Preparation and Properties of Insoluble Dietary Fiber from Marc of *Hovenia dulcis* Thunb.

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Abstract: Marc from *Hovenia dulcis* Thunb. fruit is a new cheap source of insoluble dietary fiber (IDF). To utilize this resource comprehensively, the preparation of IDF from *H. dulcis* marc by chemical methods and its bleaching with hydrogen peroxide (H_2O_2) were studied. The nutritional components and functional properties of the product were also analyzed. According to the result of single factor analysis and orthogonal test, the reasonable technology of extraction is that the marc is immersed in 1.5 mol/L NaOH at 20 ℃ for 40 min with 8∶1 (mL∶g) ratio of liquid to material, then filtered and washed to neutrality. The residue was transferred into H_2SO_4 solution of pH value 2.0 with 6∶1 (mL∶g) ratio of liquid to material and soaked at 60 ℃ for 60 min, then washed to neutrality. The optimum processing conditions of decoloration were as follows: 5 % H_2O_2, pH value 12, 45 ℃, 5 h, then dried at 65 ℃ for 5 h, milled and sieved (380 μm). Under these conditions the IDF whiteness increased from 51.63 % to 60.21 %. The IDF yield is 74.02 % and purity increased from 62.62 % to 84.97 % (on dry base). Compared with the marc, water-holding capacity, oil-holding capacity, water-retention capacity and swelling capacity of IDF product increased from 2.4 to 4.3 g/g, 2.3 to 3.9 mL/g, 2.6 to 4.7 g/g and 2.8 to 4.6 mL/g, respectively.

Key words: marc of *Hovenia dulcis* Thunb. fruit; insoluble dietary fiber; decoloration

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北枳椇果渣不溶性膳食纤维的制备及其性能特性研究

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摘 要: 对枳椇果渣不溶性膳食纤维(IDF)的化学法提取工艺、双氧水脱色工艺进行了研究,并对产品的营养成分和功能性质进行分析。通过单因素及正交试验确定最佳的化学法提取工艺为:以液料比为 8∶1(mL∶g)、浓度为 1.5 mol/L 的氢氧化钠溶液,在 20 ℃ 条件下处理 40 min 后过滤,冲洗至中性,然后取滤渣转移至液料比为 6∶1(mL∶g)、pH 值 2 的硫酸溶液中,60 ℃ 下作用 60 min,冲洗至中性,干燥。不溶性膳食纤维的提取率为 74.02 %,质量分数由果渣中的 62.62 %(干基计)提高到 84.97 %;双氧水脱色的最佳工艺条件为:5 % H_2O_2、pH 值 12、45 ℃、5 h,在此条件下不溶性膳食纤维的白度由 51.63 % 增加到 60.21 %;制备的膳食纤维产品的持水力由果渣中 2.4 g/g 增加到 4.3 g/g、持油率由 2.3 mL/g 增加到 3.9 mL/g、结合水力由 2.6 g/g 增加到 4.7 g/g、膨胀力由 2.8 mL/g 增加到 4.6 mL/g。

关键词: 枳椇果渣;不溶性膳食纤维;脱色

Hovenia dulcis Thunb. fruit has a series of pharmacological functions, such as liver protection[1], sweetness inhibition[2-3], nerve protection[4], histase release inhibition[5] and can be processed into products. This would generate a lot of marc with a high content of insoluble dietary fiber (IDF). IDF has many important physiological functions, such as reducing the risk of colon cancer[6], modulating the secretion of certain hormones, improving insulin sensitity, increasing luminal mucin contents[7], reducing the risk of developing diabetes risk, and anti-inflammatory activity[8]. An intake of 30-35 g of IDF per day is advised by the American

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Association of Dieticians[9]. To create more economic value and solve wasted marc problem, technology for the preparation of IDF from the marc of *H. dulcis* and its chemical composition were studied. The water-holding capacity(WHC), oil-holding capacity(OHC), water-retention capacity(WRC) and swelling capacity(SWC) of the product were also determined.

