

Ultrasonic-assisted extraction of dietary fibers from fenugreek seeds

H. Isleroglu and S. Dastan

Food Engineering Department, Tokat Gaziosmanpasa University, Tokat, 60150, Turkey.

*Corresponding author: hilal.isleroglu@gop.edu.tr; sedanurdstnn96@gmail.com

ABSTRACT In this study, soluble and insoluble dietary fibers were extracted from fenugreek (*Trigonella-foenum graecum L.*) seeds using the ultrasonic-assisted extraction method and the fibers were characterized. The effects of the solid–solvent ratio, ultrasonic amplitude and extraction time were investigated using the response surface methodology to obtain the highest total dietary fiber yield. In addition, functional and physicochemical properties of soluble and insoluble dietary fibers such as water holding capacity, oil holding capacity, swelling capacity, glucose adsorption index and α -amylase inhibition capacity were determined. The optimum process conditions were determined as follows: the solid–solvent ratio of 52.83 g/L, ultrasonic amplitude of 57.86 % and extraction time of 24.68 min. At these conditions, insoluble dietary fiber yield was 50.73 % and soluble dietary fiber yield was 28.67 %. According to the characterization results, insoluble dietary fiber achieved better physicochemical and functional properties compared to soluble dietary fiber.

KEYWORDS Ultrasonic-assisted extraction; Fenugreek seeds; Dietary fiber; Glucose adsorption index; Water holding capacity

1. Introduction

Dietary fiber (DF) is a carbohydrate polymer having ten or more connected monomers that cannot be hydrolyzed by human digestion system (Liu et al., 2021). DFs can reduce cholesterol and prevent diseases such as heart attack, colon cancer and diabetes (Ma and Mu, 2016; Wang et al., 2021). Moreover, DFs are effective for controlling body weight and lowering blood lipid and glucose levels (Huang et al., 2018). DFs can be categorized as soluble dietary fiber (SDF) and insoluble dietary fiber (IDF) (Du et al., 2021). In literature, it has been reported that fruits have higher content of SDF than IDF; however, cereals, seeds, legumes, and vegetables have high IDF contents (Tejada-Ortigoza et al., 2016). Fenugreek seeds, which are considered as legumes, are potential DF sources.

Fenugreek (*Trigonella-foenum graecum L.*) is a medicinal plant, and it was originated from Western Asia and Southeastern Europe (Khoja et al., 2021). Fenugreek seeds are used for the treatments of some diseases such as fever, high blood pressure and diabetes, and these seeds also have antioxidant and antibacterial properties (Madhava Naidu et al., 2011; Riasat et al., 2018). Fenugreek seeds are reported to be a good of soluble and insoluble dietary fibers (Hooda and Jood, 2005; Krishnakumar et al., 2012). The functional and physicochemical properties of DFs are mainly associated with their source, SDF and IDF percentage, matrix structure, and extraction methods (Ma and Mu, 2016). Hence, the extraction method and optimization of the extraction parameters are important topics to produce high-quality DFs (Wang et al., 2021).

In this study, ultrasonic-assisted extraction was performed to obtain IDF and SDF from fenugreek seeds. The conditions of the ultrasonic-assisted alkaline extraction technique namely solid–solvent ratio (g/L), ultrasonic amplitude (%) and extraction time (min) were optimized by a central composite design. Moreover, physicochemical and functional properties of the IDF and SDF produced at different extraction conditions were investigated.

2. Methods

2.1 Material

Fenugreek seeds were bought from a local market in Tokat, Turkey. The proximate analysis of the seeds was made according to AOAC (2000), and the carbohydrate content was determined by the difference method. Fenugreek seeds contained 6.17 ± 0.05 % moisture, 17.09 ± 0.19 % fat, 16.58 ± 0.21 % protein, 55.64 ± 0.14 % carbohydrates, and 4.52 ± 0.09 % ash. The seeds were purged of dust and impurities by hand and using a sieve, and then were grained using a rotary grinder (Sinbo SHB 3020, Turkey). Then, grinded seeds were sieved using a 630 μ m pore diameter sieve and the powder under the sieve was collected. The powdered fenugreek seeds were subjected to defatting before extraction. The powder was mixed with hexane (1:4, w/v) using a magnetic stirrer for 2 hours at room temperature. This procedure was repeated thrice with fresh hexane each time. After the defatting process, the hexane layer was discarded, and the residual hexane was removed at 50 °C overnight.

2.2 Ultrasonic-assisted extraction of soluble and insoluble dietary fiber

The extraction of soluble dietary fiber (SDF) and insoluble dietary fiber (IDF) of the defatted fenugreek seeds powder was performed by the ultrasonic-assisted extraction process using 1 M of NaOH as solvent. The ultrasonic-assisted extraction (UAE) was carried out using a labora-

Table 1: The experimental design and extraction yields.

