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Research Article

Thermal stability and degradation kinetics of the phenolics of *Trigonella-foenum graecum* L. leaf extracts

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ABSTRACT

In this study, thermal stability and degradation kinetics of the phenolics of the aqueous fenugreek leaf extracts were determined. Thermal degradations of total phenolics, total flavonoids, antioxidant activity and total saponins of the fenugreek leaf extracts were examined at different pH values (3.0, 6.0 and 9.0) and different temperatures (60, 70, 80, 90 and 100°C) for time. Moreover, degradation kinetics of the total phenolics were explained by first-order reaction kinetics. Half-life values, free energy and activation energy of the extracts for total phenolic compounds were calculated. According to the results, the extracts showed better thermal stability at pH 3.0 than the other pH values at the selected temperatures concerning total phenolics, total flavonoids, antioxidant activity and total saponins. The degradation of the total phenolics, total flavonoids and antioxidant activity followed similar trends. The phenolic extract of the fenugreek leaves had high thermal stability. The extract had antioxidant activity despite applying eight hours of thermal treatment at 100°C. Kinetic constants (k) were 0.151-0.435 h^{-1}, 0.181-0.491 h^{-1} and 0.197-0.634 h^{-1} at pH 3.0, pH 6.0 and pH 9.0, respectively. Activation and free energy values for the degradation of fenugreek phenolics were calculated in the range of 26.02-29.97 kJ/mol and 109.31-120.07 kJ/mol, respectively. The half-life values of total phenolics treated at 60-100°C were 1.59-4.59 h, 1.41-3.83 h, and 1.09-3.52 h for pH 3.0, 6.0 and 9.0, respectively.

1. Introduction

Fenugreek (*Trigonella-foenum graecum* L.) is an herbaceous and medicinal plant mainly cultivated in India and North African countries [1]. The seeds of fenugreek are used as spices and medicine, and the leaves are mainly used as green leafy vegetables in the diet. The seeds of fenugreek are valorized as food and medicine. On the other hand, the leaves of the fenugreek have the potential to be a valuable product. The leaves have an important number of vitamins and minerals [1]. Fenugreek leaves have anti-hyperglycemic and antidiabetic effects [2]. Because of the beneficial health effects of the fenugreek leaves, they are mostly consumed as fresh vegetables. However, most of the fenugreek leaves have been discarded by farmers and the food industry. Fenugreek leaves may have the potential to obtain extracts having unique properties. Isleroglu and Turker [3] reported that fenugreek leaf extracts had high antioxidant capacity, flavonoid and total phenolic compounds. The extracts are worth to be used as natural antioxidants because of their high antioxidant activity. To use these extracts in food formulations, the thermal stability of the extracts should also be investigated.

The biomaterials, such as extracts having antioxidant properties due to their phenolic content are used in food formulations as natural preservatives, coloring agents and oxidation inhibitors. The food products are subjected to thermal treatments in the range of 50 to 150°C for the inactivation of the microorganisms and/or enzymes [4]. Hence, it is vital to determine the impact of the thermal treatments on the biomaterials' stability and activity that has the potential to be used in food formulations. Moreover, other parameters, such as thermal treatment time and the medium pH level, should be investigated to determine the activity loss of biomaterial [5]. The determination of the degradation and the changes in a bioactive compound at different temperatures needs considerable laboratory work. Thus, it is expensive and time-consuming for the manufacturers and food industry to assess the changes in the bioactive compounds' activity. Here, kinetic modelling comes forward as a useful tool to predict the consequent changes in the bioactive compounds' activity [6]. The basic kinetic information can be used to calculate of activation energies and reaction

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rates [4].

The stability of the phenolic compounds mainly depends on their source [7]. The temperature has a crucial effect on the stability of the phenolic extracts, and the amount of the phenolic compounds is mainly associated with the antioxidant activity of an extract [8]. Moreover, at different pH levels, phenolic compounds may have different thermal stabilities [9]. Among the thermal stability studies in the literature, only few studies can be listed which investigated the thermal stability of the phenolic extracts at different pH levels. Gonzalez-Ortega et al. [4] investigated the effects of heat treatment on the olive leaf phenolics at different temperatures (5-90°C) and different pH levels (2.0-6.0). In another study, Liu et al. [10] studied the effect of the different pH levels and the different temperatures on blueberry anthocyanins. They revealed that anthocyanins showed better stability at pH ≤ 3.0. Amendola et al. [11] applied thermal treatment at 121°C for 15 minutes at pH 3.0, 5.0, 7.0 and 9.0 to the grape marc phenolic extracts. To our knowledge, there is no study in the literature evaluating the thermal stability of the fenugreek leaf extracts obtained.

In this study, the thermal stability of the fenugreek leaf extracts obtained using the maceration technique was determined at different temperatures (60, 70, 80, 90 and 100°C) and pH values (3.0, 6.0 and 9.0) respecting time. Thermal degradations of total phenolics, total flavonoids, antioxidant activity and total saponins were identified. In addition, the degradation kinetics of the total phenolics of the aqueous fenugreek leaf extracts were explained by first-order reaction kinetics. Half-life values, free energy and activation energy values were calculated for total phenolic compounds.

2. Materials and Methods

2.1 Material

Fenugreek leaves were provided by a local farmer at harvest time in August 2021, and the plants were provided from Kayseri Province. Before the extraction processes, the fresh leaves were pulled out from the plant stems, and then the leaves were dried at room temperature for 72 hours; the moisture content of the leaves after drying was 8.42±0.22% (wet basis). After that, the dry leaves were crumbled by hand and the samples were stored in the dark at room temperature (25°C) for further analysis. To obtain fenugreek leaf extracts, dried and cleaned leaf samples were mixed with water at the leaf–water ratio of 10 g/L, and were agitated at 400 rpm for 120 minutes at 50°C. After that, the samples were centrifuged at 7000 rpm (Hettich 320 R, Germany) for five minutes and then were filtered using a coarse filter paper. For the thermal stability study and chemical analyses, the filtered samples were used.

