Identification and characterization of soybean dreg soluble dietary fibre by combination of extrusion pre-treatment and enzymatic modification

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Abstract

OBJECTIVES: Soybean dreg is a by-product of soy milk processing, which contains high levels of soluble dietary fibre (SDF). In this study, we aimed to provide comprehensive processes of pre-treated extrusion for the improving structure and properties of soybean dreg soluble dietary fibre (SDSDF), which would be a valuable approach to enhance physiological activity.

METHODS: Here, we characteristic the functional role of SDSDF employing to extrusion pretreatment. Soybean dregs were pre-treated using the twin screw extrusion method followed by enzymatic modification using neutral protease, α- amylase, glucoamylase, and cellulose to produce SDSDF. The physical properties and antioxidant activity of SDSDF were investigated.

RESULTS: The morphology and crystal structure of SDSDF were observed that, through extrusion processing and enzymatic modification, the SDSDF yield increased by 106.28%. Moreover, the surface structure showed block-shaped or reticular formations in the extruded SDSDF, and the size of block-shaped cells was about 10 μm. Infrared spectroscopic analysis showed that a characteristic absorption peak of polysaccharide appeared at 1631 cm−1 during extrusion processing. However, after extrusion processing, decreased absorption peaks were observed for the extruded SDSDF. Furthermore, XRD analysis showed that the 2θ diffraction peak changed at 24.16° for the extruded SDSDF.

CONCLUSIONS: The overall findings suggest that the water holding capacity (WHC), oil holding capacity (OHC), expansibility, and the water solubility were significantly decreased in extruded SDSDF. In addition, the scavenging ability of 1,1-diphenyl-2-picrylhydrazyl (DPPH), -OH, O2•−, and the total reducing power were significantly improved, indicating that beneficial changes had taken place in the crystal structure of cellulose or hemicellulose to improve the physiological activity in extruded SDSDF.

Key words: Soybean dreg; Soluble dietary fibre; Extrusion; Enzymatic modification.

Introduction

Dietary fibre consists of non-starch polysaccharide substances such as hemicel lulose, pectins, mucilages, cellulose, and β-glucans. Thus, the physiological behaviour of these fibre components can be determined based on their unique chemical structures and physical properties, including water holding capacity (WHC), viscosity, bulk/volume, and fermentability (Schneeman et al., 1994). Additionally, dietary fibre is classified into soluble dietary fibre (SDF) and insoluble dietary fibre.
(IDF). Dietary fibre is defined by chemical and physical properties instead of unitary physical functions. In western countries, people consume a lot of foods containing high dietary fibre in order to have health benefits associated with lowering the risk of chronic diseases. Dietary fibres from separate sources are functionally distinct. Several meta-analyses and studies revealed the association between ingestion of cereal dietary fibres or whole-grain foods and reduced risk of type 2 diabetes and cardiovascular diseases (Salmeron et al., 1997; Abdul-Hamid and Luan, 2000; Schulze et al., 2004; Mellen et al., 2008; Kovatcheva-Datchary et al., 2015). In addition, recent studies indicated that it can lower the risk of colon or colorectal cancer (Larsson et al., 2005; Schatzkin et al., 2007). As a by-product of soybean processing, approximately 30–40% soybean dregs are produced after isolating the soybean protein and around 50% soybean dregs are produced after the processing of tofu or soybean milk. Soybean dregs are rich in nutrients, including dietary fibres and proteins. Therefore, they can be considered as a good source of dietary fibre; they contain approximately 60% dietary fibres (Dongmei et al., 2005; Mateos-Aparicio et al., 2010). The physiological activity of dietary fibres is defined by their composition and structure, and improved methods have allowed for the detection of differences in composition and structure that affect their physiological activities. The change or modification of fibre structures can affect the specific physiological functions of dietary fibres (Zhenshan et al., 2004).

Modification processes can greatly improve the characteristics of dietary fibres by altering its physicochemical properties and structural attributes, and improve its technological value. Physical, chemical, and enzymatic treatment methods can change the structure of dietary fibres or redistribute their composition to have specific physiological properties. Among these treatments, the enzymatic methods have been proved to be the most effective at improving the quality of dietary fibres (Lebesi and Tzia, 2012; Santala et al., 2014). The aim of the present study was to investigate the modification of soybean dreg soluble dietary fibre (SDSDF) by using a combination of extrusion pre-treatment and enzymatic modification. In the first stage, twin-screw extrusion was adopted for pre-treatment. SDSDF was prepared through enzymatic methods involving neutral protease, α-amylase, glucoamylase, and cellulase. The structural characteristics and physiological activities of extruded and unprocessed SDSDF (as control) were also investigated.

