Research Article

Functional Properties of Water Holding Capacity, Oil Holding Capacity, Wettability, and Sedimentation of Swell-Dried Soybean Powder

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Abstract: Functional properties of expanded granule soybean powder issued from swell-drying were studied and compared with those of conventional ground powder. Swell-dried soybeans were textured by Instant Controlled Pressure Drop DIC and subsequently airflow dried before grinding. The obtained results showed a remarkable increasing of swell-dried soybeans in both Water Holding Capacity and Oil Holding Capacity. The increasing ratios were found to be up to 51% and 57%, respectively. Furthermore, the time required for rewetting expanded granule powder was about 51 s against 10 min in the case of conventional powder. No sedimentation was observed for expanded granule powder during the first 5 min of settling, and after 1 h, sedimentation volume kept almost constant with no significant difference. Response surface methodology was used for a multi-criteria optimization of DIC treatment in terms of the best functional properties. Satured steam pressure was the dominant parameter for all studied functional properties.

Keywords: Soybean; Expanded granule powder; Instant Controlled Pressure Drop DIC; Functional properties; sedimentation.

INTRODUCTION
Consumer requirements for high quality, minimally processed products have increased remarkably in recent years. Preferences have shifted towards the fresh, healthy and rich flavored ready-to-eat foods with an enhanced shelf life. Increasingly studies aiming at defining adequate process-intensification strategy have been performed. On one hand, several food technology studies have been carried out to define new high performance processes reducing energy consumption, improving positive environment impact, and facilitating the storage of the end products. On the other hand, a useful multi-criteria optimization requires a global approach that includes all the quality characteristics of the studied products. Hence, it is necessary to optimize the quality at sensory, convenience, functional, nutritional safety, and shelf life attributes.

Soybeans have been cultivated in China for over 5000 years, to become a "miracle plant", providing protein and oil in the world [1]. Soybeans vary widely in nutrient content based on the specific variety and growing conditions, but typically they contain 35-40% protein, 15-20% fat, 30% carbohydrates, and around 5% minerals and ash [2]. Soy-based foods may be divided into four main classes namely; traditional and second-generation foods, traditional and functional ingredients [3]. To be a convenient food ingredient, soy is available as flours, powders or isolates. The two main conventional methods for producing soybean powders are spray drying and grinding after conventional airflow drying.

Spray drying is the most known method to produced powders of a wide variety of food extracts. However in some cases, spray drying is less effective owing to the high cost of both initial investment and production [4]; this leads to less competitive products in the market. The system specifically devoted for soymilk was described by Perez-Munor and Flores [5] and the conditions to improve the final powder quality were defined [6, 7]. The functional properties were reported to need too fine particle size in order to increase the specific surface area [8]. This leads to many complex problems of thermal degradation of products and also transportation, cleaning, health problems, risks of explosion, etc. [9]. Thus, it is usually important to add a final post-treatment of fluidized bed agglomeration[7]
to preserve the functional behaviors while conveniently performing storage, transporting and handling.

The technique of powder production thru the grinding process after conventional airflow drying is cheaper and easier to use than spray drying[3]. It also allows obtaining full-fat/full-fiber soy flour. However, these ground powders have generally compact-structure granule, which significantly influences their functional properties [10]. Besides, the grinding process of compact-texture beans is so difficult thus generating punctual high temperature caking.

The functional properties of powders are normally linked to the interaction between water/oil and powder. They also are associated to the properties related with the protein structure, rheological characteristics, protein surface and the compatibility with other food components [11]. These functional properties are highly dependent on the specific surface area, which is related to structural parameters (the total pore volume, porosity, mean pore radius, particle mean size, particle size distribution and presence of fine particles) [12]. Soy-based meals, concentrates, and isolates can be subjected to thermal, physical, chemical and enzymatic treatments in order to obtain the desirable structural modifications and functional properties.

