

Study on Quality of Soluble Dietary Fiber from *Jujube* (*Z. vulgaris Lam*) with Different Treatments

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Abstract. The studies focused on plant soluble dietary fiber. The apparent characteristics of SDF from the *Jujube* under different treatment methods can provide theoretical support for product development to obtain the best trait products. Optimal extraction conditions, physical properties and antioxidant activity of SDF from the *Jujube* were investigated. The yield of 20.17 ± 0.16 % for extraction of soluble dietary fiber from *Jujube* were obtained as extraction temperature was 94 °C, extraction time was 40 min and ratio of raw material to water was 1:21. The physical properties of SDF were ascertained by measuring dissolution time, rehydration, swelling ability and bulk density, respectively. And it was evaluated by FT-IR and scanning electronic microscopy (SEM). Furthermore, it was proved that the soluble dietary fiber extracted by vacuum freeze dried had higher scavenging ability than that of vacuum dried and hot dried against DPPH, ABTS⁺ and hydroxyl radical. It has good antioxidant function and can slow down intestinal aging as a basis for new food development.

1 Introduction

Jujube is the fruit of the *Ziziphus jujube Mill* of the *Rhamnaceae* [1-2]. Traditionally, it is regarded as a medicine to reduce blood sugar and blood pressure, or an edible to enhance human immunity [3-4] due to its rich in abundant vitamins [5], minerals and trace elements [6]. Dietary fiber mainly presents in the cell wall of plants including cellulose [7], hemicellulose [8], resin, pectin and lignin. It included soluble dietary fiber (SDF) and insoluble dietary fiber, (IDF) depending on the solubility of the dietary fiber [9].

Recent research pointed SDF has positive effect on human physiology including laxative [10] and hypolipidemic [11]. However, the chemical organic constituents of *jujube* were limited to the researches on the determination of nutrient components and total flavonoids. There were few researches on the extraction process and antioxidant activity of SDF. Therefore, this work mainly focused on exploring the optimal condition to extract SDF from *jujube* in order to increase the yield, as well as the antioxidant and physical properties of *jujube*.

2 Materials and methods

2.1. Materials

Jujube was harvested in October 2018 from Jinan (Long. 117°00'E; Lat. 36°40' N) of Shandong province in China. The fruits were identified as *Z. vulgaris Lam* by researcher X. Zhang (Department of Life Sciences, Shandong

Normal University). The whole *jujube* were washed with water and freeze rapidly at -37 °C with liquid nitrogen after denucleation. Finally, dried fruits were crushed by superfine grinding at 200 order to be used. The following reagents contained 1,1-diphenyl-2-picrylhydrazyl (DPPH) and diammonium salt (ABTS) were purchased from Sigma Chemical Co. (St. Louis, MO, USA).

2.2 Process of extraction of SDF

As shown in Fig. 1, *Jujube* pulp was immersed three times with petroleum ether (boiling ranged from 30 °C-60 °C) and 70 % (V/V) ethanol for 24 h at room temperature of 27 °C in order to remove oily ingredients and pigments [12]. The rest of residue was used to extract SDF by hot water with different extraction temperature, extraction time and ratio of raw material to water. The resulting solution was treated with papain (pH 6.6, 8000 u/100 mL) at 55 °C for 4 h and then filtered to remove the protein [13]. Subsequently, the saccharification enzyme (500 u/100 mL) was added and reacted at 60 °C for 2 h after it was extinguished at 80 °C with pH 4.2 of PBS (0.1 mol/L) [14]. After filtration, the supernatant was precipitated with four volumes of absolute ethanol, and the precipitate was collected by centrifugation at 9500 rpm for 25 min [15]. Finally, it was washed with diethyl ether, absolute ethanol and acetone via different dried methods to obtain SDF.

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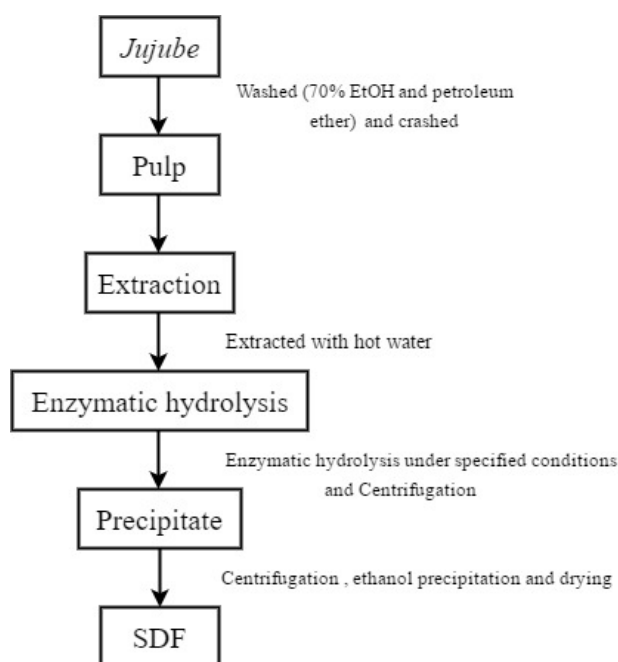