2.2 Chemicals

Aluminum chloride ($AlCl_3$), sodium carbonate (Na_2CO_3), sodium acetate (CH_3COONa), potassium persulfate ($K_2S_2O_8$), sulfuric acid (H_2SO_4) and Folin-Ciocalteau reagent were obtained from Merck Chemicals (Germany). Sodium hydroxide (NaOH), DPPH, ABTS, gallic acid, vanillin and Trolox were purchased from Sigma-Aldrich Co. (Germany). Diosgenin was obtained from Cayman Chemical (USA), and quercetin was purchased from BLD Pharmatech Ltd. (China). Ethanol (96%) and sodium nitrite ($NaNO_2$) were obtained from Tekkim Chemicals (Turkey).

2.3 Thermal stability of the extracts

For the fenugreek leaf extracts obtained by maceration technique, a thermal stability study was conducted to determine the thermal degradation of total phenolic compounds (TPC), total flavonoids (TFL), antioxidant activity (AA) and total saponins (TSC). The TPC, TFL, AA (DPPH), AA (ABTS) and TSC values of the aqueous fenugreek leaf extracts were determined at pH 3.0, pH 6.0 and pH 9.0 at varying temperatures (60, 70, 80, 90 and 100°C). Moreover, kinetic parameters were also determined for the TPC degradation. For the TPC degradation, the remaining values were calculated using Equation 1. The initial pH of the samples was ~ 6.0 and different concentrations of HCl and NaOH were used to set the pH values of the samples to 3.0 and 9.0. One mL of the samples prepared at different pH levels were incubated in a water bath at different temperatures with concerning time. After incubation, the samples were cooled rapidly in ice water.

$$\text{Remaining TPC (\%)} = \frac{\text{TPC of the sample after heat treatment}}{\text{Initial TPC of the sample (before heating)}} \times 100 \quad (1)$$

First-order reaction kinetics was observed for the degradation of the TPC of the samples and Equation 2 was used for the evaluation of the experimental data.

$$A_t = A_0 \exp(-kt) \quad (2)$$

Here, A_t is defined as the TPC of the extract at the time of t, A_0 is the initial TPC of the extract and the k is the first-order constant ($hours^{-1}$). The temperature dependence of the constant k was related to the Arrhenius equation given with Equation 3.

$$k = k_0 \exp(-E_A/RT) \quad (3)$$

Where, k_0 is the Arrhenius constant, R is the universal gas constant (8.314 J/mol.K), T is the temperature (K) and the E_a is the activation energy (J/mol). E_a is calculated by the Arrhenius plots obtained using log (k) versus 1/T. The half-life values ($t_{1/2}$) of the samples for TPC are calculated

by predetermined degradation rate constants and (Equation 4).

$$t_{1/2} = \frac{\ln(2)}{k} \quad (4)$$

Free energy values of the degradation kinetics were also calculated using Equation 5, where h was the Planck constant ($6.6262x10^{-34}$ J.s) and K was the Boltzmann constant ($1.3806\ x10^{-34}$ J/K).

$$\Delta G = -RT.\ln\left(\frac{kh}{KT}\right) \quad (5)$$

2.4. Analysis

2.4.1. Total phenolic compounds

Folin-Ciocalteau method described by Singleton and Rossi [12] was used for the determination of the TPC of the samples. 250 µL of 1:1 (v/v) diluted (with ultrapure water) Folin-Ciocalteau reagent was mixed with 250 µL of the sample. 500 µL of Na_2CO_3 solution (210 g/L) and 4 mL of ultrapure water was mixed with this mixture. The samples were centrifuged at 1456 g (Hettich Eba 21, Germany) for 10 minutes after incubation at 25°C for 25 minutes. At 760 nm, the absorbance of the supernatants was measured (T80+, PG Instruments, United Kingdom). Gallic acid was used as a standard, and the TPC were presented as mg gallic acid/g dry sample.

2.4.2. Total flavonoid content

Spectrophotometric aluminum chloride method was used to determine the TFL of the fenugreek leaf extracts [13]. The diluted samples (1:10, 1 mL) were mixed with 4 mL of ultrapure water and 5% $NaNO_2$ (0.3 mL) in a 15 mL test tube. This mixture was incubated for five minutes at 25°C. Following that, 0.3 mL of 10% $AlCl_3$ was added to the mixture and incubated for six minutes. At the end of the incubation, 2 mL of 1 M NaOH solution was added, and the volume of the solution reached 10 mL with ultrapure water. The absorbance of the samples was read at 510 nm, and total flavonoid content (TPC) was expressed as mg quercetin/g dry sample.

2.4.3. Antioxidant activity

To determine the antioxidant activity of the fenugreek leaf extracts, ABTS and DPPH methods were used. For DPPH, a slightly modified version of the method reported by Pająk et al. [14] was performed. 50 µL of the sample was mixed with 1.95 ml of 0.1 mM DPPH solution. This mixture was incubated for 30 minutes in the dark at 25°C. The absorbance of the samples at 515 nm wavelength was determined. For ABTS-reducing antioxidant activity analysis, a slightly modified analysis procedure of Pająk et al. [14] was used. Firstly, ABTS-$K_2S_2O_8$ solution was prepared using 7 mM ABTS stock solution and 2.45 mM $K_2S_2O_8$ solution. These solutions were mixed in equal proportion, and incubated in the dark for 16 hours at room temperature. After incubation, 1 mL of this solution was mixed with 54 mL of 20 mM sodium acetate buffer solution (at pH 4.5). The absorbance value of this mixture was 0.700 at 734 nm wavelength. Finally, 150 µL of the extract was mixed with 2850 µL of the final solution, and the mixture was incubated in the dark at room temperature for 30 minutes. The antioxidant activity of the fenugreek leaf extracts regarding of DPPH and ABTS methods was expressed as mg Trolox/g dry sample.

