Materials Today: Proceedings 41 (2021) 65–71

Contents lists available at ScienceDirect

Materials Today: Proceedings

journal homepage: www.elsevier.com/locate/matpr

Enhanced extraction of *Synsepalum dulcificum* (Miracle Fruit) leaves using green ultrasonication–hydrodistillation method

Nuramira Fateha Sukor, Rohayu Jusoh *, Nur Syahirah Kamarudin

Faculty of Chemical and Process Engineering Technology, College of Engineering Technology, Universiti Malaysia Pahang, Gambang, Kuantan, Pahang 26300, Malaysia

ARTICLE INFO

Article history:
Received 12 March 2020
Received in revised form 18 November 2020
Accepted 22 November 2020
Available online 26 January 2021

Keywords:
Ultrasonication–Hydrodistillation method
Synsepalum dulcificum
Phenolic compounds

ABSTRACT

In this study, the phenolic compounds of *Synsepalum dulcificum* leaves were extracted via green sonication–hydrodistillation (UHD) method. The performance of UHD method was compared with conventional hydrodistillation (HD) method with UHD method resulting three–fold higher total phenolic content (TPC) of 124.82 mg GAE/g. The UHD method was further intensified by investigating the effect of extraction temperature ranging from 100 °C to 200 °C. The most intensified temperature was at 120 °C, indicating highest extraction yield of 102.95 mg/g. Different mathematical models namely rate law, Peleg's model and Fick's model were analysed and it was found that Fick's model was successfully predict the UHD process which confirms that diffusivity is the controlling factor in extracting phenolic compounds, instead of the capacity and the rate of reaction as proposed by Peleg's model and rate law, respectively. Hence, it can be concluded that UHD method effectively enhanced the extraction efficiency to increase the extraction yield of phenolic compounds in *S. dulcificum* leaves.

1. Introduction

Synsepalum dulcificum (*S. dulcificum*) or known as miracle fruit is an evergreen shrub belongs to the Sapotaceae family. The ripened miracle fruits are red in colour and often distinguished by its most outstanding properties as it can change the taste of sour to sweet [1]. Due to its remarkable properties, *S. dulcificum* has gained widespread attention among researchers, scientists and food manufacturers due to its possible application in treating diabetic patients with insulin resistance [2], and can also be exploited for use in food, medicine and cosmetic industries as sweeteners or additives [3]. In addition, all parts of the plant are rich with polyphenols and flavonoids [4] which important for antioxidant activities.

The most common routes that are used to extract the phenolic compound using traditional methods are hydro–distillation (HD), maceration, aqueous extraction and Soxhlet [4–7]. Among these approaches, HD method has been widely implied in extracting the phenolic compound from plants [8–10]. However, the time taken for the process is way too long and the yield produced is lesser yet not economical [11]. Hence, in order to improve the extraction yield, several approaches can be done such as microwave–assisted extraction (MAE), subcritical water extraction, and ultrasonication extraction [12,13]. These methods may shorten the extraction time, improve the extraction yield and reduce the operational costs [14]. The ultrasonication extraction technique has been recognized as an interesting alternative to assist plant extraction process as compared to other methods. This is due to the cavitation bubbles collapse affected from the sonication, led to the disrupting of the plant tissues, hence allowing better penetration of solvent into the sample powder [15–17]. However, there are only few studies which combined the efficiency of both ultrasonication and HD method while the extraction efforts are mainly focused in extracting the fruits part of *S. dulcificum* [18]. Furthermore, the study employing simultaneous ultrasonication–hydrodistillation (UHD) in extracting *S. dulcificum* from its leaves is very

* Corresponding author at: Faculty of Chemical and Process Engineering Technology, Universiti Malaysia Pahang, Gambang, Kuantan, Pahang 26300, Malaysia.
E-mail address: rohayu@ump.edu.my (R. Jusoh).

https://doi.org/10.1016/j.matpr.2020.11.1008
2214-7853/

rare. Therefore, there are needs to study the performance of UHD method towards the extraction of *S. dulcificum* leaves.

The application of mathematical modelling is crucial to explain the extraction process. There are several models that can be applied including rate law, Peleg's model and Fick's model. Many previous researches have been studied on the extraction process along with certain kinetic models [19–21]. However, limited research has been carried out on the comprehensive analysis between the kinetic models with the extraction process by UHD from *S. dulcificum* leaves.

Therefore, this study aimed to extract the phenolic compound from *S. dulcificum* leaves via UHD method and the TPC value of the extract was compared with the conventional HD method. The process was then intensified by investigating the temperature effect on the extraction yield. Later, the extraction process was analysed by using different mathematical models to describe the extraction kinetics.

2. Materials and methods

2.1. Materials

S. dulcificum leaves were acquired from Jabatan Pertanian Negeri Pahang. It was first washed, cleaned, and air dried at room temperature. Then, the leaves were grinded into fine powder prior to the experiment. Deionized water was obtained from FKKSA Lab, UMP. Gallic acid was used as standard for the quatification of phenolic compound. Folin–Ciocalteu reagent and sodium carbonate were also used for TPC analysis. Acetonitrile and orthophosphoric acid (85%) were used as mobile phase for HPLC analysis. All reagents and standards were purchased from Nano Life Quest Sdn Bhd.

2.2. Extraction of phenolic compounds

2.2.1. Conventional hydrodistillation (HD) method

The sample powders of *S. dulcificum* leaves were first prepared at solid–to–solvent ratio 1:10, using deionized water as solvent. The mixture then further proceeds for extraction process under hydrodistillation using Clevenger apparatus at extraction temperature ranging from 100 °C to 200 °C for 8 h. The vaporised mixture in the distillation unit will be then routed to a process namely condensation whereby the extraction sample will be collected at the receiving vessel and stored in a sample bottle.