Fig. 1. Scheme for SDF extraction of *Jujube*.

2.3 Experimental design of response surface methodology

The influences on the extraction process including extraction time (A: 10 min, 20 min, 30 min, 40 min, 50 min and 60 min), extraction temperature (B: 50 °C, 60 °C, 70 °C, 80 °C, 90 °C and 100 °C) and ratio of raw material to water (C: 1:5 g/mL, 1:10 g/mL, 1:15 g/mL, 1:20 g/mL, 1:25 g/mL and 1:30 g/mL) were investigated. Besides, to find the best combination, the yield of SDF under these extraction factors was recorded via the Box-Behnken experimental design (BBD) with 17 experiments in order (Table 1).

2.4 Different treatments of SDF

The SDF was processed with three treatments including vacuum freeze dried (noted as T-1), hot dried (noted as T-2) and vacuum dried (noted as T-3). For T-1, the extracted wet SDF was frozen at -25 °C for 20 ± 2 h [16] in the refrigerator in advance. The actual experiments included 2 stage: the first stage was under the conditions with the temperature of -65 °C and pressure of 20 Pa for 20 h; the second stage was prepared at the temperature of -55 °C and pressure of 15 Pa for 15 h [17]. In terms of T-2, the wet SDF was kept in the oven at temperature of 85 °C with 2.5 m/s for 20 h [18] to be dried. Besides, the wet SDF was spread on a tray and dried at vacuum environment with 20 Pa at room temperature of 27 °C for the T-3 treatment.

2.5 Antioxidant capacity

The method of DPPH radical scavenging of SDF from *Jujube* was experienced via the reported essay [19]. 5 mL of prepared DPPH solution (0.1 mM in ethanol) was mixed with different concentrations of SDF samples. Then, the mixture reacted for 45 min in the dark at 27 °C [20] and detected at 517 nm [21] via UV-vis (UV-2501PC). The experiment was conducted with ascorbic acid as the control.

$$\text{DPPH (\%)} = (1 - \text{Abs}_{\text{sample}} / \text{Abs}_{\text{control}}) \times 100 \quad (1)$$

The ABTS⁺ radical scavenging ability test was mentioned by earlier report [22]. Different concentrations of SDF solutions (1 mL with pH 7.1) were added into the solution [23], which was a mixture of phosphate buffer (2.5 mL at 0.2 M with pH 6.6) and potassium ferricyanide solution (2.5 mL at 1 % (m/v)). In order to react completely, all of the samples were protected at 50 °C under water bath for 40 min in dark room [24]. Then, the ABTS⁺ radical solution was added into trichloroacetic acid solution (2.5 mL at 0.5 M) to get the steady absorbance at 700 nm. The ABTS⁺ radical scavenging ability was tested at around 700 nm with the blend of 2.5 mL distilled water and ferric chloride solution (0.5 mL at 0.05 mM) at 20 min after the initial mixing. The solution without any samples was used as control.

$$\text{Percent Inhibition (\%)} = (1 - \text{Abs}_{\text{sample}} / \text{Abs}_{\text{control}}) \times 100 \quad (2)$$

In this study, OH⁻ scavenging ability was estimated according to the method [25]. The reaction blend contained FeSO₄·7H₂O solution (2 mL at 6 mM), salicylic acid-ethanol solution (2 mL at 7.5 mM with pH 6.7), H₂O₂ (2 mL at 8 mM) and different concentrations of SDF solution (2 mL) [26]. Then, the blend reacted for 30 min at 37 °C and measured at 510 nm via UV-vis. This was ordered with ascorbic acid as the control.

$$\text{Scavenging percent (\%)} = [1 - (A - A_0) / A_1] \times 100 \quad (3)$$

A = Absorbance of extract sample; A₀ = Absorbance of control (to replace sample with water); A₁ = Absorbance of blank (to replace sample and H₂O₂ solution with water). The SDF was processed with three treatments including vacuum freeze dried

2.6 Physical and chemical properties

2.6.1 FT-IR spectroscopy

The infrared (IR) spectra of SDF was performed by the fourier transform infrared spectrophotometer (Thermo Electron Corp., Waltham, MA, USA). The SDF mixture (with 5 mg sample and 100 mg KBr) was determined from 4000 ~ 400 cm⁻¹ (mid infrared region) to analyze the characteristic groups [27].

