EFFECTS OF ULTRASOUND-ASSISTED EXTRACTION CONDITIONS ON THE RECOVERY OF TRITERPENOIDS AND ANTIOXIDANT ACTIVITY OF *Curculigo orchioides* Gaertn RHIZOMES

Phan Van Man[1], Dang Thi Cuong[1], Pham Thi Huu Hanh[2], Tran Chi Hai[3]*

[1]*Ba Ria - Vung Tau College of Technology*

[2]*Ba Ria - Vung Tau University*

[3]*Ho Chi Minh City University of Food Industry*

*Email: *haitc@hufi.edu.vn*

Received: 7 September 2022; Accepted: 28 October 2022

ABSTRACT

The objective of this study is to investigate the effects of ultrasound-assisted extraction (UAE) conditions on the recovery of triterpenoids and antioxidant activity of *C. orchioides* rhizomes using response surface methodology (RSM). The UAE parameters under study included extraction temperature (30 - 60°C), ultrasonic power (150 - 350 W), and extraction time (5-45 min). The quadratic response models were generated, and statistical analysis was performed to validate the models. The results showed that the recovery of triterpenoids and antioxidant activity (ABTS radical scavenging activity) were significantly affected by temperature and time process ($p<0.05$). The optimal conditions for the recovery of triterpenoids and ABTS were 44.90 min, 50.63 °C, and ultrasonic power of 322.50 W. Under these conditions, the experimental results agreed with the predicted values. The UAE extraction technique was found to be more efficient in the extraction of triterpenoids and antioxidant capacity in comparison with the conventional extraction method (SE). Furthermore, GC-MS analysis of optimized extract showed the presence of six components in the extracts which related to the antioxidant activity. It could be suggested that the UAE method is a promising, efficient, and green technology for the extraction of bioactive compounds from *C. orchioides* rhizomes.

Keywords: C. orchioides, triterpenoids, ultrasound-assisted extraction (UAE), response surface methodology, GC-MS analysis.

1. INTRODUCTION

Curculigo orchioides Gaertn (*C. orchioides*) is a small genus in the family of Hypoxidaceae. It is widely used as an important medicinal plant in East-Asian countries. The rhizomes of *C. orchioides* are rich in triterpenoids, which have strong antioxidant properties and antimicrobial activity [1-3]. Pharmacological studies showed that *C. orchioides* have been used for the treatment of diseases like limb numbness, jaundice, asthma, and urinary [1, 4, 5]. They are also used as a potent immunomodulatory and aphrodisiac, estrogenic and sexual behavior-modifying [6], and anti-depression [7].

The ultrasound-assisted extraction technique is commonly used to extract bioactive compounds from a wide range of materials, for example, apple [8], pomegranate flowers [9], *Rosmarinus officinalis*, and *Curcuma Zedoaria* leaves [10]. The advantage of ultrasonic extraction is less time-consuming, high extraction efficiency, and lower energy consumption [10, 11]. For UAE,

various factors (i.e., the ultrasonic power, extraction temperature, ultrasonic irradiation time, and solid-liquid ratios) affect the extraction efficiency and antioxidant activity of the extracts. It is therefore important to optimize these process variables to achieve maximum yield of bioactive compounds from *C. orchioides* and reduce the number of experimental trials. RSM also serves as a visual aid to indicate the optimization region [11-13]. However, to the best of our knowledge, there are no studies dealing with the application of the UAE process for extracting triterpenoids from *C. orchioides*.

This study aims to: (a) optimize the UAE process parameters (temperature, ultrasonic power, and time) to obtain high extraction efficiency of triterpenoids from *C. orchioides* rhizomes; b) compare the extraction efficiency of the optimized UAE conditions and the soxhlet extraction (SE) method; and (c) identify the bioactive compounds of *C. orchioides rhizomes* extracts using gas chromatography-mass spectrometry (GC-MS).

2. MATERIALS AND METHODS

2.1. Materials

Curculigo orchioides Gaertn rhizomes were collected from Quang Ngai province, Vietnam after eight months from planting. They were then washed with tap water and dried at 55°C for 5 hours to achieve 5-7% of moisture content. After that, the dried rhizomes were milled to a fine powder. The fine powder was passed through a steel mesh sieve with a pore size of 1 mm and placed in airtight plastic bags. The samples were stored at 4°C until used for further analysis.

All chemicals used in this study were supplied by Merck (Darmstadt, Germany), and they were analytical grade.

2.2. Ultrasound assisted extraction

In this study, ethanol was selected as the extraction solvent and ultrasound-assisted extraction (UAE) was carried out using an ultrasonic generator (750 W, VCX750 Vibracell; Sonic & Materials, Inc., Newtown, CT, USA). The samples (10 grams) were mixed with 100 mL of aqueous ethanol (70% ethanol concentration, 1:20 solid/liquid ratio) based on the preliminary survey showing a suitable extraction condition. The extraction was performed under three series.

First series: The ultrasonic power levels were adjusted to 150, 250, and 350 W. The extraction temperature and extraction time were fixed at 45°C for 25 min.

Second series: The ultrasonic power level was selected as 250 W. The extraction temperature was various: 30, 45, 60°C. The extraction time was fixed at 25 min.

Third series: The ultrasonic power level and time were fixed at 250 W and 25 min, respectively. The extraction time was varied from 5 to 45 min.

