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SPRAY DRYING OF DE-OILED SUNFLOWER PROTEIN EXTRACTS: FUNCTIONAL PROPERTIES AND CHARACTERIZATION OF THE POWDER

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ABSTRACT
The proteins were extracted from de-oiled sunflower seed cake and protein powder was produced using a pilot-scale spray dryer. Mild-acidic protein extraction and co-extraction of phenolic compounds were employed in order to obtain protein extracts. A pilot-scale spray dryer was used to convert protein extracts into protein powder. Micro-spherical particles were obtained having irregular surface properties. The protein powders showed poor flow behaviour having hausner ratio and angle of repose values around 1,58 and 49°, respectively. They showed poor solubility, sufficient emulsifying and oil-binding capacity. Despite their poor solubility, they had moderate wettability. The denaturation temperature of sunflower protein powder was found around 80 °C from DSC thermogram. FTIR spectrum was found to be very similar to those of soy protein and milk protein extract powders reported previously.
Keywords: sunflower protein, protein powder, de-oiled sunflower meal, spray dryer, powder properties

PÜSKÜRTMELİ KURUTUCU İLE YAĞI ALINMIŞ AYÇİÇEĞİ PROTEİN EKSTRAKTI TOZU ÜRETİMİ: FONKSİYONEL ÖZELLİKLERİ VE TOZ KARAKTERİZASYONU

ÖZ
Bu çalışmada yağı alınmış ayçiçeği çekirdeği küspesinden protein fraksiyonu ekstrakte edilerek pilot ölçekli püskürtmeli kurutucu kullanılarak protein tozu üretilmiştir. Protein ekstraktlarını elde etmek için asidik protein ekstraksiyonu kullanılmıştır. Protein ekstraktı çözeltisini protein tozuna dönüştürmek için pilot ölçekli püskürtmeli kurutucu kullanılmıştır. Elde edilen protein tozunda düzensiz yüzey özelliklerine sahip mikro küresel parçacıklar gözlemlenmiştir. Protein tozlarının, sırasıyla yaklaşık olarak 1.58 ve 49 ° Hausner oranına ve duruş açısına sahip oldukları ve zayıf akış davranışı gösterdikleri tespit edilmiştir. Protein tozları zayıf çözünürlük, orta seviyede emülsifiye etme ve yağ bağlama kapasitesi göstermişlerdir. Zayıf çözünürlüklerine rağmen, orta derecede ıslanabilirlik özelliklerine sahip oldukları anlaşılmıştır. Ayçiçeği proteini tozunun denatürasyon sıcaklığı, DSC termogramında yaklaşık 80 °C olarak belirlenmiştir. FTIR spektrumunun, daha önce bildirilen soya proteini ve süt proteini ekstraktı tozlarına çok benzer olduğu bulunmuştur.
Anahtar kelimeler: ayçiçeği proteini, protein tozu, yağsız ayçiçeği küspesi, püskürtmeli kurutucu, toz özellikleri

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INTRODUCTION

There has been an increase in demand to plant proteins due to the growing population, changing consumer preferences and increasing awareness of their importance for health and nutrition (Amagliani et al., 2016; Egerton et al., 2018). Soy and dairy are the main sources of proteins used in food industry even though proteins derived from wheat, rice, corn, pea, canola and potato are also commercially available. However, food processing by-products may have significant amount of proteins which could be utilized by food industry due to their nutritional, functional and technological properties (Pickardt et al., 2015). After extracting oil from sunflower seeds, the remaining part is called as cake or meal which are rich in proteins (Ivanova et al., 2012). Due to low amounts of antinutritive compounds and no toxic substances found in these raw materials, they can be counted as promising sources for food proteins (González-Pérez and Vereijken, 2007).

