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The estimation of kinetic parameters of the solid-liquid extraction process of the lavender flower (*Lavandula x hybrida* L.)

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ABSTRACT

The use of medicinal plants has been well documented for the past several decades. Plants from the *Lavender* genus are used for culinary purposes, but also as a part of herbal medicine, owing its benefits to essential oils and bioactive compounds. A detailed description of the extraction process dynamics is required to obtain the highest possible amount of bioactive compounds from lavender. In this study, a solid-liquid extraction of lavender flowers, using water as solvent, was performed. Total polyphenols, dry matter, conductivity, and total dissolved solids were determined in regular time intervals and experimental data was further used for mathematical modelling. Peleg's, Page's, and the logarithmic model were applied in order to describe the kinetics of bioactive compounds in lavender during solid-liquid extraction. The initial extraction rate and extraction capacity were determined for three different extraction temperatures; $T = 40$, 60, and 80 °C. The obtained results indicated that the initial extraction rate and the extraction capacity were the highest at $T = 80$ °C. The adequacy of the applied mathematical models was estimated based on R^2 and SRMSE values, which indicated that the most adequate model for solid-liquid extraction of lavender was the Peleg's model ($R^2 = 0.996$, for total polyphenols at $T = 40$ °C and $R^2 = 0.975$ for dry matter at $T = 60$ °C, respectively).

Introduction

It is well-known that wild medicinal plants, in their natural or processed form, contain a complex mixture of bioactive compounds, covering a number of demands necessary for human health (Bakkali et al., 2008). Among them, *Lavandula* species are the most used aromatic and medicinal plants in pharmaceuticals, food and flavour industries, cosmetics, perfumery, and aromatherapy (Aprotosoaie et al., 2017). Lavender essential oils have been found to exert significant clinical effects on the central nervous system (anxiolytic, sedative, anticonvulsant, analgesic, local anaesthetic activity), and also showed antioxidant, antimicrobial, anti-inflammatory, spasmolytic, and carminative properties (Hassiotis et al., 2010). Due to its high economic value, *Lavender* genus is extensively cultivated in many countries, and a large amount of waste is produced after essential oil extraction by distillation. However, these by-products are a source of polyphenolic compounds which have a wide range of physiological properties, such as antiallergenic, antiatherogenic, anti-inflammatory, antimicrobial, antithrombotic, cardioprotective, and vasodilatory (Shan et al., 2005; Torras-Claveira et al., 2007). Many of these properties are due to antioxidant activity which can be determined by different mechanisms such as free radical scavenging, electron or hydrogen atom donation, or metal cation chelation (Balasundram et al., 2006).

Efficient extraction techniques are required for the isolation and identification of polyphenolic compounds (Nkhili et al., 2009; Bart, 2011; Mustafa and Turner, 2011; Petigny et al., 2013; Rój, 2014). Solid-liquid extraction under controlled experimental conditions is important for the reproducibility of the products and the

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preservation of their bioactivity (Tsibranska and Tylkowski, 2016). In order to optimise the extraction conditions, different mathematical models are often used for the description of solid-liquid extraction processes. Kinetic modelling is of great importance for understanding complex diffusion, mass transfer, and thermodynamic parameters affecting extraction (Jurinjak Tušek et al., 2016). The most commonly used empirical model is the Peleg's model for the description of moisture sorption curves (Peleg, 1988), adapted for extraction processes (Bucić-Kojić et al., 2007; Jokić et al., 2010; Piwowarska and González-Alvarez, 2012). Other frequently used models for solid-liquid extraction processes are the modified Page's model (Jokić et al., 2010), the logarithmic model, also known as Elovich's model (Kitanović et al., 2008; Jokić et al., 2010), or the parabolic diffusion model (Kitanović et al., 2008; Piwowarska and González-Alvarez, 2012).