Materials and Methods

Materials

The wet soybean dreg samples were prepared by squeezing soybean milk (soybean:water = 1:10), and then filtered by 80-mesh sieve after desiccation. Neutral protease (60 000 U/g) and glucoamylase (100 000 U/g) were obtained from Beijing Sola-bio Science & Technology Co. Ltd. α-Amylase (3700 U/g) was purchased from Beijing Aoboxing Bio-tech Co. Ltd. Cellulose (0.57 U/mg) and 1,1-diphenyl-2-picrylhydrazyl (DPPH) was purchased from Sigma. The other reagents were of analytic grade.

Basic chemical composition analysis

The protein content in soybean dreg dietary fibres was determined by using the Kjeldahl method. A conversion factor of 6.25 was used to calculate protein on the basis of nitrogen level. The Soxhlet method using the Kjeldahl method. A conversion factor of 6.25 was used to calculate protein on the basis of nitrogen level. The Soxhlet method was used to extract fat with diethyl ether for 24 h. The moisture content was determined according to the method GB 5009.3-2010. Ash was determined by the standard method GB 5009.3-2010 that involves weighing following incineration in a furnace at 550°C. Total dietary fibre (TDF) was determined following the AACC32-06 standard method.

Extrusion pre-treatment

Extrusion experiments were performed using a laboratory scale twin screw extruder (DSE25, Brabender Technology GMBH & CO. KG, Germany). Extruder was constituted by combined sleeved (5 joints) and screws (5 joints) with a die head diameter of 2 mm × 20 mm and a screw diameter of 25 mm. The extrusion processes were carried out under optimal operating conditions for soybean dregs and adjusted based on verified experience. The screw speeds were from 0 to 530 rpm/min. The temperature of extrusion in the barrel was from 0°C to 400°C. The preliminary study shows that the moisture content in soybean dreg samples was 20% (data not shown).

Enzymatic preparation of SDSDF

Enzymatic hydrolysis (solid–liquid ratio at 1:20) with 0.5% neutral protease (50°C, pH 7) was adopted on the extruded SDSDF and the untreated negative control (without extrusion pre-treatment). After the enzymatic hydrolysis of SDSDF for 1.5 h, the enzyme (neutral protease) in the samples was inactivated in a boiling water bath for 10 min. Then α-amylase and glucoamylase (0.6% w/v) were added to the samples and incubated for 1.5 h at 60°C and pH 5.5. The enzymes were inactivated in a boiling water bath for 10 min. Then, the samples were mixed with 0.5% cellulose (50°C, pH 4.5) and incubated for 1.5 h. After enzymatic hydrolysis, the enzymes were denatured in a boiling water bath for 10 min. After centrifugation and filtration, the enzyme-treated samples were mixed with anhydrous ethanol at a ratio of 1:4 (w/v) to precipitate SDF for 1 h, and centrifuged at 4000 rpm for 5 min. The residues were freeze-dried to obtain the SDSDF samples.

Determination of physical properties of SDSDF

Determination of WHC and expansibility

The WHC of SDSDF was measured according to published methods (Qingling et al., 2014). Forty millilitres of distilled water were added to 1 g of the SDSDF sample in a 50 ml centrifuge tube and placed in a refrigerator for a night. After centrifugation at 4000 rpm for 30 min, the supernatant was discarded and the residue was weighed (m2). The WHC was calculated as grams water per gram of dry sample: (g/g) = (m2 − m1)/m1.

The expansibility of SDSDF was determined according to the method of Anping et al. (2010), with some modification. One gram of the SDSDF sample (m) was placed a 20 ml graduated test tube and compressed to flat. The volume of the dry material was then measured (V1), and the sample was vortexed with 15 ml of distilled water and placed in a refrigerator at 4°C overnight. The volume of the material was then measured (V2). The expansibility of SDSDF was calculated as follows:

\[ \text{Expansibility} \ (\text{mL/g}) = \frac{(V_2 - V_1)}{m} \]

Determination of oil holding capacity

The same protocol was followed as described for the determination of WHC, but virgin olive was used instead of water. The oil holding capacity (OHC) was expressed as grams olive oil retained per gram of dry sample.

