in food matrix from the de-oiled cake obtained by removing the oil from an oilseed. According to thermograms, glass transition values of the samples were found as 48.84 ºC with 61.47 J/kg and 42.07 ºC with 102.1 J/kg for HSMP and D-HSMP, respectively (Fig. 4).

Emulsion Properties of Powders

Proteins in the emulsion act as emulsifying agents [29]. The protein content in both powder samples is not statistically different, but since the proteins in the D-HSMP could not be adequately encapsulated by the oil, it may have been structurally denatured at the temperature and thus D-HSMP powder had a lower emulsion activity index than that of HSMP. In this study, HSMP with smaller particle size had lower emulsion stability index than that of D-HSMP (Table 1). Similar to this study Cao, Shi [30] reported that emulsion stability decreases with decreasing particle size attributing to insufficient hydrophobic/hydrophilic balance for the stabilization. Additionally, the higher hygroscopic property of D-HSMP can be shown as the reason for the higher determination of emulsion stability. Removing oil did not show any statistically significant difference on foam capacity and stability ($p > 0.05$).

Rheological Properties of Powders

The steady and dynamic properties of the reconstituted powders (1%) were carried out to examine the behavior of the powders in the food matrix. Flow curves (Fig. 5) of D-HSMP and HSMP showed that the relationship between the shear stress-shear rate were linear. This behaviour is classified as

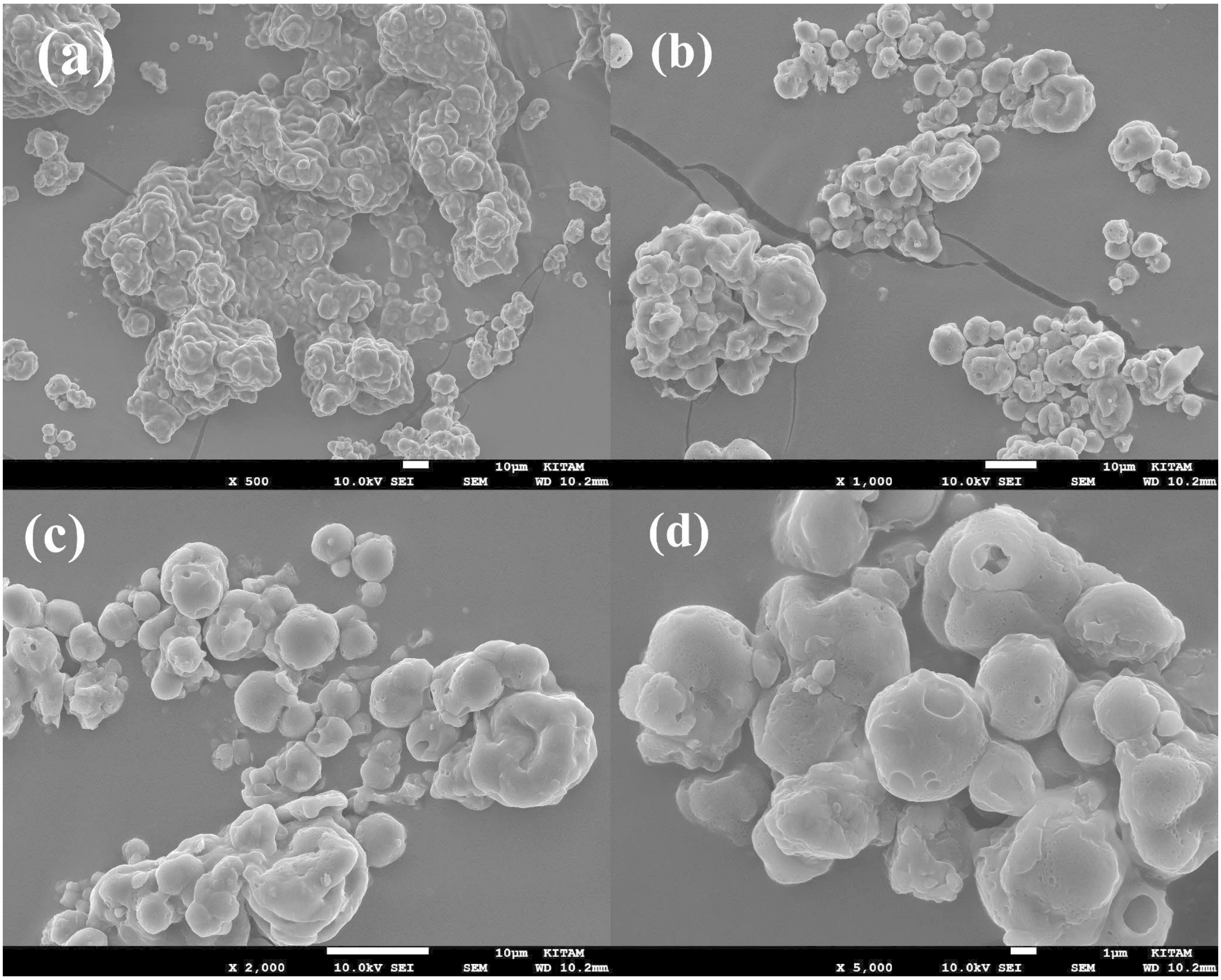
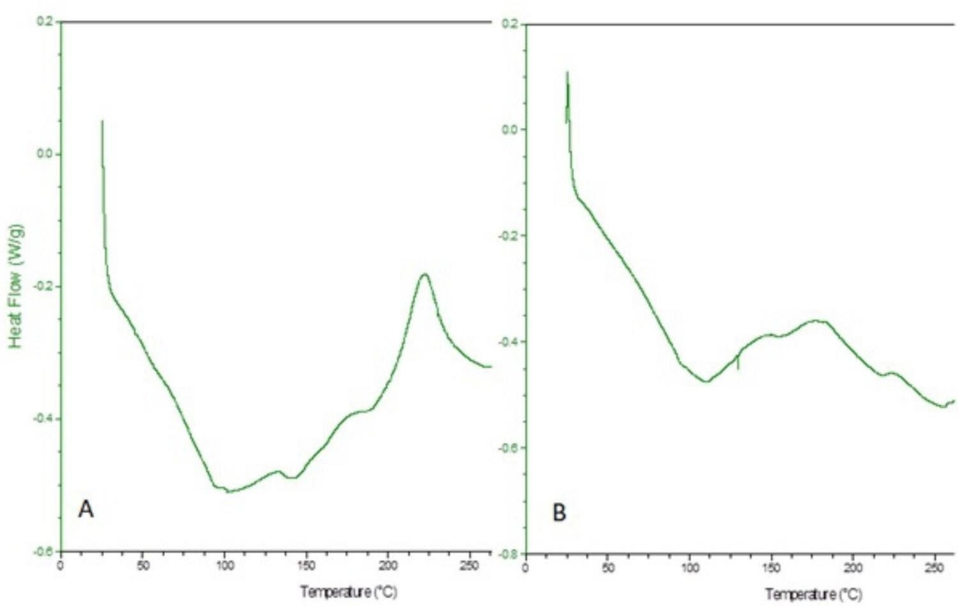