Sunflower seeds may contain from 1 to 4% phenolic compounds (Weisz et al., 2009). The phenolic compounds present in sunflower press-cakes were reported to be responsible for the dark colour, bitter taste and astringency (González-Pérez and Vereijken, 2007; Pickardt et al., 2015). For these attributes, the press-cakes have been used as low-value animal feed to date (Ivanova et al., 2012). Sunflower proteins were reported as comparable to soy and other leguminous proteins in terms of functional properties and nutritive value except low lysine content (González-Pérez and Vereijken, 2007). Therefore, removing the phenolic substances from protein extracts from de-oiled sunflower press cakes would result in high quality protein ingredient for the food industry. The technological functional properties (i.e. emulsifying capacity, solubility, oil binding capacity) of sunflower proteins need to be evaluated in order to better understand their potential use in food applications and therefore those properties need to be determined (Romero et al., 2012).

Proteins are usually produced in powder form at industrial scale to increase the shelf life and to preserve the nutritional, organoleptic and physicochemical properties and spray drying has been a widely used method for the production of protein powders due to its low cost and availability in industrial production (Amagliani et al., 2016; Khanji et al., 2018; Torres et al., 2017). The behaviour of food powders such as flowability is affected by several factors such as temperature, humidity, particle-particle interactions, particle properties, and bulk properties of powders (Fitzpatrick et al., 2004). It is crucial to understand, predict and control the behaviour of powder material to improve the effectiveness of the processes like storage, discharge, mixing, rehydration etc. Even though some of the functional characteristics have been studied, no information has been found in literature about particle and powder properties of sunflower seed cake protein powders.

This study aimed to determine the feasibility of producing protein powder from de-oiled sunflower seed cake using a selected isolation method and a pilot scale spray dryer unit. Some of the physicochemical, functional, particle and powder properties of the extracted protein isolates were investigated in order to elucidate their suitability to be used in food processing.

MATERIALS AND METHODS

Materials

De-oiled sunflower seed cakes were obtained from a local oil manufacturing company (Trakya Birlik, Edirne, Turkey). The cakes were in dry and ground form (a mix of fine and coarse particles) containing husks. The cakes were sieved using laboratory-scale sieve shaker (AS200, Retsch, Germany) and the size fractions below 500 μm were used in protein extraction process. Salt was purchased from a local market. The chemicals were obtained from Sigma Aldrich (Taufkirchen, Germany). The miracloth filter (Calbiochem, 22-25 μm pore size) was purchased from Merck (Merck KGaA, Darmstadt, Germany) and Styrene-divinylbenzene Copolymer resin (18-100 mesh size) was purchased from Sigma Aldrich (Taufkirchen, Germany).

Methods

Protein extraction and spray drying

The method given by Pickardt et al. (2015) has been used with some little modifications to extract proteins. For this purpose, the de-oiled cakes were soaked in salt (table salt) solution containing approximately 2% salt. One kg of cake was added to 9 L of salt solution (cake to water ratio was 1:9). The pH of the slurry was adjusted to 6 by using HCl or NaOH solutions and kept at room temperature (21 ± 2 °C) for one hour while stirring at 150 rpm. The liquid part containing proteins were seperated from precipitated part by centrifugation (INO HT, Inovia, Istanbul, Turkey). To remove the phenolic compounds, Styrene-divinylbenzene resin was added to the solution (around 50 g per L) and kept at room temperature for 4 hours while stirring at 150 rpm. The resins with phenolics adsorbed were removed by filtering through a miracloth filter. The solution was kept at 4-6 °C until conducting the protein recovery procedure.

HCl solution was added to the protein solution until the pH decreased to 4.0 to precipitate proteins. Precipitated proteins were seperated by using a centrifuge (3300 g for 10 min) and the seperated proteins were washed twice by using distilled water. The washed protein mass was mixed with deionized water at a ratio of 1:8 and the mixture was homogenized using a homogenizer (WiseTis HG-15A, witeg Labortechnik GmbH, Wertheim, Germany) and neutralized to pH 7.0 by adding NaOH solution. The homogenized and neutralized protein solution was spray-dried using a pilot-scale spray drying unit (Toption Instruments, TP-S100, China) equipped with a disc atomizer. The spray dryer was operated at an inlet temperature of 175 ± 5 °C and outlet temperature of 80 ± 4 °C. Atmospheric air was used as the drying gas at a flow rate of 290 kg/h. The flow rate of the protein solution was set to 2 L/h (34 mL/min).

