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Research article

From waste to base oil: Supercritical CO_2 extraction for regeneration of multi-source and saponified lubricant waste streams

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ABSTRACT

Waste lubricant oils, classified as hazardous waste, can be regenerated into valuable base oils, however, cleaner and more efficient technologies are needed to overcome the limitations of conventional processes, including safety risks, high costs, and inefficiency. While prior regeneration studies have focused on relatively homogeneous, single-source engine-drain oils, they have not addressed the challenges posed by complex, multi-source WLO blends or KOH-induced coagulation phenomena that hinder conventional re-refining routes. Here, we demonstrate for the first time that a standalone regeneration method based on supercritical CO_2 extraction can effectively recover base oils from both heterogeneous WLO mixtures and saponified (coagulated) waste streams. Process optimization using response surface methodology identified optimal conditions of 140 bar and CO_2 flow rate of 14 mL/min at 40 °C, achieving extraction yields up to 73% for non-coagulating oils and approximately 60% for coagulated samples. Regenerated oils exhibited significant improvements in physicochemical properties, including reductions in total acid number, saponification number, water content, chlorine concentration, and oxidation-related functional groups, as confirmed by ASTM methods, FTIR spectroscopy, and elemental analysis. Except for color, regenerated oils complied with technical specifications for base oils obtained from regeneration operations. These results demonstrate the ability of supercritical CO_2 extraction to overcome coagulation barriers and extend green regeneration technologies to complex WLO feedstocks previously considered unrecoverable, supporting circular economy strategies for hazardous oil waste valorization.

1. Introduction

Waste lubricant oils (WLO) are a dangerous pollutant classified as hazardous waste according to European environmental legislation (European Parliament, 2018). Since 2008, the EU has implemented guidelines for the harmonized management of waste oils, promoting effective collection and treatment strategies focused on energy recovery or recycling, typically through reprocessing or regeneration (GEIR Groupement Européen de l'Industrie de la Régénération, 2014). Using WLO for energy recovery poses issues like particulate emissions, ash residue, and carbon emissions (Pinheiro et al., 2021). Regenerating WLO is the most sustainable option, representing the best recovery route for the lubricant's life cycle and its integration into a circular economy. It reduces its environmental impact, conserving energy and resources, and provides economic and public health benefits (Kupareva et al., 2013a; Kanokkantapong et al., 2009; Abdalla et al., 2018; - et al., 2023; Sarkar et al., 2023). The regeneration process restores WLO by removing contaminants, oxidation products and additives, and the recovers the undamaged hydrocarbons molecules to achieve the base oil for the manufacture of new lubricating products.

In recent years, growing environmental awareness, resource scarcity, and legislative pressure have driven European countries to replace the cost-effective but highly polluting acid-clay method for the WLO deasphalting step of regeneration. The former uses concentrated sulfuric acid and produces large amounts of acid sludge and equipment corrosion (Dang, 1997). Commonly used cleaner technologies and processes include those based on distillation (Taiwo and Bello, 2020), solvent extraction (Sarkar et al., 2023; Pinheiro et al., 2018a, 2018b; Rincón et al., 2005), pyrolysis (Mishra et al., 2021), membrane (Widodo et al., 2020), etc. Solvent extraction methods can overcome the problems caused by acid treatments. However, these methods still require solvent evaporation and high vacuum distillation, which frequently bring process safety hazards and waste management. Additionally, there is one more operational issue, transversal to all regeneration technologies that