1 Materials and methods

1.1 Materials

The marc from *H. dulcis* in Taibai mountain which had been extracted for juices effectively was cleaned with tap water and oven-dried at 50 ℃, ground to 550 μm, sieved and stored at atmospheric temperature in sealed plastic containers prior to further analyses.

1.2 IDF preparation

The raw material was immersed in sodium hydroxide(NaOH) solution with 8∶1(mL∶g) ratio of liquid to material at appropriate temperature. It was then filtered after heating at a constant temperature for some time and rinsed to neutrality. The residue was transferred into H_2SO_4 solution of pH value 2.0 with 6∶1(mL∶g) ratio of liquid to material and soaked for some time at appropriate temperature, then washed to neutrality. The filtered residue was bleached with H_2O_2 and dried at 65 ℃ for 5 h, milled and sieved(380 μm) to obtain the product.

1.3 Determination of whiteness

Five g sample were weighted, immersed in H_2O_2 of appropriate strength with 12∶1 ratio of liquid to material. Adjusted to definite pH values and maintained for some time, washed several times until neutral. Dried at 50 ℃ in oven, whiteness was determined with a color difference meter(MINOLTA CR-310). The data was directly recorded on printing paper. The whiteness(W_h) was calculated using the following equation:

$$W_h = 100 - \sqrt{(100 - L) \times a^2 \times b^2}$$

Where: L—lightness, ranging from 0(black) to 100(white); a—ranges from 100(green) to +100(red); b—ranges from 100(blue) to +100(yellow).

1.4 Chemical composition

The moisture content was determined by gravimetric heating(100 ℃ ±5 ℃ until constant weight) using a 2-10 g sample according to Chinese standard GB/T 5009.3 - 2003. Ash, protein, fat and starch were analyzed according to GB/T 5009.4 - 2003, GB/T 5009.5 - 2003, GB/T 5009.6 - 2003 and GB/T 5009.9 - 2003 respectively. IDF was tested using GB/T 5009.88 - 2003.

1.5 Functional properties

1.5.1 Water-holding capacity(WHC)[10] WHC which is defined as the quantity of water that is bound to the fiber without the application of any external force(except gravity and atmospheric pressure), was determined by accurately weighing a dry sample(1 g) into a graduated test tube, and adding around 30 mL water, where upon it was allowed to hydrate for 18 h at ambient temperature. The supernatant was removed by passing it through a sintered glass crucible(G4) under vacuum. The hydrated residue weight was recorded then dried at 105 ℃ for 2 h to obtain the dry residue weight.

$$\text{WHC} = (\text{hydrated residue weight} - \text{dry residue weight})/\text{dry residue weight}$$

1.5.2 Oil-holding capacity(OHC)[11] OHC was determined by centrifugation as described with slight modification. Samples(0.30 g) were suspended in soybean oil(20 mL, 0.925 g/mL density). After 20 h of equilibration at room temperature, the suspension was centrifuged at 4 000 g for 20 min. The supernatants were discarded and the hydrated fiber was weighed. OHC was expressed as mL oil/g dry fibrous residue

powder. OHC = (hydrated fibers weight − 0.30)/(0.925 × 0.30)

1.5.3 Water-retention capacity (WRC)[10] WRC defined as the quantity of water that is bound to the hydrated fiber following the application of an external force (pressure of centrifugation) was determined by accurately weighting a dry sample (1 g) into a graduated centrifuge tube, adding 30 mL of water and hydrated for 18 h, after which it was centrifuged (3 000 g, 20 min) and the supernatant solution was removed by passing it through a sintered glass crucible (G4) under vacuum. The hydrated residue weight was recorded and then the sample was dried at 105 ℃ for 2 h to obtain the dry residue weight.

