

# Ultrasound-assisted alkali extraction of insoluble dietary fiber from soybean residues

**Juntao Sun\***, Zhichao Zhang, Fugang Xiao, Quanzeng Wei, Zonghui Jing

Key Laboratory of Henan Province, Food and Bioengineering College, Xuchang University, Xuchang 461000, China

\*Corresponding author, E-mail address: jtsfly@163.com

**Abstract:** Dietary fiber is a carbohydrate-based polymer with significant health benefit, soybean residues are generally discarded in food industries during the production of soybean milk and tofu. In this study, ultrasound-assisted alkali extraction technology was employed to extract insoluble dietary fiber from soybean residues. And the physicochemical properties of insoluble dietary fibers were also studied. The optimal conditions for insoluble dietary fibers were determined to be solid to liquid ratio of 1:50, alkali concentration of 0.05 mol/L, ultrasound power of 450 W, reaction time of 10 min and reaction temperature of 30 °C. The yield of insoluble dietary fiber was  $744.3 \pm 13.2$  mg/g raw soybean residues using the combination of ultrasound and alkali. In addition, the insoluble dietary fibers had higher water retention capacity, swelling capacity than those of untreated soybean residues, while the oil retention capacity of insoluble dietary fibers was lower than that of untreated soybean residues.

## 1. Introduction

Dietary fiber consists of a mixture of compounds containing carbohydrate polymers and non-carbohydrate components that is enriched in whole grains, nuts, fruits and vegetables. According to the solubility in water, total dietary fiber (TDF) can be categorised into two groups, namely soluble dietary fiber (SDF) and insoluble dietary fiber (IDF) (1). It has been suggested that high intake of dietary fiber significantly lower the risk of developing coronary heart diseases, strokes, hypertension, diabetes, obesity, and certain gastrointestinal diseases (2-4). Therefore, Dietary fibers have received increasing attention from researchers and industry due to their beneficial effects.

Soybean residue is the main by-product from soymilk and tofu preparation, which is a good dietary fiber resource (5). Approximately twenty million tons of soybean residues are produced annually in China. Unfortunately, most of this soybean residue is not fully utilized and is discarded as industry waste. This waste poses potential environmental problems because the large amounts of water and abundant nutrients that are found in soybean residues can promote the growth of microorganisms (6). Therefore, a novel food processing technique is suggested to manufacture valuable additional products, such as dietary fiber from soybean residues based on the aforementioned economic and environmental incentives (7). Dietary fiber extraction methods include chemical methods, enzymatic extraction methods and enzymatic-chemical methods (8-9). Several processing conditions contribute to considerable changes in the composition and microstructure of dietary fiber, thereby affecting its functional properties (10-11). Alkali extraction process is widely used in dietary fiber industrial production, which can remove the protein from soybean residues and obtain the high purity dietary fiber. Recently, the application of ultrasound in extraction and refining processes has found increasing attention (12-14). Ultrasound can help separating starch and protein from the isolated hemicelluloses



and splitting of  $\alpha$ -ether linkages between lignin and hemicelluloses chains, and improving the extraction yield of product (15). Ultrasound-assisted alkali extraction is an alternative method that can operate at low temperatures, require a short processing time and display more effective.

The dietary fiber in soybean residue is mainly IDF, so in the present study, the main research focus on the extraction of IDF. The solid to liquid ratio, alkali concentration, ultrasonic power, extraction time and temperature were optimized, and the physicochemical properties of IDF such as swelling, water and oil retention capacity was also studied. This study may serve as a basis for further research on the economic production of dietary fiber from soybean residues.

## 2. Materials and methods

### 2.1 Materials

The soybean residue from soymilk and tofu elaboration was obtained as a by-product from a local supplier (Xuchang, China). Soybean residues samples were dried to a constant weight at 105 °C in an oven, and milled to a particle size of less than 1.0 mm, and then defatted using a Soxhlet extractor for 6 h before extraction of dietary fiber. Alpha amylase, protease and amyloglucosidase were purchased from Beijing Aoboxing Bio-Tech Co., Ltd. (Beijing, China). All chemicals and reagents were of analytical grade and purchased from Sinopharm Chemical Reagent Co., Ltd. (Shanghai, China).