The aim of this study was to determine the most suitable kinetic model for the description of the solid-liquid extraction of polyphenolic compounds from the lavender flower (*Lavandula x hybrida* L.) at three different temperatures, in order to predict the physical (total dissolved solids, electrical conductivity, and dry matter) and chemical characteristics (total polyphenolic content) of lavender water extracts. Peleg's, Page's, and the logarithmic models were used to describe the kinetics of solid-liquid lavender extraction and to estimate the kinetic parameters of the extraction process. The adequacy of the applied mathematical models was evaluated based on the calculated determination coefficient and the scaled root mean squared error values.

Materials and methods

Plant material

Dried lavender flower (*Lavandula x hybrida* L.) was purchased from a specialized herbal store Suban d.o.o. (Strmec Samoborski, Croatia) and stored at ambient conditions until used. According to the manufacturer's specifications, the plant material was collected in the north-western part of Croatia during the flowering season of 2015, dried naturally, and the final dry matter content of the lot after drying was 86.75%.

Chemicals and reagents

The Folin-Ciocalteu reagent and sodium carbonate were purchased from Kemika (Zagreb, Croatia). Gallic acid (3,4,5-trihydroxybenzoic acid) was obtained from Aldrich (Sigma–Aldrich Chemie, Steinheim, Germany). The chemicals were of analytical reagent grade.

Extraction procedure

Lavender extracts were prepared by conventional aqueous extraction, in order to simulate the household preparation conditions and use in traditional medicine. An amount of $m = 30$ g of dry plant material was placed in a $V = 2000$ mL glass with $V = 1500$ mL of deionised water, covered with aluminium foil, and heated to a specific temperature ($T = 40, 60, 80$ °C $\pm$ 0.5 °C) using the IKA HBR4 digital oil bath (IKA-Werk GmbH & Co.KG, Staufen, Germany) at the magnetic stirrer rotational speed of rpm = 500 rev/min for $t = 90$ min. The experiment was performed with the plant material particle size range of $d = 3000 – 4000$ μm. $V = 5$ mL samples were taken from the initial glass at regular time intervals ($t = 0.5, 1, 2, 3, 4, 5, 6, 7, 8, 9, 10, 15, 20, 25, 30, 35, 40, 45, 50, 55, 60, 70, 80$ and 90 min). The samples were immediately cooled in a water-ice mixture, then filtered through a cellulose paper filter (LLG Labware, Meckenheim, Germany) with $5 – 13$ μm pore size, and stored at $T = 4$ °C until the analysis.

The selected temperatures and extraction conditions were chosen based on a set of preliminary experiments (data not shown).

Determination of total polyphenolic content, electrical conductivity, total dissolved solids, and dry matter

The total polyphenolic content (TPC) was determined spectrophotometrically using the Folin-Ciocalteu reagent, according to a modified method by Lachman et al. (1998). All analyses were carried out in triplicate and the results were expressed as mg of gallic acid equivalents (GAE) per gram of dry matter (DM) of plant material.

Electrical conductivity (G) and total dissolved solids (TDS) of water extracts were determined using a SevenCompact conductometer (Mettler Toledo, Switzerland). After cooling the lavender water extracts to room temperature, a probe was submerged into the extracts and it was left in until the electrical conductivity/total dissolved solids on the conductometer stabilized. All measurements were carried out in triplicate.

Dry matter (DM) was determined gravimetrically, using a standard AOAC method (AOAC 1990). Approximately $m = 3$ g of extracts were dried in an oven (InkoLab ST60T, Croatia) for $t = 4$ hours at $T = 105$ °C, in weighing dishes previously filled with quartz sand. All measurements were done in triplicate.

Kinetics of solid-liquid extraction

In order to describe the influence of temperature on the extraction process, mathematical modelling was applied. The kinetic parameters of Peleg's, Page's, and the logarithmic models for total polyphenolic content (TPC), dry matter (DM), total dissolved solids (TDS), and electrical conductivity (G) were estimated.

The model proposed by Peleg (1988) was adapted for the description of the solid-liquid extraction process (Eq. (1)):

$$c(t) = c_0 + \frac{t}{(K_1 + K_2 \cdot t)} \qquad (1)$$

where c_0 represents the amount of TPC/DM/TDS/G in the liquid phase at $t = 0$, $c(t)$ is the amount of TPC/DM/TDS/G in the liquid phase at the time t, K_1 is the Peleg's rate constant, K_2 is the Peleg's capacity constant, and t is extraction time.