S:S (g/L)	UA (%)	ET (min)	IDF (%)	SDF (%)	TDF (%)
60	80	22.5	50.59	24.73	75.32
100	80	5	36.00	20.27	56.27
20	30	40	41.57	21.34	62.91
100	55	22.5	47.69	25.26	72.95
60	55	22.5	50.51	28.92	79.43
60	30	22.5	47.40	24.29	71.68
20	55	22.5	47.86	28.60	76.46
60	55	5	42.57	22.76	65.33
20	55	22.5	48.76	26.95	75.71
100	30	40	40.33	21.46	61.78
100	80	40	39.38	20.13	59.51
20	80	40	38.23	24.20	62.44
60	55	22.5	49.09	29.96	79.05
20	80	5	38.92	22.45	61.37
20	30	5	34.99	19.40	54.39
60	55	40	46.30	24.18	70.47
60	55	40	48.16	23.80	71.96
60	80	22.5	48.41	26.70	75.12
60	30	22.5	47.54	24.43	71.96
60	55	5	44.42	21.15	65.56
100	30	5	33.20	17.91	51.12
100	55	22.5	46.60	25.43	72.02
60	55	22.5	50.68	28.75	79.43

S:S: solid–solvent ratio, UA: ultrasonic amplitude, ET: extraction time.

tory scale sonicator having 1/2" diameter probe (Q 500, Q Sonica, 500 W, 20 kHz, USA). An ice bath was also used to prevent samples from overheating and extraction temperature was kept at $\approx 25^\circ\text{C}$. The solid–solvent ratio (20–100 g/L), ultrasonic amplitude (30–80 %) and extraction time (5–40 minutes) were chosen as independent variables. After the extraction process, SDF and IDF fractions were separated according to the method given by Wang et al. (2021). The experimental design for the extraction of DFs is provided in Table 1. The SDF, IDF and total dietary fiber (TDF) yields were calculated by dividing the weight of individual dried dietary fiber fraction by the weight of the initial defatted fenugreek seeds powder.

2.3 Physicochemical properties of the IDF and SDF

To determine the water retention capacity (WRC), 250 mg of the extracted IDF or SDF were mixed with 15 mL of distilled water in a 50 mL of centrifuge tube. The samples were left at room temperature for 1 hour and centrifugation was applied for 20 min at 3000 g. After discarding the supernatants, the precipitates were weighed and the samples' WRC was calculated as gram of water per gram of dry sample (Sun et al., 2018).

To determine oil adsorption capacity (OAC), similar steps were used as for the WRC, only virgin olive oil was used instead of distilled water. The OAC was defined as gram of oil per gram of dry sample (Sun et al., 2018).

The swelling capacity (SC) of the IDF and SDF was determined using the method of Ma and Mu (2016) and the SC calculated using Eq. 1

$$SC \text{ (mL/g)} = (v_1 - v_0) / w_0 \quad (1)$$

where, v_1 is the volume of the sample after hydration, v_0 is the volume of the sample prior to hydration and w_0 is the initial weight of the sample.

2.4 Functional properties of the IDF and SDF

The glucose adsorption capacity (GAC) was determined using Chu et al. (2019) method. The glucose contents of the supernatants were determined using dinitro salicylic acid (DNS) (3-5 dinitro salicylic acid, CDH, India) method (540 nm) (Miller, 1959). The GAC was calculated using Eq. 2.

$$GAC \text{ (mmol/g)} = (G_1 - G_2) / \times V \quad (2)$$

where, G_1 is the glucose concentration before adsorption (mmol/g), G_2 is the glucose concentration after adsorption (mmol/g), W is the weight (g) of the samples and V is the supernatant volume in mL.

Ding et al. (2020) method was used to determine the α -amylase (α -amylase from *Aspergillus Oryzae*, ≈ 1.5 U/mL, Sigma-Aldrich, Germany) inhibition capacity (AAIC) of the IDF and SDF. The AAIC of the samples were determined according to the Eq. 3.

$$AAIC \text{ (%) } = (A_2 - A_1) / A_2 \times 100 \quad (3)$$

where, A_1 is the absorbance of the supernatant with DF, and A_2 is the absorbance of the supernatant without DF at 540 nm.

2.5 Optimization and statistical analysis

A central composite design was used for optimizing the extraction process. The conditions giving the highest TDF yield were optimized by the desirability function approach and the model used for the regression analysis is given in Eq. 4.

$$Yield \text{ (%) } = \beta_0 + \sum_{i=1}^k \beta_i X_i^2 + \sum_{i=1}^{k-1} \sum_{j=i+1}^k \beta_{ij} X_{ij} \quad k = 1, 2 \quad (4)$$

where, β_0 indicates the constant, k is the number of independent variables, X_i is the i^{th} independent variable, β_{ij} is the j^{th} coefficient of i^{th} observation, and X_{ij} indicates the j^{th} independent variable of i^{th} observation.

One-sample t-tests and univariate analysis (Duncan post hoc, 95 % confidence interval) were carried out using the SPSS 22.0 (IBM, USA) package program. The regression analysis, which was used to determine the effects of the independent process variables on the responses, response surface graph and optimization study, were done using the Design Expert 7.0 (Stat-Ease Inc., USA) package program.