### 2.2 Determination of dietary fiber content

The contents of TDF, SDF and IDF were determined according to the AOAC 991.43 enzymatic-gravimetric method (AOAC, 2005). In brief, dried powder samples were first gelatinized with heat stable  $\alpha$ -amylase (95 °C, 35 min). After gelatinization, the samples were digested with protease and amyloglucosidase to remove protein and starch in the samples. Subsequently, IDF was filtered and washed with 60 °C distilled water. The filtrate and washed water were combined and added with four volumes of 95% ethanol to precipitate the SDF. The residues were weighed after drying at 105 °C in a hot air oven. TDF was calculated as the sum of IDF and SDF.

### 2.3 Ultrasound-assisted alkali extraction of dietary fiber

Alkali extraction was carried out by incubating the reaction system in ultrasound bath using a KOAO KQ-100D (Dongguan KQAO Ultrasonic Equipment Co., Ltd, Dongguan, China). To investigate the effect of ultrasound on dietary fiber extraction, the effects of various solid material to liquid ratio (1:10, 1:20, 1:30, 1:40, 1:50 and 1:60), alkali concentration (0.05, 0.1, 0.2, 0.3, 0.4, and 0.5 mol/L), ultrasound output powers (250, 300, 350, 400, 450, and 500 W), ultrasound time (10, 20, 30, 40, 50, and 60 min) and ultrasound temperature (30 °C, 40 °C, 50 °C, 60 °C, 70 °C and 80 °C) were studied. The soybean residues digested by alkaline were then rinsed with water to neutral pH, and the water subsequently removed by centrifugation of 5000 g for 15 min. the soybean residues were dehydrated by hot air at temperature of 60 °C in a dehydrator to reach a constant weight. Finally, the dried soybean residues were ground as dietary fiber powder.

Dietary fiber production from soybean residues was also carried out without ultrasound. The conditions were as follows: solid to liquid ratio of 1:50, alkali concentration of 1 mol/L, extraction time of 80 min and temperature of 80 °C (16).

### 2.4 Physicochemical properties of dietary fiber

Physicochemical properties of dietary fiber, such swelling capacity (SC), water retention capacity (WRC) and oil retention capacity (ORC) were assessed following the experimental protocol used in European collaborative study (17).

The sample (250 mg) was weighed in a 10 mL measuring cylinder (0.1 mL graduations) and 5 mL distilled water, containing 0.02% sodium azide added. Then, it was stirred gently to eliminate trapped air bubbles and left on a level surface at room temperature overnight to allow sample to settle. Finally, the volume (mL) occupied by the sample was measured and SC was expressed as mL per g of dry

sample.

Fifteen millilitres of distilled water was added to 250 mg of sample in a 50 mL centrifuge tube. The sample was stirred and left at room temperature for 1 h. After centrifugation at 3000 g for 20 min, the supernatant was discarded, the residue was weighed and WRC was calculated as gram water per gram dry sample.

The same protocol as above was followed, but using commercial virgin olive oil instead of water. ORC was expressed as gram olive oil retained per gram dry sample.

### 3. Results and discussion

#### 3.1 Ultrasound-assisted alkali extraction of dietary fiber

The effect of solid to liquid ratio in the range of (1:10 - 1:60) on the yield of IDF was shown in Fig. 1 (A). The increase of IDF was obvious when the solid to liquid ratio was increased from 1:10 to 1:50, and the yield reached a maximum plateau at around 78%. When the solid to liquid ratio was up to 1:60, the yield declined slightly. Effects of alkali concentration on the yield of IDF were shown in Fig. 1 (B). The results indicated that the yield of IDF was decreased with increasing alkali concentration. The maximal value was happened to be 0.05 mol/L alkali concentration, after that it was reached to a level off. It was better to choose the extraction operating at 0.05 mol/L alkali concentration. Production of dietary fiber using different ultrasound power is shown in Fig. 1 (C). Increasing the ultrasound power from 200 W to 450 W slowly increased the yield of IDF, however, from 450 W to 500 W, the production of IDF was obviously decreased with the increasing ultrasound power. Thus, the suitable ultrasonic power is 450 W. The effects of ultrasonic time on the extraction yield of IDF were also explored. The results are shown in Fig. 1 (D). It was found that the yield of dietary fiber using ultrasound for 10 min is similar with that of ultrasound for 20 min, and the extraction yield of IDF decreased with increasing ultrasonic time from 20 min to 60 min. In view of saving energy, 10 min was selected as the optimum ultrasound time. Fig. 1 (E) shows the effect of ultrasound temperature on the yield of IDF. A significant decrease of the yield was observed at the ultrasound temperature range from 30 °C to 80 °C, and the yield reached a maximum at around 74 % at 30 °C.

