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RESEARCH

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Kinetic study in the extraction of xanthones from *Securidaca longepedunculata* roots by microwave-assisted-extraction

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Abstract

Background: Extraction of active compounds from plants using microwave can be utilized as an alternative solution for conventional extraction. To well understand this technology, the study of the modelization and kinetics mechanism of the extraction method is necessary. This study aimed to evaluate the suitable kinetics models for MAE of the Xanthones from *Securidaca longepedunculata* Fresen roots.

Results: The experimental data (xanthones versus time, power and ratio, respectively) were fitted to three-parameter empirical kinetics models. The second-order models appear to be the best fit to explain kinetics study of MAE than the first model. The second-order model was acceptable, with elevated value of the correlation coefficient ($R^2 = 0.9861$), showing that it perfectly relates the process.

Conclusion: Based on the results obtained, the extraction of xanthones from roots of *S. longepedunculata* is done successively in two steps, washing and diffusion of compounds from matrices as described by second-order kinetics model. The latter could report the kinetics model of extraction process from *S. longepedunculata* Fresen using microwave technology.

Keywords: Microwave-assisted extraction (MAE), *Securidaca longepedunculata* Fresen, Xanthones, Empirical kinetics model

1 Background

Securidaca longepedunculata (Fresen.) is a tree from Polygalaceae family and has been generally used as a medicinal plant in tropical Africa specially in Cameroon [1]. Commonly known as "Aalali" in the Fulfulde language, the roots of *S. longepedunculata* are used in traditional medicine to treat various ailments across to coughs, colds, fever, backache, toothache, sleeping sickness, malaria, inflammation, rheumatism [2]. The therapeutic efficacy of treatment for plant is based on the content of its bioactive components. Xanthones are the major constituents of *S. longepedunculata* [3] and have remarkable biological and medicinal activities, including antibacterial, antiviral, antihypertensive, antithrombotic, anticancer, cytotoxic, anti-oxidative and anti-inflammatory activities [4]. Xanthones play an importance role because they have functional groups which have the potential to serve as acquired of new drugs. The beneficial effects of xanthones-rich natural products have been established, and supplementing processed foods with xanthones is being practiced warranting sufficient daily intake to enhance the immune system [5]. To isolate compounds from a substance, people do extraction, and this action depends on type of plant and compound

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of interest. Currently, the production of xanthones extracts in Cameroon is still using conventional methods such as maceration. Extraction with conventional methods has several disadvantages such as large energy and solvent requirements, long extraction times, loss of several important volatile compounds and low extraction efficiency. The research has developed new methods for extracting like supercritical fluid technology and microwave-assisted extraction (MAE). Several studies show that the extraction method using microwave permits to obtain good number of compounds extracts [6]. The study of kinetics models is necessary to calculate parameters and decide mechanism that occurs from the extraction process. Experimental data are generally analyzed using kinetic models, similarly the extraction processes [7]. On the other hand, modeling is needed to predict extraction yield of the xanthones from *S. longepedunculata* obtained from separation before processing it. To the best of our knowledge, there is no information available explaining the kinetic modeling of xanthones from *S. longepedunculata.* Therefore, in this research, the effects of microwave power, extraction time, ratio of liquid to solid and extraction yield of the xanthones will be studied. The obtained data could be used for obtaining several parameters: coefficient of determination, the rate constant, the equilibrium extraction capacity and the initial extraction rate of xanthone extract explaining the kinetic model considering microwave-assisted extraction process.

2 Materials and methods

2.1 Materials

The roots of *S. longepedunculata* (Polygalaceae) were collected in Mayo Tchabal during raining season and identified by Professor Mapongmetsem, a botanist in the Department of Biological Sciences of Faculty of Science, University of Ngaoundere, Cameroon. The plant material was dried at room temperature under shed for two weeks and kept in moisture-free atmosphere. The dried roots were ground before conducting the experiments. All other chemicals and solvents used were of analytical grade purchased from Sigma-Aldrich.