Proximate analysis

Ash, moisture and dry matter contents were determined using AOAC 942.05 and 925.10 standard methods, respectively. The protein and lipid contents were determined based on total N content by Kjeldahl method (981.10) and Soxhlet method (920.39), respectively (AOAC, 2000).

Particle morphology by SEM imaging

For investigation of microstructure and surface properties of the particles, scanning electron microscopy experiments (Philips ESEM XL30 FEG, Nederlands) were performed. Before coating with gold (to avoid the charge buildup under the electron beam), particles were mounted onto a adhesive tape on a stub. The images of the particles were captured at different magnifications (100x to 10000x).

Determination of Angle of repose and Hausner Ratio values

For evaluation of the flowability of sunflower seed cake protein powder (SPP), angle of repose (Fraczek et al., 2007) and Hausner Ratio (HR) values were determined. To determine the angle of repose, a steel funnel was used. It was positioned on a stand and placed at a height of 10 cm over the flat surface of a counter. 5 gram of SPP sample was poured into the funnel to let it flow by gravity to form a heap on the counter surface. An image of cross section of the heap was captured (Figure 1) and the angle of the triangular base was measured using an image analysis software (Image J, open Java source code). The Hausner Ratio (HR) (Eq. 3) was calculated from the bulk (ϱ_b) and tapped density (ϱ_t) values (Hausner, 1967). The flow behaviour of SPP was evaluated based on the data given in Table 1 (Ermis et al., 2018).

$$HR = \varrho_t / \varrho_b \quad (1)$$

Table 1. Evaluation of flowability of powders

Hausner Ratio	Angle of repose	Flowability
1.00-1.11	<25 º	Excellent
1.12-1.18	25-30º	Good
1.19-1.25	30-38º	Satisfactory
1.26-1.34	38-45º	Poor
1.35-1.45	45-55º	Very poor
1.46-1.59	>55º	Cohesive

Source: (Ermis et al., 2018)