3. Results

3.1 Dietary fiber extraction and optimization

The experimental extraction yields and ANOVA results are given in Table 1 and Table 2, respectively. Results

showed that the highest TDF yields were obtained at the midpoint of the experimental design (Table 1). Generally, the extraction of bioactive compounds can be enhanced with a higher amount of solvent (Ding et al., 2019). On the other hand, excessive or lower dispersion of the sample may reduce the TDF extraction yield (Cheung and Wu, 2013). In this study, 60 g/L of a solid–solvent ratio provided better yields compared to that of 20 and 100 g/L (Table 1). Extraction time was one of the important independent variables affecting the yield of the IDF and SDF ($p < 0.05$, Table 2). We observed that the yields of TDF were lower at the short extraction times (5 min) compared to that of the longer extraction times (22.5 and 40 min) when the conditions of solid–solvent ratio and ultrasonic amplitude are the same (Table 1). Another parameter for the extraction yield was ultrasonic amplitude. At increasing ultrasonic amplitudes, the effect of the ultrasonic cavitation bubbles, which could have decomposed the cell wall of the products with a high rate of the mass transfer, could enhance the extraction yield (Jalili et al., 2017). Hence, the yield increased from $\approx 72\%$ to $\approx 79\%$ when the amplitude increased from 30 % to 55 % at the medium solid–solvent ratio (60 g/L) and extraction time (22.5 min). However, the yield decreased to $\approx 75\%$ when the amplitude increased to 80 % under the same conditions (Table 1). Jiang et al. (2019) reported similar results in their study that optimized extraction process of the arabinoxylan from the corn bran. They explained the reduction of the extraction yield at the higher ultrasonic power, by the high chemical decomposition (oxidation) which reduced the viscosity of arabinoxylan and led to a lower yield. Also, Sun et al. (2018) stated that fibers (mainly SDF) can degrade with the longer application of high ultrasonic treatment. The statistical results in Table 2 showed that the linear and quadratic effects of all the independent variables and the linear interactions were significant for the TDF yield ($p < 0.05$).

For the TDF yield, all generated quadratic models were significant ($p < 0.05$), and the non-significant lack of fit values were obtained ($p > 0.05$). The second-order polynomial model equation of TDF yield is expressed in Eq. 5. In addition, the response surface graph representing the effects of the independent variables on TDF yield is provided in Fig. 1. We also investigated the suitability of the model with statistical results such as R^2 , adjusted R^2 (adj- R^2), adequate precision, predicted residual error sum of squares (PRESS) and coefficient of variation (C.V., %) (Table 2). High R^2 value of the model and closeness of the R^2 and adj- R^2 values showed the goodness of fit of the generated model, which was the proof that only statistically significant terms were included in the generated model (Table 2).

$$\begin{aligned} TDF (\%) = & 15.87 + 0.31X_1 + 1.11X_2 + 1.89X_3 - \\ & 4.54 \cdot 10^{-4}X_1X_2 + 7.71 \cdot 10^{-4}X_1X_3 - 4.25 \cdot 10^{-4}X_2X_3 - \\ & 2.83 \cdot 10^{-3}X_1^2 - 8.46 \cdot 10^{-3}X_2^2 - 3.42 \cdot 10^{-2}X_3^2 \end{aligned} \quad (5)$$

For the ultrasonic-assisted extraction process of DFs from the fenugreek seeds, we carried out a numerical

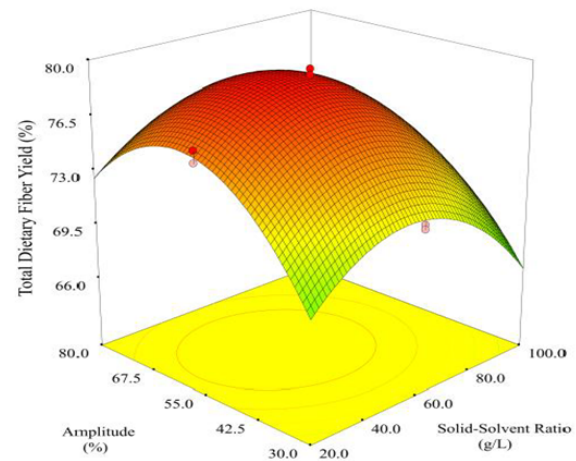


Figure 1: Response surface graph for total dietary fiber yield at the extraction time of 22.5 min.

optimization study to determine the optimum conditions in terms of the solid–solvent ratio, ultrasonic amplitude, and extraction time. We chose TDF value maximization for the optimization solutions. Only one solution calculated by the package program had a desirability value of 0.997. The optimum conditions of the extraction process were chosen as 52.83 g/L of the solid–solvent ratio, 57.86 % ultrasonic amplitude and 24.68 min of extraction time. For these conditions, the predicted TDF yield was 79.35 %, and according to the optimum point verification results, the experimental TDF yield was $79.40 \pm 0.43\%$. There was no significant difference between the experimental and predicted values of TDF yield according to the single sample t-test ($p > 0.05$). The IDF yield was $50.73 \pm 0.55\%$ and the SDF yield was $28.67 \pm 0.39\%$ at the optimum point.

3.2 The functional and physicochemical properties of IDF and SDF

The physicochemical properties of IDF and SDF for different extracts obtained at the different conditions are provided in Table 3 and Table 4, respectively. The ability to retain water when the centrifugation was applied to the DFs is defined as WRC (Ma and Mu, 2016). The highest result for the WRC of IDF was obtained for the 20 g/L solid–solvent ratio, 55 % ultrasonic amplitude and 22.5 min of extraction time (Table 3). Quite similar results were obtained for the SDF of the fenugreek seeds. At the optimum point, the WRC of the fenugreek' SDF was found as 4.93 ± 0.13 g/g (Table 4), which was higher compared to that of the banana stem and oat hull, and lower compared to that of the chia fiber and papaya peel (Alfredo et al., 2009; Jacometti et al., 2015; Zhang et al., 2017). Zhang et al. (2017) also reported that the ultrasound application significantly enhanced the WRC of the SDF samples.