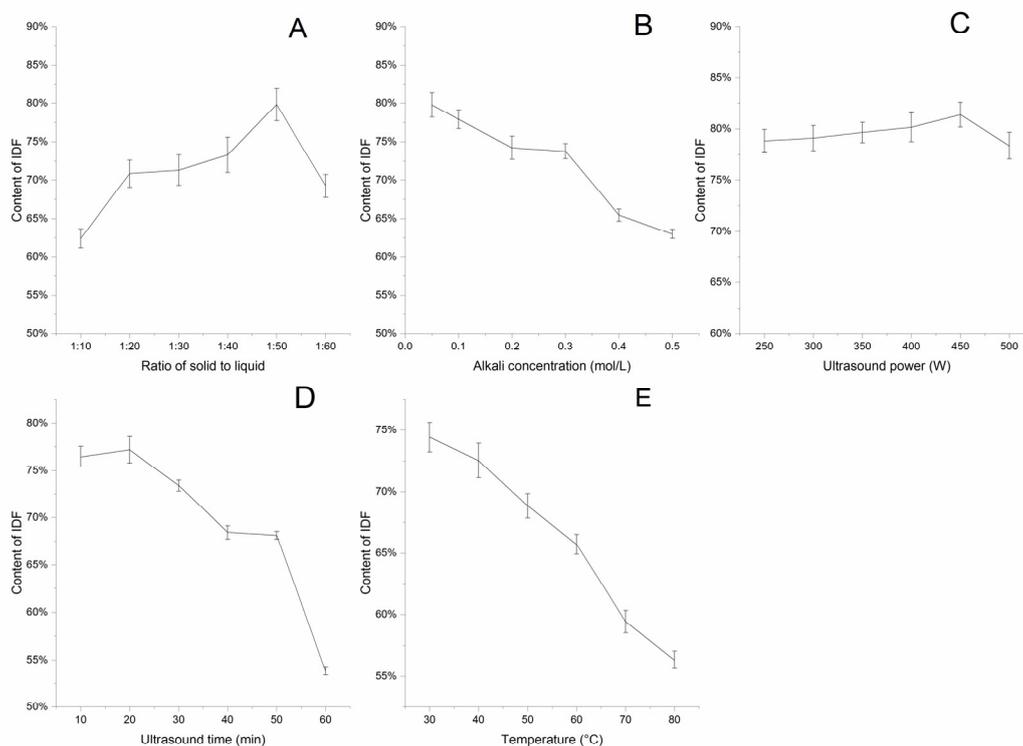


Fig.1. Effect of solid material to alkaline solution ratio, alkali concentration, ultrasound power, ultrasound time and temperature on the content of dietary fibers from soybean residues. (A) Alkali concentration, ultrasound output power, ultrasound time and ultrasound temperature were constant at 0.1 mol/L, 400 W, 30 min and 50 °C, respectively. (B) Solid to liquid ratio, ultrasound output power, ultrasound time and ultrasound temperature were constant at 1:50, 400 W, 30 min and 50 °C, respectively. (C) Solid to liquid ratio, alkali concentration, ultrasound time and ultrasound temperature were constant at 1:50, 0.05 mol/L, 30 min and 50 °C, respectively. (D) Solid to liquid ratio, alkali concentration, ultrasound power and ultrasound temperature were constant at 1:50, 0.05 mol/L, 450 W and 50 °C, respectively. (E) Solid to liquid ratio, alkali concentration, ultrasound power and ultrasound time were constant at 1:50, 0.05 mol/L, 450 W and 10 min, respectively. Error bars represent standard deviation of the means (n=3).

In summary, the optimum conditions for the ultrasound-assisted alkali extraction of IDF from soybean fibers are as follows: solid to liquid ratio of 1:50, alkali concentration of 0.05 mol/L, ultrasound power of 450 W, ultrasound time of 10 min, and ultrasound temperature of 30 °C. Under these conditions, the production of IDF was  $744.3 \pm 13.2$  mg/g raw soybean residues.