The Peleg's rate constant K_1 relates to extraction rate (B_0) at the very beginning of the extraction process ($t = t_0$):

$$B_0 = \frac{1}{K_1} \qquad (2)$$

The Peleg's capacity constant K_2 relates to maximum TPC/DM/TDS/G during the extraction process. When $t \to \infty$, Eq. (3) gives the relations between the equilibrium concentration (c_e) and the K_2 constant:

$$c(t)_{t \to \infty} = c_e = \frac{1}{K_2} \qquad (3)$$

The model proposed by Page (Jokić et al., 2010; Jurinjak Tušek et al., 2016) was used as follows:

$$c(t) = e^{-k \cdot t^n} \qquad (4)$$

where $c(t)$ is the amount of TPC/DM/TDS/G in the liquid phase at the time t, k and n are the constants of the Page's model, and t is extraction time.

The logarithmic model (Bucić-Kojić et al., 2007; Jokić et al., 2010; Jurinjak Tušek et al., 2016) was used as follows:

$$c(t) = a \log t + b \qquad (5)$$

where $c(t)$ is the amount of TPC/DM/TDS/G in the liquid phase at the time t, a and b are the constants of the logarithmic model, and t is extraction time.

Statistical analysis

The kinetic parameters of Peleg's, the logarithmic and Page's models were estimated by nonlinear regression using the Quasi–Newton method from Statistica Version 10.0 (StatSoft Inc., Tulsa, USA). Goodness of fit was assessed by the determination coefficient R^2 and the scaled root mean squared error as follows (Eq. (6)):

$$SRMSE = \frac{1}{\bar{y}_i} \cdot \sqrt{\frac{\sum_{i=1}^{n}(\hat{y}_i - y_i)^2}{n}} \qquad (6)$$

where y_i is the observed value for the i-th observation, $\bar{y}_i$ is the mean of all observed data, $\hat{y}_i$ is the predicted value, while n is the number of measurements.

Results and discussion

Since the data from preliminary experiments showed that temperature had the highest influence on the yield of the solid-liquid extraction process, different extraction temperatures were used in this study. The highest concentration of total polyphenols in lavender water extracts was obtained at $T = 80$ ºC (TPC = 81.84 mg$_{GAE}$/g$_{DM}$), while the lowest TPC concentration was observed at $T = 40$ ºC (TPC = 35.85 mg$_{GAE}$/g$_{DM}$) (Fig. 1). TPC increases as the extraction temperature rises, which is in agreement with the results from previous studies (Jurinjak Tušek et al., 2016; Valinger et al., 2017). Similar to TPC, total dissolved solids, electrical conductivity, and dry matter content increased with the increase of the extraction temperature (Fig. 2 – 4). Values of TDS, G, and DM at $T = 80$ ºC were TDS = 566 mg/L, G = 1131 µS/cm, and DM = 0.56%, respectively. The lowest TDS, G, and DM values were measured at $T = 40$ ºC (TDS = 442 mg/L, G = 884 µS/cm, and DM = 0.32%, respectively).

It can be concluded from the obtained results that an increase of the extraction time resulted in an increase of TPC, TDS, G, and DM values. The highest values of TPC, TDS, G, and DM were obtained at the end of the extraction process (Fig. 1 – 4).

By analysing the extraction curves for all the parameters used in this study, two periods of

extraction could be observed (Fig. 1 – 4). The period of rapid increase of TPC, TDS, *G,* and DM at the beginning of the process, and their slow increase as the extraction process progresses (Paunović et al., 2014; Fan et al., 2015; Jurinjak Tušek et al., 2016). Since the extraction curves describing TPC, TDS, *G,* and DM in relation to the duration of the extraction process have similar shapes, the same mathematical models (Peleg's, Page's, and logarithmic) were used to analyse the extraction kinetics. The list of estimated kinetic constants, determination coefficients, and scaled root mean squared errors at three different temperatures is given in Tables 1–4. Fig. 1–4 show the comparison of the experimentally obtained data and the predicted extraction dynamics obtained with the use of mathematical models.

