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Research Article

Investigation of conversion of sunflower oil production wastes to high value compounds by supercritical CO_2

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ABSTRACT

The evaluation of wastes from edible oil production industry has increasing importance because of resources deficiency and growing population day by day. It was aimed to investigate the recovery potential of oil and valuable components from sunflower oil production wastes by using supercritical CO_2 (SC-CO_2) extraction as a green extraction method. In this context oil amounts, total phenolic content (TPC), total flavonoid content and antioxidant activities were analyzed. The waste samples obtained from filtration processes of the oil which were composed of oily bentonite (OB) and waxy perlite (WP). Soxhlet extraction was also applied on the waste samples to calculate extraction efficiencies of the SC-CO_2. It was observed that SC- CO_2 extraction was more effective for OB (27%, v/v) than WP (11%, v/v). In addition, the efficiency increased to 37% by mixing expanded perlite and OB sample to absorb moisture content of the waste and to increase the diffusion of carbon dioxide more easily. The statistical evaluation of the experiments was also conducted to determine the effect of independent variables on the recovery efficiencys. Pressure was detected more effective variables on the recovery values than temperature. The maximum recovery efficiencies of the oil and TPC were obtained at the 50 °C, 22.1 MPa and 60 °C, 20 MPa, respectively.

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INTRODUCTION

Recovery and reuse options for food processing industry wastes have increased importance to decrease food scarcity. Sunflower oil is the most consumed oil product in the World [1]. By-products of sunflower oil contain valuable components that can be used in the food industry [2]. Bio-oil production from sunflower seed husks by hydrothermal pre-treated pyrolysis has been reported as a recovery method [3]. Another study focused on the antioxidant activities of polysaccharides from the pulp of sunflower oil production [4]. Jadhav et al. [5] investigated the use of sunflower acid oil, which is a waste from vegetable oil refinery, as glucose-containing feedstock for sophorolipid production. In recent years, supercritical CO_2 (SC-CO_2) has been used as an innovative and environmentally friendly extraction method to produce high quality product [6]. Daraee et al. [6] studied the SC-CO_2 extraction of chlorogenic acid from sunflower (Helianthus annuus) seed kernels.

In all these studies, it is aimed to contribute to the food industry. However, as far as we know, the residual oil and valuable component potential in different fillers used in sunflower oil production stages has not been studied. At the same time, while many studies focus on a single waste

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material, our study aimed to evaluate several production wastes. In addition, these wastes can not be evaluated because they are flammable and are disposed of in municipal dumps. Therefore, the recovery potential of oil and valuable components from sunflower oil production waste was investigated with SC-CO_2 extraction.

MATERIALS AND METHODS

Materials

Samples were obtained from Guzeloglu food - Zevk sunflower oil company (Konya, Türkiye). Two main wastes were obtained from the production of sunflower oil after bleaching and winterization processes and were named as oily bentonite (OB) soil and waxy perlite (WP) soil, respectively. The production process of the company consisted of 4 stages: degumming and neutralization, bleaching, winterization (dewaxing) and finally deodorization. Deacidified neutral oil is firstly heated to certain temperatures and mixed with the bleaching adsorbent (bentonite soil) under vacuum. Bentonite/oil mixture is filtered through steel plate filters to separate oil from soil and to remove coloring matters, trace metals and hydroperoxide from oil. In winterization process, waxy substances which cause turbidity in the oil and spoil the appearance of the oil at low temperatures are removed from the oil by using an auxiliary substance (perlite) in a cold environment.

Experimental Design

The effect of pressure and temperature on the SC-CO_2 extraction was studied according to the experimental design (central composite design) created by using Minitab Software statistical program for the WP and OB sample (Table 1). The results were statistically evaluated by ANOVA test. Also, the effect of mixing expanded granular perlite with the wastes on SC-CO_2 extraction was investigated.

Table 1. Ranges of independent variables in the experiments

Independent variables	Min value	-1	Midpoint	+1	Max value
Temperature (°C)	36	40	50	60	64.1
Pressure (MPa)	7.93	10	15	20	22.1

SC-CO_2 Extraction

SC-CO_2 extraction was conducted with the Superex F-500 device (Biosan, Superex, Türkiye). The extractor has 500 mL column. Pressure and temperature of device can be increased up to 34.5 MPa and 70 °C, respectively.