2.4.4. Total saponin content

The method of Akbari et al. [15] with slight modifications was used to determine the TSC of the extracts. 0.2 mL of the fenugreek leaf extract was mixed with 0.35 mL of 0.8% vanillin (prepared in 96% ethanol) and 0.8 mL of ultrapure water. 1.25 ml of 72% H_2SO_4 (v/v) was added to this mixture and gently mixed. The samples were incubated in a water bath adjusted to 60°C for 10 minutes. After incubation, all samples were taken rapidly into the ice bath. The absorbance values of the samples were measured at 544 nm. TSC values of the fenugreek leaf extracts were calculated as mg diosgenin/g dry sample.

2.5 Statistical analysis

One-way ANOVA with Duncan descriptive statistics for the thermal degradation data were carried out using the SPSS 21.0 (IBM, USA) package program. All experiments were performed with two replicates and two parallels. The evaluation of the kinetic data was made with MS Office Excel 2019. The mean absolute percentage deviation values between the experimental and predicted data (P%) were calculated to provide information about the validation of the kinetic models [16] and P% values were calculated using Equation 6. The model with a P% value lower than 10% was considered acceptable.

$$P\% = \frac{100}{N}\sum_{i}^{N}\frac{\left|C_{ei}-C_{pi}\right|}{C_{ei}} \quad (6)$$

Here, C_{ei} is defined as the experimental remaining TPC (%), C_{pi} is defined as the predicted remaining TPC (%) from the first-order reaction kinetic model, and N is the number of experimental data points.

3. Results and Discussion

The TFL, AA and TSC values of the aqueous fenugreek leaf extracts after thermal treatment at different pH values are shown in Tables 1, 2 and 3. The results showed that TFL and AA values had similar degradation trends. Chaaban et al. [17] studied the effects of heat processing on the thermal stability and antioxidant activity of different

flavonoids. They reported that the antioxidant activity values were decreased by the degradation of the flavonoids after heat treatment. Similar observations were found in our study of TFL content. The thermal degradation mechanism of the flavonoids can be explained by hydrolysis of the glycosidic linkages in pigments which leads to the formation of unstable molecules [18]. Fenugreek leaf extracts showed AA even at the 8th hour of heat treatment of 100°C, and these results showed the high thermal stability of the fenugreek leaf extracts. It was also observed that under acidic conditions (pH 3.0) (Table 1), the extracts' thermal stability was higher than that of alkaline conditions (pH 9.0) (Table 3). TSC of the fenugreek had lower thermal stability than fenugreek TFL. Nafiunisa et al. [19] reported that the extraction yield of the tea saponins had its highest level at 50°C of extraction temperature, but the degradation of the tea saponins was observed at 60°C. Heng et al. [20] also reported that DDMP saponin from peas became unstable in water at temperatures higher than 30°C.

To define the temperature dependency of fenugreek extracts, a kinetic study was evaluated for the TPC values of the extracts. The remaining TPC of the fenugreek extracts after heat treatments at different pH values was illustrated in Figure 1. When all the results were examined, it was observed that the samples incubated at pH 3.0 had the highest remaining TPC values. On the contrary, fenugreek extracts incubated at pH 9.0 showed the lowest stability at all temperatures (Figure 1). Regarding to the degradation constants, the highest half-life values were observed for pH 3.0 samples (1.59-4.59 hours) and the lowest values were obtained for pH 9.0 samples (1.09-3.52 hours) (Table 4). All samples with different pH values showed a decreasing trend of TPC when the temperature was increased, and these results can be correlated with the TFL and AA values (Table 1, 2, 3). All of the degradation kinetics were explained by the first-order kinetics for TPC (Figure 1). Singhal et al. [21] reported similar results for the thermal degradation kinetics of *Agaricus bisporus*. The researchers reported that TPC degradation could be anticipated using a first-order kinetics model.

Our samples had high thermal stability than other analyzed vegetables in the literature. Nambi et al. [22] studied the thermal stability of four different vegetables (beetroot, green pea, eggplant and green pepper) at 70-90°C. All different vegetables' TPC values were reduced by increasing temperature, and they all showed first-order reaction kinetics which finding was consistent with our study. However, the samples' phenolics were highly sensitive, and 50-52% of the TPC of the vegetables was reduced at 70°C for three minutes of heat treatment. Ismail et al. [23] reported similar results for green peas and pepper, 75-80% of TPC was lost when 15 minutes of heat treatment was applied at 90°C. This rapid TPC loss was associated with the breakdown of the phenolic compounds or dissolution of polyphenols [24]. In this study, fenugreek leaf extracts maintained their TPC about 60% for 1-hour application of 100°C.

Table 1. TFL, DPPH, ABTS and TSC values at pH 3.0.