Instant Controlled Pressure Drop DIC as a texturing process was used usually to couple the conventional hot air drying thus defining swell-drying technique [13-15]. In the case of oilseeds, it additionally has two different objectives; firstly, to effectively eliminate the anti-nutritional factors of soybean and secondly to intensify the subsequent processes such as oil extraction. Haddad and Allaf [16] showed a dramatically decreasing in soybean content from trypsin inhibitors, whose activity decreased by 94% after 1-min DIC treatment and by 99% after 6-min treatment. Similar behavior was observed on oilseed allergenicity.

On the other side, as the most of functional properties are related to the texture of powder issued from spray drying or from airflow/grinding process, DIC-texturing process is able to modify the compact granules, and subsequently improve the different functional properties, as studied by different authors [17-21].

So, the aim of this study was to use DIC process to modify soybean texture. By assessing the main functional properties (WHC, OHC, wettability index, and sedimentation index), DIC process could be compared to unprocessed soybeans and optimized.

MATERIALS AND METHODS

Raw materials

Soybeans used for this study were purchased from Tat Hui Foods Co. Ltd, China. The whole soybeans have been cleaned and the impurities were removed before using. About 400g of soybean were used for each trial.

DIC treatment

Treatment protocol

The operating protocol used for soybeans texturing by DIC is shown in
DIC reactor

DIC reactor was described in many works [13, 14, 19]. DIC is a three main parts reactor (Fig. 2) of:

- A processing vessel where high pressure/high temperature (up to 1 MPa and 180 °C) and separately vacuum can be performed,
- A vacuum tank whose volume is 100 folds greater than that of processing vessel,
- An instantaneous valve, which ensures complete connection between (1) and (3) in less than 0.050 s (Fig. 2).
Fig. 2: Schematic presentation of DIC reactor: (1) treatment vessel; (2) controlled instant pressure-drop valve; (3) vacuum tank; (A) steam generator; (B) condenser; and (C) vacuum pump

Experimental design

Table 1: Experimental design for DIC treatment of soybeans

<table>
<thead>
<tr>
<th>Trial no.</th>
<th>Coded level of variables</th>
<th>Actual level of variables</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>$x_1$</td>
<td>$x_2$</td>
</tr>
<tr>
<td>DIC 1</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>DIC 2</td>
<td>1.4142</td>
<td>0</td>
</tr>
<tr>
<td>DIC 3</td>
<td>0</td>
<td>1.4142</td>
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<tr>
<td>DIC 4</td>
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<td>DIC 6</td>
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<td>DIC 7</td>
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<td>DIC 8</td>
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<td>DIC 9</td>
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<td>DIC 10</td>
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<td>-1.4142</td>
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<tr>
<td>DIC 13</td>
<td>0</td>
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</tr>
</tbody>
</table>

The effects of various DIC treatments on the different response parameters (wettability, water and oil holding capacity, and sedimentation index) were studied. Preliminary trials were performed. They allowed selecting the most important operating parameters and defining their ranges. They were the saturated steam pressure $P$ in MPa and holding time $t$ in s. A 2-parameter, 5-level central composite rotatable experimental design was used. It comprised $N_f=2^2=4$ factorial points, $N_A=2\times 2=4$ axial points and $N_o=5$ central points (five replications). They are coded to lie at ±1 for the factorial points, 0 for the central points and ±$\alpha$ for the axial points ($\alpha = \sqrt[4]{2^5} = 1.4142$). The 13 runs were achieved in random to minimize the effects of unexpected variability which due to external factors. The analysis was carried out with the statistical program (Statgraphics, Centurion XV, USA).

Assessment protocol

Characterization of samples was normally carried out after cooling and storage at ambient conditions for 24 h in an airtight bag in order to ensure good homogenization of the powder.

Water holding capacity

Functional properties are controlled by the composition and structure of proteins and the interactions of proteins with one another and with other substances. Water-holding capacity (WHC) is an important protein–water interaction that occurs in various food systems. WHC represents the ability of a protein matrix to absorb and retain bound,
hydrodynamic, capillary, and physically entrapped water against gravity [22]. Water holding capacity of soy protein is very important as it affects the texture, juiciness, and taste of food formulations and in particular the shelf-life of bakery products [3].