For extraction experiments, 10 g sample was placed into device and falcon tube, in which the extract would accumulate, was placed in separator chamber. The CO_2 pump was started after temperature reached the desired value. The extraction process was carried out for 2 hours, the first 30 minutes being static and 90 minutes of dynamic flow mode. The extracts obtained from WP and OB were abbreviated as WPE and OBE, respectively.

Analysis Methods

Total oil content of the waste sample was analyzed by Soxhlet method [7] to determine extraction efficiency of the SC-CO_2 extraction method. Soxhlet extraction was applied to two raw samples. Extraction efficiency was calculated by dividing the amount of extract obtained by SC-CO_2 extraction by the oil content determined by the Soxhlet method.

The changes in the components of the extracts were monitored by total phenolic contents (TPC), total flavonoid contents (TFC) and antioxidant activity assays (ABTS and DPPH).

TPC of the samples were analyzed according to Singleton et al. [8] (with Folin–Ciocalteu reagent). Briefly, 20 µL of the extract was mixed with 1580 µL of methanol/water mixture and 100 µL of folin solution and then waited for 5 minutes. Then, 300 µL of sodium carbonate was added in the solution and vigorously mixed. The mixture was waited in the oven for 30 minutes at 45 °C for color development. At the end of the period, it was poured into numbered tubes and centrifuged at 4000 rpm for 5 minutes. The same procedures were performed for the witness sample. Finally, its absorbance at 765 nm was measured using a spectrophotometer (Hach-Lange, Dr 5000). A calibration chart was prepared using gallic acid as a standard. Total phenolic substance concentration (TFC) was expressed as mg gallic acid equivalents per 1 L of extract (mgGAE/L). Recovery efficiency (%RE, w/w) of TPC was calculated as the ratio of the TPC concentration in the extract to the TPC concentration obtained with the aid of n-hexane as soxhlet solvent.

TFC of the samples were analyzed according to Zhishen et al. [9]. The sample extracts were reacted with $NaNO_2$ and then with $AlCl_3$ to form a flavonoid-aluminum complex. The absorbance of these prepared solutions was read at 510 nm. Calibration chart prepared with quercetin standard was used in the calculation. The total amount of flavonoids was expressed in quercetin equivalents (mg QE/mL). TFC was calculated as mgQE/g by multiplying the TFC concentration by the volume of the extract in the mass of the extract.

Trolox equivalent antioxidant capacity were spectrophotometrically measured in respect of DPPH and ABTS radical scavenging activities (Hach-Lange, Dr 5000).

DPPH radical scavenging capacity were determined using 0.1 mM DPPH (2,2-diphenyl-1-picryhydrazyl radical) according to the Yu et al. [10]. 100 µL of sample was reacted with 1900 µL of methanolic DPPH• solution. The absorbance values of the mixture at 517 nm were read at 30 minutes after by zero with distilled water. Results were calculated with the aid of the trolox standard curve and expressed in trolox equivalents (µM TE).

ABTS radical cation scavenging capacity was determined as follows [11]; a 7 mM ABTS solution containing 2.45 mM potassium persulfate was prepared and the stock $ABTS^{\bullet+}$ solution was formed by keeping this solution in a dark environment at room temperature for 12–16 hours. $ABTS^{\bullet+}$ working solution was prepared by diluting the stock $ABTS^{\bullet+}$ solution with a water:etha-