OAC is another important physicochemical property for DFs, and it is defined as the ability of adsorbing fat (Navarro-González et al., 2011). Using the intense alkaline treatment can remove the cellulose, lignin, and

Table 2: ANOVA table and statistical parameters for extraction yield of the dietary fibers.

Source	DF	Sum of Squares			IDF	F Value		IDF	<i>p</i> - Value	
		IDF	SDF	TDF		SDF	TDF		SDF	TDF
Model	9	624.88	219.06	1536.44	52.90	17.95	624.66	< 0.0001	< 0.0001	< 0.0001
X_1	1	4.24	13.02	32.11	3.23	9.60	117.49	0.0956	0.0085	< 0.0001
X_2	1	3.52	7.79	21.80	2.69	5.75	79.75	0.1252	0.0323	< 0.0001
X_3	1	47.49	10.39	102.30	36.18	7.66	374.32	< 0.0001	0.0160	< 0.0001
X_1X_2	1	0.20	2.99	1.65	0.15	2.20	6.03	0.7044	0.1616	0.0289
X_1X_3	1	2.65	$9.9 \cdot 10^{-3}$	2.33	2.02	$7.3 \cdot 10^{-3}$	8.53	0.1792	0.9332	0.0119
X_2X_3	1	15.15	1.87	27.66	11.54	1.38	101.19	0.0048	0.2617	< 0.0001
X_{12}	1	72.88	2.27	100.90	55.53	1.68	369.19	< 0.0001	0.2178	< 0.0001
X_{22}	1	46.97	23.88	137.83	35.79	17.61	504.34	< 0.0001	0.0010	< 0.0001
X_{32}	1	190.00	89.85	541.17	144.78	66.27	1980.17	< 0.0001	< 0.0001	< 0.0001
Residual	13	17.06	17.63	3.55						
Lack of Fit	5	8.71	12.06	1.55	1.67	3.47	1.24	0.2473	0.0580	0.3746
Pure Error	8	8.35	5.56	2.00						
Total	22	641.94	236.68	1539.99						

Statistical Parameters for TDF Yield: R^2 : 0.9977, adj- R^2 : 0.9966, Adequate Precision: 81.625 PRESS: 11.41 C.V. (%): 0.76

X_1 : Solid–solvent ratio (g/L), X_2 : Ultrasonic Amplitude (%), X_3 : Extraction Time (min), DF: Degrees of Freedom, Adj- R^2 : Adjusted R^2 , PRESS: Predicted residual error sum of squares, C.V. (%): Coefficient of variation.

Table 3: Physicochemical and functional properties of insoluble dietary fiber.

S:S (g/L)	UA (%)	ET (min)	WRC (g/g)	OAC (g/g)	SC (mL/g)	GAC (mmol/g)	AAIC (%)
60	80	22.5	8.71 ± 0.35^d	4.35 ± 0.14^c	10.94 ± 0.03^e	13.41 ± 0.70^e	17.76 ± 0.88^f
100	80	5	5.89 ± 0.25^g	2.98 ± 0.01^{gh}	7.87 ± 0.18^h	9.45 ± 0.48^h	12.93 ± 0.69^i
20	30	40	5.08 ± 0.11^h	2.59 ± 0.26^i	6.46 ± 0.01^j	7.93 ± 0.24^j	10.75 ± 0.62^j
100	55	22.5	9.63 ± 0.45^{bc}	4.89 ± 0.08^{ab}	12.70 ± 0.42^{ab}	15.90 ± 0.06^b	20.56 ± 0.44^{ab}
60	55	22.5	9.68 ± 0.28^{bc}	4.76 ± 0.17^{ab}	12.16 ± 0.31^{cd}	15.04 ± 0.24^c	20.09 ± 0.22^{bcd}
60	30	22.5	5.53 ± 0.10^{gh}	2.77 ± 0.04^{hi}	6.95 ± 0.03^{ij}	8.79 ± 0.12^i	11.37 ± 0.20^j
20	55	22.5	10.02 ± 0.29^{ab}	5.03 ± 0.23^a	13.20 ± 0.32^a	16.53 ± 0.06^a	21.34 ± 0.21^a
60	55	5	5.89 ± 0.00^g	3.03 ± 0.18^{gh}	7.73 ± 0.35^h	9.37 ± 0.06^h	12.46 ± 0.45^i
20	55	22.5	9.62 ± 0.46^{bc}	4.93 ± 0.01^{ab}	11.97 ± 0.03^{cd}	14.72 ± 0.24^{cd}	19.47 ± 0.23^{cde}
100	30	40	5.16 ± 0.13^h	2.61 ± 0.19^i	6.96 ± 0.06^{ij}	8.38 ± 0.12^i	11.06 ± 0.21^j
100	80	40	7.01 ± 0.10^f	3.55 ± 0.04^f	9.18 ± 0.32^f	11.30 ± 0.12^f	14.49 ± 0.20^{gh}
20	80	40	7.03 ± 0.17^f	3.47 ± 0.17^f	9.20 ± 0.01^f	11.46 ± 0.24^f	15.11 ± 0.02^g
60	55	22.5	9.63 ± 0.34^{bc}	4.71 ± 0.04^b	12.93 ± 0.07^a	15.72 ± 0.19^b	20.87 ± 0.45^{ab}
20	80	5	6.70 ± 0.05^f	3.39 ± 0.07^f	8.70 ± 0.39^g	10.78 ± 0.18^g	14.17 ± 0.28^h
20	30	5	2.43 ± 0.11^i	1.23 ± 0.18^j	3.36 ± 0.17^k	4.12 ± 0.06^k	5.14 ± 0.22^k
60	55	40	7.93 ± 0.34^e	3.92 ± 0.04^d	10.59 ± 0.19^e	13.09 ± 0.17^e	17.13 ± 0.88^f
60	55	40	8.05 ± 0.25^e	3.93 ± 0.13^d	10.72 ± 0.02^e	13.16 ± 0.06^e	17.45 ± 0.44^f
60	80	22.5	8.67 ± 0.49^d	4.33 ± 0.15^c	11.81 ± 0.16^d	14.43 ± 0.12^d	18.85 ± 0.21^e
60	30	22.5	5.50 ± 0.10^{gh}	2.79 ± 0.09^{ghi}	7.09 ± 0.13^i	8.64 ± 0.25^i	11.21 ± 0.49^j
60	55	5	5.94 ± 0.01^g	3.06 ± 0.01^g	7.69 ± 0.39^h	9.59 ± 0.06^h	12.62 ± 0.20^i
100	30	5	2.22 ± 0.31^i	1.08 ± 0.04^j	2.61 ± 0.181	3.28 ± 0.231	4.21 ± 0.151
100	55	22.5	9.67 ± 0.15^{bc}	4.87 ± 0.06^{ab}	12.70 ± 0.39^{ab}	15.75 ± 0.18^b	20.72 ± 0.16^{ab}
60	55	22.5	9.33 ± 0.06^c	4.69 ± 0.08^b	12.08 ± 0.19^{cd}	14.80 ± 0.04^{cd}	19.31 ± 0.03^{de}
52.83	57.86	24.68	10.38 ± 0.31^a	4.84 ± 0.05^{ab}	12.35 ± 0.19^{bc}	15.74 ± 0.14^b	20.30 ± 0.48^{bc}