2.2.2. Ultrasonication–hydrodistillaion (UHD) method

The mixture containing the sample powder and water solvent (1:10) was sonicated using a 2.75L Fisherbrand Scientific ultrasonic bath by adjusting the temperature at 70 °C, respectively for 30 min [22]. Then, it undergoes the extraction process via the hydrodistillation method.

2.3. Total phenolic content (TPC) analysis

The Folin–Ciocalteu method, described by Md Salehan et al. [23] with minor modifications was implied in this study to determine the TPC of the extracted samples. 0.5 mL of sample with appropriate dilution was mixed with 2.5 mL of Folin–Ciocalteu reagent (10%). After the reaction of 5 min, 2.0 mL of sodium carbonate (0.75%) was added and reacted for 2 h at room temperature. Then, the absorbance was determined using a spectrophotometer (Perkin Elemer U–1800 UV–Vis Spectrometer, range 200–800 nm) at 765 nm. Gallic acid was used as a standard and the results were expressed as gallic acid equivalents (GAE) mg sample.

2.4. Kinetic model

2.4.1. Rate law

The general second–order model [24,25] in determining the extraction rate can be written as Eq. (1):

$$\frac{dc}{dt} = k_1(c - c_\infty)^2 \tag{1}$$

where c_∞ is the extraction capacity (g/L), c is the concentration of sample constituent in solution (g/L) at any time, t (h) and k_1 (g/mg.h) is the second order extraction rate constant.

The integrated rate law can be obtained under boundary conditions $t = 0$ to t and $c = 0$ to c, shown as Eq. (2):

$$c = \frac{c_\infty^2 k_1 t}{1 + c_\infty k_1 t} \tag{2}$$

Eq. (2) then can be further linearized to Eq. (3):

$$\frac{c}{t} = \frac{1}{\left(\frac{1}{k_1 c_\infty^2}\right) + \left(\frac{t}{c_\infty}\right)} \tag{3}$$

c/t in Eq. (3) indicates the initial extraction rate, as symbolized by h when the extraction time, t approaches zero.

$$h = k_1 c_\infty^2 \tag{4}$$

Hence, the final equation describing the concentration of solute in the extraction solvent at any time can then be described as Eq. (5):

$$c = \frac{t}{\left(\frac{1}{h}\right) + \left(\frac{t}{c_\infty}\right)} \tag{5}$$

2.4.2. Peleg's model

Peleg's model equation [26] was used to describe the kinetics of phenolic compound extraction. The equation as shown in Eq. (6):

$$C_t = C_o + \frac{t}{K_1 + K_2 t} \tag{6}$$

where C_t and C_o are the concentration of gallic acid at time t (mg/g) and initial concentration of gallic acid (mg/g) which is zero, respectively. On the other hand, K_1 is Peleg's rate constant (min.g/mg) and K_2 is Peleg's capacity constant (g/mg). The modified Peleg's equation which represents the concentration of target solute (gallic acid) in extraction solvent against time can be written as in Eq. (7):

$$C_t = \frac{t}{K_1 + K_2 t} \tag{7}$$

The graph between $1/C_t$ vs. $1/t$ can be plotted to calculate K_1 and K_2 values from the slope and intercept, respectively. C_t was also subsequently calculated using Eq. (7) at different times for checking the fitting of the model.

2.4.3. Fick's model

The unsteady-state diffusion model, based on second–order Fick's model described how diffusion causes the concentration to change with time at each stage (washing or fast and slow diffusion) as signified in Eq. (8):

$$\frac{c_\infty - c}{c_\infty} = \frac{6}{\pi^2}\left[f_1 \exp\left(-\frac{\pi^2 D_1 t}{r^2}\right) + f_2 \exp\left(-\frac{\pi^2 D_2 t}{r^2}\right)\right] \tag{8}$$

where f_1 and f_2 are the fractions of the solute, extracted from the washing or fast and slow diffusion stages with diffusion coefficients D_1 and D_2, respectively. C_∞ is concentration in equilibrium, C is concentration in time, t and r is the particle radius [27].

The parameter of slow diffusion, D_2 and fraction of solute, f_2 can be obtained from the slope and the intercept of $\ln\left[\frac{C_\infty}{C_\infty - C}\right]$ vs time,

respectively. In earlier stages of the extraction, the second exponential term is close to unity and with the addition of f_2 from the previous calculation, fast diffusion, D_1 and fraction of solute for fast extraction, f_1 can be determined.

2.5. High performance Liquid Chromatography (HPLC)

The measurements of separation and determination of phenolic compound, namely gallic acid from the extracts of *S. dulcificum* leaves were performed using High Performance Liquid Chromatography (HPLC) system Agilent Series 1100 equipped with diode array detection (DAD) and a column Phenomenex Prodigy 5µ (250 X 4.60 mm) [28]. The wavelength for detection of gallic acid was set at 270 nm. Separation was achieved by flow rate of 1 mL/min with 3.0% Phosphoric acid (90%) / Acetonitrile (10%), in an isocratic programme. The injection volume was 10 µl. Each sample and standard were filtered with nylon syringe filter (pore size of 0.22 µm). For standard preparation, the mobile phase of phosphoric acid and acetonitrile were prepared, degassed in an ultrasonic bath and injected through the chromatographic column.