According to the calculated values for R^2 and SRMSE, the Peleg's model gave the most suitable description of the experimental data for TPC extraction from lavender at $T = 40$ and $T = 60$ ºC. The determination coefficients were high in these two experiments ($R^2 = 0.996$ and $R^2 = 0.991$, respectively) and the scaled root mean squared errors were SRMSE = 0.049 and SRMSE = 0.071, respectively. Page's model also gave a good concordance between experimental and calculated data at $T = 40$ and $T = 60$ ºC ($R^2_{40\ ºC} = 0.947$ and $R^2_{60\ ºC} = 0.943$, respectively), while TPC extraction at $T = 80$ °C was best described by the logarithmic model ($R^2 = 0.927$, SRMSE = 0.093) (Table 1).

Table 1. Values of estimated model parameters for solid-liquid extraction of lavender for total polyphenolic content (TPC)

| T/ºC	Peleg's model				Logarithmic model				Page's model			
	K_1/g$_{DM}$ min/mg$_{GAE}$	K_2/g$_{DM}$/mg$_{GAE}$	R^2	SRMSE	a	b	R^2	SRMSE	k	n	R^2	SRMSE
40	0.667±0.025	0.02±0.0005	0.996	0.049	19.207±1.191	-4.044±1.541	0.922	0.192	-1.628±0.111	0.183±0.017	0.947	0.172
60	0.484±0.027	0.014±0.0006	0.991	0.071	27.415±1.672	-5.007±2.162	0.924	0.183	-2.014±0.112	0.157±0.014	0.943	0.170
80	0.066±0.009	0.013±0.0005	0.915	0.103	31.390±1.876	21.936±2.426	0.927	0.093	-3.403±0.069	0.061±0.005	0.885	0.118
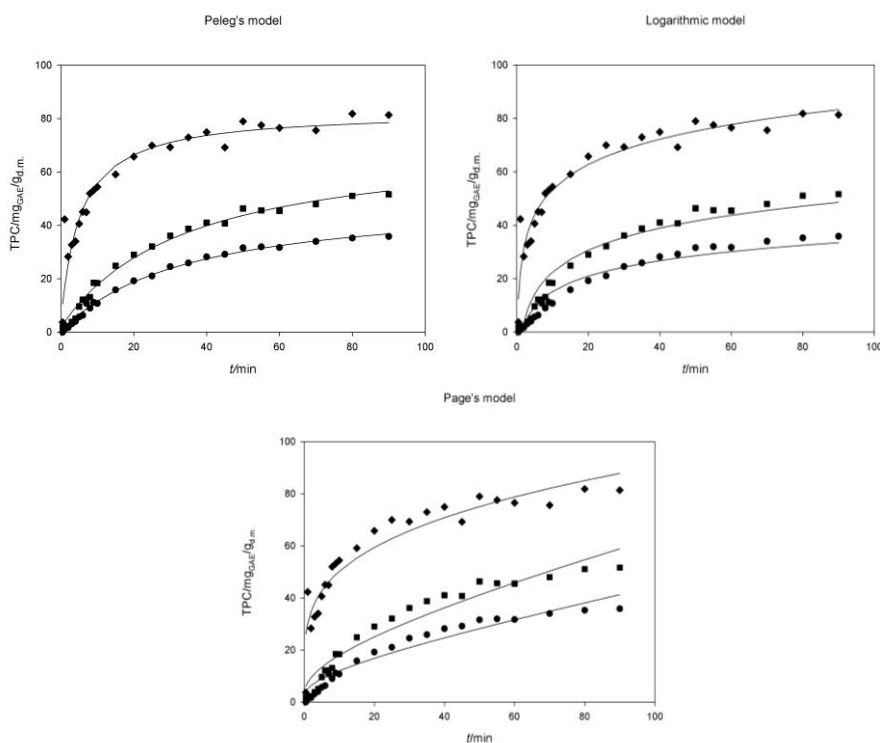
Fig. 1. Comparison between the experimental data and the kinetic model simulations for solid-liquid extraction of total polyphenolic content (TPC) from lavender at (●) *T* = 40 ºC, (■) *T* =60 ºC, and (♦) *T* =80 ºC. All experiments were carried out in triplicate and the average values are reported (symbols: experimental data; lines: represent approximation according to Peleg's, the logarithmic, and Page's models).