At the end of the extraction, the extracts were filtered, and the solvent was evaporated using vacuum evaporation. The extracts were stored at 4ºC for further analysis.

2.3. Optimisation of UAE extraction conditions

RSM based on Box Behnken design was used to find the optimal extraction condition using Design Expert software version 8.0.6.1. The three independent extraction parameters included extraction temperature (X_1: 30 – 60 °C), ultrasonic power (X_2: 150 – 350 W), and extraction time (X_3: 5 – 45 min). The individual independent variables were coded as −1, 0, and +1 (low, center, and high) (Table 1). The experimental design consisted of 17 runs, including three center point replicates and two responses (Y_1 and Y_2) (Table 2).

Table 1. Independent variables and their levels used in the response surface design

Factor	Code units	Variables levels		
		−1	0	+1
Extraction temperature (°C)	X_1	30	45	60
Sonication power (W)	X_2	150	250	350
Extraction time (min)	X_3	5	25	45

Table 2. Box Behnken design of experimental and predicted values for the recovery of triterpenoids and antioxidant capacity of *C. orchioides* rhizomes extracted using UAE

Exp No	Temp-erature (X_1, °C)	Sonic-ation power (X_2, W)	Sonic-ation time (X_3, min)	Experimental value (Y_1, mg UA/g d.m)	Predicted value (Y_1, mg UA/g d.m)	Experimental value (Y_2, mg TE/g)	Predicted value (Y_2, mg TE/g)
1	45	150	45	120.8±1.65	126.8±1.1	45.01±0.59	45.64±1.51
2	45	250	25	159.4±1.31	160.1±1.33	79.53±1.65	80.58±1.05
3	45	350	5	119.9±2.01	123.4±2.24	43.67±1.73	45.13±1.02
4	45	250	25	150.1±1.15	153.3±1.19	68.0±1.65	67.91±1.09
5	60	250	45	129.9±1.09	132.9±1.23	52.98±0.93	53.01±1.01
6	45	250	25	159.0±0.89	159.5±0.81	68.09±1.52	66.19±1.07
7	30	250	45	120.9±1.21	123.9±1.11	45.15±1.56	49.05±0.99
8	45	350	45	148.0±1.45	149.2±1.55	59.06±0.56	60.16±1.06
9	45	250	25	150.5±1.50	153.1±1.54	69.01±1.85	69.31±1.01
10	30	250	5	100.7±1.55	106.5±1.31	38.02±2.05	39.23±1.55
11	30	150	25	89.0±0.98	91.0±0.99	36.46±1.85	37.49±0.95
12	60	250	5	129.8±0.99	130.3±1.09	54.15±1.48	55.41±0.98
13	60	350	25	121.4±1.13	124.4±1.32	47.15±1.52	49.19±1.02
14	45	150	5	129.8±1.41	134.3±1.14	54.98±1.55	55.01±1.03
15	60	150	25	114.9±1.19	115.4±1.12	40.99±1.59	41.02±0.99
16	30	350	25	119.8±1.38	123.3±1.39	42.78±1.04	43.48±1.14
17	45	250	25	155.6±0.95	159.5±0.99	68.99±1.52	69.12±1.02

The multiple regression equation was used to fit the quadratic polynomial equation (Eq. 1) based on the experimental data:

$$Y = \beta_0 + \beta_1 X_1 + \beta_2 X_2 + \beta_3 X_3 + \beta_{11} X_1^2 + \beta_{22} X_2^2 + \beta_{33} X_3^2 + \beta_{12} X_1 X_2 + \beta_{23} X_2 X_3 + \beta_{13} X_1 X_3 \quad (1)$$

where, Y is the predicted response for the recovery of triterpenoids, or ABTS radical scavenging capacity; X_1, X_2, X_3 are the independent variables, β_0 is a constant, β_1, β_2, β_3, β_{11}, β_{22}, β_{33} and β_{12}, β_{23}, β_{13} represent the regression coefficients of the linear, quadratic, and interaction effects respectively.

2.4. Conventional solvent extraction (SE)

Soxhlet extraction was used as the standard technique being also an ultrasound-assisted extraction Murali and Kuttan (2016) [14]. The powdered *C. orchioides* was extracted with

70% ethanol (v/v) in a Soxhlet apparatus for 24 hours. The extracts were centrifuged at 5500 rpm for 5 min, and the residual solvent was evaporated using a rotary evaporator (550 mbar, 2 hours). The solutions were collected and stored below 4°C for further analysis.

2.5. Determination of total triterpenoid content (TC)

The total triterpenoid content (TC) was determined by the colourimetric assay following the procedure outlined in Tran et al. (2021) with some modifications [15]. Briefly, a 0.16ml extract was mixed with 0.4 mL vanillin/glacial acetic acid (5% w/v) in the test tube, and then added 1.0 mL of perchloric acid solution. The mixture was incubated in a water bath at 60°C for 30 min. The mixture was rapidly cooled and added 5.0 mL of glacial acetic acid. The absorbance at 573 nm was measured using a UV spectrophotometer/NIR (Shimazu, UV-2600, Japan). For triterpenoid analysis, ursolic acid (0.1–1.0 g/100 mL in methanol) was used as the standard solution. The results were expressed in mg of ursolic acid equivalents per g of d.m. All samples were analysed in triplicate.