### 3.2 Comparative study on alkali extraction with and without ultrasound

Alkali extraction was performed with and without ultrasound assistance to investigate the influence of ultrasound on the extraction. The result was shown in Table 1. The content of SDF and TDF obtained through ultrasound-assisted condition were 1.94 and 1.04-fold higher, respectively, than those obtained through without ultrasound. But the content of IDF under ultrasound-assisted alkali extraction was 1.02-fold lower than that of without ultrasound. Ultrasound and alkali exhibited a synergistic effect in lowering the diffusion-limiting barrier between alkali and the substrate. During the collapse of the ultrasound-induced cavitation bubbles, powerful jet streams created in the liquid media act as a transport mechanism, lowering the diffusion-limiting barrier surrounding the soybean residues (18), the protein can easily remove by alkali from soybean residues to obtain high pure IDF, and ultrasound can increase the extraction rate of the SDF. To obtain similar or higher yields of dietary fibers fractions,

the ultrasound-assisted procedures could reduce extraction temperature, time and alkali concentration. The current findings corroborate previous studies on the synergistic role of ultrasound in liquid–liquid-phase chemical reactions (19-21).

Table 1 Extraction of dietary fiber from soybean residues with and without ultrasound

	Without ultrasound	With ultrasound
IDF (%)	76.12 ± 1.49	74.43 ± 1.32
SDF (%)	5.25 ± 0.15	10.19 ± 0.13
TDF (%)	81.37 ± 1.74	84.62 ± 1.83

All assays were performed in triplicate.

In the present paper, the efficiency of the ultrasound-assisted alkali extraction procedures exceeded that of the classical procedures. To obtain similar or higher yields of dietary fibers fractions, the extraction temperature could be reduced from 80 °C to 30 °C, the extraction time could be shortened from 80 min of the classical extraction to 10 min of ultrasound-assisted extraction, and the consumption of alkali can be reduced by about 95% yield. These advantages support the importance and great potential of ultrasound-assisted extraction processes applied for the isolation of industrially important dietary fiber from soybean fibers.

### 3.3 Physicochemical properties of dietary fiber

The SC, WRC and ORC of IDF in functional foods significantly affect the rheological properties of the final products. Thus, these properties affect the mouth-feel experienced by consumers (22). The water-related properties were investigated here to explore the possibility of utilizing it as a healthy food additive in cooked wheat-based food or sauces. The SC, WRC and ORC of IDF and soybean residues are listed in Table 2. The results indicated that the SC (4.00 g/g) and WRC (1.63 g/g) of IDF from soybean residues were significantly higher than the SC (2.89 g/g) and WRC (1.03 g/g) of untreated soybean residues, while the ORC of IDF was lower than that of untreated soybean residues. The possible reason for this result may be that ultrasound-assisted alkali treatment changed the three-dimensional structure of dietary fiber and increased the amount of short-chain and surface area of dietary fiber (7).

Table 2 Physicochemical properties of IDF

	SC (mL /g)	WRC (g /g)	ORC (g/g)
Soybean residues	2.89 ± 0.06	1.03 ± 0.03	1.11 ± 0.02
IDF	4.00 ± 0.09	1.63 ± 0.04	0.82 ± 0.03

All assays were performed in triplicate.

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### References

- [1] Vasanthan T, Gaosong J, Yeung J, Li J. Dietary fiber profile of barely flour as affected by extrusion cooking. *Food Chem.* 77: 35-40 (2002)
- [2] Elleuch M, Bedigian D, Roiseux O, Besbes S, Blecker C, Attia H. Dietary fibre and fibre-rich by-products of food processing: Characterisation, technological functionality and commercial applications: A review. *Food chem.* 124: 411-421 (2011)
- [3] Huang Z, Ye R, Chen J, Xu F. An improved method for rapid quantitative analysis of the insoluble