Water Holding Capacity (WHC) was determined according to Heywood et al. [23] and Traynham et al. [24] but with some modifications. 2.5g soybean powder was weighed in pre-weighed 30 mL plastic centrifuge tubes. For each sample 10 mL of distilled water were added and well mixed with the sample. Samples stood at room temperature (22 ± 2°C) for 30 min. The mixture was centrifuged at 1200 g (3709 rpm) for 30 min (Model 3K 15, motor 11133, SIGMA, Germany). Just after centrifugation, the supernatant was carefully decanted and the new mass of the sample was recorded. WHC (g water / g powder) was calculated as:

\[
\text{WHC} = \frac{\text{Total water mass}}{\text{Dry matter mass}} \quad \text{Eq. 1}
\]

Oil holding capacity

Oil Holding Capacity (OHC) was calculated according to Chakraborty [25] with slight modifications. 2g soybean powder was weighed in pre-weighed 30 mL plastic centrifuge tube. For each sample, 20 mL of refined vegetable oil (sunflower oil, density = 0.89877 g. ml\(^{-1}\)) were added and well mixed with the sample using a Vortex mixer at the highest speed, the samples were subsequently allowed to stand at room temperature (22 ± 2°C) for 30 min. Sample-oil mixture was centrifuged at 1200 g (3709 rpm) for 30 min (Model 3K 15, motor 11133, SIGMA, Germany), the supernatant was carefully decanted and the new mass of the sample was recorded. OHC (g oil / g dry powder) was calculated as:

\[
\text{OHC} = \frac{(m_{\text{oil}e} - m_d)}{m_d} \quad \text{Eq. 2}
\]

Where \(m_d\) and \(m_{\text{oil}e}\) are the mass of dry material and the mass of sample including held oil, respectively.

Wettability index (WI)

The term “wettability” describes the capacity of the particles to absorb water on their surface, thus initiating reconstitution. Wettability is defined as liquid penetration into a porous system due to capillary action or the ability of powder to be penetrated by the liquid [10], while wettability index reflects how long, the particles become wet during reconstitution process reflects.

The wettability index is defined as the time (in seconds) required for wetting all particles of a specified amount of powder (sink under the water surface) when placed on the surface of the water at a specified temperature. It is determined by the method described by NIRO [26] but with slight modifications. 100 ml of distilled water at 25°C were poured into a 400 ml beaker (diameter 70mm). A glass funnel (height 100 mm, lower diameter 40 mm, upper diameter 90 mm) was placed and maintained on the upper edge of the beaker. A test tube was placed within the funnel to block the lower opening of the funnel. 3g powder is placed around the test tube; while the timer is started, the tube is simultaneously elevated. Finally, the time is recorded when the powder is completely wet (visually assessed that all powder particles have diffused into the water). The measurement is performed at least twice for each sample and until the relative difference between the two results do not exceed 20%.

Sedimentation index (SI)

Sedimentation is simply the process in which a suspension is allowed to settle under the gravity action. Sedimentation index is determined according to Naega [27] but with some modifications, about 1.3g of soybean powder was transferred into a 25 ml graduated cylinder. 20ml of distilled water were added, the mixture is stirred for 5 min using vortex shaker (Rotolab OSI) with an interval of 30 sec. After agitation, the cylinder is immediately held vertically and the sedimentation volume is recorded (mL) every 10 min during one hour except the first 5 min.