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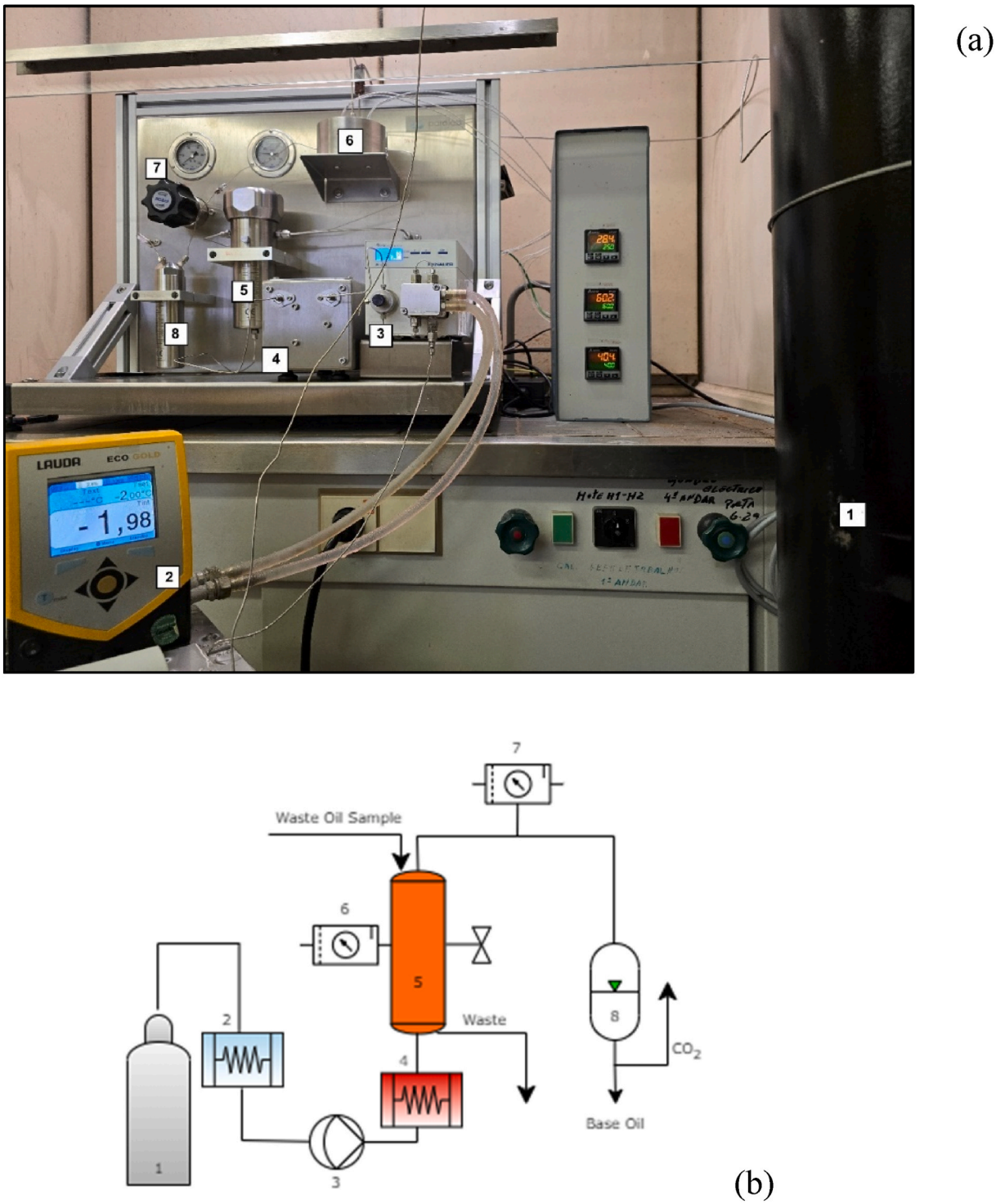


Fig. 1. SFE experimental setup (a) and schematic description (b). Legend: 1- CO_2 cylinder; 2- Thermostatic bath; 3- Pump; 4- Pre-heating; 5- Extraction Vessel; 6- Back Pressure regulator for extraction vessel; 7- Back Pressure regulator for separator; 8- Separator.

include a distillation step (and/or high temperature) and that prevents WLO from being regenerated depending on its composition (Yokoyama and Iwama, 2014). This issue is caused by chemical treatment, a usual stage done prior to deasphalting aimed at the enhancement of contaminant removal through the addition of potassium hydroxide. KOH destabilizes the colloidal system by promoting particle aggregation and flocculation (Cosgrove, 2009), resulting in lower plant fouling, neutralization of acids and chlorine, and precipitation of low-solubility metal salts (Yokoyama and Iwama, 2014; Kajdas, 2000a, 2000b). However, the addition of KOH can also cause coagulation issues (Pinheiro et al., 2017a, 2018b), due to the saponification reactions with long chain fatty acids and or esters normally present in synthetic or semi-synthetic based lubricant oils (Chakrabarty, 2003), decreasing WLO fluidity and leading to plant shutdowns. Therefore, there is a clear need to combine viable, efficient, environment friendly, composition independent, affordable treatment and high yield method.