WRC = (hydrated residue weight − dry residue weight)/dry residue weight

1.5.4 Swelling capacity (SWC)[12] For SWC determination, 0.1 g of sample of 380 um was hydrated in 10 mL of distilled water in a calibrated cylinder (15 cm diameter) at room temperature (30 ℃). After equilibration for 18 h, the bed volume was recorded and expressed as mL/g of the original sample dry weight.

SWC = volume occupied by sample/original sample dry weight

2 Results and discussion

2.1 Effects of various factors on IDF extraction rate

2.1.1 Effect of alkalinity Five samples of same weight were added to 0.5, 1.0, 1.5, 2.0, 2.5 mol/L NaOH separately with 8∶1 (mL∶g) ratio of liquid to material. It was maintained at 40 ℃ for 100 min in a water bath, and then washed to neutrality. Transferred the sample into H_2SO_4 solution of pH value 2.0 and soaked for 120 min at 70 ℃. Thereafter, the filter was washed to neutrality, dried and measured. The results are given in Fig. 1(a). We can see that appropriate alkalinity can hydrolyze giant molecules effectively to small molecules, and high alkalinity may lead to IDF hydrolysis. The alkalinities of 1.0, 1.5, 2.0 mol/L were used in the orthogonal experiment.

2.1.2 Effect of time of alkali treatment This single factor was determined under the same conditions except in 1.5 mol/L NaOH solution for 20, 40, 60, 80 and 100 min separately. The results are given in Fig. 1(b). IDF extraction rate is high at 40 min, which was adopted for further research.

2.1.3 Effect of temperature of alkali treatment This single factor was determined under the same conditions using 1.5 mol/L NaOH solution maintained in 20, 40, 60, 80, 100 ℃ separatly for 100 min. The results are given in Fig. 1(c). Around 60 ℃ the IDF extraction rate was high, so we used 40, 60 and 80 ℃ to design the orthogonal experiment.

2.1.4 Effect of temperature of H_2SO_4 treatment This single factor was determined under the same conditions using 1.5 mol/L NaOH solution maintained 40 ℃ for 100 min. Transferred the sample in H_2SO_4 solution of pH value 2.0 and soaked at 40, 50, 60, 70, 80 ℃ separately. The results are given in Fig. 1(d). At 60 ℃ the IDF extraction rate is high, but a too high temperature may lead to IDF hydrolysis, so we adopted 50, 60 and 70 ℃ for further research.

2.1.5 Effect of time of H_2SO_4 treatment This single factor was determined under the same conditions using 1.5 mol/L NaOH solution at 40 ℃ for 100 min. Transferred the sample in H_2SO_4 solution of pH value 2.0 and soaked at 70 ℃ for 30, 60, 90, 120, 150 min separately. The results are given in Fig. 1(e). The IDF extraction rate was high at 60 and 90 min, so we used 60 min for further research.

2.2 Orthogonal experiment analysis

Weighed 5.0 g of sample accurately for each group, choose significance factors, adopt $L_9(3^4)$ orthogonal experiment; the design is given in Table 1.