Based on the calculated K_1 and K_2 constants of the Peleg's model, it can be noticed that the values of the extraction rate constant (K_1) and the capacity constant (K_2) showed a tendency of decrease with the increase in extraction temperature. Higher temperatures favour the extraction process and the highest amount of TPC is extracted at the beginning of the process (Fig. 1). The same trend for K_1 and K_2 constants of the Peleg's model has been observed for solid-liquid extraction of TPC from chamomile, yarrow, marigold, and dandelion extracts (Jurinjak Tušek et al., 2016), and for solid-liquid extraction of total polyphenols from grape seeds (Bucić-Kojić et al., 2007).

According to the high R^2 and low SRMSE values in all experiments, these models showed to be suitable for model solid-liquid extraction of total polyphenols from lavender (Fig. 1). A good agreement between the experimental data and the data estimated by Peleg's, the logarithmic, and Page's models were also observed in the study by Jokić et al. (2010) and Jurinjak Tušek et al. (2016).

The experimental data for total dissolved solids (TDS) was also best described by the Peleg's model for all analysed temperatures ($R^2_{40\ °C} = 0.993$, $R^2_{60\ °C} = 0.982$, and $R^2_{80\ °C} = 0.973$, respectively), with values of scaled root mean squared errors from SRMSE = 0.045 to SRMSE = 0.068. The logarithmic model also gave a good prediction of TDS for solid-liquid extraction, with R^2 values from 0.953 to 0.974 (SRMSE ranged from 0.045 to 0.111). The values of the determination coefficient for Page's model were lower, compared to Peleg's and the logarithmic model, and were in the range from 0.913 to 0.949. SRMSE values of the Page's model were in the range from 0.083 to 0.120 (Table 2).

Table 2. Values of estimated model parameters for solid-liquid extraction of lavender for total dissolved solids (TDS)

T/ºC	Peleg's model				Logarithmic model				Page's model			
	K_1/L min/mg	K_2/L/mg	R^2	SRMSE	a	b	R^2	SRMSE	k	n	R^2	SRMSE
40	0.037±0.001	0.002±0.00004	0.993	0.045	214.676±10.134	17.577±13.104[*]	0.953	0.111	-4.508±0.083	0.071±0.005	0.949	0.120
60	0.036±0.003	0.002±0.0001	0.982	0.068	222.901±10.472	53.874±13.54	0.954	0.098	-4.805±0.081	0.06±0.005	0.938	0.115
80	0.008±0.0006	0.002±0.00004	0.973	0.046	194.454±6.735	211.644±8.708	0.974	0.045	-5.541±0.048	0.032±0.002	0.913	0.083