Table 1 Design-expert design scheme and experimental results

Experiments	A:Extraction time(min)	B:Exaction temperature(°C)	C:Ratio of raw material to water	Actual yield (%)	Predict value (%)
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1	-1(30)	-1(80)	0(1:20)	17.15±0.13	16.97
2	1(50)	-1(80)	0(1:20)	18.34±0.23	18.37
3	-1(30)	1(100)	0(1:20)	18.30±0.16	18.27
4	1(50)	1(100)	0(1:20)	19.56±0.33	19.48
5	-1(30)	0(90)	-1(1:15)	17.79±0.21	17.90
6	1(50)	0(90)	-1(1:15)	19.89±0.16	19.84
7	-1(30)	0(90)	1(1:25)	18.69±0.19	18.79
8	1(50)	0(90)	1(1:25)	19.87±0.21	19.76
9	0(40)	-1(80)	-1(1:15)	17.81±0.18	17.88
10	0(40)	1(100)	-1(1:15)	19.56±0.10	19.48
11	0(40)	-1(80)	1(1:25)	18.50±0.11	18.50
12	0(40)	1(100)	1(1:25)	19.70±0.19	19.63
13	0(40)	0(90)	0(1:20)	19.72±0.16	19.84
14	0(40)	0(90)	0(1:20)	19.71±0.17	19.84
15	0(40)	0(90)	0(1:20)	19.89±0.21	19.79
16	0(40)	0(90)	0(1:20)	19.88±0.22	19.84
17	0(40)	0(90)	0(1:20)	19.99±0.14	19.84

Table 2 The analysis of variance (ANOVA) of the regression model for the yield response

Variable	SS	DF	F-value	P-value
Model	13.52	9	56.41	<0.0001 ^a
A	4.10	1	154.16	<0.0001 ^a
B	3.54	1	132.89	<0.0001 ^a
C	0.37	1	13.73	0.0076 ^b
AB	0.24	1	0.046	0.8363
AC	0.21	1	7.95	0.0258
BC	0.076	1	2.84	0.1358
A ²	1.87	1	70.26	<0.0001 ^a
B ²	2.93	1	110.01	<0.0001 ^a
C ²	0.052	1	1.97	0.2036
Residual	0.19	7		
Lack of Fit	0.13	3	2.96	0.1609
Pure Error	0.058	4		
Cor Total	13.70	16		
C.V.%	0.86			

^a Significant at 0.001 level. ^b Significant at 0.01 level.

Table 3 The chemical composition of SDF with different treatments

Items	T-1	T-3	T-2
Protein (%)	0.16±0.06 ^b	0.22±0.12 ^a	0.13±0.36 ^b
Carbohydrate (%)	51.61±2.02 ^a	82.56±2.71 ^b	74.32±2.21 ^c
Total flavonoid (mg GAE/100 mg)	0.16±0.03 ^b	0.14±0.06 ^c	0.13±0.05 ^a
Uronic acid (%)	7.15±0.15 ^a	12.39±1.68 ^b	11.25±1.24 ^c

the different lowercases indicated significant difference ($P_b < 0.05$) in the same line.

Table 4 Physical properties of SDF under the T-1, T-2, T-3 treatments. *

	Dissolution time (s)	bulk density (g/mL)	swelling ability (mL/g)	Rehydration
T-1	0.210±0.020 ^b	7.067±0.145 ^a	32.000±3.000 ^c	0.480±0.036 ^a
T-2	0.320±0.027 ^a	3.667±0.208 ^b	80.667±3.056 ^a	0.410±0.036 ^b
T-3	0.227±0.021 ^b	3.937±0.208 ^b	52.337±3.786 ^b	0.397±0.021 ^b

*Result were present as means ± standard deviation (n=3). Means with different uppercase letter superscripts in the same column were significant different at $P < 0.05$.

2.6.2 Scanning Electronic Microscopy of SDF

The scanning electronic microscopy of SDF has been reported by the scanning projection electron microscope (Thermo Prisma E., Waltham, MA, USA). A small amount of the SDF sample was detected after it was dried by an infrared lamp for 10 min [28-29].