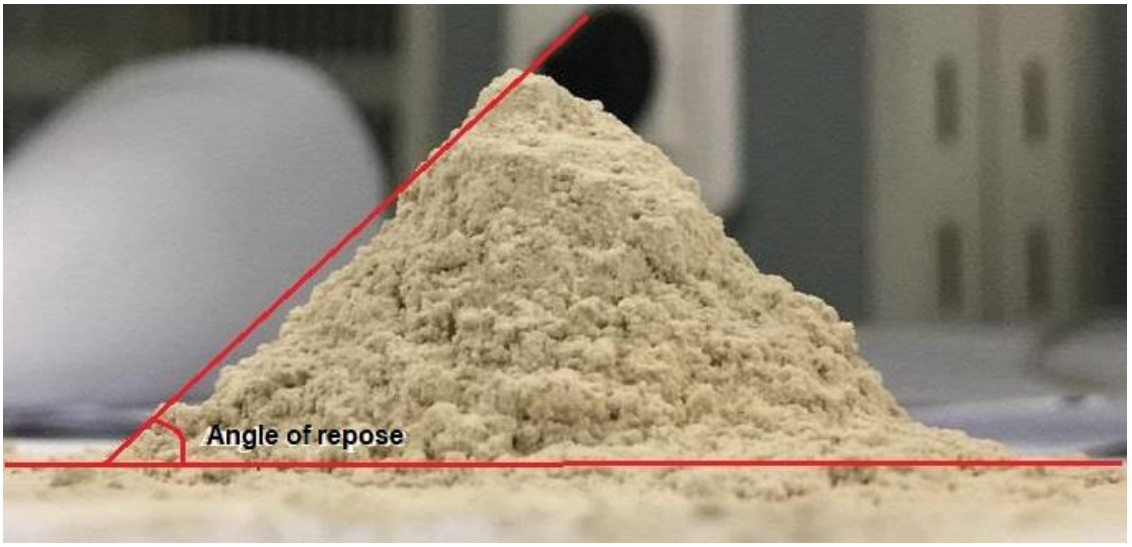

Figure 1. Angle of repose profile of SPP

Determination of the emulsifying properties

The methods given by previous researchers (Sila et al., 2014; Zhang and Zhao, 2013) were used to determine emulsion stability index (ESI) and emulsion activity index (EAI) with slight modifications. One gram of SPP sample was dissolved in distilled water (10 mL) to final concentration of 10% (w/v). SPP solution was mixed with 5 mL of olive oil and homogenized for one min at room temperature. 50 µL of the emulsion was taken immediately after the homogenization and 10 min after. The aliquots were diluted with 0.1% SDS solution (w/v) at a ratio of 1:100 and mixed for 10 s. The absorbance was measured using a Shimadzu UV-1280 UV-VIS spectrophotometer (Shimadzu Co. Ltd., Kyoto, Japan) at 500 nm wavelength.

The ESI and EAI were calculated using the equations given below (Egerton et al., 2018; Sila et al., 2014; Zhang and Zhao, 2013):

$$EAI\ (m^2/g) = (2 \times 2.303 \times A_0) / (0.25 \times \text{protein weight (g)} \quad (2)$$

$$ESI\ (\%) = (A_{10} / A_0) \times 100 \quad (3)$$

Where A_0 and A_{10} were absorbance values at 0 min and 10 min, respectively.

Determination of the oil binding capacity

Oil binding capacity (OBC) of SPP was determined by using a method described by Egerton et al. (2018) and Zhang and Zhao (2013) with some modifications. 1 g of SPP solution was taken into a tube containing 10 mL of refined olive oil and kept for 30 min at room temperature. The mixture was centrifuged (5000 x g) for 25 min at room temperature and the volume of supernatant was determined. OBC was calculated using equation given below:

$$OBC\ (mL/g\ \text{proteins}) = (V_1 - V_2) / (\text{protein weight (g)} \quad (4)$$

where V_1 was the initial volume of oil and V_2 was the volume of supernatant after centrifugation

Solubility Analysis

The solubility of SPP was evaluated using the method given by Shittu and Lawal (2007) and Zungur Bastıoğlu et al. (2016). For this purpose, 1 g was taken from SPP sample and mixed with 10 mL distilled water in a tube. The mixture was homogenized at 50 Hz for 10 min. The supernatant was transferred into an eppendorf tube and centrifuged at 6000 rpm for 10 min. After centrifugation, the supernatant was taken to an aluminium plate and dried for 24 h at 105 °C. The solubility percentage (mass of soluble powder / mass of total powder) was calculated by the weight difference.

Wettability analysis

Dynamic wettability was evaluated using the Washburn method given by Washburn (1921) (Ji et al., 2015) with small modifications. The method measures the wettability of powder based on capillary rise. To measure the dynamic wettability, 1 g of SPP was taken into a plastic tube (2 cm diameter) which is covered by filter paper fixed with parafilm at the bottom to prevent powder discharge. The weight of whole tube containing powder sample was determined (m1). The tube was placed and fixed on the surface of distilled water (21 ± 2 °C) to allow capillary rise for 10 min. The tube was weighed after 10 min (m2). The amount of water absorbed by SPP was calculated per g of powder [(m^2-m1)/g sample].

Fourier-transform infrared spectroscopy (FTIR) and differential scanning calorimetry (DSC) characterization

The chemical structure of SPP was observed using FT-IR (Fourier Transform Infrared) Spectroscopy. A Shimadzu IRTracer-100 (Shimadzu Co. Ltd., Kyoto, Japan) spectrophotometer equipped with an ATR (Attenuated Total Reflectance) (Shimadzu MIRacle) accessory was used to obtain the spectrum. The spectra of SPP was recorded with a resolution of 2 cm^{-1} (accumulating 16 scans per spectra). The absorbance values were obtained from 4000 cm^{-1} to 600 cm^{-1}. Crystal surface was cleaned using a piece of a soft tissue paper wetted with ethanol and a background (air) scan was done prior to analysis (Ermis et al., 2018). The thermal behaviour of SPP was characterized using a Shimadzu differential scanning calorimeter, DSC-60 Plus (Shimadzu Co. Ltd., Kyoto, Japan). Around 2-3 milligrams of sample were sealed in a hermetic aluminium pan and heated from 25 to 100 °C at a rate of 10 °C/min. An empty aluminium pan was considered as reference (Ermis et al., 2018).