3 Methods

3.1 Microwave-assisted extraction

Extraction experiments were carried out using a modified domestic microwave oven apparatus (DAE WOO, KOG-360, Combi Grill) adapted to a condenser. 100 g of dried *Securidaca longepedunculata* powder was extracted with 1000 mL solvent (acetone 95%) under different MAE conditions. After extraction, the vessels were allowed to cool at room temperature before opening. Microwave power (200, 400, 600, 800, 100 W), extraction time (20 to140 s, with an interval of 20 s) and solid–liquid ratio (0.25 to 2 g/20 mL with an interval of 0.25 g/20 mL) were evaluated for the extraction of xanthone from *S. longepedunculata*. The final extract was evaporated and dissolved in dimethyl sulfoxide before UV–Vis spectrophotometric (Spectroquant® Pharo 100 M) analysis. Three replicates were performed in each extraction.

3.2 Quantification of the total xanthones by colorimetric method

Colorimetric method followed by visible spectroscopy technique is one of the common analytical techniques used for the determination of a group of components in a mixture. This method is especially useful for the analysis of natural products which are very complicated mixtures [8]. Thus, this method may provide a solution for the quantification of the xanthones the *S. longepedunculata* extracts. The principle of this analysis was based on the oxidation of xanthones by the sodium acetate [9], which leads to the formation of yellow complex absorbing at 410 nm. 250 µL microwave extract and 75 µL of sodium acetate (5%) were mixed. This mixture was kept at ambient temperature for 30 min in order to gain a maximum of yellow color. The absorbance of the upper phase was read at 400 nm on an UV/Vis spectrophotometer (Spectroquant® Pharo 100 M) using glass cuvettes against blank in the number one test tube. Calibration curve was developed from 0 to 0.08 µL with an interval of 0.02 µL in different test tube with standard solution standard of 2-hydroxy-1,7-diméthoxyxanthone. Total xanthone were expressed as mg 2-hydroxy-1,7-diméthoxyxanthone equivalent per gram of dry weight extract (EDMX/gP).

3.3 Empirical kinetic models

in this study, kinetics of microwave-assisted-extraction of xanthones from *S. longepedunculata* roots is performed using first-order and second-order models, it could be known rate of extraction from xanthones.

3.4 Hervas et al. model [10]

The kinetics mechanism proposed by Hervas et al. was used to study the extraction process under equilibrium conditions, as shown in Eq. (1)

$$\frac{dC}{dt} = k(C_0 - C) \tag{1}$$

where C is the concentration of xanthones (µg EDMX/mL) produced at any time t, C_0 is the initial concentration of xanthones (µg EDMX/mL) present, and k is the effective diffusion coefficient (µL/µg EDMX s).

Integrating Eq. (1) between the initial moment and a given point at time t gives Eq. (2) with the boundary conditions as $Ct|t=0=0$ and $Ct|t=t=Ct$

$$C(t) = C_0\left(1 - e^{-kt}\right) \tag{2}$$

3.5 Peleg model [11]

The model proposed by Peleg was adapted for the extraction and used in the form:

$$C(t) = \frac{t}{K_1 + K_2 * t} \tag{3}$$

where $C(t)$ is the concentration of xanthones at time t (μg EDMX/g), t is the extraction time (s), K_1 is the Peleg rate constant (s g/μg EDMX), and K_2 is the Peleg's capacity constant (g/μg EDMX).

The Peleg rate constant K_1 relates to the extraction rate (B_0) at the very beginning ($t = t_0$)

$$B0 = \frac{1}{K1} \tag{4}$$

The Peleg capacity constant K_2 relates to maximum extraction yield, i.e., equilibrium concentration of xanthones extracted (*ce*) when $t \to \infty$. Equation (5) gives the relation between equilibrium concentration and K_2 constant

$$C0 = \frac{1}{K_2} \tag{5}$$

Thus, the extraction rate coefficient can be written as in Eq. (6)

$$k = \frac{K_2}{K_1} \tag{6}$$

3.6 Gaussian model

The model proposed by Gauss was adapted for the extraction and used in the form:

$$Y = a \exp\left[-0,5((x - x_0)/k)^2\right] \tag{7}$$

Y is the extraction rate, a is the maximum value of the extraction rate, x is the variable studied in the extraction, x_0 is the variable corresponding to the maximum value of the extraction rate a, and k is the constant of the extraction rate.

3.7 Statistical analysis

All analyses were performed in triplicate. The results are presented as average values with standard error and were analyzed statistically using one-way ANOVA. Statistical significance was accepted at a level of $p < 0.05$ using the statistical program Statgraphics centurion 12.