2.6. ABTS radical scavenging assay

The ABTS assay was described according to Weremfo's method with some modifications [16]. 7 mM ABTS solution was mixed with 2.45 mM potassium persulfate and then incubated at room temperature for 16 h. After that, the mixture (1 mL) was diluted by mixing with ethanol (60 mL) to adjust the absorbance to 0.70 ± 0.01 at 734 nm. The extract (1 mL) was allowed to react with 1 mL of ABTS solution and stored in a dark place for 10 min. The absorbance of the mixture was taken at a wavelength of 734 nm using a UV-Vis spectrophotometer (Jasco V630; Kyoto, Japan). Trolox (TE) was used as a standard, and the results are expressed as mg TE/g d.m.

2.7. Gas chromatography–mass spectrometry (GC-MS) analysis

GC-MS analysis was conducted following the previous method with slight modifications [17]. The extracts were dissolved in ethanol and injected into an Agilent 7890A GC system equipped with an MS (Agilent technologies). The separation of the samples was conducted on a DB-5MS column (30 m length × 0.25 mm diameter ×0.25 μm film thickness). The GC-MS operating conditions were as follows: oven temperature increased from 50°C to 260°C at a rate of 10°C/min and then hold isothermally for 10 min. The sample was injected in the splitless mode with 2 μL, and helium, a carrier gas was at 1 mL/min. The mass spectrometer was operated at 70 eV, and the total running time of the GC was 50 min. The compounds identified by GC-MS analysis were compared with compounds in NIST library data.

2.8. Statistical analysis

The significance of the data was evaluated using analysis of variance (ANOVA) followed by Tukey's HSD (honest significant difference) test with $\alpha = 0.05$. Design Expert software version 8.0.6.1 (Stat-Ease, Minneapolis) was employed for the regression analysis and optimization. All experiments were performed in triplicate and presented as mean ± SD (standard deviation).

3. RESULTS AND DISCUSSION

3.1. Effects of extraction conditions on the recovery of triterpenoids and ABTS radical scavenging activity

The influence of extraction conditions on the recovery of triterpenoids and antioxidant acitvity of *C. orchioides* extract were presented in Fig 1. It can be seen that the recovery of triterpenoids and antioxidant activity varied from 120.8 to 151.4 mg UA/g d.m, and 54.14 to

79.93 mg TE/g d.m, respectively. Under UAE, given identical sonication time (25 min), increased extraction temperature (30, 45, 60 °C) and ultrasonic power (150, 250, 350 W) significantly increased the recovery of triterpenoids and their antioxidant activity in the extracts ($p<0.05$). Specifically, when the ultrasound power increased from 150 W to 250 W, the the recovery of triterpenoids and antioxidant activity increased from 120.8 to 151.4 mg UA/g and 54.14 to 79.93 mg TE/g, respectively (Fig 1a). In Fig 1b, the extraction temperature changed from 30 to 45°C, the triterpenoids and their antioxidant activity significantly increased from 139.5 to 153.3 mg UA/g and 62.98 to 79.93 mg TE/g, respectively. The reason was that an increase in ultrasonic power and temperature processes promoted cell membrane breaking, thus enhancing the dissolution of bioactive compounds into the solvent. Similar findings have been reported by Oludemi et al. (2018) for the extraction of bioactive compounds from *Ganoderma lucidum* [18]. However, the contents of triterpenoids and antioxidant activity values slightly decrease as ultrasonic power and temperature processes increased from 250 to 350W and 45 to 60°C, respectively. Phan et al. (2020) reported that the cell membrane may break into small fragments under high temperature and ultrasonic power processes, which leads to increase impurities being extracted, thus a decrease in the recovery of bioactive compounds extracted [19]. Tran et al. (2021) reported that high temperature (over 60°C) destroyed the triterpenoid molecular structure of the five rings, resulting in a low level of triterpenoids and making these compounds less effective [15].

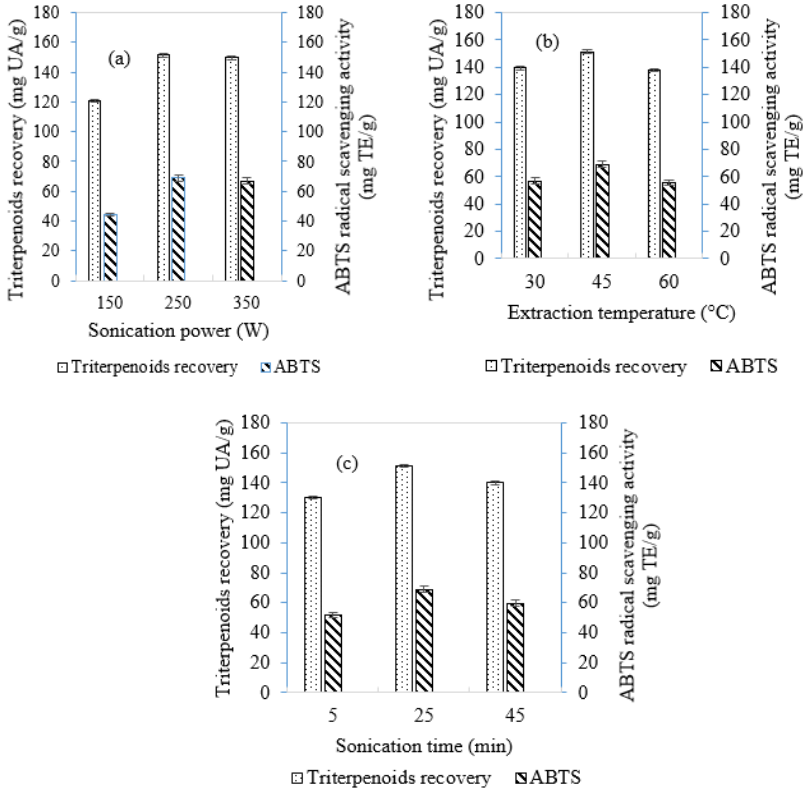

Figure 1. Effects of ultrasound-assisted extraction conditions: (a) ultrasonic power; (b) extraction temperature; and (c) sonication time on the recovery of triterpenoids and ABTS activity.