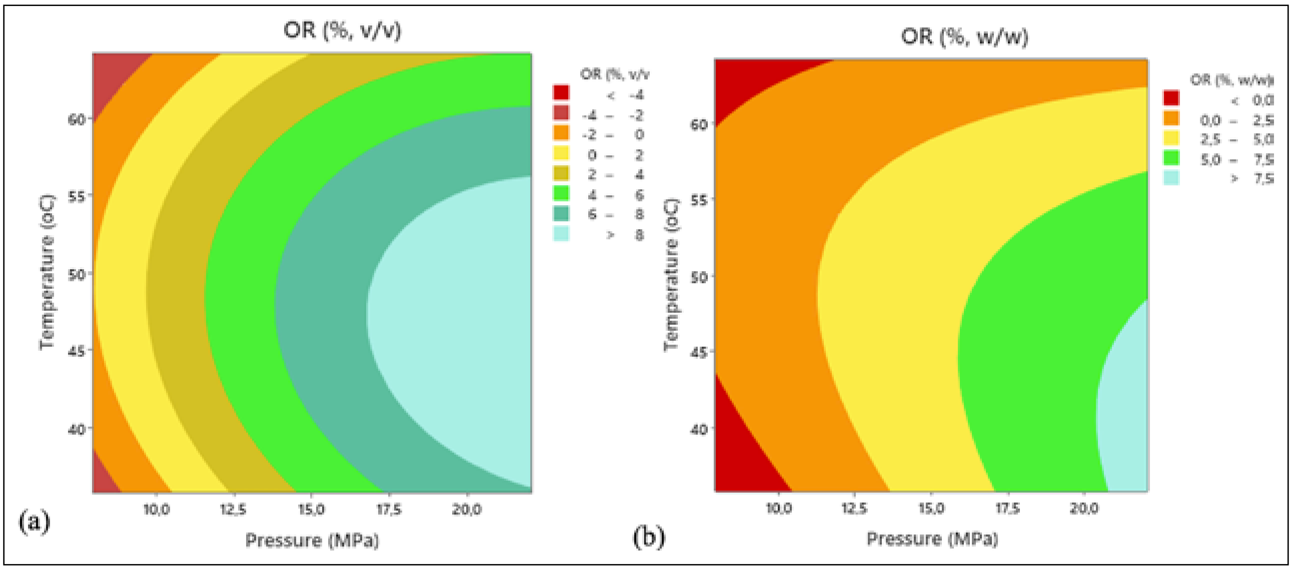

Figure 1. Binary effects of temperature and pressure on the oil recovery (OR) efficiency calculated by using **(a)** extract volume of WP and **(b)** extract mass of WP.

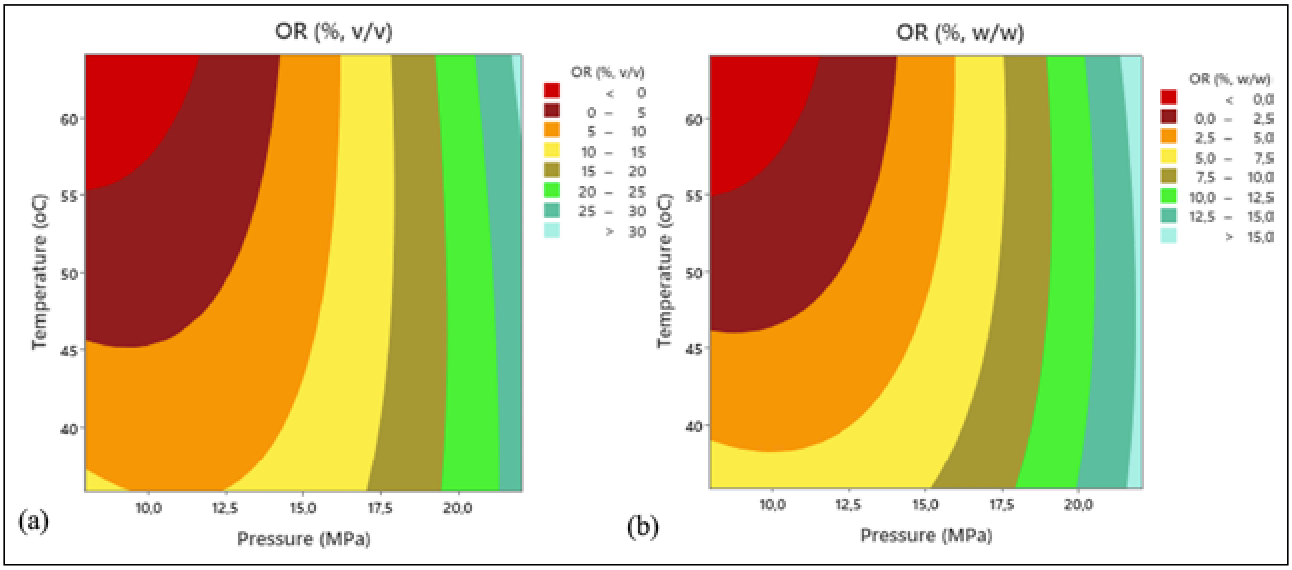

Figure 2. Binary effects of temperature and pressure on the oil recovery (OR) efficiency calculated by using **(a)** extract volume of OB and **(b)** extract mass of OB.