Statistical analysis

The mean values and standard deviations were calculated using the data obtained from the experiments performed in triplicate.

RESULTS AND DISCUSSION

Proximate composition of press cake

The grounded cake with husks contained around 35±4% protein (conversion factor=5.6), 6.9 ± 0.6% ash and 1.4 ± 0.4% crude fat content based on dry matter (around 90 ± 1% total dry matter).

Protein extraction

Production of sunflower protein extracts from de-oiled sunflower seed cake has been demonstrated by some previous researchers on a pilot plant scale Pickardt et al., (2011) and Salgado et al., (2012) while some of the research works have been performed on a laboratory scale (Pickardt et al., 2009; Taha et al., 1981). However, those researchers have not charaterized the particle and powder properties of protein powders they obtained. The optimised conditions given by Pickardt et al. (2011) and Weisz et al. (2010) were used for absorption of phenolic substances to obtain a light- colored protein powder with improved economic viability.

The low yield of precipitated protein from de-oiled sunflower seed cake protein extracts (around 30% of total proteins) was noted in this study and this could be attributed to decreased protein solubility due to denaturation occurring during oil production (Ivanova et al., 2012). According to a recent study (Pickardt et al., 2011), approximately 3/4 of the extracted protein could be precipitated. In addition, Pickardt et al. (2015) reported that the washing precipitated proteins with water account for losses up to 17% of the precipitated proteins. Despite the loss of proteins during precipitation and purification, the percentage of protein fraction in precipitated proteins was quite high (around 90% in dry basis) and was in agreement with the previous researchers' findings (González-Pérez et al., 2002; Pickardt et al., 2015; Shchekoldina and Aider, 2012).

Microstructure analysis

The morphology of protein particles was investigated using scanning electron images (Figure 2). The SEM images revealed that the SPP consisted of varied size fractions of particles. As can be seen in Figure 3, the particles had irregular

and wrinkled surface properties. Amagliani et al. (2016) reported similar particle properties of rice protein concentrate powder. They also reported that the particle properties such as irregular, fractured, hollow, wrinkled and porous were typical of spray-dried protein powders.