4 Results

4.1 Effect of extraction time on xanthones extraction yield

Kinetics extraction of xanthones from *S. longepedunculata* by MAE will be studied. Figure 1 shows the curve of xanthones concentration extracts with the extraction time. In this figure, we can observe rapid augmentation of xanthones concentration during eighty seconds and then more controlled increase during twenty seconds and near-saturation during later stages of extraction.

Therefore, to understand the extraction kinetics, two mathematical models proposed by Harvest and Peleg were used.

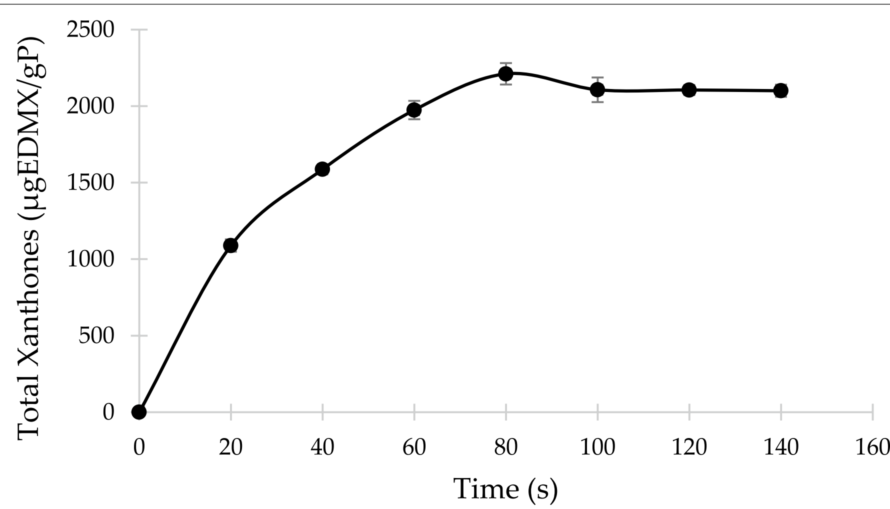

Fig. 1 Effect of extraction time on the concentration of xanthones during microwave-assisted extraction

Table 1 Hervas model of microwave-assisted extraction of xanthones

C_0 (µg EDMX/mL)	K (µg EDMX/mL s)	R^2
523	0.00017	0.63

Table 2 Peleg model of microwave-assisted extraction of xanthones

C_0 (µg EDMX/mL)	K_1 (s g/µg EDMX)	K_2 (µg EDMX/mL)	B_0 (µg EDMX/g)	R^2
2500	0.009	0.0004	111.11	0.98

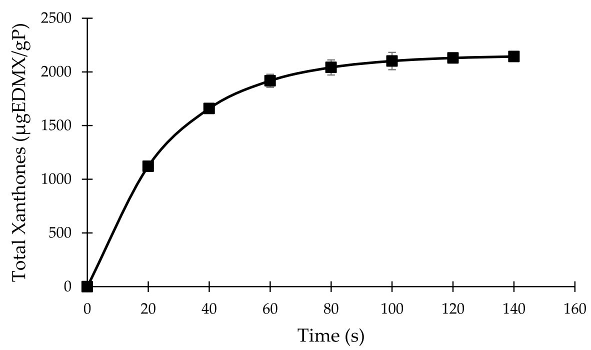

Fig. 2 Suitability of Peleg model for the kinetics of microwave-assisted extraction of xanthones from *S. longepedunculata*

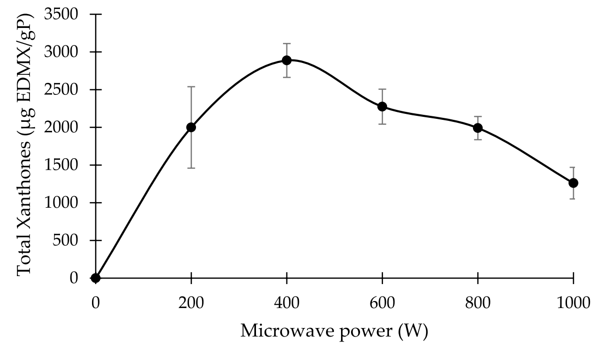

Fig. 3 Effect of microwave power on the concentration of xanthones during microwave-assisted extraction