Supercritical fluid cleaning using carbon dioxide is a capable alternative for industrial cleaning processes, namely as a replacement of

conventional cleaning solvents, reducing the pollution associated with the operations that would otherwise require significant amounts of volatile organic compounds. Carbon dioxide in supercritical condition is inert, non-toxic, non-flammable and is readily availability in high purity at a relatively low cost. Once its supercritical conditions are reached (p_c = 7.38 MPa, T_c = 31 °C), carbon dioxide converts into a fluid with the density of a liquid but the ability to expand like a gas (Gaponenko et al., 2020; Guo et al., 2020) and, when used in supercritical extraction, the separation of carbon dioxide from the product can be easily achieved by simple depressurization. The low viscosity of supercritical carbon dioxide coupled with its high solute diffusivity, enhances mass transfer, enabling it to penetrate solid microstructures and efficiently extract target compounds. Its ability to dissolve a wide range of both polar and non-polar substances further contributes to its classification as a green solvent. Based in all the above, supercritical carbon dioxide appears like an obvious good alternative to conventional extraction or SFE solvents (Rincón et al., 2005, 2007; Laitinen and Kaunisto, 1999; Tomasko et al., 2003; Liu et al., 2005). SFE with $scCO_2$ has great potential to overcome the above-mentioned issues, because it achieves deep and clean separation at low temperatures, making it appropriate for thermally unstable hydrocarbons and preventing column fouling, eliminating solvent residues and overriding the major reason that excludes regeneration: it allows to regenerate WLOs that coagulate, where conventional technologies fail. To our knowledge, recent studies exploring supercritical CO_2 in waste lubricant oil treatment remain limited. Huang et al. demonstrated the selective extraction of lighter saturated hydrocarbons using $scCO_2$ in a multi-stage fractionation approach, achieving approximately 60% recovery while retaining heavy polar compounds and heteroatom-rich fractions in the residues, thus requiring subsequent refining steps (Huang et al., 2024). Earlier studies involving $scCO_2$ extraction (Rudyk and Spirov, 2016; Gourgouillon et al., 2000; Pavlova et al., 2022) have employed it for remediation purposes or as a viscosity-reducing agent in membrane-assisted separations, rather than as a standalone regeneration technology. These studies typically focus on single-source used oils and overlook the complexity and variability of multi-source WLO blends commonly encountered in real-world collection systems. Moreover, they do not address the challenges posed by KOH-induced coagulation, which can render oils unrecoverable by conventional methods. In this work, we address these gaps by demonstrating that $scCO_2$ extraction alone can effectively regenerate base oils from both complex, multi-source WLO blends and from samples exhibiting saponification-driven coagulation. We detail the use of $scCO_2$ as a green solvent to establish a new standalone methodology for the regeneration of used lubricant oils, outlining strategies to extract base oils and reintegrate them into the lubricant life cycle—fully aligned with circular economy principles and aimed at preserving product value while minimizing waste.

2. Materials and methods

2.1. Materials and samples

Waste lubricant oil samples were collected by SOGILUB S.A, the national organization responsible for the Portuguese waste lubricant oil management system and recovered from SISAV unit in Santarém. Two representative samples were taken, both consisting of a complex blend of waste lubricant oil originated from several types of producers such as garages, industry and other producers (e.g. transportation, construction, agriculture or public sectors, among others) and were selected considering their response stability to alkaline treatment (Pinheiro et al., 2017a). Sample 1 is an oil mixture unaffected by the addition of KOH while Sample 2 is a sample that has tested positive for saponification (it coagulates) preventing it to be treated by conventional regeneration methods that involve distillation. Both samples were used as received, after steps of dehydration and filtration treatment in the SISAV unit. They were stored in plastic containers at room temperature, in darkness, to preserve their integrity until needed.