S:S: solid–solvent ratio, UA: ultrasonic amplitude, ET: extraction time. Uncommon superscripts within a column are significantly different ($p < 0.05$).

hemicellulose. Ultrasound treatment can expose more of the functional groups, which may enhance the DFs' oil retention (Huang et al., 2018; Zhang et al., 2020). The OAC of the IDFs varied between 1.08 and 5.03 g oil/g sam-

ple. The lowest values were observed for conditions of the lowest ultrasonic amplitude and the lowest extraction time. On the other hand, 55 % of the ultrasonic amplitude and 22.5 min of the extraction time provided the best

Table 4: Physicochemical and functional properties of soluble dietary fiber.

S:S (g/L)	UA (%)	ET (min)	WRC (g/g)	OAC (g/g)	SC (mL/g)	GAC (mmol/g)	AAIC (%)
60	80	22.5	4.23±0.18 ^{ef}	2.23±0.06 ^{cdef}	5.49±0.34 ^e	6.94±0.06 ^{de}	9.19±0.22 ^{def}
100	80	5	3.33±0.14 ^g	1.72±0.05 ^j	4.23±0.01 ^g	5.16±0.18 ^h	6.70±0.22 ⁱ
20	30	40	1.94±0.23 ^h	0.96±0.18 ^k	2.49±0.01 ^h	3.44±0.06 ⁱ	4.52±0.22 ^j
100	55	22.5	4.48±0.09 ^{bcd}	2.25±0.18 ^{cdef}	5.85±0.20 ^{bcd}	7.43±0.06 ^{abc}	9.81±0.22 ^{abcd}
60	55	22.5	4.61±0.12 ^{abcd}	2.51±0.10 ^{ab}	6.10±0.15 ^{ab}	7.70±0.12 ^a	9.97±0.44 ^{abcd}
60	30	22.5	1.96±0.05 ^h	1.08±0.01 ^k	2.61±0.19 ^h	3.24±0.06 ^{ij}	4.21±0.22 ^j
20	55	22.5	4.66±0.13 ^{abc}	2.21±0.19 ^{cdef}	6.07±0.19 ^{ab}	7.59±0.06 ^{ab}	10.12±0.22 ^{abc}
60	55	5	3.43±0.04 ^g	1.95±0.08 ^{ghi}	4.47±0.02 ^{fg}	5.41±0.41 ^h	7.17±0.44 ^{hi}
20	55	22.5	4.41±0.20 ^{bcd}	2.06±0.16 ^{fgh}	5.61±0.17 ^{de}	6.81±0.18 ^{ef}	8.72±0.88 ^{efg}
100	30	40	2.07±0.06 ^h	1.09±0.10 ^k	2.62±0.17 ^h	3.31±0.18 ⁱ	4.36±0.44 ^j
100	80	40	4.10±0.01 ^f	2.12±0.08 ^{efg}	5.37±0.17 ^e	6.47±0.12 ^{fg}	8.57±0.22 ^{fg}
20	80	40	4.23±0.01 ^{def}	2.15±0.12 ^{defg}	5.42±0.17 ^e	6.74±0.06 ^{ef}	9.19±0.22 ^{def}
60	55	22.5	4.48±0.06 ^{bcd}	2.20±0.18 ^{cdef}	5.84±0.02 ^{bcd}	7.11±0.12 ^{cde}	9.35±0.44 ^{cdef}
20	80	5	3.63±0.22 ^g	1.85±0.12 ^{hij}	4.72±0.17 ^f	5.39±0.24 ^h	7.32±0.22 ^{hi}
20	30	5	1.03±0.06 ⁱ	0.47±0.02 ^l	1.37±0.18 ⁱ	1.67±0.18 ^k	2.02±0.22 ^k
60	55	40	4.34±0.21 ^{cdef}	2.20±0.10 ^{cdef}	5.71±0.01 ^{cde}	6.77±0.12 ^{ef}	8.57±0.22 ^{fg}
60	55	40	4.30±0.46 ^{cdef}	2.26±0.04 ^{cdef}	5.57±0.11 ^{de}	6.34±0.06 ^g	7.94±0.22 ^{gh}
60	80	22.5	4.60±0.34 ^{abcde}	2.38±0.08 ^{bcd}	5.97±0.03 ^{abc}	6.89±0.18 ^{de}	9.50±0.44 ^{cde}
60	30	22.5	1.93±0.09 ^h	1.01±0.04 ^k	2.48±0.01 ^h	2.93±0.18 ^j	3.89±0.22 ^j
60	55	5	3.41±0.11 ^g	1.75±0.09 ^{ij}	4.45±0.04 ^{fg}	5.28±0.18 ^h	7.01±0.22 ⁱ
100	30	5	1.07±0.02 ⁱ	0.53±0.02 ^l	1.37±0.18 ⁱ	1.81±0.12 ^k	2.34±0.44 ^k
100	55	22.5	4.60±0.09 ^{abcde}	2.40±0.02 ^{bc}	5.97±0.03 ^{abc}	7.24±0.06 ^{bcd}	9.66±0.88 ^{bcd}
60	55	22.5	4.76±0.06 ^{ab}	2.34±0.09 ^{bcd}	6.21±0.03 ^a	7.61±0.12 ^a	10.44±0.22 ^{ab}