2.6. Fourier Transform Infrared (FTIR)

The functional groups of bioactive compounds were identified using Fourier Transform Infrared (FTIR) Spectrometry (Perkin Elmer Spectrum GX FTIR Spectrometry) using KBr method with a scan range 500–4000 cm^{-1}.

3. Results and discussion

3.1. Method selection: Total phenolic content (TPC)

The performance of UHD method and conventional HD were compared in obtaining the highest extraction yield of phenolic compound in *S. dulcificum* leaves extract. Fig. 1 indicates the total phenolic content (TPC) of *S. dulcificum* leaves acquired from both methods. The result revealed that the TPC obtained from UHD method was 124.82 mg GAE/g, three–fold higher as compared to the conventional HD, which only attained a value of 41.37 mg GAE/g. The simultaneous implementation of UHD process provided a much better yield as ultrasonication induced cavitation effect towards the sample materials which provides disrupt the plant tissues and increase mass transfer [29]. Later, the HD process occurs in which the phenolic compound exerts equilibrium pressure temperatures, causing enhanced extraction of phenolic compound. Meanwhile, the conventional HD technique only focuses on the phenolic compound leaving the plant material based on temperatures elevation without disrupting the plant tissue, thus producing much lower extraction yield. Therefore, this simultaneous UHD method remarkably manages to increase the extraction product yield than the conventional extraction techniques.

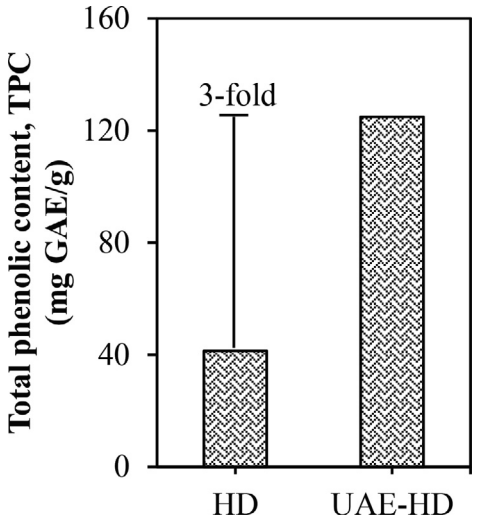

Fig 1. Total phenolic content corresponding to different extraction methods namely HD and UHD methods.

On top of that, past studies on the TPC values from various plants of Sapotaceae family species via different extraction method as tabulated in Table 1 indicated the high amount of phenolic content that exhibits high antioxidant activity [30–35]. The results are comparable depends on the different extraction methods used, showing the potential of UAE method in obtaining higher amount of phenolic contents compared to other methods.

3.2. Process intensification at different extraction temperatures

Fig. 2 demonstrated the influence of extraction temperature ranging from 100 °C to 200 °C on the yield of phenolic compounds. It can be observed that the extraction yield increased significantly from 77.02 mg/g to 102.95 mg/g as the temperature increased from 100 °C to 120 °C. It was noticed that an increase in temperature improves the extraction efficiency due to the decreasing solvent viscosity and thus, facilitating its diffusion into plant tissues. These eventually enhanced both solubility and desorption of the phenolic compound from the plant material [36,37]. However, the extraction yield started to decrease as the temperature was further increased to 150 °C and 200 °C. This may due to the degradation of phenolic compounds at too high temperature [38].

Past study on the effect of extraction temperature towards the amount of total phenolic and flavonoid compounds from pomegranate (*Punica granatum* L.) peel, gave comparable result of 130 °C obtaining 314.65 mg gallic acid equivalent/g and 153.66 mg rutin equivalent/g [39]. Thus, 120 °C is the optimum extraction temperature for the extraction of phenolic compounds from *S. dulcificum* leaves.

3.3. Kinetic analysis

In this study, three different kinetic models were compared in finding the controlling factor to describe the nature of phenolic compounds extraction via UHD from *S. dulcificum* leaves. The results of experimental data, calculated data, coefficient of determination (R^2) and root mean squared deviation (RMSD) at different extraction temperatures (100 °C–200 °C) were analysed for rate law, Peleg's model and Fick's model. R^2 and RMSD were used as

Table 1
Table of comparison of TPC values from various plants of Sapotaceae family species.

Extraction method	TPC (mg GAE/g)	References
S. dulcificum (HD)	41.37	This study
S. dulcificum (UHD)	124.82	This study
S. dulcificum (maceration)	58.67	Obafemi et al. (2017)
Chrysophyllum boivinianum (maceration)	8.05	Rakotoniaina et al. (2018)
Manilkara hexandra (solvent extraction)	128.10	Dutta and Ray (2018)
Mimusops elengi (solvent extraction)	98.0	Shahwar and Raza (2012)
Baillonella toxisperma (accelerated solvent extraction)	80.0	Saha et al. (2013)
Vitellaria paradoxa (maceration)	18.48	Talla et al. (2016)
Argania spinosa (UAE)	221.39	Dakiche et al. (2016)

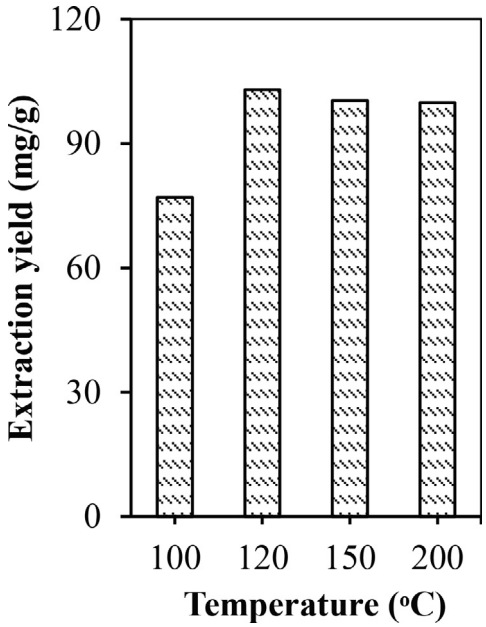

Fig 2. Extraction yield of phenolic compound at different extraction temperature from *S. dulcificum* leaves [UHD method; solid-to-solvent ratio 1:10; solvent deionized water].

the statistical measure that indicated the goodness of fit of a model and to measure the differences between values predicted by a model with the values obtained from the experiments, respectively. High R^2 near to 1 and small RMSD value represented a good correlation between the experimental and calculated data.