[*]estimated parameters are not statistically significant ($p > 0.05$)
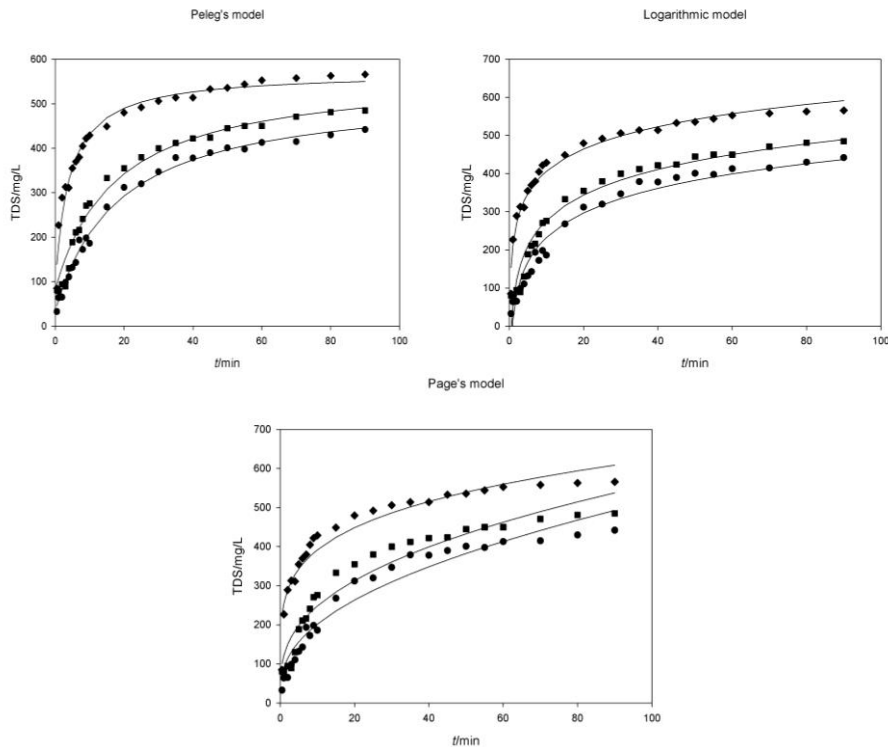

Fig. 2. Comparison between the experimental data and the kinetic model simulations for solid-liquid extraction of total dissolved solids (TDS) from lavender at (●) T =40 ºC, (■) T =60 ºC, and (♦) T =80 ºC. All experiments were carried out in triplicate and the average values are reported (symbols: experimental data; lines: represent approximation according to Peleg's, the logarithmic, and Page's models).

K_1 and K_2 constants of the Peleg's model for total dissolved solids were lower in comparison with K_1 and K_2 constants calculated for total polyphenolic content, indicating that the initial extraction is faster for TPC than for TDS (Table 2).

According to the data presented in Fig. 2, it can be seen that the applied models follow the trend of the experimental data and can be used for TDS modelling during the solid-liquid extraction process.

The values of estimated model parameters for solid-liquid extraction of lavender for electrical conductivity (*G*) are shown in Table 3, while the comparison between the experimental data and the kinetic model simulations is presented in Fig. 3. Electrical conductivity (*G*) was best described by the Peleg's model. The determination coefficients were high in all experiments ($R^2 = 0.973 – 0.992$) and the scaled root mean squared errors were in the range from SRMSE = 0.050 to 0.068, which implied a good agreement between experimental and calculated data. According to the results of the Page's model, it is visible that the values of determination coefficients were lower than in Peleg's and the logarithmic models, and were in the range from $R^2 = 0.912$ to $R^2 = 0.953$. The values of determination coefficients obtained by the logarithmic model were in the range from $R^2 = 0.946$ to $R^2 = 0.973$. The logarithmic model also gave a good concordance between experimental and calculated data at $T = 80$ °C with the lower SRMSE value (0.045), as compared to other models (Table 3).

Table 3. Values of estimated model parameters for solid-liquid extraction of lavender for electrical conductivity (*G*)

T/ºC	Peleg's model				Logarithmic model				Page's model			
	K_1/cm min/µS	K_2/cm/µS	R^2	SRMSE	*a*	*b*	R^2	SRMSE	*k*	*n*	R^2	SRMSE
40	0.021±0.001	0.001±0.00003	0.992	0.051	426.963±21.533	42.334±27.842[*]	0.946	0.118	-5.196±0.078	0.063±0.004	0.953	0.113
60	0.018±0.001	0.001±0.00004	0.983	0.068	446.365±21.301	104.269±27.543	0.952	0.100	-5.478±0.078	0.054±0.004	0.939	0.115
80	0.004±0.0003	0.001±0.00002	0.973	0.050	388.821±13.724	422.823±17.745	0.973	0.045	-6.232±0.048	0.029±0.002	0.912	0.083