2.6.3 Determination of dissolution time

To maintain 50 mL mixture (2 g sample with 50 mL water) under 23 ± 2 °C until it was completely dissolved. The dissolution time was noted as T_{sp} . No precipitation was formed when standing for 30 seconds after the complete dissolution can be regarded as completely dissolution determination [30].

2.6.4 Determination of bulk density

The SDF was scattered through a funnel in a 5 mL graduated cylinder. Quality was recorded as m_0 when the mass was at a volume of 5 mL.

$$\rho = m_0/5 \quad (4)$$

m_0 =the mass of SDF scattered 5mL, g; ρ =the bulk density, g/mL.

2.6.5 Determination of swelling ability

SDF was spread into the bottom of scaled tube with water to be fully dissolved (at 27 °C for 48 ± 2 h). The ratio of difference volume of the sample after and before water absorption to sample mass can be used to estimate the swelling ability.

$$P = (V - V_0)/m \quad (5)$$

V =volume after water absorption, mL; V_0 =unabsorbed dry weight volume, mL; m =sample quality, g; P =the swelling ability, mL/g.

2.6.6 Determination of rehydration

The sample was centrifuged at 4 °C with 11000 r/min for 25 min. The water on sample surface was washed with a small amount of absolute ethanol. The ratio of wet to dried weight was the index of rehydration[30].

$$R = m/m_0 \quad (6)$$

R = rehydration ratio; m = mass after washing, mg; m_0 =mass of dry sample, mg.

2.6.7 Formatting the title

0.1 g of the sample was dissolved in 40 ml ethanol and agitated well to be measured (Beckman CoulterLS 13320, MA, USA).

2.7 Statistical analysis

The BBD design and data analysis of RSM was performed using Software Design-Expert (V8.0.6). The significance of diversities was assessed by one-way ANOVA analysis (with the significance of 2 levels). All tests were repeated for three groups, with three parallel tests of each group ($n=3 \times 3$). Data of triplicate parallel experiences were reported as means \pm standard deviations by Microsoft Office (15.0).

3 Results and discussion

3.1 Extraction factor analysis

According to Fig. 2 (C), the extraction yield of SDF increased significant ($P < 0.05$) with the increasing ratio of material to water, and the maximum value is consistent remained at 18.7 ± 0.84 % when it was 1:20 g/mL. It might be dissolved more completely with the action of dilution when the ratio of material to water was lower [31]. On the contrary, higher ratio would increase the specific heat capacity of the system [32], which may increase the destruction of the SDF.

To explore the influence of extraction temperature on yield of SDF, the extraction temperature conditions of 50 °C, 60 °C, 70 °C, 80 °C, 90 °C, and 100 °C was studied, just as shown in the Fig. 2 (B). In the certain temperature range from 50 °C to 100 °C, the total extraction yield of SDF reached a maximum ($P < 0.05$) of 18.9 ± 0.52 % when the extraction temperature reached 90°C. This situation might be due to the high temperature causing decomposition of SDF [33].

In this part, it can be seen from Fig. 2 (A) that the extraction yield of SDF increased significant ($P < 0.05$) with the increase of extraction time from 10 min to 40 min. Longer extension time did not change the extraction rate of the SDF after 40 min, which may be because its fully solubility in the solvent reach at 19.29 ± 0.35 %.

3.2 Statistical analysis and model fitting

The relationship between extraction yield of SDF and potential covariates including extraction temperature, extraction time, ratio of raw material to water were measured using the following second order regression equation: $Y = 19.84 + 0.72A + 0.67B + 0.21C + 0.017AB - 0.23AC - 0.14BC - 0.67A^2 - 0.83B^2 - 0.11C^2$.

According to the Table 2 analysis, the regression model ($P < 0.001$) and missing term ($P > 0.1$) indicated that the data fitted the regression model well, and the experimental error was low [34]. Thus, it can be used to predict the extraction yield of SDF in *jujube* under given extraction conditions.