The calculated parameters of rate law including the extraction rate constant, k_1, initial extraction rate, h, RMSD and R^2 were tabulated in Table 2. The k_1 values was observed to be increased from 3.34 g/mg.h to 4.86 g/mg.h as the temperature rise from 100 °C to 120 °C and decreased subsequently as the temperature was further increased. Higher value of k_1 at temperature 120 °C indicated higher extraction rate at that particular temperature compared to others.

Besides, the h values also demonstrated similar trend resulting highest value at temperature 120 °C at 5.57E^4. In relation to the constant k_1 and extraction parameter h, these values are supposedly to increase with increased extraction temperature [40], indicated increased in the extraction rate of phenolic acids [25]. This is in agreement with previous experiment resulting 120 °C as the optimum temperature for this extraction process as too high temperature might lead to a degradation of phenolic compounds. However, despite a literally high R^2 values ($0.97 > R^2 > 0.99$), too high RMSD values ranging from 3.26 mg/g to 8.76 mg/g at varies temperature was impropriated to fully explain the model on the process. In addition, Fig. 3A showed a less fit curve of the rate law model compared to the experimental data, further confirmed the incompetent of the model in explaining the extraction process of phenolic compounds via UHD from *S. dulcificum* leaves.

Fig. 3B illustrated a less scattered experimental values to the Peleg's curve compared to previous rate law model. Peleg's model parameters explained on the constants K_1 and K_2, which related to extraction rate at the beginning process and the maximum phenolic acids concentration during the extraction process, respectively. All the K_1 and K_2 constants, R^2 and RMSD values at varies temperature were tabulated in Table 3. As the temperature increased from 100 °C to 120 °C, both K_1 and K_2 constants decreased from 0.0099 min.g/mg to 0.0082 min.g/mg and 0.0120 g/mg to 0.0089 g/mg, respectively. These constants have tendency to decreased with the increased of temperature as higher temperature favour the extraction process and higher amount of phenolic compounds extracted at the beginning of the process [41]. However, too high extraction temperature up to 200 °C increased K_1 and K_2 values as it does not favour the process, might be due to the damaged of the organic compounds at too high temperature. This is also in line with previous experiment result obtaining 120 °C as optimum extraction temperature. Even though this model acquired a literally high R^2 values ranging from 0.70 to 0.97, a quite high RMSD values ranging from 0.60 mg/g to 3.58 mg/g were still unable to fully describe the extraction process.

The diffusion coefficients for fast (D_1) and slow (D_2) are the core values in explaining an extraction process using Fick's model as Fick's claimed that the process occur in two stages namely fast diffusion or washing and slow diffusion. The diffusion coefficients, R^2 and RMSD were tabulated in Table 4. Highest D_1 value of 0.51 m^2/s was observed at temperature 120 °C indicated higher diffusion occur at this stage, while it decreased significantly as the extraction was increased up to 200 °C. High temperatures significantly lead to a kinetic improvement, however, it is limited by the fact that phenolic compound are sensitive to high temperatures. Thus, although heat treatments can improve extraction kinetics, they reduce the amount of phenolic content [22]. On the other hand, there was not much difference can be seen from the D_2 values at varies temperature. On top of that, Fig. 3C illustrated well–fitted values between the Fick's curve and experimental results. High R^2 values ranging from 0.89 to 0.98 with low RMSD values < 0.05 mg/g, remarkably reflect the compatibility of this Fick's model in explaining the extraction process of phenolic compounds from *S. dulcificum* leaves.

3.4. Fourier Transform Infrared (FTIR) analysis

The FTIR results of the extraction samples from *S. dulcificum* leaves via different methods namely HD and UHD were presented in Fig. 4Ai and 4Aii, respectively. Peak at 3397 cm^{-1} confirmed the presence of compounds with hydroxyl (–OH) groups, usually occurs between 2900 and 3550 cm^{-1} [42]. It can be observed that the hydroxyl peak was intense when the extraction was done via UHD instead of HD, indicated higher number of phenolic compounds were released during the UHD process. Apart from that, carbonyl (C = O) groups that appeared at peak 1701 cm^{-1} [43], further confirmed the existence of phenolic compounds. In addition, peaks 1508 cm^{-1} and 1154 cm^{-1} attributed to the aromatic rings C = C and C–H, respectively.

Fig. 4B summarized important functional groups to provide better understanding on the different peak intensities towards the extraction yield. Peaks 1701, 1154 and 1508 cm^{-1} indicated functional groups carbonyl C = O, aromatic C–H and C = C, respectively.

Table 2
Rate law kinetic model analysis for fitting experimental data via UHD method of phenolic compounds from *S.dulcificum* leaves at different extraction temperature.