52.83	57.86	24.68	4.93±0.13 ^a	2.69±0.10 ^a	6.21±0.12 ^a	7.75±0.18 ^a	10.64±0.39 ^a

S:S: solid–solvent ratio, UA: ultrasonic amplitude, ET: extraction time. Uncommon superscripts within a column are significantly different ($p < 0.05$).

(Table 3). Same as IDF, the SDF of the samples had the lowest results when sonication amplitude and extraction time were lower (Table 4). (Krishnakumar et al., 2012) revealed that the SDF of fenugreek seeds had 20 mL/g WRC and 3 mL/g OAC, and their OAC results were close to the results found in our study.

SC is the ratio of the volume of DFs after applying excessive amounts of water – later then the equilibrium state – to the actual weight of DFs (Ma and Mu, 2016).

It was also reported that the alkaline treatment may improve the SC of DFs due to disrupting cellulose chains and forming porous structures. Further, ultrasonic treatments can enhance the SC of DFs (Huang et al., 2018; Meng et al., 2019). In this study, SC values of the IDF were measured between 2.61 and 13.20 mL/g (Table 3). At the optimum point, the SC of the IDF was 12.35 ± 0.19 mL/g, which was higher compared to that of peas and chickpeas (Tosh and Yada, 2010), but lower compared to that of garlic straw IDF and coconut IDF (Raghavarao et al., 2008; Huang et al., 2018). Different SC results can be associated with the extraction conditions and extraction method, which may lead to an increase or decrease of SC of the DF samples (López et al., 1996).

The GAC and AAIC values of the IDF and SDF samples, which were defined as the functional properties, are also provided in Table 3 and Table 4, respectively. The

GAC is used as a tool to determine the impact of DFs on dietary carbohydrates as an in vitro index, and GAC is considered one of the mechanisms that can explain the hypoglycemic action (Chau et al., 2003; Jia et al., 2020). The GAC of the IDF and SDF of the fenugreek seeds at the optimum point was 15.74 ± 0.14 mmol/g and 7.75 ± 0.18 mmol/g, respectively. Similarly, Wang et al. (2021) extracted DF from kiwifruit and according to their results, IDFs (2.04 ± 0.03 mmol/g) had more GAC than SDF samples. Begum and Deka (2019) extracted IDF from banana bracts and reported the GAC value as 7.95 ± 0.13 mmol/g when they applied the ultrasonic-assisted extraction method. They also reported that the sonication procedure might have helped to reduce the fibers' particle size, so that glucose molecules could be trapped, thereby reducing its release, which leads to higher GAC levels. Dong et al. (2020) reported relatively higher GAC results (162.40 mmol/g) for SDF from coffee peel. The results of GAC for DFs found in the literature showed that GAC values can vary due to the extraction method, extraction conditions and material source that was used for the extraction.