RESULTS AND DISCUSSION

Water holding capacity WHC

The obtained results show that water holding capacity WHC is increased under all DIC processing conditions (Table 2), it is generally increased by 43% in case of expanded granule powder compared to those issued from grinding soybean. The higher the saturated steam pressure, the higher the WHC up to 51%, this was obtained with 0.63 MPa as saturated steam pressure, the higher the saturated steam pressure, the higher the WHC increased under all DIC processing conditions (Table 2), it is generally increased by 43% in case of expanded granule powder compared to those issued from grinding soybean. The higher the saturated steam pressure, the higher the WHC up to 51%, this was obtained with 0.63 MPa as saturated steam pressure, the higher the WHC increased under all DIC processing conditions (Table 2), it is generally increased by 43% in case of expanded granule powder compared to those issued from grinding soybean. The higher the saturated steam pressure, the higher the WHC up to 51%, this was obtained with 0.63 MPa as saturated steam pressure, the higher the WHC increased under all DIC processing conditions (Table 2), it is generally increased by 43% in case of expanded granule powder compared to those issued from grinding soybean. The higher the saturated steam pressure, the higher the WHC up to 51%, this was obtained with 0.63 MPa as saturated steam pressure, the higher the WHC increased under all DIC processing conditions (Table 2), it is generally increased by 43% in case of expanded granule powder compared to those issued from grinding soybean. The higher the saturated steam pressure, the higher the WHC increased under all DIC processing conditions (Table 2), it is generally increased by 43% in case of expanded granule powder compared to those issued from grinding soybean. The higher the saturated steam pressure, the higher the WHC increased under all DIC processing conditions (Table 2), it is generally increased by 43% in case of expanded granule powder compared to those issued from grinding soybean. The higher the saturated steam pressure, the higher the WHC increased under all DIC processing conditions (Table 2), it is generally increased by 43% in case of expanded granule powder compared to those issued from grinding soybean. The higher the saturated steam pressure, the higher the WHC increased under all DIC processing conditions (Table 2), it is generally increased by 43%

Comparing the operating parameters of DIC, saturated steam pressure and thermal holding time, the later has no detrimental effect on WHC while, saturated
steam pressure is the most influencing parameter (Fig. 3) as mentioned above. These findings agree with those found by He et al. [28], he found an increase of 29.46% as maximum value in WHC of PPI (peanut protein isolate) treated by high pressure HP at 200MPa for 5 min. This increasing was gradually as a result of pressure increasing which confirms our results; the higher saturated steam pressure, the higher WHC of soybean powder as a result of pressure drop (mechanical effect). But when comparing the two products, SD and HP, we can assume that there are other factors influence the WHC regardless the chemical composition which differ from one to other product. In case of SD soybean powder, the product is subjected to two effects; thermal and mechanical effect influencing more in protein modifications compared to HP products, in which the effect is limited to only mechanical one. Heating generally lessens protein WHC, because the denaturation/aggregation reduces the availability of polar amino groups for hydrogen bonding with water molecules [29]. However, heating can also unfold the protein and expose side chains that can bind water resulting in improved WHC. Kinsella [30] reported that the major factors affecting WHC are protein denaturation and unfolding, and presence of carbohydrates and non-protein components.

The obtained regression model for water holding capacity is as following with $r^2 = 94\%$.

$$WHC(\%) = 2.21 - 2.56P + 3.101P^2 + 0.012Pt - 0.0080t + 0.00002t^2$$  Eq. 3
Fig. 3: Impact of DIC operating parameters; saturated steam pressure (MPa) and thermal holding time (s) on the WHC (g water/ g powder) of expanded granule soybean powder (WHC of non-treated soybean powder=1.429 g/g)

These results corroborate those of several studies, which aimed at improving functional properties of the protein products. They were carried out through thermal processes used as pretreatment[31], as well as alkylation (Lys, Cys, Me, His and Try), oxidation (Cys, Me, His and Try), acylation (Lys and Try), esterification and amide formation (Glu and Asp) [11]. Sugar addition was also used to enhance whipping properties of oilseed proteins [32]. Besides, the addition of polysaccharides can form biopolymers, which are essential in stabilizing food formulations as foams, emulsions, and dispersions against heat and pressure [33]. Enzymatic modifications were used to break peptidic bonds leading to peptides with desired size, charge and surface properties. Similar to DIC treatment, enzymatic hydrolysis could increase protein solubility [11, 34]. Trypsin treatment of protein products resulted in higher solubility and water holding capacity than untreated products [35-37]. DIC impact can be compared to high pressure treatment, which also improves the functional properties of soybean proteins [38]. Other thermo-mechanical texturing ways, such as cooking-extrusion treatment, were proposed. Hojilla-Evangelista and Evangelista [39] obtained similar effect on functional properties (higher water holding,....) of milkweed proteins depending on temperatures and screw pressing.