Temperature (°C)	Experimental (mg/g)	k_1 (g/mg.h)	h (g/mg.h)	Calculated (mg/g)	R^2	RMSD (mg/g)
100	77.02	3.34	1.98E^4	70.51	0.99	3.26
120	102.95	4.86	5.57E^4	96.01	0.99	3.47
150	100.33	4.68	4.71E^4	91.62	0.98	4.36
200	99.85	4.59	5.15E^4	82.33	0.97	8.76

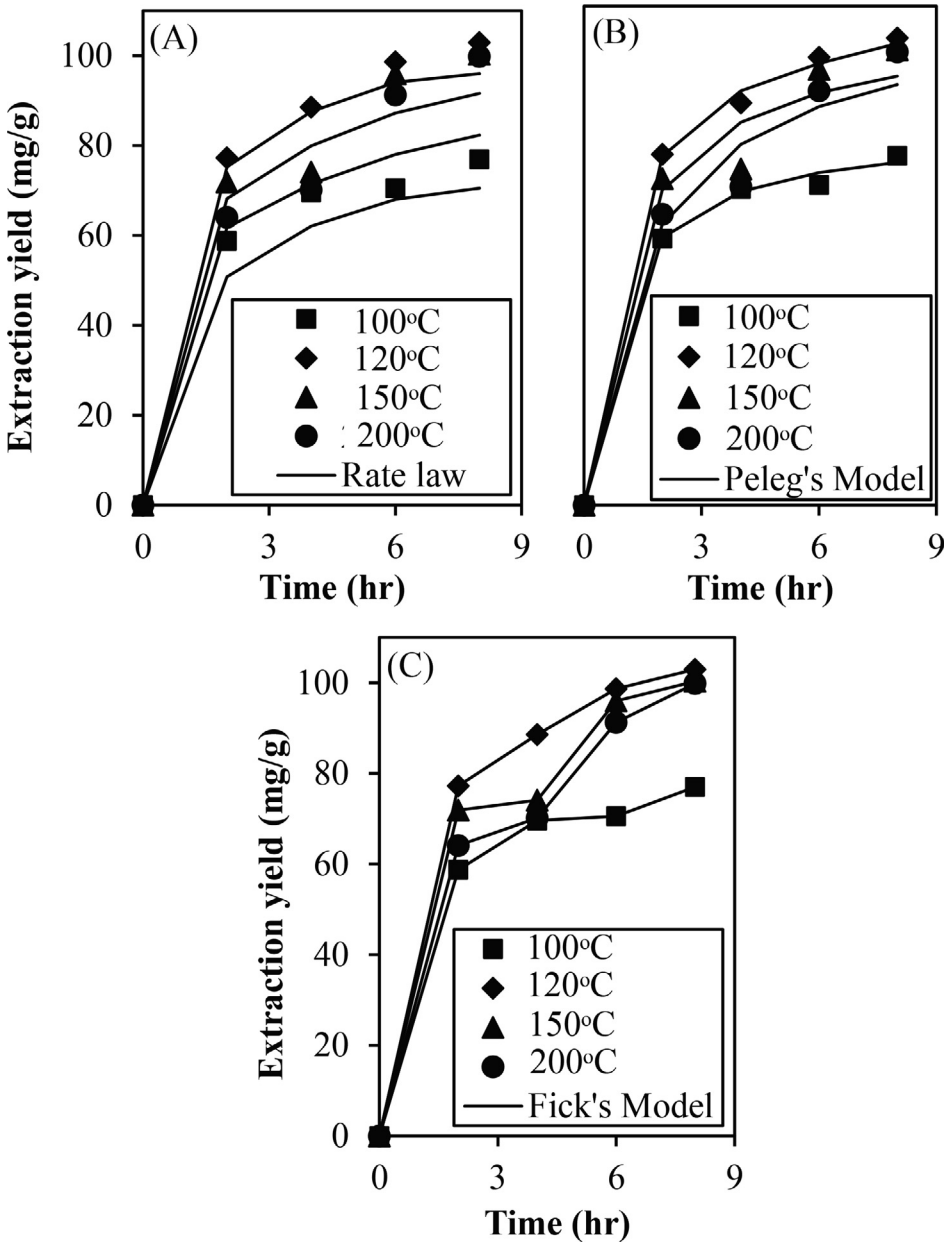

Fig. 3. Extraction kinetic of (A) rate law, (B) Peleg's model and (C) Fick's model at different extraction temperature.

Table 3
Peleg's kinetic model analysis for fitting experimental data via UHD method of phenolic compounds from *S.dulcificum* leaves at different extraction temperature.

Temperature (°C)	Experimental (mg/g)	K_1 (min g/mg)	K_2 (g/mg)	Calculated (mg/g)	R^2	RMSD (mg/g)
100	77.02	0.0099	0.0120	75.54	0.96	0.74
120	102.95	0.0082	0.0089	101.76	0.97	0.60
150	100.33	0.0102	0.0093	94.56	0.71	2.89
200	99.85	0.0143	0.0090	92.70	0.82	3.58

Table 4
Fick's kinetic model analysis for fitting experimental data via UHD method of phenolic compounds from *S. dulcificum* leaves at different extraction temperature.