T (°C)	t (h)	TFL (mg quercetin/g dry sample)	AA (DPPH) (mg Trolox/g dry sample)	AA (ABTS) (mg Trolox/g dry sample)	TSC (mg diosgenin/g dry sample)
60	0	12.90 (±0.04)^{a}	1.25 (±0.01)^{a}	9.89 (±0.05)^{a}	19.01 (±0.13)^{a}
	1	11.62 (±0.04)^{b}	1.14 (±0.01)^{b}	9.03 (±0.06)^{b}	15.45 (±0.12)^{b}
	2	9.57 (±0.05)^{e}	0.94 (±0.01)^{e}	7.27 (±0.04)^{e}	12.40 (±0.08)^{f}
	4	7.83 (±0.07)^{h}	0.73 (±0.01)^{j}	5.75 (±0.05)^{j}	8.24 (±0.12)^{k}
	6	5.72 (±0.08)^{l}	0.56 (±0.01)^{n}	4.11 (±0.05)^{n}	5.14 (±0.11)^{n}
	8	3.92 (±0.04)^{p}	0.41 (±0.01)^{q}	3.22 (±0.08)^{p}	3.31 (±0.10)^{p}
	10	2.64 (±0.03)^{s}	0.29 (±0.00)^{r}	1.99 (±0.06)^{r}	1.90 (±0.11)^{t}
70	0	12.90 (±0.04)^{a}	1.25 (±0.01)^{a}	9.89 (±0.05)^{a}	19.01 (±0.13)^{a}
	1	10.91 (±0.04)^{c}	1.06 (±0.01)^{c}	8.44 (±0.08)^{c}	14.53 (±0.05)^{c}
	2	8.54 (±0.06)^{g}	0.82 (±0.00)^{h}	6.57 (±0.07)^{g}	11.61 (±0.05)^{g}
	4	5.98 (±0.05)^{k}	0.59 (±0.01)^{m}	4.48 (±0.06)^{m}	7.71 (±0.13)^{l}
	6	3.12 (±0.03)^{q}	0.30 (±0.00)^{r}	2.60 (±0.06)^{q}	4.41 (±0.06)^{o}
	8	2.18 (±0.06)^{u}	0.21 (±0.01)^{u}	1.55 (±0.04)^{t}	2.72 (±0.09)^{r}
	10	1.23 (±0.06)^{x}	0.11 (±0.01)^{w}	0.91 (±0.08)^{w}	1.74 (±0.12)^{u}
80	0	12.90 (±0.04)^{a}	1.25 (±0.01)^{a}	9.89 (±0.05)^{a}	19.01 (±0.13)^{a}
	1	10.59 (±0.05)^{d}	1.00 (±0.01)^{d}	7.82 (±0.06)^{d}	13.83 (±0.09)^{d}
	2	7.83 (±0.03)^{h}	0.76 (±0.01)^{i}	6.01 (±0.04)^{i}	10.57 (±0.08)^{h}
	4	5.42 (±0.06)^{m}	0.51 (±0.01)^{o}	4.06 (±0.08)^{n}	5.90 (±0.03)^{m}
	6	2.51 (±0.04)^{t}	0.26 (±0.01)^{s}	1.76 (±0.05)^{s}	3.35 (±0.13)^{p}
	8	1.73 (±0.05)^{v}	0.15 (±0.00)^{v}	1.34 (±0.06)^{u}	2.21 (±0.08)^{s}
	10	1.08 (±0.04)^{y}	0.12 (±0.00)^{w}	0.68 (±0.07)^{x}	1.44 (±0.06)^{v}
90	0	12.90 (±0.04)^{a}	1.25 (±0.01)^{a}	9.89 (±0.05)^{a}	19.01 (±0.13)^{a}
	1	9.12 (±0.05)^{f}	0.90 (±0.01)^{f}	6.93 (±0.06)^{f}	13.02 (±0.06)^{e}
	2	6.83 (±0.07)^{i}	0.68 (±0.01)^{k}	5.10 (±0.06)^{k}	9.52 (±0.08)^{i}
	3	4.67 (±0.03)^{n}	0.47 (±0.01)^{p}	3.62 (±0.07)^{o}	5.11 (±0.09)^{n}
	4	2.79 (±0.03)^{r}	0.24 (±0.02)^{t}	1.97 (±0.09)^{r}	2.85 (±0.09)^{r}
	6	1.57 (±0.03)^{w}	0.15 (±0.00)^{v}	1.14 (±0.05)^{v}	1.46 (±0.11)^{f}
	8	0.81 (±0.05)$^{\alpha}$	0.09 (±0.01)^{x}	0.65 (±0.03)^{x}	0.89 (±0.09)^{x}
100	0	12.90 (±0.04)^{a}	1.25 (±0.01)^{a}	9.89 (±0.05)^{a}	19.01 (±0.13)^{a}
	1	8.57 (±0.06)^{g}	0.85 (±0.01)^{g}	6.40 (±0.07)^{h}	12.39 (±0.08)^{f}
	2	6.26 (±0.06)^{j}	0.61 (±0.01)^{l}	4.66 (±0.07)^{l}	8.76 (±0.10)^{j}
	3	4.25 (±0.05)^{o}	0.41 (±0.01)^{q}	3.23 (±0.05)^{p}	4.43 (±0.06)^{o}
	4	2.55 (±0.03)^{t}	0.23 (±0.01)^{t}	1.92 (±0.06)^{r}	2.29 (±0.05)^{s}
	6	0.93 (±0.03)^{z}	0.09 (±0.00)^{x}	0.72 (±0.05)^{x}	1.18 (±0.13)^{w}
	8	0.40 (±0.03)$^{\beta}$	0.04 (±0.00)^{y}	0.30 (±0.03)^{y}	0.71 (±0.13)^{y}

T: Temperature, t: Time, TFL: Total Flavonoid Content AA: Antioxidant Activity, TSC: Total Saponin Content

$^{a-\beta}$Means with uncommon superscripts in a colon are statistically significant ($p<0.05$). After alphabet (a-z), α and β were used as subsequent letters.

Table 2. TFL, DPPH, ABTS and TSC at pH 6.0.