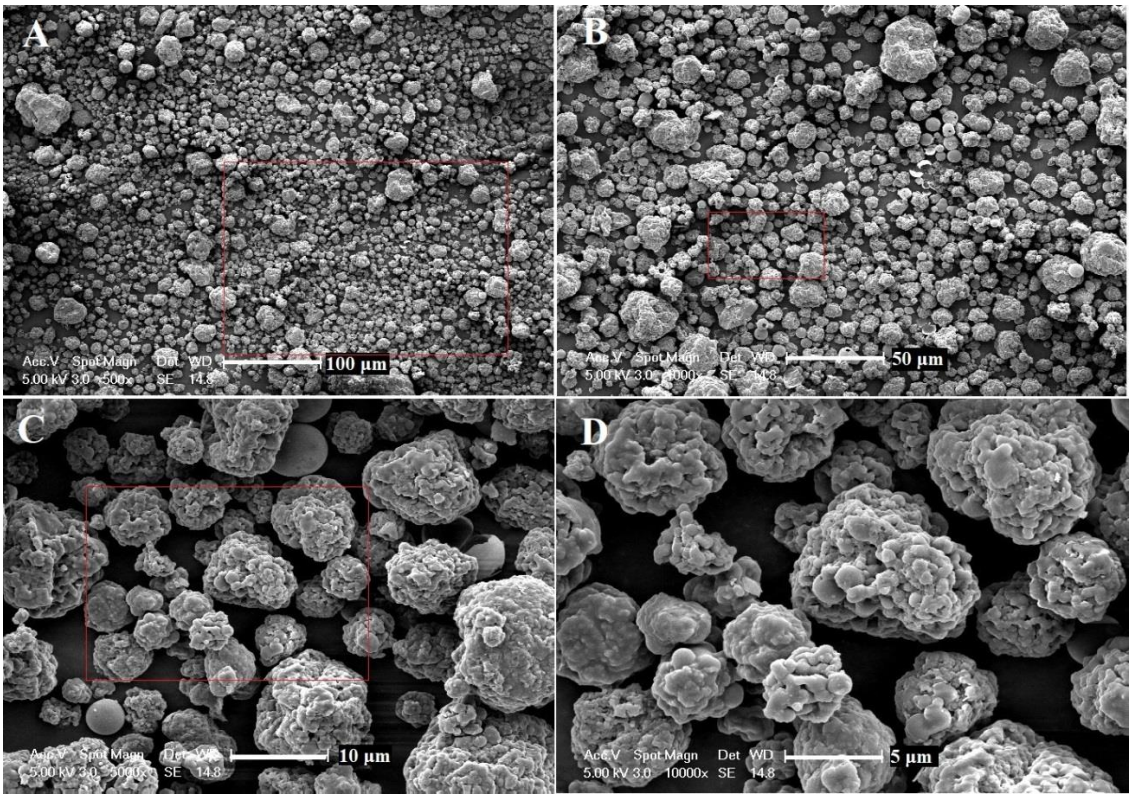

Figure 2. SEM images (A:500x , B:1000x, C:5000x, D:10000x).

Flowability and compressibility

Determination of flow properties is needed to predict the behaviour of a powder material during processing steps such as storage, handling, transportation and discharge to prevent flow problems (i.e. rat-holing, arching, erratic flow and flooding) (Ermis et al., 2018). The compressibility of SPP was found to be high and this might be attributed to the closer particle packing, irregular particle properties and increase in inter-particle surface contact (Amagliani et al., 2016). It was reported by Babu et al. (2018) that the higher protein content, irregular particle shape, existence of interparticle voids and surface irregularity/roughness resulted in high degree of compressibility and cohesion (as a result of Van der Waals forces and mechanical interlocking) which agreed with the findings of this study. High cohesive behaviour and high compressibility lead to poor flowability. The values of tapped density (ϱ_t) and bulk (poured) density (ϱ_b) determined for SPP were around 639 and 403 kg.m^{-3}, respectively. The hausner ratio (ϱ_t/ϱ_b) of SPP was found to be around 1.58 which corresponded to cohesive attribute and hence very poor flow behaviour (Table 1). The degree of angle of repose (around 49º) also confirmed that the flowability of SPP was very poor. Therefore SPP might have a greater tendency to compress under self-weight during storage in silos, which may adversely affected their handling properties (Crowley et al., 2014).

Emulsifying properties

Due to the diversity of methods used and pretreatments applied in the literature,

quantitative comparison with other studies was found to be difficult in terms of emulsifying properties and oil binding capacity. It has been previously reported that sunflower proteins can form stable emulsions (González-Perez et al., 2005). In addition, it has been shown that sunflower proteins showed similar or better emulsifying properties compared to soy proteins, carotenoproteins, skim milk powder and egg powder (González-Pérez and Vereijken, 2007; Shchekoldina and Aider, 2012; Sila et al., 2014). The emulsifying properties (EAI and ESI) for soy protein isolate were reported as 99.7 $m^2.g^{-1}$and 12.1%, respectively (Zhang and Zhao, 2013). In this study, those values (EAI and ESI) were determined as around 74 $m^2.g^{-1}$ and 5.25%, respectively (Table 2). Even though EAI of SPP was found lower than that of soy protein isolate, the data derived from this study indicated that SPP can be potentially used in food formulations.