CO_2 with purity higher than 99.995% (water content <40 ppm) was supplied by Messer.

2.2. Regeneration experiments of samples with $scCO_2$

Regeneration of waste lubricant oil is done in a customized supercritical fluid extraction (SFE) laboratory apparatus, showed in Fig. 1, equipped with a 300 mL stainless-steel vessel designed to work at high pressure and followed by a 20 mL stainless-steel separator to recuperate base oil, built by Paralab. CO_2 exiting from the cylinder is cooled down, to ensure its liquid state, both by a stainless-steel coil submerged into a thermostatic circulating water bath set to −2 °C (Lauda Eco RE415G) and a heat exchanger, mounted in an HPLC analytical pump (AZURA P 4.1S from Knauer) that impels CO_2 into the system. CO_2 is then heated with a heat exchanger to the extraction temperature, to attain supercritical conditions. $ScCO_2$ enters, from below, the cleaning vessel, packed with 5 mm glass beads and loaded with the sample of WLO to regenerate. Pressure is controlled with two back pressure regulators. A first diaphragm back pressure regulator, Equilibar U6L is mounted over the pressure vessel, controlling the pressure over extraction. BPR pressure is operated using a N_2 cylinder with manual regulation. A second manual back pressure regulator from Fitok is mounted between the extraction vessel and the separator and is meant to control the pressure release over the latter.

During the experiment the contaminants are retained in the pressure vessel while CO_2 and the base oils move to the separator. CO_2 dissolving capacity can be modulated by altering experimental conditions (p and T). Cleaned sample is recovered in the separator, by depressurization of CO_2.

2.3. Analytical procedures

2.3.1. Physicochemical characterization

Samples were characterized regarding the density at 40 °C, kinematic viscosity at 40 °C and 100 °C, viscosity index, total acid number (TAN), saponification number (SN), water content, chlorine and sulfur content. Properties were determined using reference ASTM methods.

Kinematic viscosity was obtained at 40 °C and 100 °C according to ASTM D7042, using an Anton Parr-SVM 3000 Stabinger Viscosimeter. The density at 15 °C and 40 °C was determined according to ASTM D4052, using a digital densimeter, Mettler Toledo DM40.

Total acid number (TAN) was determined by potentiometric titration according to ASTM D664. Titration solvent consisted of a mixture of toluene, isopropanol and water (50, 49.5 and 0.5% v/v, respectively), while titrant was a solution of alcoholic potassium hydroxide (0.1 M) to neutralize the lubricant acidic components. TAN was expressed in milligrams of KOH required to neutralize the acidic constituents per gram of oil.

Saponification number (SN) was determined by potentiometric titration following ASTM D94. Oil samples were dissolved in a mixture of a KOH alcoholic solution (0.5 M), butanone (50 mL) and White Spirit solution (25 mL), the latter added to facilitate the dissolution of lubricant oil and additives. The mixture was refluxed for 30 min, and the condenser was rinsed with 50 mL of naphtha to eliminate any remaining sample. The titrant was HCl (0.5 M). The saponification number (SN) corresponds to the milligrams of KOH required to saponify fatty material present in 1 g of oil.

The water content was measured using coulometric Karl Fischer titration, following ASTM D6304 guidelines, using an Metrohm 870 KF Titrino Plus automatic titrator. The oil sample was dissolved in Aquametric Solvent CM for Karl Fisher analysis and titrated with Hydranal Composite 5. Once all the water was titrated, excess iodine, produced at the anode during the Karl Fischer reaction, was detected by an electrometric endpoint detector, signaling the end of the titration. The quantity of water is proportional to the total integrated current

Table 1
Levels of factors used in the 3^2 factorial design.

Symbol	Independent Variable	Levels		
		−1	0	+1
X_1	Pressure/bar	80	110	140
X_2	Flow rate/mL/min	6	10	14

according to Faraday's Law, considering that 1 mol of iodine reacts with 1 mol of water.