T (°C)	t (h)	TFL (mg quercetin/g dry sample)	AA (DPPH) (mg Trolox/g dry sample)	AA (ABTS) (mg Trolox/g dry sample)	TSC (mg diosgenin/g dry sample)
	0	13.06 (±0.04)^{a}	1.28 (±0.00)^{a}	10.01 (±0.05)^{a}	19.94 (±0.14)^{a}
	1	11.48 (±0.05)^{b}	1.09 (±0.01)^{b}	8.53 (±0.04)^{b}	14.84 (±0.08)^{b}
	2	9.16 (±0.10)^{e}	0.88 (±0.01)^{e}	6.81 (±0.04)^{e}	11.70 (±0.11)^{f}
60	4	7.14 (±0.07)^{i}	0.70 (±0.01)^{h}	5.19 (±0.03)^{h}	7.80 (±0.15)^{k}
	6	4.96 (±0.05)^{m}	0.47 (±0.00)^{l}	3.63 (±0.07)^{j}	4.37 (±0.13)^{n}
	8	3.38 (±0.03)^{q}	0.30 (±0.00)^{n}	2.62 (±0.06)^{m}	2.57 (±0.08)^{s}
	10	2.06 (±0.03)^{u}	0.21 (±0.00)^{p}	1.51 (±0.06)^{qr}	1.54 (±0.12)^{v}
	0	13.06 (±0.04)^{a}	1.28 (±0.00)^{a}	10.01 (±0.05)^{a}	19.94 (±0.14)^{a}
	1	10.29 (±0.11)^{c}	1.03 (±0.01)^{c}	7.82 (±0.07)^{c}	13.84 (±0.06)^{c}
	2	8.10 (±0.04)^{g}	0.76 (±0.00)^{g}	5.98 (±0.04)^{f}	10.65 (±0.12)^{g}
70	4	5.10 (±0.05)^{l}	0.47 (±0.01)^{l}	3.65 (±0.08)^{j}	6.64 (±0.11)^{l}
	6	2.78 (±0.03)^{s}	0.24 (±0.01)^{o}	2.00 (±0.05)^{o}	3.80 (±0.08)^{q}
	8	2.10 (±0.05)^{tu}	0.17 (±0.01)^{q}	1.36 (±0.07)^{s}	2.20 (±0.07)^{t}
	10	1.06 (±0.04)^{x}	0.11 (±0.01)^{r}	0.86 (±0.06)^{t}	1.46 (±0.09)^{v}
	0	13.06 (±0.04)^{a}	1.28 (±0.00)^{a}	10.01 (±0.05)^{a}	19.94 (±0.14)^{a}
	1	9.68 (±0.06)^{d}	0.93 (±0.01)^{d}	7.23 (±0.05)^{d}	13.49 (±0.08)^{d}
	2	6.83 (±0.03)^{j}	0.67 (±0.01)^{i}	5.37 (±0.05)^{g}	10.25 (±0.10)^{h}
80	4	4.66 (±0.04)^{o}	0.44 (±0.01)^{l}	3.42 (±0.07)^{k}	5.38 (±0.18)^{m}
	6	2.15 (±0.06)^{t}	0.20 (±0.02)^{pq}	1.75 (±0.07)^{p}	2.97 (±0.06)^{r}
	8	1.22 (±0.05)^{w}	0.12 (±0.01)^{r}	0.90 (±0.06)^{t}	1.50 (±0.06)^{v}
	10	0.66 (±0.05)^{z}	0.06 (±0.01)st	0.46 (±0.06)^{w}	0.81 (±0.09)^{x}
	0	13.06 (±0.04)^{a}	1.28 (±0.00)^{a}	10.01 (±0.05)^{a}	19.94 (±0.14)^{a}
	1	8.56 (±0.05)^{f}	0.85 (±0.01)^{f}	6.82 (±0.05)^{e}	12.03 (±0.08)^{e}
	2	5.82 (±0.05)^{k}	0.61 (±0.01)^{j}	4.52 (±0.05)^{i}	8.42 (±0.08)^{j}
90	3	4.31 (±0.09)^{p}	0.44 (±0.01)^{l}	3.26 (±0.05)^{l}	4.19 (±0.12)^{o}
	4	2.08 (±0.05)^{tu}	0.19 (±0.03)^{pq}	1.57 (±0.05)^{q}	2.31 (±0.08)^{t}
	6	0.92 (±0.03)^{y}	0.10 (±0.00)^{r}	0.70 (±0.04)^{u}	1.02 (±0.08)^{w}
	8	0.49 (±0.03)$^{\alpha}$	0.05 (±0.01)st	0.38 (±0.03)^{x}	0.65 (±0.10)^{y}
	0	13.06 (±0.04)^{a}	1.28 (±0.00)^{a}	10.01 (±0.05)^{a}	19.94 (±0.14)^{a}
	1	7.66 (±0.01)^{h}	0.83 (±0.05)^{f}	5.90 (±0.07)^{f}	12.10 (±0.08)^{e}
	2	4.84 (±0.05)^{n}	0.58 (±0.07)^{k}	3.62 (±0.06)^{j}	8.03 (±0.05)^{j}
100	3	3.10 (±0.05)^{r}	0.41 (±0.08)^{m}	2.09 (±0.05)^{n}	3.98 (±0.12)^{p}
	4	1.79 (±0.03)^{v}	0.19 (±0.01)^{pq}	1.47 (±0.16)^{r}	1.84 (±0.08)^{u}
	6	0.66 (±0.04)^{z}	0.07 (±0.00)^{s}	0.57 (±0.03)^{v}	0.79 (±0.11)^{xy}
	8	0.21 (±0.03)$^{\beta}$	0.03 (±0.01)^{t}	0.19 (±0.03)^{y}	0.36 (±0.05)^{z}

T: Temperature, t: Time, TFL: Total Flavonoid Content AA: Antioxidant Activity, TSC: Total Saponin Content

$^{a\text{-}\beta}$Means with uncommon superscripts in a colon are statistically significant ($p<0.05$). After alphabet (a-z), α and β were used as subsequent letters.

Table 3. TFL, DPPH, ABTS and TSC values at pH 9.0.