In this model, extraction time (A, $P < 0.05$), extraction temperature (B, $P < 0.05$), ratio of raw material to water (C, $P < 0.05$), the extraction temperature quadratic term (A^2 , $P < 0.05$) and the extraction time quadratic term (B^2 , $P < 0.05$) have a significant effect on extraction yield of SDF. The conditions of parameters which was optimized after combined with practical experimental feasibility was

extraction time of 45 min, extraction temperature at 94 °C and ratio of raw material to water with 1:21 (g/mL). The

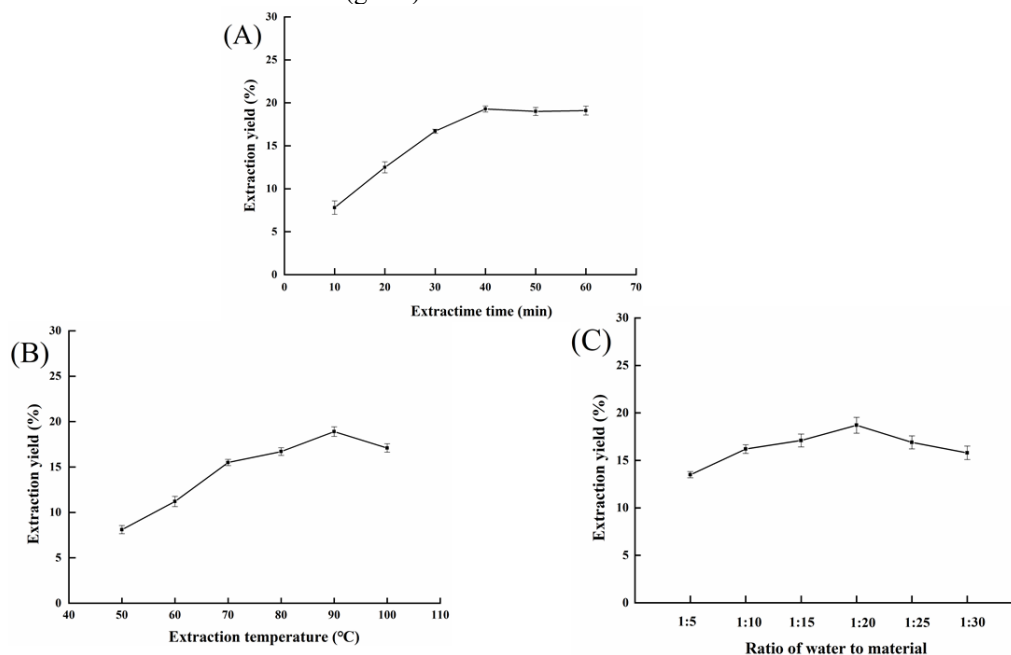


Fig. 2. Effect of extraction time (A), extraction temperature (°C) (B) and ratio of raw material to water (g/mL) (C) on the extraction yield of SDF.