Fig. 3 SEM images of HSMP: 500 kV (a), 1000 kV (b), 2000 kV (c), 5000 kV (d). HSMP: Hempseed milk powder

Fig. 4 DSC heating flow curves of the reconstituted sprayed powder. HSMP: Hempseed milk powder, D-HSMP: De-oiled hempseed milk powder



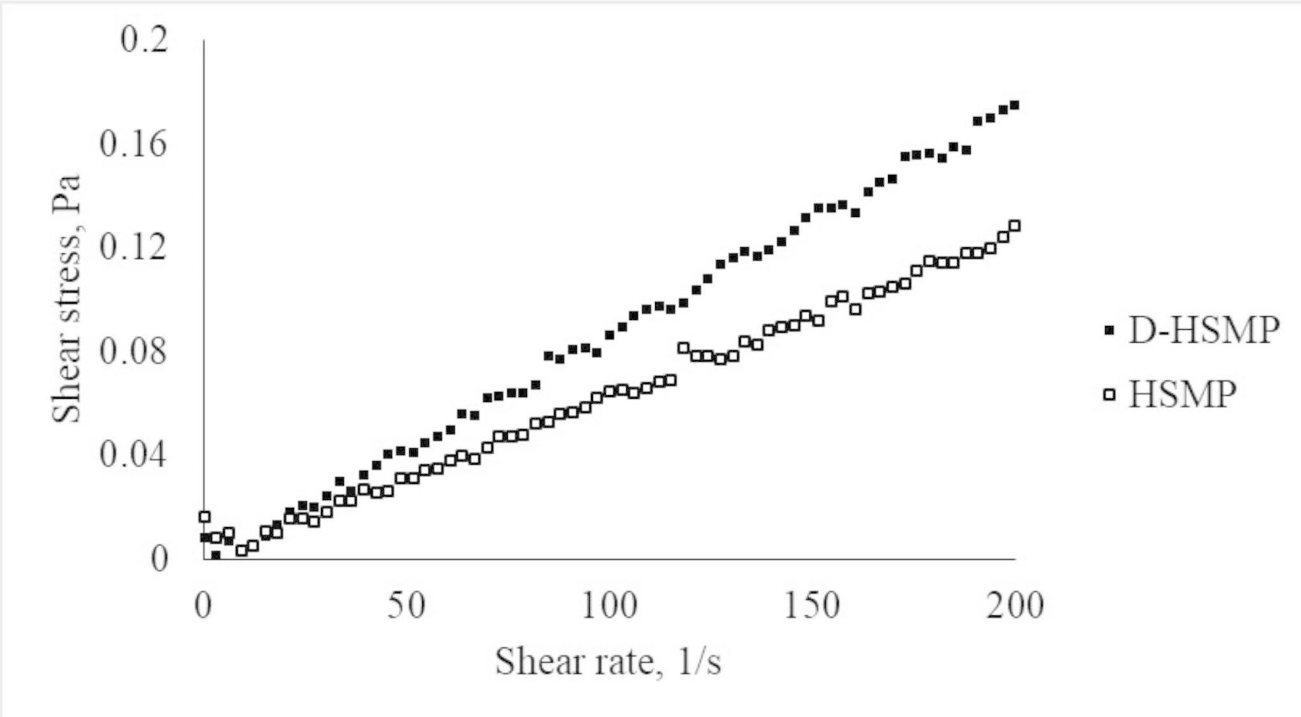


Fig. 5 Flow curves of reconstituted powder (1%). HSMP: Hempseed milk powder, D-HSMP: De-oiled hempseed milk powder