- dietary fiber in common cereals and some sorts of beans. *J. Cereal Sci.* 57: 270-274 (2013)
- [4] Ding HH, Cui SW, Goff HD, Wang Q, Chen J, Han NF. Soluble polysaccharides from flaxseed kernel as a new source of dietary fibres: Extraction and physicochemical characterization. *Food Res. Int.* 56: 166-173 (2014)
- [5] Mateos-Aparicio P, Mateos-Peinado C, Ruperez P. High hydrostatic pressure improves the functionality of dietary fiber in okara by-product from soybean. *Innov. Food Sci. Emerg.* 11:445-450 (2010)
- [6] O'Toole DK. Characteristics and use of okara, the soybean residue from soy milk production – a review. *J. Agr. Food Chem.* 47: 363-371(1999)
- [7] Ye C, Ran Y, Luo Y, Ning Z. Novel blasting extrusion processing improved the physicochemical properties of soluble dietary fiber from soybean residue and in vivo evaluation. *J. Food Eng.* 120: 1-8 (2014)
- [8] Englyst NH, Quigley ME, Hudson GJ. Determination of dietary fiber as non-starch polysaccharides with gas-liquid chromatographic, highperformance liquid chromatographic or spectrophotometric measurement of constituent sugars. *Analyst.* 119:1497-1509(1994)
- [9] Galanakis CM, Tornberg E, Gekas V. A study of the recovery of the dietary fibers from olive mill wastewater and the gelling ability of the soluble fiber fraction. *LWT – Food Sci. Technol.* 43:1009-1017(2010)
- [10] Peerajit P, Chiewchan N, Devahastin. Effects of pretreatment methods on health-related functional properties of high dietary fibre powder from lime residues. *Food Chem.* 132: 1891-1898 (2012)
- [11] Ma MM, Mu TH, Sun HN, Zhang M, Chen JW, Yan ZB. Optimization of extraction efficiency by shear emulsifying assisted enzymatic hydrolysis and functional properties of dietary fiber from deoiled cumin (*Cuminum cyminum L.*). *Food Chem.* 179: 270-277 (2015)
- [12] Wang A, Wu L, Li X. Optimization of ultrasonic-assisted preparation of dietary fiber from corn pericarp using response surface methodology. *J. Sci. Food Agr.* 93: 2922-2926 (2013)
- [13] Wang J, Sun B, Liu Y, Zhang H. Optimisation of ultrasound-assisted enzymatic extraction of arabinoxylan from wheat bran. *Food Chem.* 150: 482-488 (2014)
- [14] Govindarajan Ramadoss, Karuppan Muthukumar. Ultrasound assisted ammonia pretreatment of sugarcane bagasse for fermentable sugar production. *Biochem. Eng. J.* 83: 33-41(2014)
- [15] Hollmann J, Elbegzaya N, Pawelxik E, Lindhauer MG. Isolation and characterization of glucuronoarabinoxylans from wheat bran obtained by classical and ultrasound-assisted extraction methods. *Qual. Assur. Saf. Crop.* 1: 231-239 (2009)
- [16] Li N, Ning Z, Zhu Z, Li L. Preparation and characterization of dietary fibers from soybean dregs. *Food Sci.* 30: 251-254 (2009). (China Journal)
- [17] Robertson JA, de Monredon FD, Dysseler P, Guillon F, Amado R, Thibault JF. Hydration properties of dietary fiber and resistant starch: A European collaborative study. *LWT-Food Sci. Technol.* 33:72-79 (2000)
- [18] Easson MW, Cindon B, Dien BS, Iten L, Slopek R, Yoshioka-Tarver M. The Application of Ultrasound in the Enzymatic Hydrolysis of Switchgrass. *Appl. Biochem. Biotech.* 165: 1322-1331 (2011)
- [19] Wang YH, Zhang JC. A novel hybrid process, enhanced by ultrasonication, for xylan extraction from corncobs and hydrolysis of xylan to xylose by xylanase. *J. Food Eng.* 77: 140-145 (2006)
- [20] Siwek M, Bari Noubar A, Bergmann J, Niemeyer B, Galunsky B. Enhancement of enzymatic digestion of Antarctic krill and successive extraction of selenium organic compounds by ultrasound treatment. *Anal. Bioanal. Chem.* 384: 244-249 (2006)
- [21] Kang KE, Jeong GY, Park DH. Rapeseed-straw enzymatic digestibility enhancement by sodium hydroxide treatment under ultrasound irradiation. *Bioproc. Biosyst. Eng.* 36: 1019-1029 (2013)
- [22] Singh N, Singh J, Kaur L, Sodhi NS, Gill BS. Morphological, thermal and rheological properties of starches from different botanical sources. *Food Chem.* 81: 219-231 (2003)