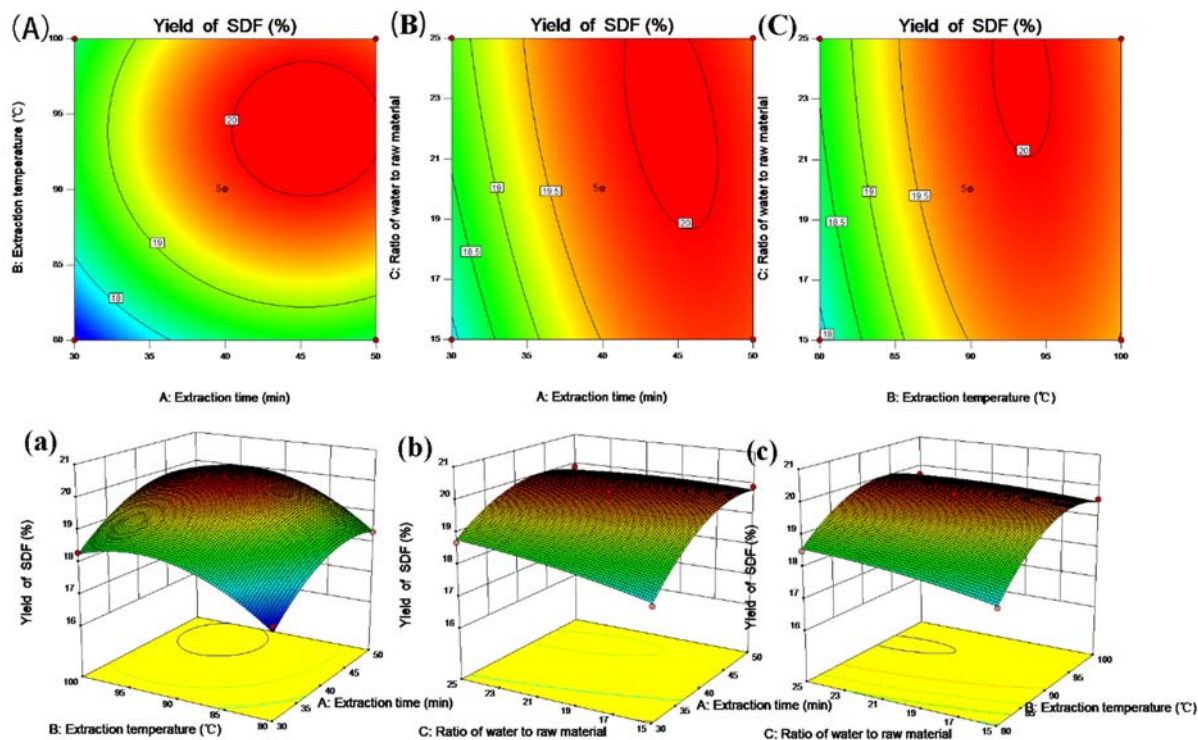


Fig. 3. Response surface plots of the interaction of various factors on the yield of SDF. (A) and (a) : The factors of time and temperature. (B) and (b) : The factors of time and ratio of raw material to water. (C) and (c) : The factors of temperature and ratio of raw material to water.

extraction yield of SDF under this condition remained 20.17 ± 0.16 %.

3.3 Analysis of response surface

The influence of the interaction between the reaction factors with the extraction yield of SDF can be realized by the steepness of the vertical surface of the response

surface. As shown in Fig. 3, the order of the influence between each factor on extraction yield of SDF was extraction time > extraction temperature > ratio of raw material to water according to the steepness degree from high to low. The shape of contour difference between extraction time and extraction temperature tends to be elliptical, which indicated that the interaction between the quadratic term of these was significant ($P < 0.05$).

3.4 Chemical composition results

The contents of carbohydrates, proteins, uronic acids and total flavonoids in SDF with different treatments were summarized in Table 3. The results indicated that the higher content of these SDF was carbohydrate, which were $51.61 \pm 2.02\%$, $82.56 \pm 2.71\%$ and $74.32 \pm 2.21\%$, respectively. The T-1 contained lower uronic acid content ($7.15 \pm 0.15\%$) while it was absent of protein ($0.16 \pm 0.06\%$). The phenomenon of this difference in composition indicated that the enzyme produced good deproteinization effect. Thus, SDF can be considered a relatively pure substance. In addition, the deficient of flavonoids and protein detection determined the independence of the antioxidants of the SDF.

3.5 Physical characterization

3.5.1 FT-IR spectrum analysis

The IR analysis spectrum of the soluble dietary fiber was shown in Fig. 4. The absorption peaks around 1680 to 1639 cm^{-1} belonged to the -C=O carboxy symmetric stretching vibration of the uronic acid groups [34]. The groups of -C-O on the soluble dietary fiber were indicated by the absorption band at 1137 and 1139 cm^{-1} [35]. The adsorption band at 1072 cm^{-1} was attributed to the C-O-C stretching vibration [36] of the pyranose pentacyclic ring. It showed that this long chain cellulose with strengthen O-H and short -CH_2 absorption peak contained a glycosidic-bonded pyranose ring.

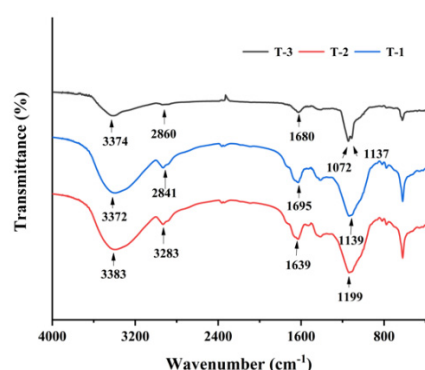


Fig. 4. FT-IR spectra of SDF.

3.5.2 SEM shape

There were a large number of obvious pore structures on the surface under the T-1 treatment mode, while the

surface of the T-2 was relatively gentle at the same magnification in Fig. 5. The reason for these phenomena could be that the water of SDF slowly evaporated under the action of high temperature, which caused the structure collapsed [37-38] and compacted into an inseparable structure [39]. In addition, the moisture removal efficiency of different drying methods was also an important factor [40] in the coarse pore structure of the SDF surface.

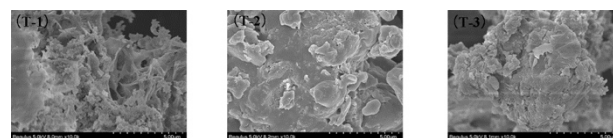


Fig. 5. SEM of SDF with different treatments.