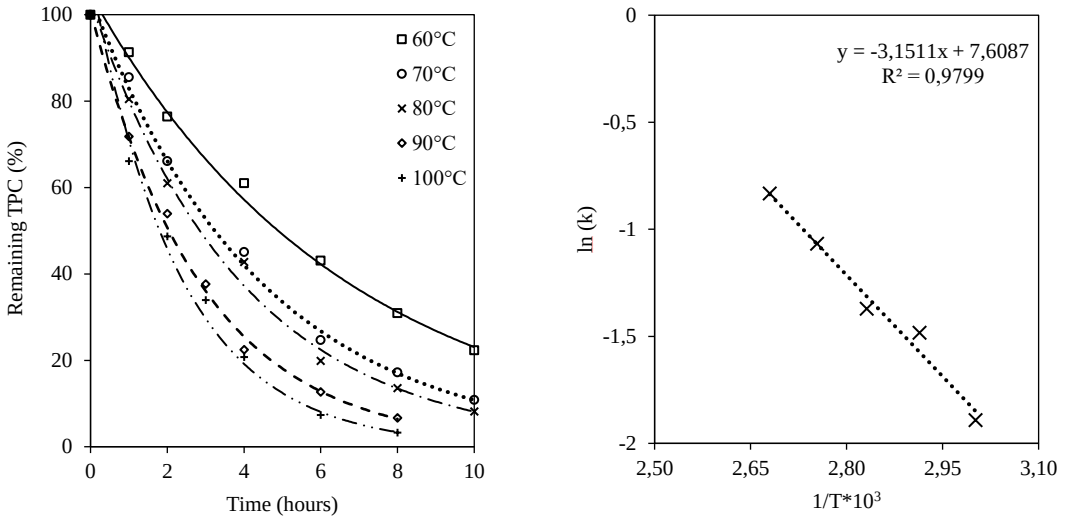
T (°C)	t (h)	TFL (mg quercetin/g dry sample)	AA (DPPH) (mg Trolox/g dry sample)	AA (ABTS) (mg Trolox/g dry sample)	TSC (mg diosgenin/g dry sample)
	0	12.79 (±0.04)^{a}	1.24 (±0.01)^{a}	9.83 (±0.07)^{a}	18.64 (±0.12)^{a}
	1	11.39 (±0.03)^{b}	1.06 (±0.00)^{b}	8.28 (±0.05)^{b}	14.37 (±0.08)^{b}
	2	9.03 (±0.07)^{e}	0.88 (±0.01)^{e}	6.60 (±0.04)^{f}	11.30 (±0.09)^{f}
60	4	6.52 (±0.04)^{j}	0.61 (±0.01)^{j}	4.92 (±0.05)^{j}	7.00 (±0.08)^{j}
	6	3.81 (±0.05)^{o}	0.35 (±0.01)^{k}	2.81 (±0.05)^{o}	4.05 (±0.14)^{m}
	8	2.83 (±0.07)^{q}	0.26 (±0.01)^{mn}	1.98 (±0.05)^{q}	2.20 (±0.07)^{q}
	10	1.78 (±0.04)^{t}	0.19 (±0.00)^{o}	1.34 (±0.05)^{t}	1.18 (±0.09)^{s}
	0	12.79 (±0.04)^{a}	1.24 (±0.01)^{a}	9.83 (±0.07)^{a}	18.64 (±0.12)^{a}
	1	10.28 (±0.06)^{c}	0.97 (±0.01)^{d}	7.65 (±0.04)^{c}	13.21 (±0.11)^{c}
	2	7.56 (±0.04)^{g}	0.71 (±0.00)^{h}	5.73 (±0.06)^{g}	9.86 (±0.07)^{g}
70	4	4.95 (±0.04)^{l}	0.47 (±0.01)^{k}	3.67 (±0.04)^{l}	5.48 (±0.08)^{k}
	6	2.39 (±0.04)^{r}	0.24 (±0.01)^{p}	1.75 (±0.06)^{r}	2.58 (±0.08)^{p}
	8	1.38 (±0.03)^{w}	0.12 (±0.01)^{r}	1.12 (±0.06)^{o}	1.78 (±0.10)^{r}
	10	0.80 (±0.04)^{z}	0.07 (±0.00)^{s}	0.57 (±0.03)^{x}	1.17 (±0.08)^{s}
	0	12.79 (±0.04)^{a}	1.24 (±0.01)^{a}	9.83 (±0.07)^{a}	18.64 (±0.12)^{a}
	1	9.55 (±0.03)^{d}	0.91 (±0.01)^{d}	7.39 (±0.04)^{d}	12.97 (±0.10)^{d}
	2	6.64 (±0.03)^{i}	0.65 (±0.01)^{h}	5.09 (±0.06)^{i}	9.61 (±0.11)^{h}
80	4	3.95 (±0.04)^{n}	0.35 (±0.01)^{k}	3.03 (±0.05)^{n}	4.88 (±0.14)^{l}
	6	1.43 (±0.05)^{v}	0.15 (±0.01)^{p}	1.42 (±0.04)^{s}	2.62 (±0.11)^{p}
	8	0.91 (±0.05)^{y}	0.09 (±0.01)^{r}	0.73 (±0.04)^{w}	1.13 (±0.06)^{s}
	10	0.44 (±0.02)$^{\alpha}$	0.04 (±0.01)^{s}	0.36 (±0.03)^{y}	0.51 (±0.06)^{u}
	0	12.79 (±0.04)^{a}	1.24 (±0.01)^{a}	9.83 (±0.07)^{a}	18.64 (±0.12)^{a}
	1	8.84 (±0.02)^{f}	0.87 (±0.00)^{e}	6.93 (±0.05)^{e}	11.50 (±0.14)^{e}
	2	5.25 (±0.03)^{k}	0.47 (±0.01)^{j}	4.02 (±0.05)^{k}	7.70 (±0.08)^{i}
90	3	3.10 (±0.03)^{p}	0.29 (±0.01)^{l}	2.40 (±0.05)^{p}	3.31 (±0.12)^{n}
	4	1.66 (±0.05)^{u}	0.17 (±0.01)^{op}	1.28 (±0.04)^{t}	1.85 (±0.10)^{r}
	6	0.77 (±0.02)^{z}	0.07 (±0.01)^{r}	0.70 (±0.07)^{w}	0.78 (±0.10)^{t}
	8	0.25 (±0.03)$^{\beta}$	0.03 (±0.01)^{s}	0.21 (±03.03)^{z}	0.40 (±0.11)^{u}
	0	12.79 (±0.04)^{a}	1.24 (±0.01)^{a}	9.83 (±0.07)^{a}	18.64 (±0.12)^{a}
	1	7.38 (±0.03)^{h}	0.84 (±0.07)^{f}	5.52 (±0.07)^{h}	11.29 (±0.06)^{f}
	2	4.08 (±0.03)^{m}	0.45 (±0.03)^{j}	3.28 (±0.06)^{m}	6.99 (±0.11)^{j}
100	3	2.22 (±0.03)^{s}	0.27 (±0.04)^{lm}	1.78 (±0.04)^{r}	2.96 (±0.05)^{o}
	4	1.09 (±0.03)^{x}	0.16 (±0.02)^{p}	0.93 (±0.05)^{v}	1.40 (±0.08)^{s}
	6	0.28 (±0.02)$^{\beta}$	0.03 (±0.00)st	0.23 (±0.03)^{z}	0.48 (±0.07)^{u}
	8	0.07 (±0.02)$^{\gamma}$	0.01 (±0.00)^{t}	0.06 (±0.02)$^{\alpha}$	0.10 (±0.05)^{v}