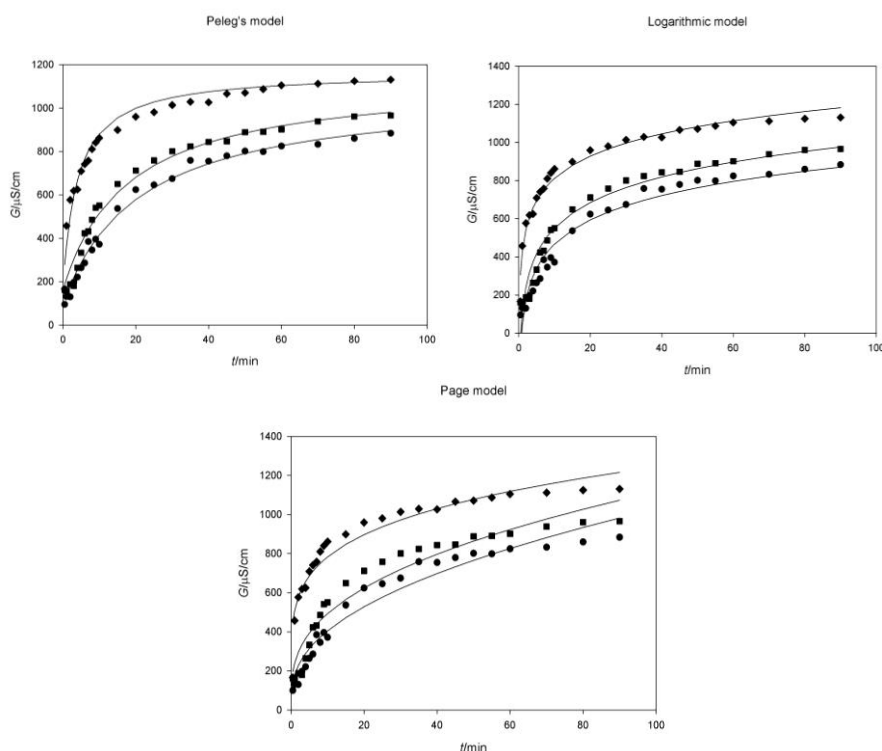[*]estimated parameters are not statistically significant ($p > 0.05$)

Fig. 3. Comparison between the experimental data and the kinetic model simulations for solid-liquid extraction of electrical conductivity (*G*) from lavender at (●) *T* =40 ºC, (■) *T* =60 ºC, and (♦) *T* =80 ºC. All experiments were carried out in triplicate and the average values are reported (symbols: experimental data; lines: represent approximation according to Peleg's, the logarithmic, and Page's models).

As in the case of total dissolved solids, it can be seen that differences in the initial extraction rate (K_1) and capacity constant (K_2) of G were not as distinctive as for the total polyphenolic content. This is to be expected, since electrical conductivity is in correlation with total dissolved solids.

The applied models follow the trend of the experimental data obtained for solid-liquid extraction of electrical conductivity from lavender extracts (Fig. 3).

Based on the calculated determination coefficients and scaled root mean squared errors for solid-liquid extraction of dry matter from lavender at three different temperatures, Peleg's and Page's models were chosen as the most suitable for the description of the extraction dynamics at T =40 ºC and T =60 ºC (R^2> 0.947; SRMSE = 0.092 – 0.156).

The logarithmic model gave the best prediction of dry matter solid-liquid extraction at 80 ºC, with R^2 = 0.957 and SRMSE = 0.072, compared to Peleg's and Page's models (Table 4).

As in the case of total polyphenolic content (TPC), the values of K_1 and K_2 constants of the Peleg's model decreased with the increase in extraction temperature, indicating that higher temperatures favour the extraction process (Table 4). The same trend of Peleg's constants for solid-liquid extraction of dry matter from chamomile, yarrow, marigold, and dandelion has been observed in the study by Jurinjak Tušek et al. (2016).

As in previous experiments, the applied models follow the trend of the experimental data and can be used to model solid-liquid extraction kinetics of dry matter (DM) from lavender extracts (Fig. 4).