3.6 Antioxidant capacity analysis

The principle of DPPH free radical scavenging activity depended on the reports [41-42]. As shown in Fig. 6 (A), it can be seen that the T-1, T-2 and T-3 show a significant ($P < 0.05$) dose-dependent effect on better free radical scavenging ability in the concentration range from 0.2 mg/mL to 4.0 mg/mL . Comparing these three treatments, it can be found that T-1 and T-3 had better oxidation resistance than T-2, which may attribute to that the higher temperature environment destroyed the internal structure of SDF, resulting in a decrease in electron binding ability to DPPH radicals [43]. High temperature treatment also promoted the decomposition of some functional components in SDF (such as polysaccharides and brass) [44] to reduce their hydrogen reduction ability.

The absorbance of ABTS can be determined at 734 nm to calculate the total antioxidant capacity of the samples [45]. As shown in Fig. 6 (B), the ABTS^+ radical scavenging activity of T-1, T-2 and T-3 were positively correlated ($P < 0.05$) with the sample concentration range. The scavenging activities were in turn of $\text{T-1} > \text{T-3} > \text{T-2}$. These data demonstrated that the ABTS^+ free radical scavenging activity of SDF obtained by different drying methods depended on their structural integrity and molecular size [46], which was no different from previous reports.

Gene transcriptional expression process was blocked when the hydroxyl group rapidly reacted with intracellular DNA across the biofilm system [47]. As shown in the Fig. 6 (C), it could be seen that the hydroxyl radical scavenging activity of T-1, T-2 and T-3 were concentration dependent, and it followed the order of $\text{T-1} > \text{T-3} > \text{T-2}$. The factor for this phenomenon may be explained by that T-1 provided a amount of active hydroxyl groups such as polysaccharides under the low temperature, which was consistent with previous reports [48].

3.7 Physical property analysis

Dissolution time was an important indicator to measure the shrinkage, porosity and cell damage of products, which can be directly present the rehydration rate of the dried SDF in this test [49]. As shown in Table 4, the

minimum dissolution time of SDF under vacuum freeze dried was 32 s ($P < 0.05$).

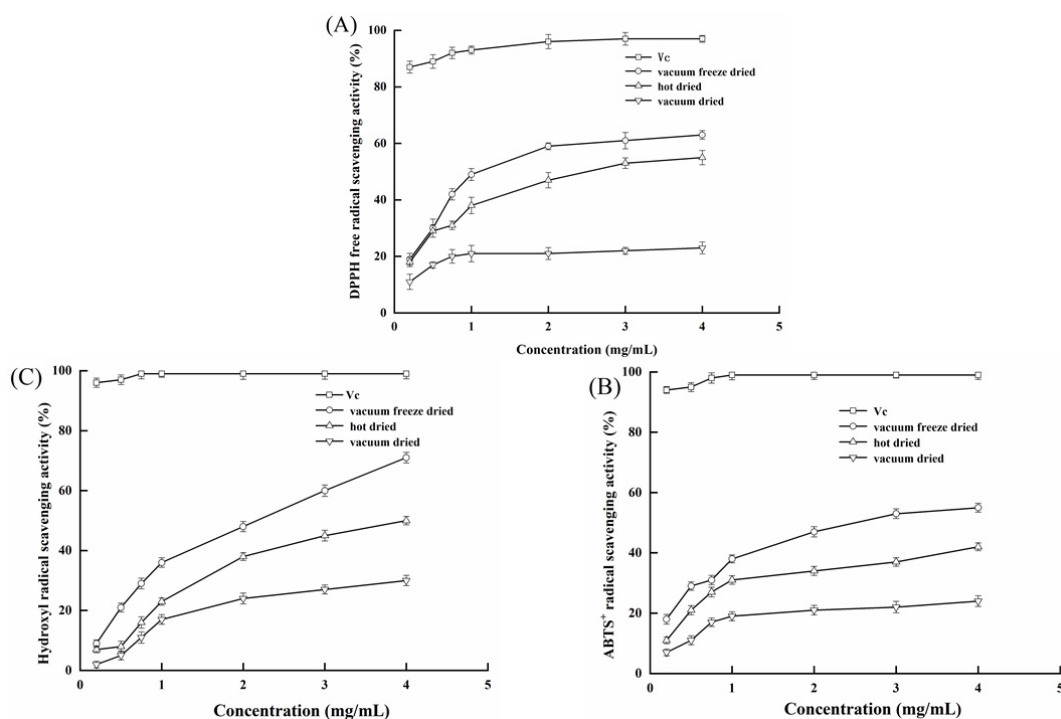


Fig. 6. Antioxidant activities on DPPH free radical scavenging activity (A), ABTS⁺ radical scavenging activity (B) and Hydroxyl radical scavenging activity (C) of SDF. Values were present as means±SD.