The results also noted that the duration of treatment affected triterpenoids in all treated samples; 25 min resulted in extraction of higher triterpenoids and antioxidant activity compared to 5 and 45 min at the same sonication power and extraction temperature (Fig 1c). Therefore, the independent variables (temperature, ultrasonic power, and extraction time) have

significant effects on the recovery of triterpenoids and the ABTS radical scavenging activities of *C. orchioides* extract.

3.2. Model Fitting

In this study, the Box Behnken design under the RSM was used to study the interaction among these parameters including extraction temperature (X_1), sonication power (X_2), and extraction time (X_3) to optimize the recovery of triterpenoids and antioxidant activity (Y_1 and Y_2). The adequacy and reliability of the regression model were presented in Table 3. This study found that the respective values of R^2 and adj-R^2, and predict R^2 for the recovery of triterpenoids (0.98, 0.95, and 0.83, respectively) and ABTS (0.95, 0.88, and 0.70, respectively) were close 1, which indicated good agreement between the experimental and predicted results [19,20]. The high p-values of lack of fit tests proved that the models are generated well. Furthermore, our results also showed that the regression model was highly significant with a high F-value (35.47 and 14.40) and a small p-value (<0.01). Thus, the results showed that the two RSM models were highly significant ($p<0.05$) for triterpenoids and ABTS radical scavenging activity, and the response of each variable is explained as follows.

3.3. Optimization of extraction conditions

The individual, interactions, and quadratic effects of extraction temperature (X1), sonication power (X2), and extraction time (X3) level on the recovery of triterpenoids and ABTS are shown in Table 3. For the triterpenoids response (Y1), the individual parameters including X1, X2, and X3 positively influenced Y1, and the most significant factor was X1. The interaction of X2×X3 also had a positive influence on Y1. These results implied that X1, X2, X3, and X2×X3 were effect by increasing Y1. However, the interaction effect of X1×X3 on Y1 was insignificant ($p>0.05$) and thus eliminated. The final mathematical model was expressed by the following equation:

$$Y_1 = -286.59 + 12.56X_1 + 0.99X_2 + 0.86X_3 - 0.18X_1^2 - 0.0018X_2^2 - 0.02X_3^2 - 0.004X_1 \times X_2 - 0.046X_2 \times X_3 \quad (2)$$

For ABTS response (Y_2), the linear terms of X_2 and X_3 and interaction terms of $X_1 \times X_2$ and $X_2 \times X_3$ were insignificant ($p>0.05$), indicating that the linear terms of X_2 and X_3 and interaction terms of $X_1 \times X_2$ and $X_2 \times X_3$ did not affect Y_2. Its mathematical model was described in Eq. 3:

$$Y_2 = -172.06 + 6.85X_1 + 0.04X_1 \times X_3 - 0.08X_1^2 - 0.0013X_2^2 - 0.018X_3^2 \quad (3)$$

Furthermore, the influences and their mutual interactions of three independent variables (X_1, X_2, and X_3) on Y_1 and Y_2 can also be visualized on the generated 3D response surface curves. As shown in Fig. 2, it can be seen that extraction temperature (X_1) was the most significant factor that affected Y_1, while Y_2 depended on extraction temperature (X_1) and extraction time (X_3). The longer the extraction time, the better the ABTS scavenging activity of the extract. Meanwhile, at high levels of the extraction time increasing extraction temperature (50–60°C) resulted in the decrement of the triterpenoid content and ABTS value (Figs. 2 a-b). A similar observation was reported by Tran et al. from *Ganoderma lucidum* [15].

The optimal results using Design Expert software indicated that the optimal conditions recommended in the extraction process of *C. orchioides* rhizomes were temperature of 50.63 °C, sonication power of 322.50 W, and extraction time of 44.90 min. Under these conditions, the contents of predicted triterpenoids (Y_1) and the ABTS radical scavenging activity (Y_2) were 161.17mg UA/g d.m and 79.69 mg TE/g d.m, respectively. However, considering the practical operability, the optimal extraction conditions are modified as follows: extraction temperature of 51 °C, ultrasonic power of 323 W, and sonication time of 45 min. The experiments were then conducted under the modified optimal conditions, and the results showed that the experimental values of triterpenoids and ABTS radical scavenging

activity were 163.41 ± 1.27 mg UA/g d.m (n = 3), and 82.12± 1.12 mg TE/g d.m (n = 3), which were statistically close to predicted values at 95% confidence level, and consequently confirming the validity of the predicted extraction models.