T: Temperature, t: Time, TFL: Total Flavonoid Content, AA: Antioxidant Activity, TSC: Total Saponin Content

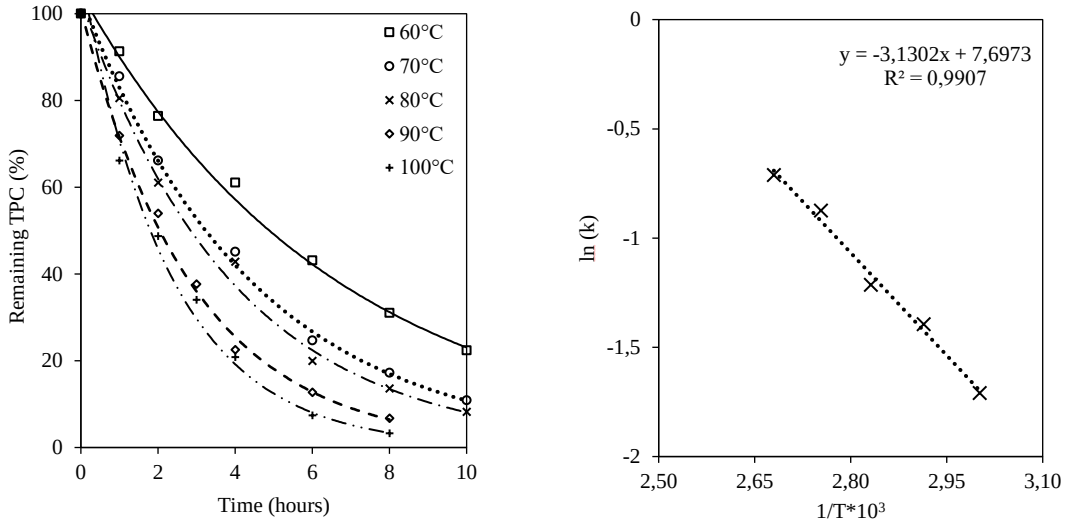
$^{a\text{-}\gamma}$Means with uncommon superscripts in a colon are statistically significant ($p<0.05$). After alphabet (a-z), α, β and γ were used as subsequent letters.

The difference in the thermal stability of the extracts obtained from different sources may vary because of the other components extracted with phenolics, such as gums, and these components may improve the thermal stability of the phenolics [25].

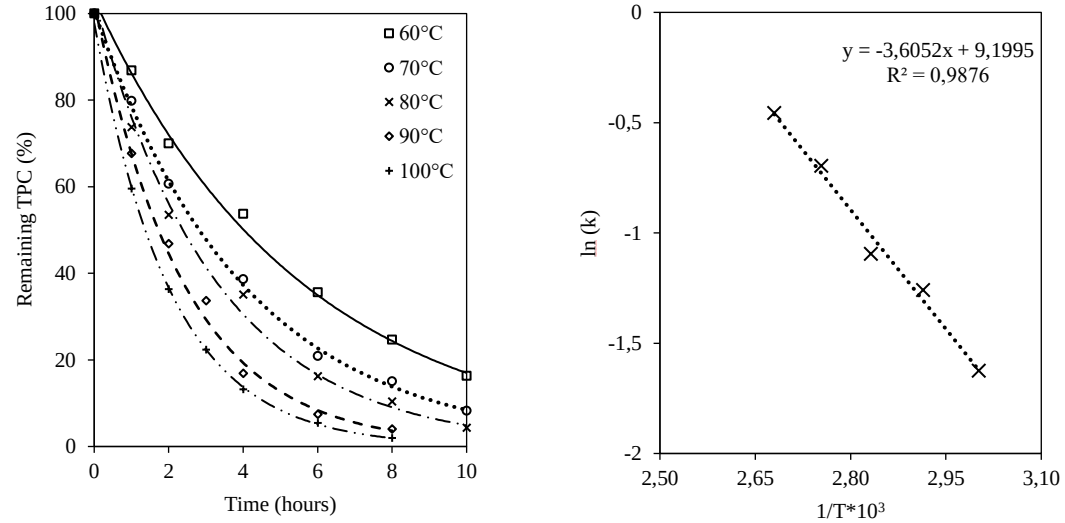
Table 4 also shows the E_a and ΔG values of the phenolic extracts. E_a can be defined as the energy required to activate a reaction. The higher values of E_a can be related to the smaller values of the change in temperature required to degrade phenolic compounds [26].