The α -amylase is an essential enzyme to form monosaccharides from oligosaccharides. Suppressing α -amylase can prevent the conversion of blood glucose, so that the increment of the blood glucose levels can be retarded (Hua

et al., 2020). Therefore, it is vital to provide inhibition of α -amylase to overcome type 2 diabetes (Im and Yoon, 2015). According to our results, the IDF and SDF had an α -amylase inhibitory effect, which IDF samples had higher AAIC levels compared to that of SDF samples (Table 3 and Table 4). Functional groups (such as hydroxyl groups), glucose content and molecular weight may affect AAIC values (Jiang et al., 2021). Begum and Deka (2019) reported similar results for the IDF of defatted banana bracts (AAIC value of 18.32 %). They also revealed that the samples' AAIC activity could be associated with the cellulose's crystalline structure. Therefore, DFs can absorb the enzyme and the fibrous network can trap the starch molecules. This mechanism leads to the starch and α -amylase contacting, so that the amount of glucose in the serum reduces (because of the inhibition of the starch molecules).

4. Conclusions

In this study, IDF and SDF were obtained using the ultrasonic-assisted extraction method from fenugreek seeds. The optimum extraction conditions ensuring the maximum TDF were determined by the response surface methodology. Also, physicochemical and functional properties of the IDF and SDF produced at different conditions were achieved. Results showed that DFs from fenugreek seeds can be obtained at shorter extraction times with high yields using the ultrasonic-assisted extraction process. The functional and physicochemical properties of the fenugreek seeds' DFs obtained at the optimum conditions showed high quality; therefore, it is evident that fenugreek seeds have a great potential as a DFs source. Moreover, these DFs that have different properties can be used in different food formulations to produce functional foods.

5. Acknowledgments

This study was financially supported by Tokat Gaziosmanpaşa University Scientific Research Projects Committee (Project No: 2020/125).

References

- Alfredo, V.-O., Gabriel, R.-R., Luis, C.-G., and David, B.-A. (2009). Physicochemical properties of a fibrous fraction from chia (*Salvia hispanica* L.). *LWT - Food Science and Technology*, 42(1):168–173.
- AOAC (2000). *Official methods of analysis of the AOAC (17th ed.)*. Association of Official Analytical Chemists, Rockville, MD, USA.
- Begum, Y. A. and Deka, S. C. (2019). Effect of processing on structural, thermal, and physicochemical properties of dietary fiber of culinary banana bracts. *Journal of Food Processing and Preservation*, 43(12).
- Chau, C.-F., Huang, Y.-L., and Lee, M.-H. (2003). In Vitro Hypoglycemic Effects of Different Insoluble Fiber-Rich Fractions Prepared from the Peel of Citrus *Sinensis* L. cv. Liucheng. *Journal of Agricultural and Food Chemistry*, 51(22):6623–6626.
- Cheung, Y.-C. and Wu, J.-Y. (2013). Kinetic models and process parameters for ultrasound-assisted extraction of water-soluble components and polysaccharides from a medicinal fungus. *Biochemical Engineering Journal*, 79:214–220.
- Chu, J., Zhao, H., Lu, Z., Lu, F., Bie, X., and Zhang, C. (2019). Improved physicochemical and functional properties of dietary fiber from millet bran fermented by *Bacillus natto*. *Food Chemistry*, 294:79–86.
- Ding, Q., Li, Z., Wu, W., Su, Y., Sun, N., Luo, L., Ma, H., and He, R. (2020). Physicochemical and functional properties of dietary fiber from *Nannochloropsis oceanica*: A comparison of alkaline and ultrasonic-assisted alkaline extractions. *LWT*, 133:110080.
- Ding, Q., Wu-Chen, R. A., Wu, Q., Jiang, H., Zhang, T., Luo, L., Ma, H., Ma, S., and He, R. (2019). Kinetics of ultrasound-assisted extraction of flavonoids and triterpenes and structure characterization of chinese northeast black bee propolis. *Chiang Mai Journal of Science*, 46(1):72–92.
- Dong, W., Wang, D., Hu, R., Long, Y., and Lv, L. (2020). Chemical composition, structural and functional properties of soluble dietary fiber obtained from coffee peel using different extraction methods. *Food Research International*, 136:109497.
- Du, X., Wang, L., Huang, X., Jing, H., Ye, X., Gao, W., Bai, X., and Wang, H. (2021). Effects of different extraction methods on structure and properties of soluble dietary fiber from defatted coconut flour. *LWT*, 143:111031.
- Hooda, S. and Jood, S. (2005). Organoleptic and nutritional evaluation of wheat biscuits supplemented with untreated and treated fenugreek flour. *Food Chemistry*, 90(3):427–435.
- Hua, M., Sun, Y., Shao, Z., Lu, J., Lu, Y., and Liu, Z. (2020). Functional soluble dietary fiber from ginseng residue: Polysaccharide characterization, structure, antioxidant, and enzyme inhibitory activity. *Journal of Food Biochemistry*, 44(12).
- Huang, L., Ding, X., Zhao, Y., Li, Y., and Ma, H. (2018). Modification of insoluble dietary fiber from garlic straw with ultrasonic treatment. *Journal of Food Processing and Preservation*, 42(1):e13399.
- Im, H. and Yoon, K. (2015). Production and characterisation of alcohol-insoluble dietary fibre as a potential source for functional carbohydrates produced by enzymatic depolymerisation of buckwheat hulls. *Czech Journal of Food Sciences*, 33(5):449–457.
- Jacometti, G. A., Mello, L. R., Nascimento, P. H., Sueiro, A. C., Yamashita, F., and Mali, S. (2015). The physicochemical properties of fibrous residues from the agro industry. *LWT - Food Science and Technology*, 62(1):138–143.
- Jia, F., Yang, S., Ma, Y., Gong, Z., Cui, W., Wang, Y., and Wang, W. (2020). Extraction optimization and constipation-relieving activity of dietary fiber from *Auricularia polytricha*. *Food Bioscience*, 33:100506.
- Jiang, G., Bai, X., Wu, Z., Li, S., Zhao, C., and Ramachandraiah, K. (2021). Modification of ginseng insoluble dietary fiber through alkaline hydrogen peroxide treatment and its impact on structure, physico-