Table 2. Some physico-chemical and powder properties of SPP

Angle of repose (°)	Hausner Ratio	Bulk density ($kg.m^{-3}$)	Tapped density ($kg.m^{-3}$)	Solubility (%)	Wettability (g water)	OBC (mL/g)	Emulsifying properties	
							EAI (m^2/g)	ESI (%)
49±2	1.58±0.11	403±14	639±19	3.75±0.52	0.39±0.03	2.85±0.43	73.69±0.44	5.25±0.15

EAI: emulsion activity index, ESI: emulsion stability index, OBC: oil binding capacity

The oil binding capacity (OBC) of proteins contribute to functional characteristics and taste of the food products particularly for meat and confectionery products (Pickardt et al., 2015). In addition, OBC is closely related to texture of food product and OBC is affected by bulk density of protein powder (Tanuja et al., 2012). The OBC value obtained from this study (2.85 $mL.g^{-1}$) was found to be lower than that of soy protein isolate (4.00 $mL.g^{-1}$) (Zhang and Zhao, 2013).

Solubility and wettability

Since the solubility of proteins is as an important phenomenon in terms of functional and technological properties, it needs to be determined and evaluated (González-Pérez and Vereijken, 2007; Saeed and Cheryan, 1988). Functional properties such as emulsifying and foaming properties are directly affected by solubility and lowest solubility of proteins was reported at around pH 4.0 (Karayannidou et al., 2007; Sila et al., 2014). Solubility may vary due to net charge of peptides and surface hydrophobicity (Sila et al., 2014). SPP had the solubility value around 3.75% in water (21 ± 2 °C) at pH 7.0. This is very low when compared with the data reported previously (Pickardt et al., 2011, 2015). They report the solubility of sunflower proteins in the range from 7 to 13% depending on the type of production process they applied. The denaturation of proteins due to thermal and chemical treatment during processing, binding of phenolics during extraction and precipitation and formation of firm aggregates during precipitation and separation might be considered to be responsible for low solubility of SPP (González-Pérez and Vereijken, 2007; Ivanova et al., 2012; Pickardt et al., 2011). It was reported that the increase in protein content resulted in decreased solubility (Babu et al., 2018) which explains the low solubility obtained from SPP in this study.

Wettability of powders are influenced by the size of the particles, the size of the pores in between and also contact angles and powders with high protein content lead to poor wettability and dispersibility (Ji et al., 2015). The wettability for milk protein isolate powder containing around 86% protein was reported as 0.236 g water (Ji et al., 2015). The wettability of SPP was found to be higher (0.390 g water) than that value in this study. The reason might be attributed to the variation of size of the particles used, particle surface properties and the gaps between the particles.

FTIR characterization

The FTIR spectrum of SPP is given in Figure 3. As can be seen in Figure, the peak around 3290 cm^{-1} was assigned to O-H and N-H group stretching vibration. The peak located around 3080 cm^{-1} was assigned to C-H stretching vibration. A component close to 2960 cm^{-1} was assigned to CH_3 asymmetrical stretching. Peaks around 1640 cm^{-1} and 1535 cm^{-1} were assigned to Amide I (protein C=O stretch) and Amide II (protein N-H bend, C-N stretch) groups, respectively. In addition, peaks around 1450 cm^{-1} and 1240 cm^{-1} were assigned to CH_2 bending vibration and C-N stretching properties, respectively (Chen et al., 2013; Kher et al., 2007). The FTIR spectrum of SPP exhibitied similar peaks to those of soy proteins (Chen et al., 2013) and milk protein concentrate powders (Kher et al., 2007).