(a)



(b)



(c)

Figure 1. Thermal degradation and Arrhenius plots at (a) pH 3.0, (b) pH 6.0 and (c) pH 9.0

E_a values of the fenugreek leaf extracts at different pH values were determined as 26.20, 26.02, and 29.97 kJ/mol for pH 3.0, 6.0 and 9.0, respectively. pH 3.0 and pH 6.0 samples showed quite similar E_a values, but pH 9.0 samples had higher E_a values which suggests that pH 9.0 samples are more heat-labile.

Table 4. Kinetic constants (k, h^{-1}), R^2, P%, half-life values, free energy and activation energy of the extracts

pH	T (°C)	k (h^{-1})	R^2	P%	$t_{1/2}$ (hours)	ΔG (kJ/mol)	E_a (kJ/mol)
3.0	60	0.151 (±0.004)^{j}	0.9935	2.53	4.59 (±0.11)^{a}	109.82 (±0.07)^{k}	26.20 (±0.24)^{a}
	70	0.227 (±0.003)^{h}	0.9952	3.46	3.05 (±0.03)^{d}	112.03 (±0.03)^{i}	
	80	0.254 (±0.006)^{g}	0.9941	4.89	2.73 (±0.07)^{e}	115.05 (±0.07)^{g}	
	90	0.344 (±0.006)^{d}	0.9967	4.75	2.01 (±0.04)^{h}	117.48 (±0.05)^{d}	
	100	0.435 (±0.015)^{c}	0.9922	7.57	1.59 (±0.05)^{i}	120.07 (±0.11)^{a}	
6.0	60	0.181 (±0.003)^{i}	0.9962	2.96	3.83 (±0.06)^{b}	109.31 (±0.04)^{m}	26.02 (±0.88)^{a}
	70	0.248 (±0.007)^{g}	0.9988	4.27	2.79 (±0.08)^{e}	111.78 (±0.08)^{j}	
	80	0.297 (±0.006)^{e}	0.9961	9.65	2.33 (±0.05)^{g}	114.59 (±0.06)^{h}	
	90	0.417 (±0.010)^{c}	0.9952	9.19	1.66 (±0.04)^{i}	116.90 (±0.07)^{e}	
	100	0.491 (±0.010)^{b}	0.9996	1.94	1.41 (±0.03)^{j}	119.70 (±0.06)^{b}	
9.0	60	0.197 (±0.002)^{i}	0.9919	5.36	3.52 (±0.03)^{c}	109.47 (±0.02)^{l}	29.97 (±1.37)^{b}
	70	0.284 (±0.009)^{f}	0.9968	4.04	2.44 (±0.08)^{f}	111.72 (±0.09)^{j}	
	80	0.335 (±0.007)^{d}	0.9979	3.21	2.07 (±0.05)^{h}	114.52 (±0.07)^{h}	
	90	0.499 (±0.006)^{b}	0.9924	6.02	1.39 (±0.02)^{j}	116.58 (±0.04)^{f}	
	100	0.634 (±0.045)^{a}	0.9949	7.52	1.09 (±0.07)^{k}	119.06 (±0.21)^{c}	

$^{a-m}$ Means with uncommon superscripts within a column are significantly different ($p<0.05$).

Oncea and Drăghici [27] reported similar results for the Romanian red onion extracts, and they revealed that alkaline conditions significantly lowered the stability of the phenolic extracts. On the other hand, acidic conditions had a positive effect on the thermal stability of the fenugreek leaf extracts. This phenomenon can be explained by the interaction of the co-pigments which can ensure higher extract stability [28]. Nambi et al. [20] reported 17.24 to 25.48 kJ/mol activation energy for different kinds of vegetables. ΔG is an important criterion to determine the type of reaction if it is spontaneous or not. There were smaller differences between the samples incubated at different pH levels, which indicated that there was an increase in the total energy of the system. The ΔG values of all different samples were positive, demonstrating that the degradation reactions of different samples were not spontaneous [29]. Zahir et al. [30] and Nambi et al. [20] have found consistent results for different kinds of vegetables' TPC extracts. As shown in Table 4, ΔG values increased with increasing temperature, which is consistent with the study reported by Zahir et al. [30].

4. Conclusions

In this study, aqueous fenugreek leaf extracts were subjected to the thermal stability study. The results showed that fenugreek leaf extracts had high heat tolerance at long incubation times, and the extracts had antioxidant activity even at higher incubation times at 90 and 100°C. The resistance of the fenugreek leaf phenolics to the high temperatures at long incubation times was higher than that of the phenolics obtained from different sources such as green pea and green pepper. The fenugreek leaf extract was more stable at pH 3.0 and was more labile at pH 9.0. The heat-tolerant nature of the aqueous fenugreek leaf extract having high phenolic compounds and antioxidant activity has the potential to be used in different food formulations. The half-life values of 1.09-4.59 hours also showed that the fenugreek leaf phenolics could be subjected to thermal treatments applied to food products with minor activity losses. Our data can be helpful for the determination of the degradation levels of bioactive compounds found in the fenugreek leaf extracts for an application of the thermal process.

Declaration

The authors declared no potential conflicts of interest with respect to the research, authorship, and/or publication of this article. The authors also declared that this article is original, was prepared in accordance with international publication and research ethics, and ethical committee permission or any special permission is not required.

Author Contributions

H. Isleroglu contributed to developing of the methodology, conducting the study, evaluating the results, and writing the article. I. Turker performed the analysis, interpreted the results, and contributed to writing the article.

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