nol (1:1, v/v) mixture such that the total mixture had an absorbance of 0.70 at 734 nm. In the creation of the calibration chart and sample analysis; 1980 µL ABTS•$^{+}$ working solution was added to 20 µL sample and mixed rapidly with the help of vortex, and its absorbance at 734 nm was determined after waiting for 6 minutes. Calibration chart was prepared using trolox as standard. The radical cation scavenging capacity was expressed in trolox equivalents (µM TE).

RESULTS AND DISCUSSION

Oil Recovery from the Waste Materials

Volumetric (v/v) and gravimetric (w/w) oil recovery (OR) efficiencies for SC-CO_2 extraction of the WP ranged from 1.5% to 11% and from 0.3% to 8%, respectively (Fig. 1). It was observed that amount of the extracts increased with the increase of pressure and decrease of temperature, and it was maximizing above 20 MPa. Similar to our results, it was reported that increased pressure (28–34 MPa) of SC-CO_2 and milling time of chia seeds increased oil yield, but higher temperature (60–80 °C) could decrease the yield [12].

Higher extraction efficiencys were obtained for SC-CO_2 extraction of the OB waste samples than WP. Volumetric and gravimetric oil recovery (OR) efficiencies for SC-CO_2 extraction of the OB wastes ranged from 0.2% to 27% (v/v) and from 0.5% to 16% (w/w), respectively (Fig. 2). The effect of expanded granular perlite (10 g) addition on the OB sample (20 g) was also investigated to increase the amount of extract at 40 °C and 20 MPa. Oil recovery efficiency in-