- chemical and functional properties. *LWT*, 150:111956.
- Jiang, Y., Bai, X., Lang, S., Zhao, Y., Liu, C., and Yu, L. (2019). Optimization of ultrasonic-microwave assisted alkali extraction of arabinoxylan from the corn bran using response surface methodology. *International Journal of Biological Macromolecules*, 128:452–458.
- Khoja, K. K., Aslam, M. F., Sharp, P. A., and Latunde-Dada, G. O. (2021). In vitro bioaccessibility and bioavailability of iron from fenugreek, baobab and moringa. *Food Chemistry*, 335:127671.
- Krishnakumar, I., Ravi, A., Kumar, D., Kuttan, R., and Maliakel, B. (2012). An enhanced bioavailable formulation of curcumin using fenugreek-derived soluble dietary fibre. *Journal of Functional Foods*, 4(1):348–357.
- Liu, Y., Zhang, H., Yi, C., Quan, K., and Lin, B. (2021). Chemical composition, structure, physicochemical and functional properties of rice bran dietary fiber modified by cellulase treatment. *Food Chemistry*, 342:128352.
- López, G., Ros, G., Rincón, F., Periago, M. J., Martínez, M. C., and Ortuño, J. (1996). Relationship between physical and hydration properties of soluble and insoluble fiber of artichoke. *Journal of Agricultural and Food Chemistry*, 44(9):2773–2778.
- Ma, M.-m. and Mu, T.-h. (2016). Effects of extraction methods and particle size distribution on the structural, physicochemical, and functional properties of dietary fiber from deoiled cumin. *Food Chemistry*, 194:237–246.
- Madhava Naidu, M., Shyamala, B., Pura Naik, J., Sulochanamma, G., and Srinivas, P. (2011). Chemical composition and antioxidant activity of the husk and endosperm of fenugreek seeds. *LWT - Food Science and Technology*, 44(2):451–456.
- Meng, X., Liu, F., Xiao, Y., Cao, J., Wang, M., and Duan, X. (2019). Alterations in physicochemical and functional properties of buckwheat straw insoluble dietary fiber by alkaline hydrogen peroxide treatment. *Food Chemistry: X*, 3:100029.
- Miller, G. L. (1959). Use of dinitrosalicylic acid reagent for determination of reducing sugar. *Analytical Chemistry*, 31(3):426–428.
- Navarro-González, I., García-Valverde, V., García-Alonso, J., and Periago, M. J. (2011). Chemical profile, functional and antioxidant properties of tomato peel fiber. *Food Research International*, 44(5):1528–1535.
- Raghavarao, K. S. M. S., Raghavendra, S. N., and Rastogi, N. K. (2008). Potential of coconut dietary fiber. *Indian Coconut Journal*, 51:2–7.
- Riasat, M., Heidari, B., Pakniyat, H., and Jafari, A. A. (2018). Assessment of variability in secondary metabolites and expected response to genotype selection in fenugreek (*Trigonella* spp.). *Industrial Crops and Products*, 123:221–231.
- Sun, J., Zhang, Z., Xiao, F., Wei, Q., and Jing, Z. (2018). Ultrasound-assisted alkali extraction of insoluble dietary fiber from soybean residues. *IOP Conference Series: Materials Science and Engineering*, 392:052005.
- Tejada-Ortigoza, V., Garcia-Amezquita, L. E., Serna-Saldívar, S. O., and Welti-Chanes, J. (2016). Advances in the functional characterization and extraction processes of dietary fiber. *Food Engineering Reviews*, 8(3):251–271.
- Tosh, S. M. and Yada, S. (2010). Dietary fibres in pulse seeds and fractions: Characterization, functional attributes, and applications. *Food Research International*, 43(2):450–460.
- Wang, K., Li, M., Wang, Y., Liu, Z., and Ni, Y. (2021). Effects of extraction methods on the structural characteristics and functional properties of dietary fiber extracted from kiwifruit (*Actinidia deliciosa*). *Food Hydrocolloids*, 110:106162.
- Zhang, W., Zeng, G., Pan, Y., Chen, W., Huang, W., Chen, H., and Li, Y. (2017). Properties of soluble dietary fiber-polysaccharide from papaya peel obtained through alkaline or ultrasound-assisted alkaline extraction. *Carbohydrate Polymers*, 172:102–112.
- Zhang, Y., Liao, J., and Qi, J. (2020). Functional and structural properties of dietary fiber from citrus peel affected by the alkali combined with high-speed homogenization treatment. *LWT*, 128:109397.