Wettability index

In soybean food applications, wettability is one of the properties that may influence the overall reconstitution and/or mixing characteristics. Many of conventional soy powders need long time to wet reflecting poor wettability, because of low specific surface area and particle’s texture/microstructure, and chemical composition. DIC expanded granule powder (0.63 MPa during 105s) needed about 1min (51s) to rehydrate against 10 min in case of conventional dried powder (Table 2), the decreasing in time (improvement) is subsequently found to be 91.5%. However, under some DIC conditions (P: 0.45 MPa, t: 70s), a maximum decreasing in time is observed; it is found to be 96%.

Comparing the operating parameters of DIC texturing, we found that the most manipulated parameter was the saturated steam pressure; wettability time was continuously declined as long as the saturated steam pressure was increased till declining a maximum, and restart increased after declining a maximum. While, the influence of thermal holding time was slight compared to saturated steam pressure (Error! Reference source not found.).
This phenomenon may be due to:

1. Oil expulsion of soy bean under some conditions (soft conditions: low pressures) and its accumulation on the surface when the saturated steam pressure was increased, it involved soybeans defatting; oil removing from beans surface after pressure dropping. This results agrees with those assumed by Moure et al. [11] who mentioned that defatting of meals lead to increase OHC; Honig and Rackis [40] also showed losses in volatile fatty acid (lipooxygenase) of soybean when the pressure and processing time are sufficiently high, the hydrophobic binding sites that are blocked by this fatty acid are released, and usually available for binding hydrophobic substances.

2. Textural modifications occurred thru DIC processing as a result of swelling. Texture characteristics were studied by many authors [9, 15, 19-21] who mentioned that expanded granule powder has a spongy texture with numerous vacuoles within the particles. The open texture allows absorbing water quickly; moreover DIC texturing induces beans roasting which improves their wettability. Roasting can eliminate some of oil contained in beans, which may act as barrier inhibiting the reaction with water especially if this oil is found at particle’s surface.

The obtained regression model for wettability index is as following with $r^2 = 91\%$.

$$WI = 311.18 - 797.179P - 2.402t + 541.198P^2 + 3.640Pt + 0.0039t^2 \quad \text{Eq. 4}$$

### Table 2: Wettability, Water Holding Capacity WHC and Oil Holding Capacity OHC of soybean powders issued from dried raw material and DIC textured materials

<table>
<thead>
<tr>
<th>Parameter</th>
<th>WI (s)</th>
<th>WHC (g/g db)</th>
<th>OHC (g/g db)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Control</td>
<td>602</td>
<td>1.429</td>
<td>0.698</td>
</tr>
</tbody>
</table>
Oil holding capacity OHC

The obtained results show that oil holding capacity OHC is decreased under soft conditions of DIC (low saturated steam pressures) but, it is increased, after decreasing a maximum, with increasing the pressure of saturated steam. It is increased by 57% compared to unprocessed powder; this increasing in OHC is obtained under DIC conditions of P: 0.63 MPa as saturated steam pressure during 105 s as thermal holding time (Table 2). Soybean powder with high oil capacity is desirable for use in cold meat industry particularly for sausages, where the protein can bond the fat and water in these products [41].

These results can be explained by oil expulsion from soybean during DIC texturing inducing sort of beans defatting, these results agree with those assumed by Moure et al. [11], the author mentioned that defatting increased the water and oil holding capacities. Furthermore, the oil absorption is commonly attributed to the physical entrapment of fat by the protein [42]. On the other hand, OHC increasing at high saturated steam pressure can explain why the wettability index was increased under the same conditions; in fact the presence of oil on the external surface of particles acts as barrier layer inhibiting/delaying the interaction between particle surface and water.

Comparing the two operating parameters of DIC processing (Figure 5); saturated steam pressure and thermal holding time, the obtained results from Pareto Chart did not reveal a clear effect of these parameters, because the limits of these parameters were well defined and their values were close. He et al. [28] found that OHC of PPI (peanut protein isolate) treated by high pressure HP at 5 - 200 MPa were remarkably higher than the commercial soybean protein isolates.