Temperature (°C)	Experimental (mg/g)	D_1 (m^2/s)	D_2 (m^2/s)	Calculated (mg/g)	R^2	RMSD (mg/g)
100	77.02	0.42	0.18	≈77.02	0.89	< 0.05
120	102.95	0.51	0.13	≈102.95	0.98	< 0.05
150	100.33	0.48	0.08	≈100.33	0.90	< 0.05
200	99.85	0.34	0.16	≈99.85	0.94	< 0.05

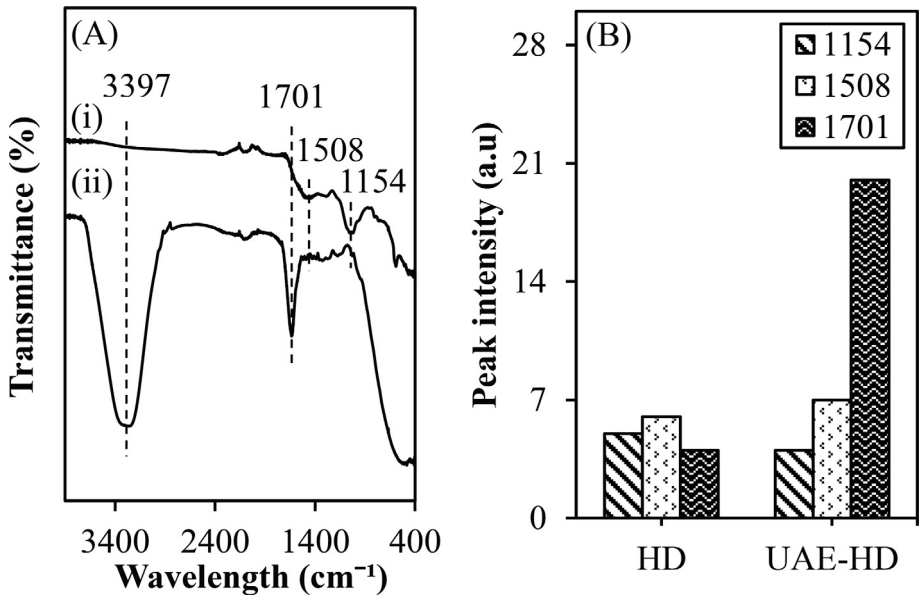

Fig 4. (A) FTIR spectra of *S. dulcificum* leaves after extraction via different methods namely (i) HD, (ii) UHD and (B) Peak intensity of different functional groups after extraction via HD and UHD methods.

The rise of peaks intensities after extraction process via UHD method could be clearly observed as compared to the one using HD method. The physical stressing from UHD method indeed plays an important role in extracting the phenolic compounds from the *S. dulcificum* leaves as almost all the target compounds were able to extract out, resulted to higher peak intensities. This is in agreement with the previous TPC result showing highest amount of phenolic compounds (gallic acid) in the plant extract using UHD method. Thus, the observed characteristic fingerprint pattern showing literally high intensity peaks reflected high amount of functional groups of phenolic compounds in plant extract.

4. Conclusions

The potential of UHD method in extracting phenolic compounds from *S. dulcificum* leaves was successfully recognized based on the TPC value of 124.82 mg GAE/g, three–fold higher as compared to the conventional HD. Further intensification of UHD method at different extraction temperature remarkably enhanced the extraction yield to a maximum value of 102.95 mg/g at 120 °C. The result is corresponding with the Fick's kinetic model that successfully predict the UHD process, confirmed that diffusivity is the controlling factor in extracting phenolic compounds, instead of the capacity and the rate of reaction as proposed by Peleg's model and rate law, respectively. Significantly, it is believed that further investigation in this area may contribute to understanding the potential of UHD method in extracting the phenolic acids which can contribute in the future development of extraction technology.

CRediT authorship contribution statement

Nuramira Fateha Sukor: Conceptualization, Methodology, Investigation, Writing - original draft. **Rohayu Jusoh:** Validation, Resources, Writing - review & editing, Supervision. **Nur Syahirah Kamarudin:** Formal analysis, Software.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgement

The authors are grateful for the support from Jabatan Pertanian Negeri Pahang, the Fundamental Research Grant Scheme from Ministry of Higher Education, Malaysia (Grant No. RDU160154) and internal university grant by Universiti Malaysia Pahang, Malaysia (Grant No. RDU180353).

References

[1] Z. He, J.S. Tan, O.M. Lai, A.B. Ariff, Optimization of conditions for the single step IMAC purification of miraculin from *Synsepalum dulcificum*, Food Chem. 181 (2015) 19–24.
[2] T.O. Obafemi, A.C. Akinmoladun, M.T. Olaleye, S.O. Agboade, A.A. Onasanya, Antidiabetic potential of methanolic and flavonoid-rich leaf extracts of *Synsepalum dulcificum* in type 2 diabetic rats, J. Ayurveda Integrat. Med. 8 (2017) 238–246.
[3] Z. He, J.S. Tan, S. Abbasiliasi, O.M. Lai, Y.J. Tam, A.B. Ariff, Phytochemicals, nutritionals and antioxidant properties of miracle fruit Synsepalum dulcificum, Ind. Crops Prod. 86 (2016) 87–94.
[4] V. Osabor, R. Etiuma, M. Ntinya, Chemical Profile of Leaves and Roots of Miracle Fruit, Synsepalum dulcificum, Am. Chem. Sci. J. 12 (2016) 1–8.
[5] T.O. Obafemi, A.C. Akinmoladun, M.T. Olaleye, A. Onasanya, K.C. Komolafe, J.A. Falode, M.L. Athayde, High Performance Liquid Chromatography HPLC) fingerprinting, mineral composition and in vitro antioxidant activity of methanol leaf extract of Synsepalum dulcificum (Sapotaceae, J. Appl. Pharmaceut. Sci. 7 (2017) 110–118.
[6] H.Y. Sintim, A. Burkhardt, A. Gawde, C.L. Cantrell, T. Astatkie, A.E. Obour, V. Schlegel, Hydrodistillation time affects dill seed essential oil yield, composition, and bioactivity, Ind. Crops Prod. 63 (2015) 190–196.
[7] L.P. Stanojević, N.S. Radulović, T.M. Djokić, B.M. Stanković, D.P. Ilić, M.D. Cakić, V.D. Nikolić, The yield, composition and hydrodistillation kinetics of the essential oil of dill seeds (*Anethi fructus*) obtained by different hydrodistillation techniques, Ind. Crops Prod. 65 (2015) 429–436.
[8] L.A. Conde-Hernández, J.R. Espinosa-Victoria, A. Trejo, J. Guerrero-Beltrán, CO^2-supercritical extraction, hydrodistillation and steam distillation of essential oil of rosemary, Rosmarinus officinalis, J. Food Eng. 200 (2017) 81–86.
[9] N. Jeyaratnam, A.H. Nour, R. Kanthasamy, A.H. Nour, A.R. Yuvaraj, J.O. Akindoyo, Essential oil from *Cinnamomum cassia* bark through hydrodistillation and advanced microwave assisted hydrodistillation, Ind. Crops Prod. 92 (2016) 57–66.