Determination of water solubility

To determine the water solubility of SDSDF, the weighted sample powder (m) was mixed with 50 ml of distilled water in a 200 ml conical flask under stirring, and then the mixture was vortexed continuously at 90°C for 30 min in a constant temperature oscillation water bath.
Identification and characterisation of SDSDF, 2017, Vol. 1, No. 2

The chemical composition of dried soybean dregs is listed in Table 1. The soybean dregs contained 69.41% of total dietary fibres, and thus it can be considered as a natural source of dietary fibre. The preparation of soybean dregs is of great importance in soybean processing. The present study could provide useful information to utilize by-products of soybean processing.

Antioxidant activity of SDSDF

**DPPH radical-scavenging ability**

Antioxidant capacity was evaluated based on radical-scavenging activity using the DPPH (2,2-diphenyl-1-picrylhydrazyl hydrate) method as reported by Qingling et al. (2014). The DPPH solution was made by mixing anhydrous ethanol and DPPH to a final concentration of 0.2 mmol/l. Four millilitres of DPPH solution were added to different concentrations of the SDSDF solution and mixed. The mixtures were kept in the dark for 30 min and transferred to cuvettes. The measurements were done in triplicate at the wavelength of 517 nm. DPPH was used as control (A0), and the blank contained methanol (A1). The antioxidant activity was determined by

\[
\text{Clearance ratio of DPPH} = \left( \frac{A_0 - (A_1 - A_2)}{A_1} \right) \times 100 / (A_1 \text{ control})
\]

Determination of SDSDF -OH scavenging rate

Four millilitres of phosphate buffer solution (pH 7.4) and 1.5 ml of adjacent concentration nitrogen solution (5 mmol/l) were mixed. Then, 1.0 ml of FeSO4 solution (7.5 mmol/l), 1.0 ml of different concentrations of SDSDF solution and control solution, and 1.5 ml of double distilled water were added. Finally, 1.0 ml of H2O2 solution was added into tubes, and the mixture was then incubated in a water bath at 37°C for 1 h (Zhike and Shen, 2009). The measurements were done in triplicate at the wavelength of 536 nm.

\[
\text{Clearance ratio of -OH} = \left( \frac{A_1 - A_2}{A_1 - A_2} \right) \times 100
\]

where \( A_1 \) represents the light absorption value of the solution containing SDSDF samples and \( A_2 \) is the light absorption value of \( \text{H}_2\text{O}_2 \). \( A_3 \) represents the light absorption value of solutions without SDSDF sample and \( \text{H}_2\text{O}_2 \).

Determination of \( \text{O}_2^- \) scavenging rate of SDSDF

Five millilitres of phosphate buffer solution (0.075 mmol/l, pH 8.2) were mixed with 1.0 ml of different concentration SDSDF solution and control, then shaken in a water bath at 25°C for 20 min. One millilitre of pre-heated pyrogallic acid (4.5 mmol/l) was added to each sample and mixed. The mixtures were then incubated in a water bath at 25°C for 4 min. The reaction was terminated by the addition of HCl (8 mol/l). Finally, the absorbance was measured at the wavelength of 420 nm (A1), measuring the light absorption value of pyrogallic acid with deionized water (A0), and measuring the light absorption value (A2) of SDSDF samples (Taoyun and Juan, 2010). The \( \text{O}_2^- \) scavenging rate of SDSDF was then calculated as follows.

\[
\text{Clearance ratio of } \text{O}_2^- / (\%) = \left( A_0 - (A_1 - A_2) \right) / A_0 \times 100
\]

**Determination of total reducing power in SDSDF**

For total reducing power analysis, 2.5 ml of 0.2 M phosphate buffer (pH 6.6) containing 1% ferrocyanide was added to different concentrations of SDSDF sample.

The mixture was incubated at 50°C for 20 min. Then 10% trichloroacetic acid (TCA, 2.5 ml) was added to a portion of this mixture (5 ml) and centrifuged at 3000 rpm for 10 min. The supernatant was separated and mixed with distilled water (2.5 ml) containing 1% ferric chloride (0.5 ml) solution. The absorbance of the mixture (A) was measured at 700 nm. The negative control (A0) was prepared in a similar manner, excluding the addition of SDSDF sample (Taoyun and Juan, 2010).

\[
A_p = A - A_0
\]

where \( A_p \) represents the total antioxidant capacity.