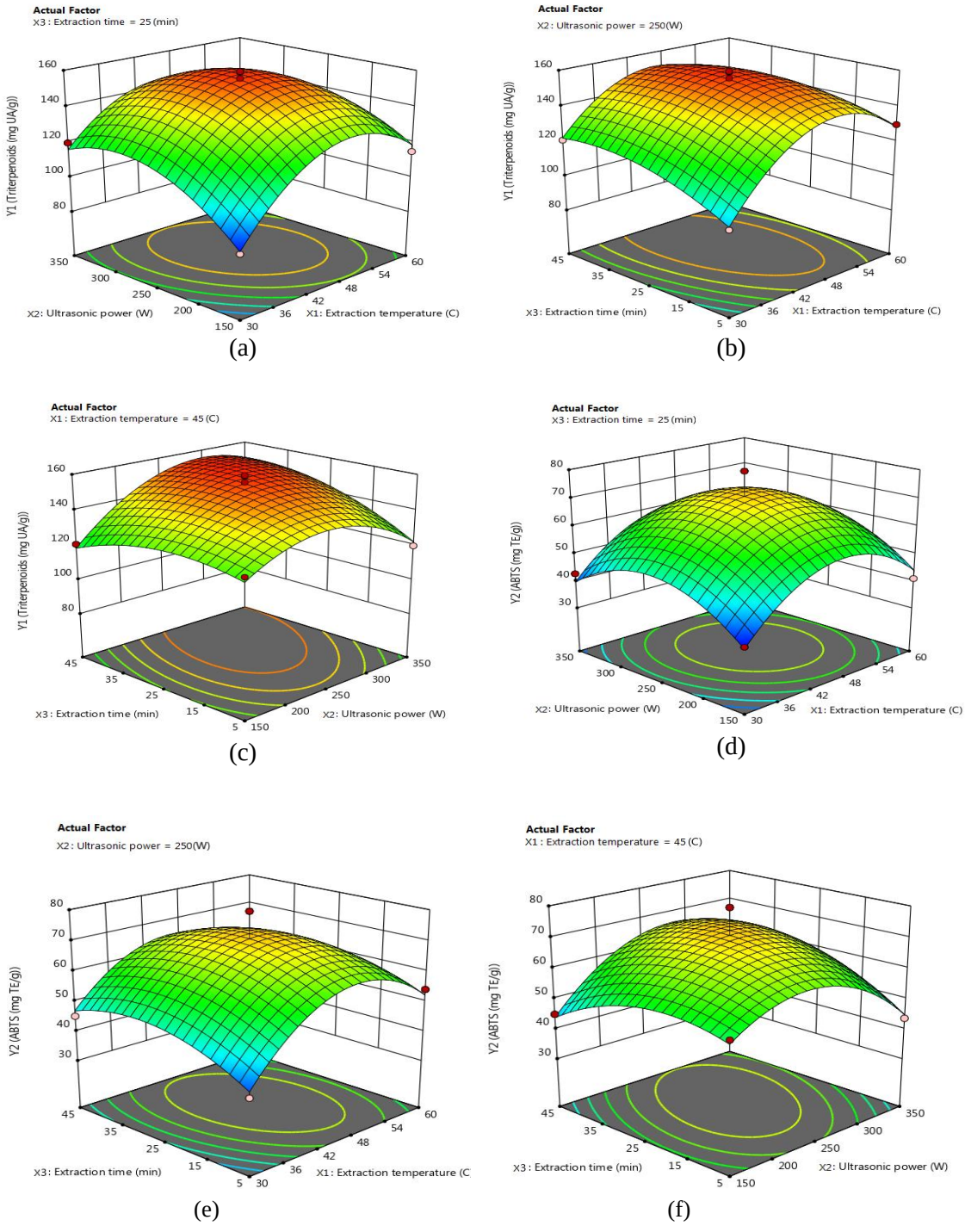

Figure 2. 3D surface plot of the effects of extraction parameters: (a and d) extraction temperature and ultrasonic power; (b and e) extraction temperature and extraction time; (c and f) extraction time and ultrasonic powder on the recovery of triterpenoids and ABTS.

Table 3. Analysis of variance for fitted quadratic model of extraction of the recovery of triterpenoids and ABTS radical scavenging activity

Source	**Sum of Squares**	**df**	**Mean Square**	**F-value**	**p-value**	
The recovery of triterpenoids (Y_1)						
Model	6574.55	9	730.51	35.47	< 0.0001	significant
X_1	537.92	1	537.92	26.12	0.0014	
X_2	372.65	1	372.65	18.09	0.0038	
X_3	194.05	1	194.05	9.42	0.0181	
X_1X_2	147.62	1	147.62	7.17	0.0317	
X_1X_3	101.00	1	101.00	4.90	0.0624*	
X_2X_3	344.10	1	344.10	16.71	0.0046	
X_1^2	2950.71	1	2950.71	143.27	< 0.0001	
X_2^2	1241.66	1	1241.66	60.29	0.0001	
X_3^2	277.79	1	277.79	13.49	0.0079	
Residual	144.17	7	20.60			
Lack of Fit	64.23	3	21.41	1.07	0.4554*	not significant
Pure Error	79.95	4	19.99			
R^2	0.97	**Predicted R^2**	0.83			
Adjusted R^2	0.95					
ABTS radical scavenging activity (Y_2)						
Model	2555.85	9	283.98	14.40	0.0010	significant
X_1	134.97	1	134.97	6.84	0.0346	
X_2	28.96	1	28.96	1.47	0.2650*	
X_3	16.19	1	16.19	0.8206	0.3951*	
X_1X_2	0.0064	1	0.0064	0.0003	0.9861*	
X_1X_3	17.22	1	160.78	8.15	0.0245	
X_2X_3	160.78	1	17.22	0.8730	0.3812*	
X_1^2	1076.82	1	1076.82	54.58	0.0002	
X_2^2	699.26	1	699.26	35.45	0.0006	
X_3^2	215.67	1	215.67	10.93	0.0130	
Residual	138.09	7	19.73			
Lack of Fit	40.25	3	13.42	0.5484	0.6755*	not significant
Pure Error	97.85	4	24.46			
R^2	0.95	**Predicted R^2**	0.70			
Adjusted R^2	0.88					