[10] A. Oreopoulou, E. Papavassilopoulou, H. Bardouki, M. Vamvakias, A. Bimpilas, V. Oreopoulou, Antioxidant recovery from hydrodistillation residues of selected Lamiaceae species by alkaline extraction, J. Appl. Res. Med. Aromat. Plants 8 (2018) 83–89.
[11] M. Vinatoru, T.J. Mason, I. Calinescu, Ultrasonically assisted extraction (UAE) and microwave assisted extraction (MAE) of functional compounds from plant materials, TrAC - Trends Analyt. Chem. 97 (2017) 159–178.
[12] E.M. Garcia-Castello, A.D. Rodriguez-Lopez, L. Mayor, R. Ballesteros, C. Conidi, A. Cassano, Optimization of conventional and ultrasound assisted extraction of flavonoids from grapefruit (*Citrus paradisi* L.) solid wastes, LWT - Food Sci. Technol. 64 (2015) 1114–1122.
[13] M.T. Munir, H. Kheirkhah, S. Baroutian, S.Y. Quek, B.R. Young, Subcritical water extraction of bioactive compounds from waste onion skin, J. Cleaner Prod. 183 (2018).
[14] T.V. Fonteles, A.K.F. Leite, A.R.A. Silva, A.P.G. Carneiro, E.D.C. Miguel, B.S. Cavada, S. Rodrigues, Ultrasound processing to enhance drying of cashew apple bagasse puree: Influence on antioxidant properties and in vitro bioaccessibility of bioactive compounds, Ultrason. Sonochem. 31 (2016) 237–249.
[15] H. Jian, F. Qiao, J. Chen, N. He, Physicochemical Characterisation of Polysaccharides from the Seeds and Leaves of Miracle Fruit (*Synsepalum dulcificum*) and Their Antioxidant and α-Glucosidase Inhibitory Activities in Vitro, J. Chem. 2017 (2017).
[16] M.M.A. Rashed, Q. Tong, M.H. Abdelhai, M.A.A. Gasmalla, J.B. Ndayishimiye, L. Chen, F. Ren, Effect of ultrasonic treatment on total phenolic extraction from *Lavandula pubescens* and its application in palm olein oil industry, Ultrason. Sonochem. 29 (2016) 39–47.
[17] J. Švarc-Gajić, Z. Stojanović, A. Segura Carretero, D. Arráez Román, I. Borrás, I. Vasiljević, Development of a microwave-assisted extraction for the analysis of phenolic compounds from *Rosmarinus officinalis*, J. Food Eng. 119 (2013) 525–532.
[18] I. Khemakhem, M.H. Ahmad-Qasem, E.B. Catalán, V. Micol, J.V. García-Pérez, M. A. Ayadi, M. Bouaziz, Kinetic improvement of olive leaves' bioactive compounds extraction by using power ultrasound in a wide temperature range, Ultrason. Sonochem. 34 (2017) 466–473.
[19] R. Fan, F. Yuan, N. Wang, Y. Gao, Y. Huang, Extraction and analysis of antioxidant compounds from the residues of *Asparagus officinalis* L, J. Food Sci. Technol. 52 (2015) 2690–2700.
[20] F.J. Segovia, J.J. Corral-Pérez, M.P. Almajano, Avocado seed: Modeling extraction of bioactive compounds, Ind. Crops Prod. 85 (2016) 213–220.
[21] Y. Tao, Z. Zhang, D.W. Sun, Kinetic modeling of ultrasound-assisted extraction of phenolic compounds from grape marc: Influence of acoustic energy density and temperature, Ultrason. Sonochem. 21 (2014) 1461–1469.
[22] N.F. Sukor, R. Jusoh, S.A. Rahim, N. Kamarudin, Ultrasound assisted methods for enhanced extraction of phenolic acids from *Quercus Infectoria* galls, Mater. Today:. Proc. 5 (2018) 21990–21999.
[23] N.A. Md Salehan, A.Z. Sulaiman, A. Ajit, Effect of temperature and sonication on the extraction of gallic acid from *Labisia Pumila* (Kacip Fatimah), ARPN J. Eng. Appl. Sci. 11 (2016) 2193–2198.
[24] P.S. Chiang, D.J. Lee, C.G. Whiteley, C.Y. Huang, Extracting antioxidant phenolic compounds from compressional-puffing pretreated *Pinus morrisonicola*: Effects of operational parameters, kinetics and characterization, J. Taiwan Inst. Chem. Eng. 75 (2017) 70–76.
[25] Z. Pan, W. Qu, H. Ma, G.G. Atungulu, T.H. McHugh, Continuous and pulsed ultrasound-assisted extractions of antioxidants from pomegranate peel, Ultrason. Sonochem. 19 (2012) 365–372.
[26] A.L.B. Dias, C.S. Arroio Sergio, P. Santos, G.F. Barbero, C.A. Rezende, J. Martínez, Effect of ultrasound on the supercritical CO^2 extraction of bioactive compounds from dedo de moça pepper (*Capsicum baccatum* L. var. pendulum), Ultrason. Sonochem. 31 (2016) 284–294.