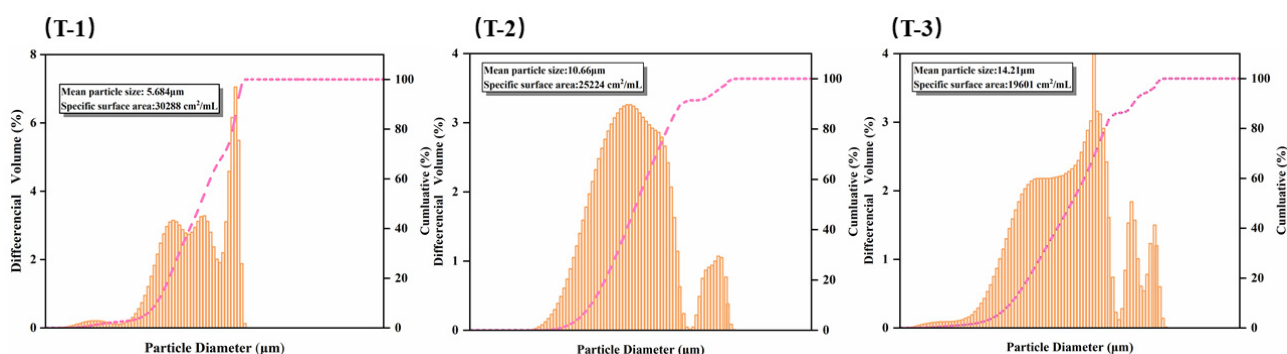


Fig. 7. FT-IR spectra of SDF.

It can be found that the dissolution time under hot dried was significantly longer than vacuum freeze dried (T-1) and vacuum dried (T-3) ($P < 0.05$). The bulk density was one of the most important parameters reflecting the texture of the powder [50]. The bulk density of SDF was significantly higher ($P < 0.05$) than vacuum dried (T-2) and vacuum dried (T-3) by vacuum freeze dried (T-1) according to Table 4. That maybe contribute to that the average particle size of SDF became smaller by the method of hot dried (T-2), when the volume became smaller under the same mass. It maintained the original loose porous structure of the tissue by methods of vacuum dried (T-1) and vacuum freeze dried (T-3), which adopted vacuum freeze sublimation dehydration technology. It was significant difference ($P < 0.05$) on the swelling ability with these three methods. The order of swelling ability was given in the table as follows: vacuum dried > hot

dried > vacuum dried. This may be due to that the molecule structure (especially high molecular polymers such as starch) was destroyed and connected with particles, which eventually led to a lower swelling capacity under the high temperature during hot dried. The relative increase in the action of water and gelation led to high swelling ability under the treatment of vacuum freeze dried [51]. The effect of different treatments on rehydration of SDF were significant ($P < 0.05$). This phenomenon may due to that it maintained the better original structure of the products and the material structure was loose after drying with the method of vacuum freeze dried (T-1) [52]. In addition, the experiments have been supplemented as you suggested about average particle size. The mean particle size of T-1, T-2 and T-3 were 5.864 µm, 10.66µm and 14.21µm, and the specific surface area were 30288 cm²/mL, 25224

cm²/mL and 19601 cm²/mL, respectively. However, the rehydration ratio of SDF obtained by hot air drying was less than fifty percentage of that of vacuum dried products. The reason was that hot dried had a high rate of water loss at the initial stage and the structure was seriously damaged.

4 Conclusion

In this study, the SDF was purified by enzymatic hydrolysis with papain and glucoamylase after extracted with hot water and precipitated in ethanol from jujube. BBD was adopted to increase the extraction yield of SDF, and the optimal conditions were obtained: extraction temperature of 94 °C, extraction time of 40 min and ratio of raw material to water of 1:21 g/mL. It was coincided with the model predictions under these conditions, which the extraction yield of SDF was 20.17 ± 0.16%. SEM and FT-IR clearly showed that the characteristic structure of SDF has a uronic acid groups. The bioactivity assays indicated that the SDF treated by vacuum freeze dried showed relatively higher antioxidant activity in vitro than that by hot dried.

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Compliance with ethics guidelines

The authors declare that they have no conflict of interest. This article does not contain any studies with human or animal subjects performed by any of the authors.

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