**Structural characterization of SDSDF**

**Ultrasound observation of SDSDF**

To examine the discrepant surface structure of SDSDF and untreated SDSDF control, scanning electron microscopy (SEM, S3400-N, Hitachi, Japan) was performed following the gold spraying process using the sputtering deposition method. The dried SDSDF samples were placed on a carbon tape mounted to the SEM holder and SDSDF samples were observed at 20 kV voltage.

**Infrared spectrum detection of SDSDF**

Infrared spectrum of treated SDSDF and non-treated control samples were obtained using Fourier transform infrared spectroscopy (L1600401, Liantrisant, UK). Two milligrams of the SDSDF sample were ground with KBr (100 mg, spectroscopic grade), sheeted to one slice, and scanned with a blank KBr background. To compare the structural changes in the treated SDSDF and the negative control of non-treated samples, Fourier transform infrared spectroscopy was performed at the spectral range from 400 to 4000 cm\(^{-1}\).

**XRD analysis**

XRD analysis of SDSDF was carried out using a JDX-10P 3A diffractometer (Japan Electron Optics Laboratory, Tokyo, Japan). The XRD patterns were obtained by using a copper target to examine the change in crystal structure. Test conditions were as follows: operating voltage, 40 kV; current, 40 mA; fixed divergence slit; scanning range, 5–35°; scanning step, 0.013°; scanning speed, 32.895; scanning mode, continuous. The XRD patterns of sample and standard pdf cards of each material were compared using the Jade 5.0 software to analyse phases.

**Statistical analysis**

All measurements were performed at least in triplicate. All statistical analyses were carried out by using SAS 8.0 for windows. \( P < 0.05 \) was considered to be significant by Duncan’s test.

**Results and Discussion**

**Chemical composition of soybean dregs**

The chemical composition of dried soybean dregs is listed in Table 1. The soybean dregs contained 69.41% of total dietary fibres, and thus it can be considered as a natural source of dietary fibre. The present study could provide useful information to utilize by-products of soybean processing.
Extraction rate analysis of SDSDF

The SDF content produced from the non-extruded soybean dregs was 6.21%, and close to double amount of SDSDF (12.41%) was produced from the extruded samples. The result suggested that the pre-treatment of extrusion process significantly increased the yield of SDSDF. This might be because fibre carbohydrates can be degraded at high temperatures, especially when the samples move to the last stage of the extrusion process where they are exposed to 240°C to 400°C temperatures. Also high temperature and shear of screw extruder would break the glycosidic bond, that is some C–O C–C bonds were broken during extrusion process, resulting in an increase of soluble fibre contents. Therefore, the results suggested that the yield of SDSDF was mainly affected by temperature during extrusion. The dehydrated glycosyl as fibre carbohydrate could promote solubility of dietary fibre, and SDF yield also increased by shear stress (Huiqin et al., 2012).

Physical property analysis of SDSDF

The results showed that the WHC, OHC, expansibility, and water solubility of the SDSDF were reduced to different degrees after extrusion pre-treatment and enzymatic modification (Table 2). The SDSDF level rises in samples with the combined extrusion and enzymatic treatment relative to control. Large molecules were cracked easily, which amplifies the degree of decomposition of fibre molecules, and thus transformed into smaller molecules with high solubility in water. The parameters of extrusion processing had some influence on the physical properties of SDF. When the moisture content of the sample is higher, expansibility and WHC were all reduced with increasing extrusion temperatures (Altan et al., 2009; Ying et al., 2014).

Oxidation resistance analysis of SDSDF

Detection of DPPH radical-scavenging ability of SDSDF

The results showed that the DPPH scavenging activity rate of SDSDF was increased with increasing sample concentrations, especially at high concentrations of SDSDF. The SDSDF in the extruded samples exhibited a significantly higher scavenging activity rate compared with the SDSDF in the control non-treated samples (Figure 1). The radical scavenging rate of DPPH was up to 76.20% in the extruded SDSDF, which was 5.57% greater than control non-treated samples. The results indicated that extrusion pre-treatment enhanced the radical scavenging ability of SDSDF by modifying the molecular and structural properties of SDSDF, and this was positively correlated with the SDSDF concentrations.