[27] C.H. Chan, R. Yusoff, G.C. Ngoh, Modeling and kinetics study of conventional and assisted batch solvent extraction, Chem. Eng. Res. Des. 92 (2014) 1169–1186.
[28] N.F. Sukor, R. Jusoh, N.S. Kamarudin, N.A.A. Halim, A.Z. Sulaiman, S.B. Abdullah, S.B. Abdullah, Synergistic effect of probe sonication and ionic liquid for extraction of phenolic acids from oak galls, Ultrason. Sonochem. 62 (2019) 104876.
[29] J. Deng, Z. Xu, C. Xiang, J. Liu, L. Zhou, T. Li, C. Ding, Comparative evaluation of maceration and ultrasonic-assisted extraction of phenolic compounds from fresh olives, Ultrason. Sonochem. 37 (2017) 328–334.
[30] H. Dakiche, M. Khali, A.K. Abu-el-Haija, A. Al-Maaytah, Q.A. Al-Balas, Biological activities and phenolic contents of *Argania spinosa* L. (Sapotaceae) leaf extract, Trop. J. Pharm. Res. 15 (2016) 2563–2570.
[31] S. Dutta, S. Ray, Comparative assessment of total phenolic content and in vitro antioxidant activities of bark and leaf methanolic extracts of *Manilkara hexandra* (Roxb.) dubard, J. King Saud Univ. – Sci. (2018).
[32] E.N. Rakotoniaina, D. Donno, D. Randriamampionona, H.L. Harinarivo, H. Andriamaniraka, N.R. Solo, G.L. Beccaro, Insights into an endemic medicinal plant species of Madagascar and Comoros: The case of Famelona, Chrysophyllum boivinianum (Pierre) Baehni, Sapotaceae family, S. Afr. J. Bot. 117 (2018) 110–118.
[33] J.B.T. Saha, D. Abia, S. Dumarçay, M.K. Ndikontar, P. Gérardin, J. Ngamveng Noah, D. Perrin, Antioxidant activities, total phenolic contents and chemical compositions of extracts from four Cameroonian woods: Padouk (Pterocarpus soyauxii Taubb), tali (Erythrophleum suaveolens), moabi (Baillonella toxisperma), and movingui (Distemonanthus benthamia), Ind. Crops Prod. 41 (2013) 71–77.
[34] D. Shahwar, M.A. Raza, Antioxidant potential of phenolic extracts of *Mimusops elengi*, Asian Pacific J. Trop. Biomed. 2 (2012) 547–550.
[35] E. Talla, Z. Stojanović, A. Segura Carretero, D. Arráez Román, I. Borrás, I. Vasiljević, Antioxidant Activity and a New Ursane-type Triterpene from *Vitellaria paradoxa* (Sapotaceae) Stem Barks, Eur. J. Med. Chem. 16 (2016) 1–20.
[36] M. Dent, V. Dragović-Uzelac, M. Penić, M. Brñić, T. Bosiljkov, B. Levaj, The effect of extraction solvents, temperature and time on the composition and mass fraction of polyphenols in dalmatian wild sage (*Salvia officinalis* L.) extracts, Food Technol. Biotechnol. 51 (2013) 84–91.
[37] S. Ferarsa, W. Zhang, N. Moulai-Mostefa, L. Ding, M.Y. Jaffrin, N. Grimi, Recovery of anthocyanins and other phenolic compounds from purple eggplant peels and pulps using ultrasonic-assisted extraction, Food Bioprod. Process. 109 (2018) 19–28.
[38] N.R. Jawade, A.R. Chavan, Ultrasonic -assisted extraction of aloin from aloe vera gel, Procedia Eng. 51 (2013) 487–493.
[39] Y. Linlin, C. Yungang, Z. Guangyao, Optimization of subcritical water extraction of phenolic antioxidants from pomegranate (*Punica granatum* L.) peel by response surface methodology, Anal. Methods 1 (2013) 8–14.
[40] S. Trapani, C. Breschi, L. Cecchi, L. Guerrini, N. Mulinacci, A. Parenti, B. Zanoni, Indirect indices of oxidative damage to phenolic compounds for the implementation of olive paste malaxation optimization charts, J. Food Eng. 207 (2017) 24–34.
[41] A.M. Cvetkovic, T. Jurina, D. Valinger, A. Jurinjak Tusek, M. Benkovic, J. Gajdos Kljusuric, The estimation of kinetic parameters of the solid-liquid extraction process of the lavender flower (*Lavandula x hybrida* L.), Croatian, J. Food Sci. Technol. 10 (2018) 64–72.
[42] J. Wang, J. Wang, J. Ye, S.K. Vanga, V. Raghavan, Influence of high-intensity ultrasound on bioactive compounds of strawberry juice: Profiles of ascorbic acid, phenolics, antioxidant activity and microstructure, Food Control 96 (2019) 128–136.
[43] E. Müsellim, M.H. Tahir, M.S. Ahmad, S. Ceylan, Thermokinetic and TG/DSC-FTIR study of pea waste biomass pyrolysis, Appl. Therm. Eng. 137 (2018) 54–61.