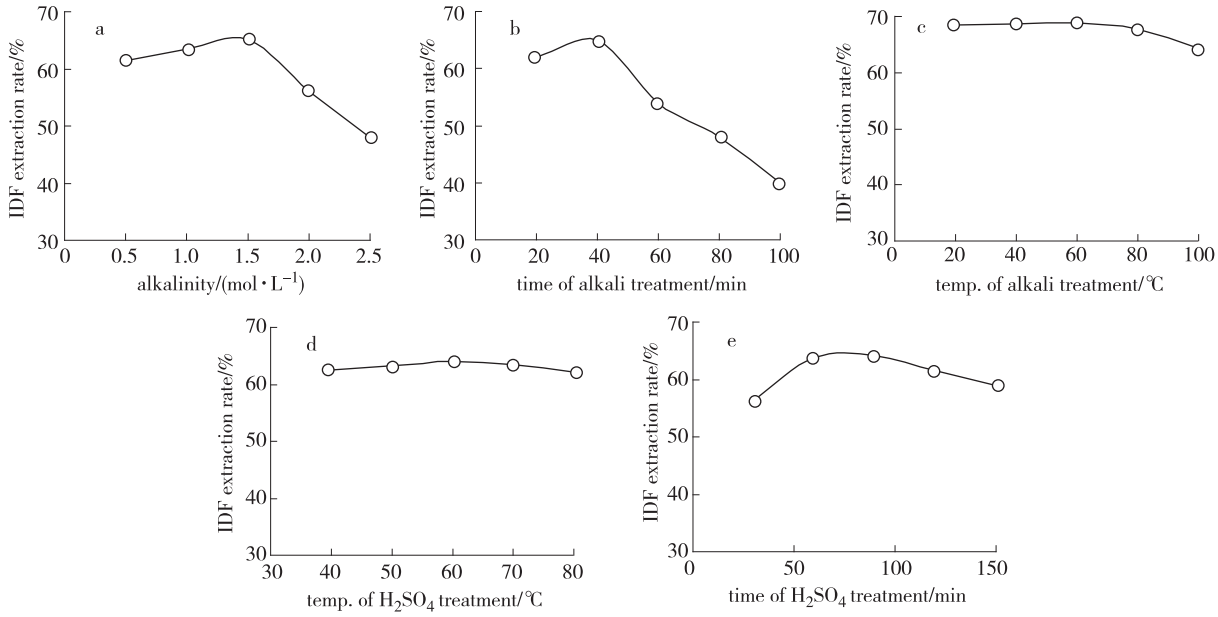

Fig. 1 Effects of different reaction conditions on IDF extraction rate

Table 1 Orthogonal experimental results and analysis of IDF extraction rate

No.	A alkalinity/(mol·L^{-1})	B temp. of alkali treatment/℃	C temp. of H_2SO_4 treatment/℃	IDF weight/g
1	1.0	20	50	3.4408
2	1.0	40	60	3.6201
3	1.0	60	70	3.4126
4	1.5	20	60	3.6812
5	1.5	40	70	3.6322
6	1.5	60	50	3.5278
7	2.0	20	70	3.4359
8	2.0	40	50	3.4621
9	2.0	60	60	3.4562
k_1	3.4912	3.5193	3.4769	
k_2	3.6137	3.5878	3.5858	
k_3	3.5525	3.4655	3.4935	
R	0.1225	0.1223	0.1089	

The significance of IDF extraction from the range analysis is: A > B > C. The best combination is $A_2B_2C_2$, and the weight of the IDF is 3.701 0 g corresponding to IDF extraction rate 74.02 %.

2.3 Bleaching

A series of major factors affecting bleaching were chosen in designing the orthogonal experiment. The factors and results are presented in Table 2.

As we can see, the significance of IDF H_2O_2 bleaching from the range analysis is: A > D > B > C. The better assemble for W_h value is $A_4B_4C_1D_4$, which is very close to $A_4B_4C_3D_4$, and for L value is $A_4B_4C_3D_4$. So $A_4B_4C_3D_4$ was chose, and under such conditions the L value is 60.21 and W_h value is 51.63.

The color of extracted insoluble dietary fiber powder is from yellow to white after H_2O_2 bleaching with whiteness of 60.21. This means that the product can be widely used in food manufacture.

2.4 Chemical composition

The compositions of marc and extracted insoluble dietary fiber from *H. dulcis* were presented in Table 3.