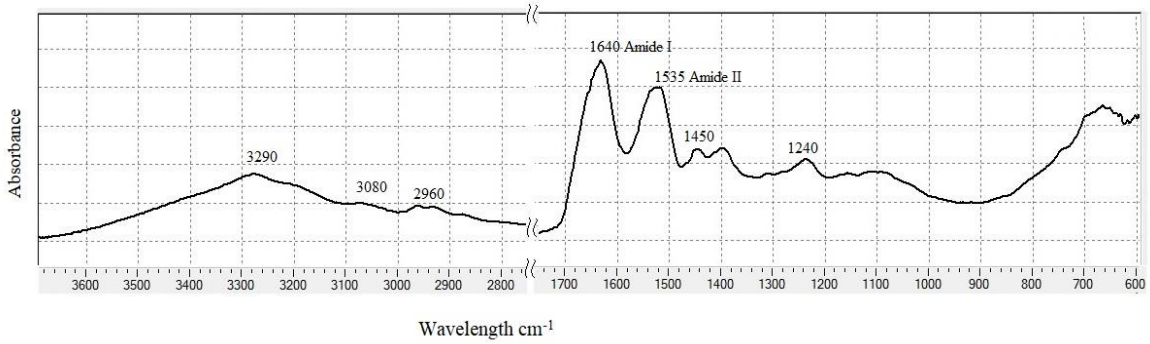

Figure 3. FTIR spectra of SPP

DSC characterization

The thermal properties of SPP was determined using differential scanning calorimetry and DSC curve is shown in Figure 4. SPP showed an endothermic peak at around 80 ºC which was in agreement with the findings of previous researchers (Amagliani et al., 2016; Fitzsimons et al., 2007). The curve in the temperature range of 40–80 °C, indicated denaturation of the protein structures (Hashimoto et al., 2004) and thus it could be used to evaluate the thermal stability.

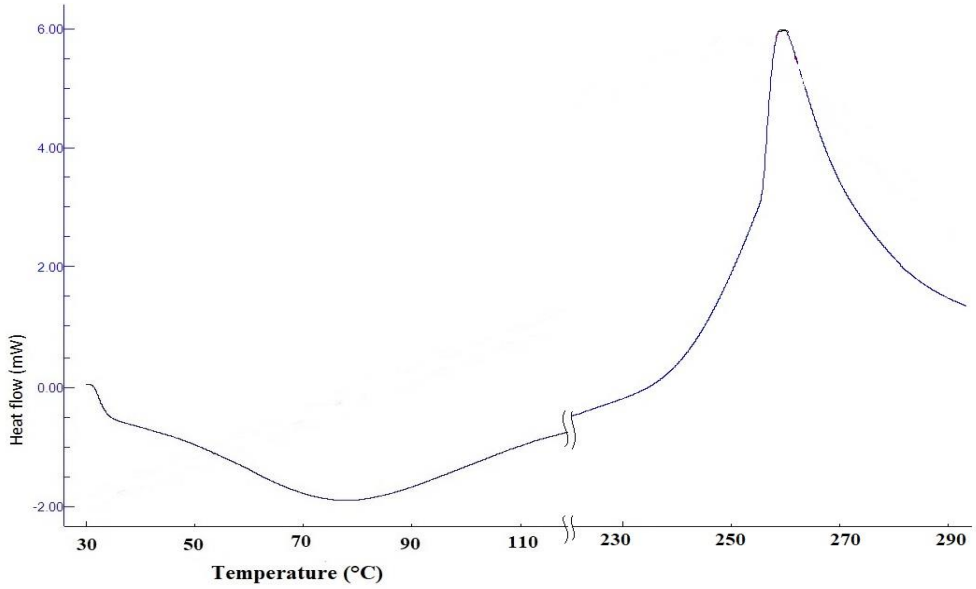

Figure 4. DSC thermogram of SPP

CONCLUSION

In this study, proteins were extracted from de-oiled sunflower seed cake (a by-product of industrial oil production from heat-treated sunflower seeds), precipitated and its powder form was obtained using a pilot scale spray dryer. Some physical, functional and microstructural attributes were investigated. The powders exhibited varying physical and functional characteristics which adversely affected the flowability. Low flowability indicated the challenges in bulk flow of the powders and the need for higher energy to make the powder flow at unconfined conditions. The powders exhibited low solubility while the emulsifying and wettability properties were found to be satisfying. The results obtained in this study provided useful information for the evaluation of the behaviour of SPP at industrial applications such as storage, handling and processing and of potential use in formulations of vegan, confectionary and meat products.

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Declaration of interest statement

The authors declare that there is no conflict of interest related to this study.

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