Determination of SDSDF ·OH scavenging rates

Similar to DPPH scavenging activity, the ·OH scavenging activity of SDSDF increased with increasing sample concentrations. The assay of ·OH scavenging rate showed that the impression of extrusive SDSDF had a prominent scavenging rate compared with the control group (Figure 2). The ·OH scavenging rate of the extruded SDSDF was 20.24% at concentration 1 mg/L, which was 221.27% higher than the control group. This can be explained by the fact that the antioxidant activity depends on the structure of SDSDF, in particular the number and positions of the hydroxyl groups and the nature of substitutions on the aromatic rings (Balasundram et al., 2006). After extrusion and enzymatic hydrolysis, the hydroxyl group in the treated SDSDF increased the ability of reaction, resulting in increased scavenging activity of SDSDF. The results suggest that the ·OH scavenging rate reached the highest level with high concentrations of SDSDF.

Table 1. Component and mass fraction in dry weight of soybean dregs.

| Component | Protein (mass per cent) (%) | Fat (mass per cent) (%) | Water (mass per cent) (%) | Ash (mass per cent) (%) | TDF (mass per cent) (%) |
|-----------|-----------------------------|-------------------------|---------------------------|-------------------------|------------------------|
|           | 12.07 ± 0.27                | 5.52 ± 0.16             | 9.91 ± 0.13               | 3.06 ± 0.11             | 69.41 ± 0.5            |

Table 2. Functional properties of dreg soluble dietary fibre.

|                      | WHC (g/g) | OHC (g/g) | Expansive ability (ml/g) | Water solubility (%) |
|----------------------|-----------|-----------|--------------------------|----------------------|
| Non-extruded SDSDF  | 2.10 ± 0.03 | 3.42 ± 0.02 | 12.50 ± 0.03             | 34.00 ± 0.08         |
| Extruded SDSDF      | 1.70 ± 0.01 | 2.18 ± 0.04 | 10.50 ± 0.02             | 30.00 ± 0.11         |
Determination of O$_2^-$ scavenging rate
The results showed that O$_2^-$ scavenging activity of SDSDF increased with increasing sample concentrations of SDSDF. The O$_2^-$ scavenging activity in the extruded SDSDF was higher than the control group when the concentrations of SDSDF were greater than 0.6 mg/ml, but no significant difference was found at lower concentrations (Figure 3). When the concentration reached to 1.0 mg/ml, the O$_2^-$ scavenging rate of the extruded SDSDF was 53.85% (12.23% > control). These results indicate that the O$_2^-$ scavenging rate was highly related to the concentration of SDSDF.

Detection of total reducing force in SDSDF
The results revealed that the total reducing force in SDSDF increased with increasing SDSDF concentrations and that the extruded SDSDF was always higher than the control group. The extrusion pre-treatment increased the total reducing force by 193.81% compared with the control group (Figure 4). The results suggested that the total reducing force was positively correlated with the concentration of SDSDF. It is possible that the oxidation resistance increased in different degrees after degradation of polysaccharides (Jeddou, 2016; Xizhen et al., 2016). In this study, oxidation resistance of extruded SDSDF increased in a great degree, especially for the radical scavenging and the total reducing force. The results demonstrated that the polysaccharides in the SDSDF was degraded after extrusion, resulting in an increase in their oxidation resistance.

Ultrastructure of SDSDF
To investigate the ultrastructures of SDSDF, pre-treatment of extrusion and the enzymatic hydrolysis were carried out. As shown in Figure 5, the surface structure of the extruded SDSDF has a notable change. Furthermore, loose structure, regular comb holes, and bigger superficial area were observed in the extruded SDSDF compared with the control (Figure 5A–D). Larger changes were also observed in the crystal structures of the extruded SDSDF. There were many block-shaped or dot-like spherical and network-like structures on the surface of the extruded SDSDF, and the size of the dot-like spherical cell was around 10 μm, indicating that the unit was with mutual cross-linking by chemical bonds to assemble into the new micro-structure (Figure 5E–H). Therefore, these results suggested that the water solubility of SDSDF was improved by extrusion pre-treatment, and other functional properties may also change.