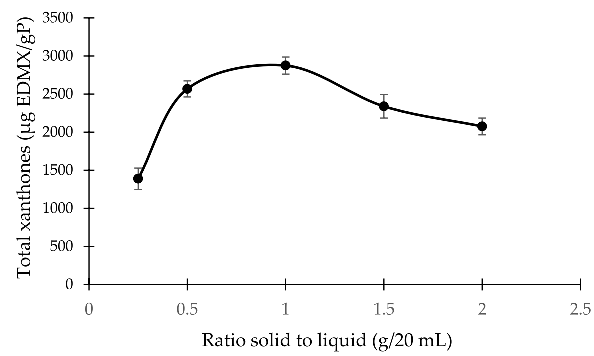

Fig. 4 Effect of solid-to-liquid ratio on the concentration of xanthones during microwave-assisted extraction

- Hervas model

Table 1 presents the extraction kinetics data obtained for the solid–liquid extraction using the Hervas method. The constant, k, C_S and correlation of coefficient R^2 were calculated (Table 1).

- Peleg model

The same data were analyzed using the model of Peleg. The extraction constant, correlation of coefficient R^2 and the coefficient (K_1, K_2 and k) for the extraction using the method were calculated and are mentioned in Table 2.

The plot of curve (Fig. 2) shows that kinetics extraction of xanthones of *S. longepedunculata* can be represented.

4.2 Effect of microwave power on xanthones extraction yield

Figure 3 shows the effect of microwave power on xanthone content during extraction. It can be clearly seen in this figure that there is a rapid increase in total xanthone at the power ranged from 200 to 550 W after which it reaches a threshold value. However, at 600 W we observe decrease in total xanthone content. The xanthone content at 200 and 550 W is found to be 1998.712 and 2900.7161 µg EDMX/gP, respectively.

4.3 Effect of solid-to-liquid ratio on xanthones extraction yield

Figure 4 shows the effect of solid-to-liquid ratio on the concentration of xanthones during MAE. It can be clearly seen in these figures that there is a steady increase in xanthone content at the ratio of solid to liquid from 0.25 to 1 g/20 mL after there is a significant decrease in the xanthone content and the yield decreases.

4.3.1 Gaussian model

The microwave power and solid–liquid ratio data were analyzed by using a Gaussian model of extraction. The maximum value of extraction rate, coefficient of determination R^2 and the coefficient (x_o and k) for the extraction

Table 3 Gaussian model of microwave-assisted extraction of xanthones

Coefficient	Effect of microwave power	Effect of solid–liquid ratio
a	2792.41 (µg EDMX/gP)	2822.41 (µg EDMX/gP)
K	320.86 (W)	0.935 (g/20 ml)
x_o	592 (W)	1.02 (g/20 ml)
R^2	0.89	0.69

using the method were calculated and are mentioned in Table 3.

5 Discussion

5.1 Effect of extraction time on xanthones extraction yield

Kinetics extraction of xanthones from *S. longepedunculata* by MAE will be studied. Figure 1 shows the curve of xanthones concentration extracts with the extraction time. In this figure, it is observed that xanthones concentration increased rapidly with time during eighty seconds, corresponding to initial phase of MAE, followed by twenty seconds of more controlled increase and near-saturation during later stages of extraction. This phenomenon may be explained by the rapid dissolution of dissolvable substance on particle area through washing and followed by undesirable element removal by diffusion [12]. The extraction curves (concentration of xanthones versus time) have a similar shape as the sorption curves (moisture content vs. time), and due to that fact, all these curves can be described using mathematical models of the mass transfer. Therefore, to describe the extraction kinetics, two mathematical models proposed by Harvest and Peleg model were used.

- Hervas model

Table 1 presents the extraction kinetics data obtained for the solid–liquid extraction using the Hervas method. These results presented low value of correlation coefficients. Thus, the process does not act in accordance with evolution of a first-order kinetic model of extraction, although the beginning of the extraction agreed with this order. The same data were analyzed by using a Peleg model of extraction. The extraction constant, correlation coefficient R^2 and the coefficient (K_1, K_2 and k) for the extraction using the method were calculated and are mentioned in Table 2. Compared with the harvest model, Peleg model presents very high coefficients and may be used to explain the microwave-assisted extraction process. During the microwave-assisted extraction of xanthones, an intense dissolution during the initial first stage and a strong scrubbing of the most soluble molecules (normal extraction) in the second stage are observed. The second stage is much slower because of the of transfer phenomenon of other molecules and the modification of the solid structure. This stage corresponds essentially to an external diffusion that concerns the remaining soluble matter [13].