** Statistically insignificant at p>0.05.*

The extraction efficiency and antioxidant activities of UAE extract at the optimal extraction conditions were compared with the SE method (Fig. 3). The results showed that the triterpenoids content and ABTS of the extract obtained using UAE were nearly 2-fold higher than that of SE ($p<0.05$). Compared with the SE method, the UAE method is less time-consuming, thus reducing solvent usage. Therefore, UAE is considered a promising extraction method for extracting triterpenoids from *C. orchioides* rhizomes.

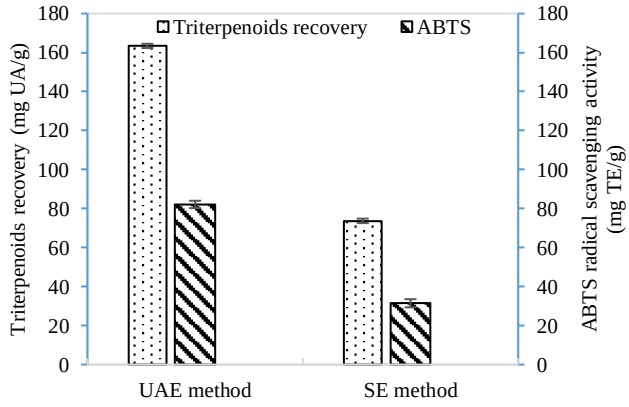

Figure 3. Comparison of the extraction efficiency of UAE with SE method.

3.4. GC-MS Analysis of phenolic compounds in *C. orchioides* extract

The bioactive compounds of *C. orchioides* rhizomes extracts were analyzed using the GC-MS method. The results showed that 6 compounds (triterpenoids, phenolics, and aromatic compounds) were screened under different retention times (RT) (Fig. 4, Table 4). The most abundant compounds identified in the extracts were curculigosaponin G, and curculigosaponin I, followed by curculigoside B while the least abundant compound was 13-Octadecenal. Our results were comparable with the qualitative studies of Wang, Li, and Li (2021) and Niu et al. (2020) [1,21]. These authors identified that curculigosaponin G, curculigosaponin I, and curculigoside B are the most abundant bioactive compounds of *C. orchioides*. According to some previous studies, curculigoside B and ethyl iso-allocholate exhibited antimicrobial, antiosteoporotic, and control of human tumors [3,22]. In addition, curculigosaponin G and curculigosaponin I also exhibited immunomodulatory effects, and antioxidant activities [14,23,24]. This study found that the identified compounds could be responsible for the ABTS radical scavenging activity of the extract obtained by the UAE method. However, some unknown compounds were detected at a retention time of 11.45, 25.61, and 27.54 min, respectively, this will require further analysis for characterization.

Table 4. Quantification of biochemical compounds of the *C. orchioides* rhizomes extract

Peak	Compound	RT [min]	Formula	Peak area (%)	Chemical type
1	Curculigoside B	7.041	$C_{21}H_{24}O_{11}$	16.12	Phenolic glycosides
2	-	11.45	-	1.56	Unknow compound
3	13-Octadecenal	11.89	$C_{18}H_{34}O$	14.34	Aromatic compound
4	Curculigosaponin G	12.87	$C_{18}H_{18}O_6$	18.64	Triterpene glycosides
5	Curculigosaponin H	16.14	$C_{22}H_{44}O_2$	14.45	Triterpene glycosides
6	Curculigosaponin I	19.79	$C_{22}H_{44}O_2$	17.54	Triterpene glycosides
7	Ethyl iso-allocholate	21.04	$C_{23}H_{26}O_{10}$	15.56	Phenolic glycosides
8	-	25.61	-	0.94	Unknow compound
9	-	27.54	-	0.85	Unknow compound