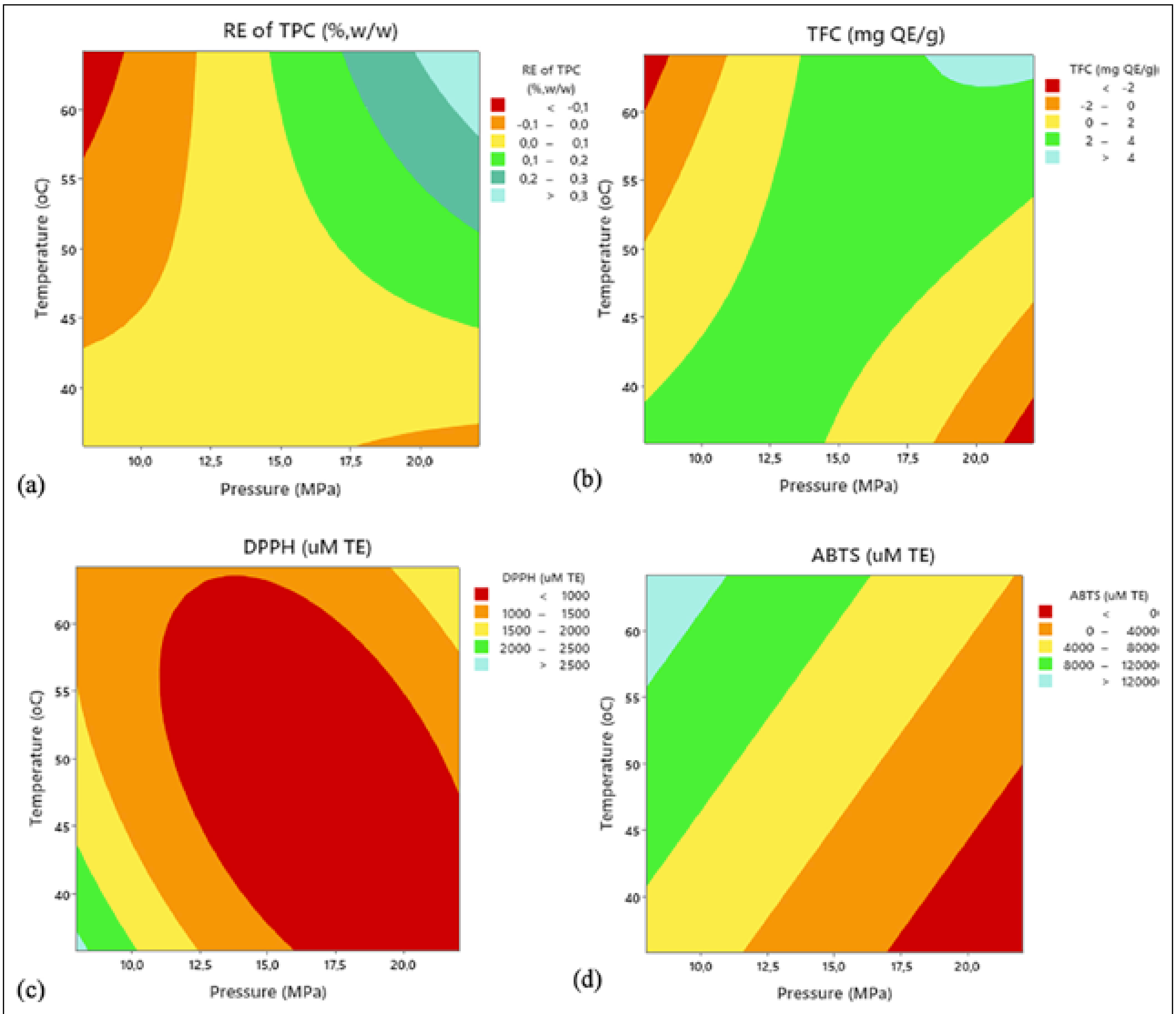

Figure 3. Binary effects of the experimental variables on the valuable components of WPE; **(a)** TPC value, **(b)** TFC value, **(c)** DPPH and **(d)** ABTS radical scavenging activity.

creased from 27% to 37% (v/v) and 16% to 19% (w/w) by using the expanded perlite. Expanded perlite might be absorb more easily the moisture content of the waste and also increase carbon dioxide diffusion with its porous structure.

Although higher extract amount usually can be obtained by using conventional chemicals such as hexane [12], it should be stated that SC-CO_2 extraction provides more stable, healthy extracts and lower toxic residue. However, it was reported in the literature that oil recovery efficiency of SC-CO2 could be comparable with Soxhlet extraction which used hexane [13]. This situation was caused by factors such as experimental variables or solvent selection. Similar to our study, the highest extract efficiency was obtained only at 40 °C and 25 MPa operating condition with SC-CO_2, and the extract efficiency obtained in Soxhlet extraction with hexane was achieved [13]. In the same study, it was stated that the sample obtained by SC-CO_2 extraction exhibited a higher oxidative induction time than that extracted with hexane.

The Change of Valuable Content

TPC recovery efficiencies of WPE were ranged between 0.001–0.3% (Fig. 3). The highest TPC recovery efficiency of WPE was above of 20 MPa and 60 °C. TFC/TPC ratio of the extracts were generally high and near to 1 showing high flavonoids content. TFC in the extract increased at the high pressure and temperature for WP extracts, while similar results were also obtained at lowest conditions (Fig. 3). Similarly, maximum TFC concentration for OBE was obtained as 3.94 mg QE /g at the condition of 60 °C and 20 MPa. It can be said that the increase in polarity and density of CO_2 at high pressure increases the solubility of some phenolic compounds [14]. Flavonoids are expected to be more soluble in SC-CO_2 than other phenolics because of their high molecular weight and relatively low polarity [15–17].