5.2 Effect of microwave power on xanthones extraction yield

Figure 3 shows the effect of microwave power on xanthone content with extraction time. It can be clearly seen in these figures that there is a steady increase in xanthone content at the power ranged from 200 to 550 W after which it reaches a threshold value. The xanthone content at 200 and 550 W is found to be 1998.712 and 2900.7161 µg EDMX/gP, respectively. This extraction of xanthones by increasing microwave power can be associated with the direct effects of microwave energy on molecules by ionic conduction and dipole rotation which results in power dissipated in volumetric basis inside the solvent and plant material which generates molecular movement and heating [14]. Microwave irradiation energy can enhance the penetration of solvent into the matrix, and the heat generated in the system could cause softening of plant tissue, disruption of compounds of interests and increase in their solubility [15]. As shown in Fig. 3, the steep decrease at 600 W is due to the degradation of interest compounds at higher microwave power range. As the experiments are conducted in dry matter, as is usually the case, chances of degradation due to drying or evaporation at a higher microwave power intensity are ruled out [16]. Similar results of decrease in extraction yield of astragalosides from Radix astragali at high power due to disorderly molecular interactions have been reported in the optimization study of MAE of four main astragalosides in *Radix astragali* [17].

5.3 Effect of solid-to-liquid ratio on xanthones extraction yield

As shown in Fig. 4, the total xanthone increased with increasing liquid-to-solid ratio. When the liquid-to-solid ratio increased from 0.25 g/20 mL:1 to 1 g/20 mL, the xanthone total also increased, which was probably due to the fact that more solvent could enter cells, while more xanthones compounds could permeate into the solvent under the higher solid-to-liquid ratio conditions. With further increase in liquid-to-solid ratio, a decline xanthone content was observed. Effect of solid-to-liquid ratio on the concentration of xanthones during MAE was investigated. Pompeu et al. have reported that extraction of phenolics compounds was highly liquid–solid ratio dependent [18]. They have reported that liquid–solid ratio of 40:1 (mL/g) was sufficient to extract high quantities of phenolics from fruits of E. oleracea. Gan and Latiff reported that liquid–solid ratio (20 mL/g) played a significant role in the yield of phenolics, while extraction temperature did not make any significant contribution toward total phenolics content [19].

5.4 Gaussian model

The microwave power and solid–liquid ratio data were analyzed using a Gaussian model of extraction. The maximum value of extraction rate, coefficient of determination R^2 and the coefficient (x_o and k) for the extraction using the method were calculated and are mentioned in Table 3. The Gaussian model presents very high coefficients of and may be used to explain the effect of power and solid–liquid ratio from microwave-assisted extraction process. This model with an axis of symmetry shows the increase and decrease in our interesting compounds of interest. The maximum of xanthones were obtained at a microwave power of 600 W and a solid–liquid ratio of 1 g/20 mL. These models which can give us the maxima of xanthones can be used to contain the experimental domain.

6 Conclusion

The study of kinetic extraction of xanthones from *S. longepedunculata* using microwave-assisted extraction (MAE) has been done. This study confirms that microwave power and extraction time could affect the microwave-assisted extraction process. Further, the first- and second-order models were evaluated and we have obtained a remarkable fitting between experimental data and predicted model. The second-order model of kinetics extraction can well represent extraction models. These studies provide knowledge about mechanisms involved in the extraction kinetic of xanthones from *S. longepedunculata* using microwave. This indicates the potential of utilization of microwave technology for an industrial scale or commercial purposes.

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Author contributions
MBN was involved in conceptualization; GEDT and RKTT contributed to data curation and resources; GEDT was involved in formal analysis, investigation and writing—original draft; and SDS and MBN contributed to project administration and were involved in supervision. All authors read and approved the final manuscript.

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Availability of data and materials
The datasets analyzed during the current study are available from the corresponding author on reasonable request.

Declarations

Ethical approval and consent to participate
Not applicable.

Consent for publication
Not applicable.

Competing interests
The authors declare that they have no competing interests.

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