Table 2　Results of orthogonal test of IDF hydrogen peroxide bleaching

No.	A pH value	B H_2O_2 dose/%	C temperature/℃	D time/h	W_h	L
1	6	2	35	2	40.02	43.12
2	6	3	40	3	40.03	45.24
3	6	4	45	4	44.77	49.11
4	6	5	50	5	45.21	51.28
5	8	2	40	4	45.72	52.08
6	8	3	35	5	49.24	55.29
7	8	4	50	2	44.38	50.78
8	8	5	45	3	45.62	51.94
9	10	2	45	5	48.78	55.51
10	10	3	50	4	46.89	51.40
11	10	4	35	3	47.51	52.61
12	10	5	40	2	46.70	53.48
13	12	2	50	3	47.76	56.75
14	12	3	45	2	47.65	56.00
15	12	4	40	5	49.25	59.02
16	12	5	35	4	50.65	57.91
k_1	42.51	45.57	46.86	44.69		
k_2	46.25	45.96	45.42	45.23		
k_3	47.47	46.48	46.71	47.01		
k_4	48.83	47.05	46.07	48.12		
R	6.28	1.48	1.44	3.43		
k'_1	47.19	51.87	52.23	50.85		
k'_2	52.53	51.99	52.46	51.64		
k'_3	53.26	52.88	53.15	52.63		
k'_4	57.43	53.66	52.56	55.28		
R'	10.24	1.79	0.92	4.43		

Table 3　Chemical compositions of marc and IDF product from *H. dulcis*　%

samples	moisture	ash	protein	fat	starch	IDF(on dry basis)
marc	11.59	3.71	8.45	7.30	6.62	62.62
IDF	8.59	4.35	2.38	3.06	—	84.97

The purity of IDF product increased from 62.62 % to 84.97 % in marc. It is much higher than similar products on the market from other materials, for example, okara 55.48 % and soybean seeds 24.37 %[13], rice bran 18.3 %–30.5 %[14], date fresh 14.4 %–18.4 %[15] respectively. This shows that technology for preparation of insoluble dietary fiber is reasonable and scientific.

2.5　Functional properties

The determined results of WHC, OHC, WRC and SWC from marc and IDF product were presented in Table 4.

Table 4　The functional properties of marc and IDF product from *H. dulcis*

functional properties	WHC/(g·g^{-1})	WRC/(g·g^{-1})	OHC/(mL·g^{-1})	SWC/(mL·g^{-1})
marc	2.4	2.6	2.3	2.8
IDF	4.3	4.7	3.9	4.6

As shown in Table 4, IDF product from *H. dulcis* has a higher WHC(4.3 g/g) than the results reported previously, such as 3.10 g/g in wheat and 3.50 g/g in pea. The WRC(4.7 g/g) was similar to 5.33 g/g in coconut fiber and 3.10 g/g in carrot[16-17]. The OHC(3.9 mL/g) is close to mango peel dietary fiber(DF) (4 g oil/g dry sample) and in citrus peel ber(2.35–5.09 g oil/g dry sample)[18-19]. This shows that *H. dulcis* insoluble dietary fiber have a good market prospect.

3　Conclusions

3.1　The marc from *Hovenia dulcis* Thunb. is a new cheap source of insoluble dietary fiber. The reasonable

technology of extraction is that the marc is immersed in 1.5 mol/L, 20 ℃ NaOH solution for 40 min with 8∶1 (mL∶g) ratio of liquid to material, then filtered and washed to neutrality. The residue was transferred in 60 ℃ H_2SO_4 solution of pH value 2.0, and soaked for 60 min, thereafter, filtered and washed to neutrality. The filtered residue was bleached with H_2O_2. The optimum condition was pH value 12, H_2O_2 strength 5 % in 45 ℃ for 5 h. Then dried at 65 ℃ for 5 h, milled and sieved(380 μm).

3.2 The IDF product presents albescent. Its whiteness is 60.21 %. IDF extraction rate is 74.02 % and purity increased from 62.62 % to 84.97 % (on dry base), which was much higher than that of IDF from many other fruits and vegetables on the market. The IDF powder presented 4.3 g/g water-holding capacity, 3.9 mL/g oil-holding capacity, 4.7 g/g water retention capacity and 4.6 mL/g swelling capacity. This shows that the technology for preparation of IDF powder was simple, reasonable and scientific, and functional properties of the product are conspicuous. It can be used as either a functional food or a functional food ingredient.

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