Antioxidant activities of WPE in respect of DPPH and ABTS radicals increased significantly by SC-CO_2 extraction compared to conventional extraction method

Table 2. Comparison of TPC, TFC and antioxidant activity values in literatures

Studies	Raw material	Extraction method	TPC (mg GAE/g)	TFC (mg QE /g)	Antioxidant activity (µMTE/g)	
In this study	WPE OBE	SC-CO_2	0–0.77 0–6.55	0–3.9 0.0–21	ABTS: 89–1878 72–9975	DPPH: 57–220 13–668
Neves et al. [18]	Sunflower cake (Shell and raw material mix)	Conventional extraction + Microwave-assisted extraction	12–14	–	ORAC: 180–260	
Ye et al. [19]	Sunflower florets	Pure solvents with increasing polarity (ethyl acetate, ethanol, methanol and water)	1.3–25	–	ABTS: 8.36–265.77	DPPH: 3.85–137
Weisz et al. [20]	-Dehulled sunflower kernels -Dehulled sunflower shells	Extracted twice by stirring aqueous methanol (60%) after n-hexane in a soxhlet extractor	29.38–41.8 0.4–0.86	–	–	–
Pajak et al. [21]	Sunflower seeds - sprouts	Shaking with methanol	4–9	25–45	ABTS: 32–72	DPPH: 24–48
Abdalla et al. [22]	Sunflower seeds	Shaking with methanol after n-hexane	22.2–33.1	1.02–3.34 mg RE/g	ABTS: 78–128.5	DPPH: 90.3–161.4

Table 3. ANOVA results for oil recovery (OR) efficiency and extract quality parameters of the WPE

ANOVA terms	OR (%, v/v)		OR (%, w/w)		TPC (%RE, w/w)		$DPPH^{\cdot}$ µM TE		$ABTS^{\cdot+}$ µM TE		TFC mg QE/g	
	F	p	F	p	F	p	F	p	F	p	F	p
Source	**Quadratic**		**Quadratic**		**Linear+ Interactions**		**Quadratic**		**Linear**		**Quadratic**	
Model	6.83	0.043	41.9	0.002	5.8	0.033	0.88	0.565	4.31	0.060	38.67	0.002
Temperature	1.66	0.267	28.5	0.006	2.03	0.204	0.01	0.927	2.93	0.131	10.94	0.030
Pressure	27.8	0.006	147	0.0002	10.72	0.017	1.23	0.329	5.68	0.049	9.63	0.036
Lack of fit	3.58	0.366	–	–	5.54	0.311	56.75	0.097	70.83	0.091	0.59	0.715
Std. dev.	1.83		0.53		0.072		524.6		4407.5		0.30	
R-squared	0.90		0.98		0.74		0.52		0.55		0.98	

(Soxhlet). The highest antioxidant activity of WPE was achieved at 50 °C and 7.93 MPa. Pressure was determined as the most effective experimental variable like for TFC results.

Antioxidant activity of the WPE was between 890 and 18780 µM TE in respect of ABTS radical, while it was found between 570–2200 µM TE for DPPH radical. Less antioxidant activity was found for WPE compared to the OBE sample. It was reported that antioxidant activity in the extracts of sunflower by-product obtained by using microwave-assisted extraction was in the range of 180 to 266 µM TE/g [18]. Although we obtained higher than the literature, the antioxidant activity showed a wide range of variation (Table 2). This may be a result of variation of the extracted compounds, which may be responsible for the antioxidant activity, under different experimental conditions. Valuable component measurement results obtained for various sunflower extraction methods show that TPC and TFC results of this study are comparable to the other studies, even though they have used conventional, more polar and hazardous solvents (Table 2).

The highest recovery of TPC (0.6%) and TFC (21.3 mg QE/g) for OBE was obtained at the operating conditions higher than 60 °C and 20 MPa (Fig. 4). TPC and TFC recovery of OBE was quite high compared to WPE. Although we obtained a lower total phenolic content compared to many studies, it was also seen that similar results were obtained (Table 2). It could be said that the reason for the TPC values similar to our results were the choice of raw material and solvent used. Abdalla et al. [22] achieved similar results with WPE in terms of TFC, but Pajak et al. [21] obtained much higher results than OBE. This may be due to the fact that methanolic extraction was performed without recovering the oil.