Previously, Gaoshuang et al. (2012) study indicated that the quantity of SDSDF increased gradually as its particle size decreased. After soybean dregs were pre-treated with superfine grinding processing, the WHC, expansibility, water retaining capacity, and OHC were decreased, because the high temperature, high pressure, and high shear force caused the chemical bonds of high polymer dissociated (Chuanfu et al., 2008). In the present study, the results showed that the WHC, HOC, expansibility, and water solubility of SDSDF were decreased because the external state and internal molecular structure of SDSDF were changed by high swelling pressure which was generated by combined extrusion and enzymatic hydrolysis with neutral protease, α-amylase and glucoamylase.

Analysis of infrared spectrum of SDSDF
The SDSDF produced from soybean dregs is mainly formed from soybean polysaccharides, a kind of pectin polysaccharide substance that contains lots of galacturonic acid. According to infrared spectroscopic analysis, there were characteristic absorption peaks of polysaccharides with and without extrusion processing (Figure 6), which appeared between 3000 and 3700 cm$^{-1}$ were due to O–H stretching vibration of the hydroxyl group in the sugar ring. The peaks that appeared at 2900 cm$^{-1}$ nearby were due to C–H asymmetric stretching vibration of C–CH$_2$–C, and the peaks appeared at 1650 cm$^{-1}$ close by were due to C=O stretching vibration of the aldehyde group. There was an intensive absorption appeared at 1631 cm$^{-1}$ nearby (Figure 6), that is CO–NH absorption peak, in the control group, indicating that sugar amine condensation bonds actually exist.

Lining et al. (2009) reported that protein denaturation occurred after extrusion processing of soybean. The absorption peak decreased significantly in SDSDF after extrusion processing, indicating that protein denaturation could be the result of the reduction of hydroxylamine condensation bonds under high temperature and pressure conditions.

Analysis of XRD
Cellulose-type materials were made up by 70% orderly crystalline cellulose, 30% disorderly amorphous cellulose, and hemicellulose...
The XRD analysis showed that there were crystal structures and amorphous structures in SDSDF (Figure 7). The destruction of diffraction peak intensity was not serious, compared with the nearby crystalline region. In the process of twin screw extrusion, the order of the natural crystal structure of SDSDF was changed to some extent, which could help its enzyme degradation. In general, the diffraction peak represents cellulose and hemicellulose that have a range of $2\theta$ between 15° and 25° (Gidley, 1992). The result revealed that the diffraction peak of the extruded SDSDF was changed by 24.16°, indicating that the crystalline and amorphous structure of SDSDF were changed by extrusion pretreatment, which is consistent with the results of SEM detection.

Conclusions

Based on all the experimental data, the physicochemical properties and surface structure of SDSDF was proposed. The SDSDF yield increases by 106.28% with extrusion processing. The functional properties such as WHC, OHC, expansibility, and water solubility were lower in the extruded SDSDF compared with the control non-extruded samples. The detection of oxidizability and reducing power showed that scavenging ability of DPPH, $\cdot$OH, and $\text{O}_2^-$ were significantly improved by extrusion processing of SDSDF. In the extruded SDSDF, the scavenging ability of $\cdot$OH increased by 221.21%, and the total reducing power increased by 193.81% on average. Moreover, the observation of surface structure using electron microscopy revealed that there were many regular honeycomb holes, loose arrangement, and increasing superficial area in the extruded SDSDF. Additionally, the surfaces present rules structure of the size of rod cell was about 10 μm. Furthermore, infrared spectroscopic analysis showed that a characteristic absorption peak of polysaccharide appeared with/without extrusion processing. An absorption peak appeared at 1631 cm$^{-1}$ nearby, while the absorption peak decreased significantly in SDSDF after extrusion processing. The results suggested that protein denaturation could be caused by reducing hydroxylamine condensation bonds under high temperatures and pressures. In addition, XRD analysis showed that 20 diffraction

Figure 5. Ultrastructure of SDSDF. A. Un-extrusive SDSDF, 500 times; B. Extrusive SDSDF, 500 times; C. Un-extrusive SDSDF, 2000 times; D. Extrusive SDSDF, 2000 times; E. Un-extrusive SDSDF, 4000 times; F. Extrusive SDSDF, 4000 times; G. Un-extrusive SDSDF, 8000 times; H. Extrusive SDSDF, 8000 times.
Conflict of interest statement. None declared.

Identification and characterization of SDSDF, 2017, Vol. 1, No. 2

peak changed at 24.16° in the extruded SDSDF compared with the control group, indicating that some changes have taken place in the crystal structure of cellulose or hemicellulose.

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