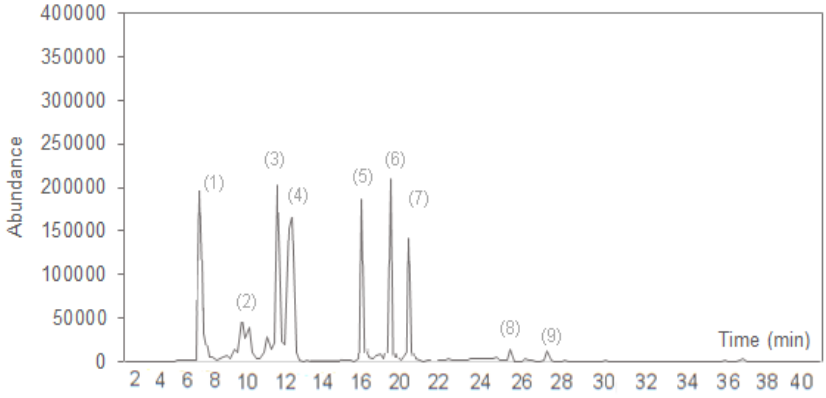

Figure 4. GC-MS profile of the *C. orchioides* rhizomes extract

4. CONCLUSION

In this study, the RSM based on the Box-Behnken design was successfully used to study the optimum extraction condition (temperature, ultrasonic power, and extraction time) for the recovery of triterpenoids and ABTS of *C. orchioides*. The results showed that the optimized extraction conditions included a temperature of 50.63°C, ultrasonic power of 322.50W, and sonication time of 44.90 min. Under these optimal conditions, the experimental recovery of triterpenoids and their antioxidant activity were 163.41 ± 1.27 mg UA/g d.m (n = 3) and 82.12± 1.12 mg TE/g d.m (n = 3), respectively, which was closely agreed with the predicted values. The GC-MS analysis indicates that the extracts are composed of triterpene and phenolic glycoside forms and have significant antioxidant activity. According to these findings, UAE was found to be the best efficient technology in the exploitation of bioactive compounds from *C. orchioides* rhizomes.

Conflict of Interest: We have no conflict of interest.

REFERENCES

1. Wang Y., Li J., and Li N. - Phytochemistry and Pharmacological Activity of Plants of Genus *Curculigo*: An Updated Review since 2013. Molecules **26** (11) (2021) 3396.
2. Zabidi N. A., Ishak N. A., Hamid M., and Ashari S. E. - Subcritical Water Extraction of Antioxidants from *Curculigo latifolia* Root. Journal of Chemistry **2019** (2019) 8047191.
3. Asif M. - A review on phytochemical and ethnopharmacological activities of *Curculigo orchioides.* Mahidol University Journal of Pharmaceutical Sciences **39** (3-4) (2012) 1-10.
4. Ramchandani D., Ganeshpurkar A., Bansal D., Karchuli M. S., and Dubey N. - Protective Effect of *Curculigo orchioides* Extract on Cyclophosphamide-Induced Neurotoxicity in Murine Model. Toxicology International **21** (3) (2014) 232–35.
5. Zhang B., Lu C., Xu Z., Guo H., Zhang G., and Hao Y. - *Curculigo orchioides* Polysaccharides Extraction, Characterization, and Their Protective Effects against Femoral Head Necrosis. Arabian Journal of Chemistry **15** (1) (2022) 103414.
6. Chauhan N. S., Rao Ch. V., and Dixit V. K. - Effect of *Curculigo orchioides* Rhizomes on Sexual Behaviour of Male Rats. Fitoterapia **78** (7–8) (2007) 530-534.

7. Ge J. F., Gao W. Ch., Cheng W. M., Lu W. L., Tang J., Peng L., Li N., and Chen F. H. - Orcinol Glucoside Produces Antidepressant Effects by Blocking the Behavioural and Neuronal Deficits Caused by Chronic Stress. European Neuropsychopharmacology **24** (1) (2014) 172–80.
8. Virot M., Tomao V., Bourvellec C. L., Renard C. M. C. G., and Chemat F., Towards the Industrial Production of Antioxidants from Food Processing by-Products with Ultrasound-Assisted Extraction. Ultrasonics Sonochemistry **17** (6) (2010) 1066–74.
9. Wu W., Jiang S., Liu M., and Tian S. - Simultaneous Process Optimization of Ultrasound-Assisted Extraction of Polyphenols and Ellagic Acid from Pomegranate (*Punica Granatum L.*) Flowers and Its Biological Activities. Ultrasonics Sonochemistry **80** (2021) 105833.
10. Azahar N. F., Gani S. S. A., and Mokhtar N. F. M. - Optimization of Phenolics and Flavonoids Extraction Conditions of *Curcuma Zedoaria* Leaves Using Response Surface Methodology. Chemistry Central Journal **11** (1) (2017) 1–10.
11. Li Z. J., Yang F. J., Yang L., and Zu Y. G. - Ultrasonic Extraction of Oil from *Caesalpinia Spinosa* (Tara) Seeds. Journal of Chemistry **2016** (2016) 1–7.
12. Phan V.M., Junyusen T., Liplap P., and Junyusen P. - Effects of Ultrasonication and Thermal Cooking Pretreatments on the Extractability and Quality of Cold Press Extracted Rice Bran Oil. Journal of Food Process Engineering **42** (2) (2019) 1–8.
13. Carrera C., Ruiz-Rodríguez A., Palma M., and Barroso C. G. - Ultrasound Assisted Extraction of Phenolic Compounds from Grapes. Analytica Chimica Acta **732** (2012) 100–104.
14. Murali V. P., and Kuttan G. - *Curculigo Orchioides* Gaertn Effectively Ameliorates the Uro- and Nephrotoxicities Induced by Cyclophosphamide Administration in Experimental Animals. Integrative Cancer Therapies **15**(2) (2016) 205–15.
15. Tran D. D., Nguyen D. V., Phan L. T. M. , Nguyen T. L., Vuong H. T., Nguyen T. T. L., Nguyen T. K. N. - The Application of Ethanolic Ultrasonication to Ameliorate the Triterpenoid Content Extracted from Vietnamese *Ganoderma Lucidum* with the Examination by Gas Chromatography. Chemistry Select **6** (10) (2021) 2590–2606.
16. Weremfo A., Adulley F., and Adarkwah-yiadom M. - Simultaneous Optimization of Microwave-Assisted Extraction of Phenolic Compounds and Antioxidant Activity of Avocado (*Persea Americana Mill.*) Seeds Using Response Surface Methodology. Journal of Analytical Methods in Chemistry **2020** (2020) 7541927.
17. Daffodil E. D., Uthayakumari F. K., Mohan V. R. - GC-MS determination of bioactive compounds of *Curculigo orchioides* Gaertn. Science Research Reporter **2**(3) (2012) 198-201.
18. Oludemi T., Barros L., Prieto M. A., Heleno S. A., Barreiro M. F., and Ferreira I. C. F.R. - Extraction of Triterpenoids and Phenolic Compounds from: *Ganoderma Lucidum*: Optimization Study Using the Response Surface Methodology. Food and Function **9** (1) (2018) 209–226.
19. Phan V.M., Junyusen T., Liplap P., and Junyusen P. - Optimization and Kinetics of Ultrasound-Assisted Solvent Extraction of Gamma Oryzanol from Dried Rice Bran Soapstock. Agriculture and Natural Resources **54** (2020) 309-316.
20. Zhao Y., Hou Y., Tang G., Cai E., Liu S., Yang H., Zhang L., and Wang S. - Optimization of Ultrasonic Extraction of Phenolic Compounds from *Epimedium brevicornum* Maxim Using Response Surface Methodology and Evaluation of Its