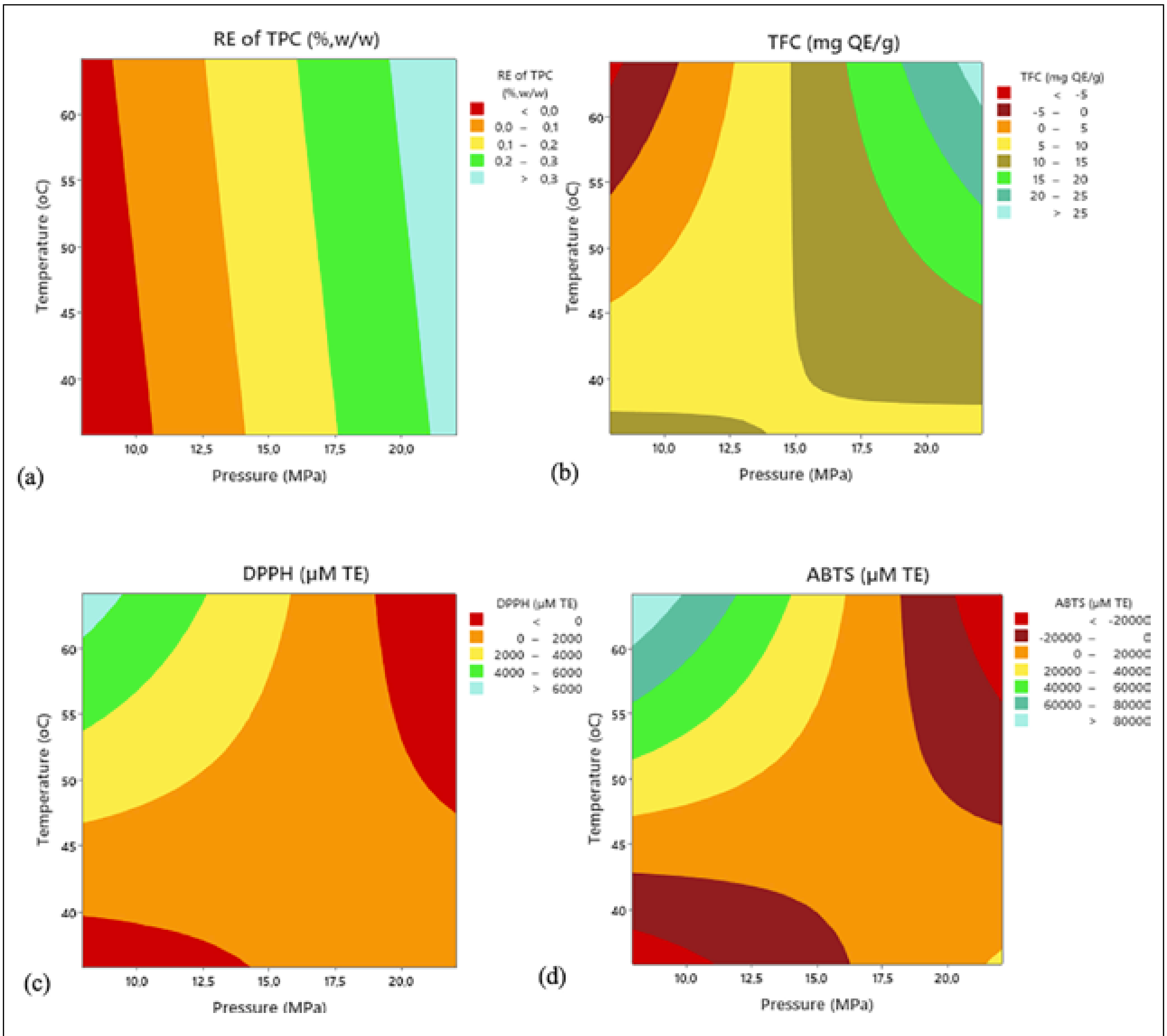

Figure 4. Binary effects of the experimental variables on the valuable components of OBE; **(a)** TPC value, **(b)** TFC value, **(c)** DPPH and **(d)** ABTS radical scavenging activity.