Table 4. Values of estimated model parameters for solid-liquid extraction of lavender for dry matter (DM)

T/ºC	Peleg's model				Logarithmic model				Page's model			
	K_1/min/%	K_2/1/%	R^2	SRMSE	a	b	R^2	SRMSE	k	n	R^2	SRMSE
40	91.676±11.735	2.085±0.259	0.950	0.156	0.163±0.015	-0.024±0.019[*]	0.846	0.268	4.912±0.414	-0.34±0.024	0.947	0.156
60	59.612±5.832	1.84±0.132	0.975	0.097	0.214±0.014	-0.009±0.018[*]	0.911	0.174	3.93±0.258	-0.351±0.019	0.976	0.092
80	11.414±1.194	1.988±0.067	0.955	0.074	0.211±0.01	0.15±0.012	0.957	0.072	1.865±0.094	-0.261±0.017	0.955	0.075

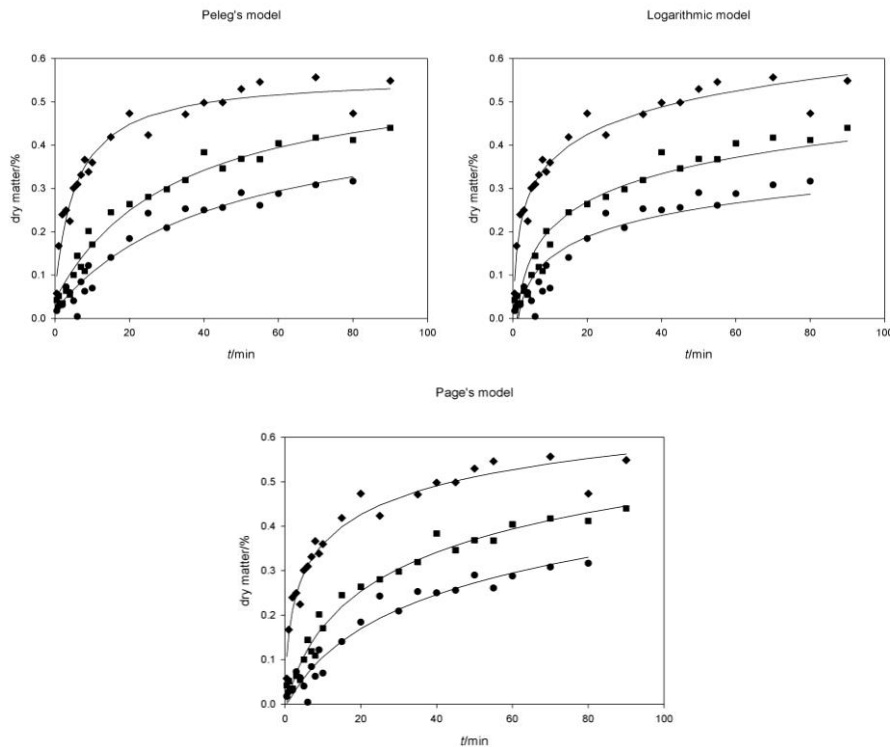[*]estimated parameters are not statistically significant ($p > 0.05$)

Fig. 4. Comparison between the experimental data and the kinetic model simulations for solid-liquid extraction of dry matter from lavender at (●) T =40 ºC, (■) T =60 ºC, and (♦) T =80 ºC. All experiments were carried out in triplicate and the average values are reported (symbols: experimental data; lines: represent approximation according to Peleg's, the logarithmic, and Page's models).

Conclusions

The experimental results showed that temperature had a significant influence on the solid-liquid extraction process of total polyphenols, total dissolved solids, electrical conductivity, and dry matter from dried lavender flowers. The highest extraction efficiency of TPC, TDS, *G,* and DM was achieved at 80 ºC and after 90 minutes of extraction. Most of the polyphenols and dry matter are extracted from a lavender flower to the solvent in the beginning of the extraction process.

According to the calculated determination coefficients and scaled root mean squared errors, the Peleg's model was chosen as the most suitable model for the description of the experimental data for total polyphenolic content, total dissolved solids, electrical conductivity, and dry matter content for lavender water extracts. The logarithmic and Page's models also showed a good agreement between experimental and model calculated data, which allows the application of the above mentioned models for the purpose of modelling and optimisation of solid-liquid extraction of dried lavender flowers.

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