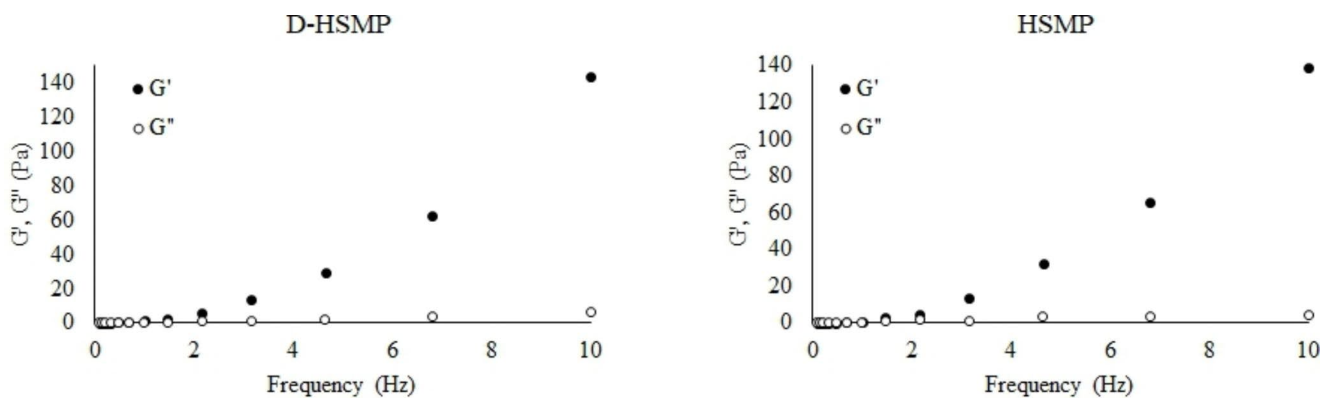


Fig. 6 Dynamic rheological properties of the reconstituted sprayed powder. HSMP: Hempseed milk powder, D-HSMP: De-oiled hempseed milk powder

Newtonian flow type. Apparent viscosity (η at shear rate 50 s^{-1}, η_{50}) of D-HSMP was 0.841 ± 0.14 mPa, while that of HSMP was 0.76 ± 0.04 mPa and there was no statistical differences in viscosity values (p = 0.396) (Table 1). The flow behaviour index (n) values of reconstituted D-HSMP (n = 1.034) and HSMP (n = 1.044) were close to 1 because of exhibiting Newtonian fluid characteristics as can be seen in Fig. 5. In Newtonian behaviour apparent viscosity of the sample is constant with increase in shear rate. This means that when powder samples are added to the food matrix at 1% concentration, no increase or decrease in viscosity will occur within a certain shear rate range (0-200 s^{-1}). When it was examined the dynamic rheological properties, Fig. 6 shows the storage (G′) and loss (G″) modulus as a function of frequency (Hz). The G′ values of the samples were higher than the G″ values over the whole frequency range. This indicates an elastic structure of both samples rather than a viscous structure.

Conclusion

In this study the potential to produce plant-based milk powder from a whole and de-oiled hemp seed without any carrier agent using spray dryer was investigated. According to results, using milk from de-oiled hemp cake significantly increased the spray dryer process efficiency. Furthermore apparent viscosity, porosity, particle size, hygroscopicity, solubility and emulsion stability index were significantly increased with de-oiled. However the flowability properties of the powder were adversely affected by the oil removal. The rheological properties did not change significantly and 1% solutions of both powder samples showed Newtonian flow behavior. The results can be guide for the further research for the vegetable based milk powder possibilities based on their whole seeds and their cold press cake. In addition, it can be utilized from physicochemical properties and characterization of hempseed milk powder instead of whey protein and milk powder for vegan based products.

Supplementary Information The online version contains supplementary material available at https://doi.org/10.1007/s11130-023-01053-x.

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Author Contributions M.M.: Methodology, Investigation, Formal analysis, Writing – original draft; **Z.T.**: Formal analysis; **M.K.**: Formal analysis; **A.B.**: Writing – review & editing; **F.Y.**: Supervision.

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Data Availability Data is available upon request.

Declarations

Ethics Statement Not applicable.

Consent to Participate Not applicable.

Consent for Publication Not applicable.

Conflict of Interest There are no conflicts of interest.

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