Table 4. ANOVA results for oil recovery (OR) efficiency and extract quality parameters of the OBE

ANOVA terms	OR (%, v/v)		OR (%, w/w)		TPC (%RE, w/w)		DPPH· μM TE		ABTS·+ μM TE		TFC mg QE/g	
	F	p	F	p	F	p	F	p	F	p	F	p
Source	**Quadratic**		**Quadratic**		**Linear**		**Linear+ Interactions**		**Linear+ Interactions**		**Linear+ Interactions**	
Model	15.6	0.010	13.1	0.014	4.16	0.064	4.68	0.052	3.05	0.114	7.76	0.017
Temperature	2.74	0.173	3.81	0.123	0.10	0.764	3.29	0.12	2.21	0.188	0.04	0.851
Pressure	66.8	0.001	55.6	0.002	8.23	0.024	6.05	0.049	2.91	0.139	17.39	0.006
Lack of Fit	0.16	0.913	0.23	0.872	1.04	0.635	3.77	0.372	99.85	0.076	0.29	0.875
Std. dev.	3.18		1.90		0.142		1314.26		23663		3.70	
R-squared	0.95		0.94		0.54		0.70		0.60		0.80	

The highest antioxidant activity of OBE was achieved at 60 °C and 10 MPa for DPPH (6683 μM TE) and ABTS (99748 μM TE) radical. In addition, the extract of the expanded perlite-OB mixture had lower antioxidant capacity than OBE, while it had average TFC value. But RE of TPC (2.2%, w/w) is considerably higher than OBE.

Statistical Analysis

OR efficiencies for WPE were statistically significant in respect of the volumetric recovery (p value: 0.043) and mass recovery (p value: 0.002) (Table 3). The most significant variable on OR efficiency was pressure (p: 0.006 for volumetric recovery; p: 0.0002 for mass recovery). However, the temperature variable was significant only in mass recovery efficiency (p: 0.006). Similar results have reported in literature regarding the significance of experimental variables for extraction of sunflower seed kernels by SC-CO_2 extraction method [6].

It was also concluded that recovery efficiencies of TPC and TFC were found statistically significant (p: 0.033 and p: 0.002, respectively). Temperature and pressure provided the same level of significance for TFC while pressure was significant only for TPC.

Statistical importance of the experimental variables on the antioxidant activities was not significant (p: 0.565 for DPPH and p: 0.06 for ABTS). However, it was observed that pressure has significant effect on the ABTS antioxidant activity values of the extracts (p: 0.049).

The OR efficiencies for OBE were found to be statistically significant in terms of volumetric recovery (p: 0.010) and mass recovery (p: 0.014). Similar to WPE, the most significant variable was found to be pressure (p: 0.001 for volumetric recovery and p: 0.002 for mass recovery). The recovery efficiency of TPC for OBE was found as statistically insignificant (p: 0.064) while TFC (mg QE/g) was significant (p: 0.017). Contrary to WPE, DPPH antioxidant activity was found close to statistical significance (p: 0.052) while ABTS antioxidant activity was insignificant (p: 0.114) in OBE (Table 4).

CONCLUSION

It has been observed that SC-CO_2 extraction was more effective on the oily bentonite (OB) wastes to recover oil and valuable components than waxy perlite (WP) waste. The ANOVA tests of the developed models resulted in high coefficient of determination for oil recovery of WPE and OBE values for extract volume (R^2=90% and 95%) and mass (R^2=98% and 94%). It was detected that pressure variable was the most important variable affecting the significance for all results. A significant correlation was found between the TFC value of both samples and the independent variables of SC-CO_2 extraction. Oil recovery significantly increased up to 37% with the addition of expanded perlite into the waste sample before SC-CO_2 extraction. Although sunflower oil production industry waste samples were used in this study, results similar to the literature were obtained for TPC and TFC. Due to the high antioxidant content, TPC and TFC in the extracts, it may be possible to use these products in different sectors (cosmetics, pharmaceuticals, food, etc.).

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DATA AVAILABILITY STATEMENT

The authors confirm that the data that supports the findings of this study are available within the article. Raw data that support the finding of this study are available from the corresponding author, upon reasonable request.

CONFLICT OF INTEREST

The authors declared no potential conflicts of interest with respect to the research, authorship, and/or publication of this article.

ETHICS

There are no ethical issues with the publication of this manuscript.

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