Antioxidant Activities in Vitro. Journal of Analytical Methods in Chemistry **2014** (2014) 864654.

21. Niu C., Zhang Z. Z., Yang L. P., Zhai Y. Y., Li S. N., Hao S. Y., Chen X. Y., Wang J. H., and Wang Z. H. - Chemical Constituents of *Curculigo orchioides*. Chemistry of Natural Compounds **56** (5) (2020) 957–59.
22. Boligon A. A., Piana M., Schawnz T. G., Pereira R. P., Rocha J. B. T., and Athayde M. L. - Chromatographic Analysis and Antioxidant Capacity of *Tabernaemontana catharinensis*. Natural Product Communications **9** (1) (2014) 61–64.
23. Singh R., and Gupta A. K. - Antimicrobial and Antitumor Activity of the Fractionated Extracts of Kalimusli (*Curculigo orchioides*). International Journal of Green Pharmacy **2** (1) (2008) 34.
24. Bafna A. R., and Mishra S. H. - Immunostimulatory Effect of Methanol Extract of *Curculigo orchioides* on Immunosuppressed Mice. Journal of Ethnopharmacology **104** (1–2) (2006) 1–4.

TÓM TẮT

ẢNH HƯỞNG CỦA SÓNG SIÊU ÂM ĐẾN QUÁ TRÌNH THU NHẬN TRITERPENOIDS VÀ HOẠT TÍNH CHỐNG OXY HÓA CỦA RỄ SÂM CAU *C. orchioides*

Phan Văn Mẫn[1], Đặng Thị Cương[1], Phạm Thị Hữu Hạnh[2], Trần Chí Hải[3*]
[1]*Trường Cao đẳng Kỹ thuật Công nghệ Bà Rịa - Vũng Tàu*
[2]*Trường Đại học Bà Rịa - Vũng Tàu*
[3]*Trường Đại học Công nghiệp Thực phẩm TP.HCM*
*Email: *haitc@hufi.edu.vn*

Mục tiêu của nghiên cứu này là nghiên cứu ảnh hưởng của sự hỗ trợ sóng siêu âm đến quá trích ly để thu hồi triterpenoids và hoạt tính kháng oxy hóa của rễ sâm cau *C. orchioides* sử dụng phương pháp bề mặt đáp ứng (RSM). Những thông số của quá trình trích ly có sự hỗ trợ của siêu âm (UAE) trong nghiên cứu đã được khảo sát gồm nhiệt độ trích ly (30 - 60°C), công suất siêu âm (150 - 350 W), và thời gian siêu âm (5 - 45 phút). Mô hình bề mặt bậc hai đã được thiết lập và xử lý thống kê được sử dụng để đánh giá mô hình. Kết quả cho thấy hiệu suất thu nhận triterpenoids và hoạt tính kháng oxy hóa (khả năng bắt gốc tự do ABTS) bị ảnh hưởng đáng kể bởi nhiệt độ và thời gian ($p<0.05$). Điều kiện tối ưu cho quá trình thu hồi triterpenoids và ABTS là nhiệt độ 50,63^{o}C, công suất 322,5W và thời gian siêu âm 44,9 phút. Tại điều kiện này, kết quả thực nghiệm tương tự với kết quả dự đoán trên mô hình. So với phương pháp trích ly truyền thống thì phương pháp UAE cho giá trị hàm lượng triterpenoid và ABTS cao hơn. Khi phân tích GC-MS, dịch chiết có 6 hợp chất có khả năng kháng oxy hóa. Điều này có thể cho thấy phương pháp UAE là một phương pháp đầy hứa hẹn, hiệu quả và kỹ thuật xanh để trích ly các hợp chất có hoạt tính sinh học từ rễ sâm cau *C. orchioides*.

Từ khóa: C. orchioides, triterpenoids, trích ly hỗ trợ sóng siêu âm (UAE), phương pháp bề mặt đáp ứng, phân tích GC-MS.