The obtained regression model for oil holding capacity is as following with $r^2 = 82\%$.

$$OHC = 2.0957 - 3.157P - 0.015t + 2.96434P^2 + 0.0062Pt + 0.000079t^2$$  \hspace{1cm} Eq. \hspace{1cm} 5
**Fig. 5: Impact of DIC operating parameters; saturated steam pressure (MPa) and thermal holding time (s) on the OHC (g oil/g powder) of expanded granule soybean powder (OHC of non-treated soybean powder=0.698 g/g)**

**Sedimentation index**

*Sedimentation* property is important for soymilk prepared from powder because it reflects the stability of this milk versus time of settling. It reflects the suspension stability and particles properties in terms of hydrodynamic interaction between particles and surrounding medium [43, 44]. The gravity sedimentation is linked to the difference between the solid and liquid densities, but also depends on particle shape and size, particle size distribution, the solid's surface characteristics, and the liquid viscosity [45]. Thus, DIC expanded granule soybean powder presented no sedimentation particularly during the first 5 min except some points compared to unprocessed powder. This later recorded 2 ml from the interface for a total volume of 20 ml. While the optimal DIC treated powder had 10 ml of sediment volume just 5 min after settling. This sediment volume kept almost constant with no significant difference (8 ml) after 1 h.

This phenomenon can be explained by the compactness of unprocessed soybean powder, which encourages their settling during the first 5 min. However under severe conditions of DIC texturing, the thermal treatment induces the formation of aggregates. Since these aggregates are heavy, they settle quickly with the time mainly because of water absorption which contributes in increasing their weight.
The effects of the two operating parameters of DIC processing on the sedimentation index at 10 min are shown in Fig. 6. This figure shows that both the steam pressure and the thermal holding time are significant at greater than 95% confidence level, among them the steam pressure has a stronger influence than treatment time; the higher the pressure and the treatment time, the higher the sedimentation index. In other words, the appearance of the interface between a clear liquid and slurry phase takes place more slowly. This means that the stability of the soymilk is increased. The obtained regression model for sedimentation index is as following with $r^2=96\%$.

$$SI = 14.738 - 39.46P - 0.124t + 36.84P^2 + 0.22Pt + 0.000321t^2$$  \text{Eq. \ 6}$$

**Multi-criteria optimization**

The main quality attributes of DIC treated soybean powders were identified as closely depending on DIC treatment parameters. Optimization should normally be done versus the required quality, which depends on the industrial uses and the consumer needs. In the present case, we defined them as to be the highest values of Water Holding Capacity WHC, Oil Holding Capacity OHC, and Sedimentation Index, with the Wettability Index WI. In this study with the defined ranges of saturated steam pressure $P$ (from 0.2 MPa to 0.7 MPa), and treatment time $t$ (from 20 s to 120 s), DIC treatment was optimized to be DIC 5 ($P=0.63$ PMa and $t=105$ s). This allows WHC, OHC, SI, and WI to be: 2.15 (g/g db), 1.099 (g/g db), 10 ml (at 10 min) and 51 s, respectively; while they were: 1.429 (g/g db), 0.698 (g/g db), 2.3 ml (at 10 min) and 602 s, respectively for raw material.

**CONCLUSION**

Functional properties of soybean powders are controlled by the composition and structure of proteins. They also depend on the interactions between proteins and other substances. This study shows that DIC texturing of soybean can improve the functional properties of issued powder (expanded granule powder) thru the modification of protein texture and structure. Expanded granule powder has high WHC and OHC compared to unprocessed powder. Moreover, it does not need long time to be wetted. A large difference of sedimentation is recorded during the first 5 min between the two powders, while a non-significant difference is observed after 1 hour of settling. Comparing the operating parameters, we found that the saturated steam pressure was the dominant parameter for all the studied properties. The obtained results show also the flexibility of DIC texturing which means that DIC operating parameters can be modified and optimized in order to meet the industry and consumer needs and requirements.

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