2.3.2. Composition analysis

2.3.2.1. FTIR spectroscopy. The vibrational analysis of the sample was conducted using Fourier Transform Infrared Spectroscopy (FTIR), by reflectance, employing the non-destructive sampling technique of Attenuated Total Reflectance (ATR). The infrared spectrum of the solid sample at room temperature was recorded, in the range of 4000-550 cm^{-1}, using a Perkin Elmer Frontier spectrometer (FT NIR/MIR), equipped with an FR-DTGS detector and a KBr beam splitter. The spectra were recorded with a resolution of 4.0 cm^{-1} with 32 accumulations. A Perkin Elmer sampling accessory, the universal module for ATR (Attenuated Total Reflectance - UATR) with a diamond/ZnSe crystal, was used, applying a constant force of 10 N in all recordings.

2.3.2.2. Elemental analysis. Determination of chlorine and sulfur content was made by wavelength-dispersive X-ray fluorescence spectrometry, according to ISO 15597, using an X-Supreme 8000 EDXRF spectrometer from Oxford Instruments.

Quantification of total carbon, hydrogen and nitrogen was done using a ECS 8040 CHNS-O elemental analyzer from NC Technologies according to the standard test procedures. The technique used for the determination of CHNS/O was based on the quantitative "dynamic flash combustion" method.

2.4. Design of experiments

Design of Experiments (DoE) is widely used to optimize engineering processes and identify which input variables significantly influence the output. In a factorial design, each factor or variable is assessed at specific levels. In a three-level factorial design, each factor takes on one of three values, typically representing a high, intermediate, and low level for continuous variables. Crucially, DoE can also reveal interactions between factors, enabling more efficient process optimization compared to analyzing variables individually. While SFE improves efficiency by adjusting temperature and pressure, other variables like residence time and flow rate are also key (Dejaegher and Vander Heyden, 2011; Sharif et al., 2014). Preliminary tests identified pressure (X_1) and flow rate (X_2) as the main factors. After determining their variation ranges through preliminary screening tests, a full factorial design with two variables at three levels was conducted to study their interactions, using yield as the target response. The experimental matrix included nine runs, each performed in duplicate. The levels of the independent variables used in the experimental design are shown in Table 1.

The optimization strategy of the extraction process and the statistical analysis was carried out using STATISTICA7 software (StatSoft, 2004) (Inc. STATSOFT). A second-order polynomial regression model for predicting the optimal point was expressed according to Eq. (1):

$$Y = \beta_0 + \sum_{i=1}^{3} \beta_i X_i + \sum_{i=1}^{3} \beta_{ii} X_i^2 + \sum_{1 \le i \le j}^{3} \beta_{ij} X_i X_j \quad (1)$$

where Y is the response, β_0 the intercept, X_i and X_j are the factors, and β_i, β_{ii}, and β_{ij} are regression coefficients of the linear, quadratic, and

Table 2
Comparative physicochemical properties and compositional indicators of waste lubricant oils (WLO) and corresponding regenerated oils (R), including reference specifications for WLO eligibility for regeneration and regenerated base oils according to Portuguese regulations.

Property	Units	Test method	WLO acceptance specs[a)]	Sample 1 (WLO)	Sample 1R[d)] (regenerated)	Sample 2 (WLO)	Sample 2R[d)] (regenerated)	Base oil specs[e)]
Density at 15 °C	kg/m^3	ASTM D1298	800–1000	871	854	877	865	850–880
Kinematic viscosity at 40 °C	cSt	ASTM D445	10–100	55	21.5	58	31	15–40
Kinematic viscosity at 100 °C	cSt	ASTM D445	—	11	9.5	9.5	4.5	—
Viscosity Index	—	ASTM D2270	—	197	123	148	126	>90–95
Water content	wt %	ASTM D6304	<1	0.41	0.2	0.30	0.2	—
Total acid number (TAN)	mgKOH/g	ASTM D664	0.43–4.48[b)]	2.26	1.26	2.86	1.11	—
Saponification number (SN)	mgKOH/g	ASTM D94	0.8–64[c)]	21.10	8.57	44.17	13.40	—
Coagulation	—	ASTM D94	Negative	Negative	Negative	Positive	Positive	
Color	—	ASTM D1500	—	—	3–4	—	3–4	<2–2.5
Elemental content								
Chlorine (Cl)	ppm	—	<2000	56.00	19.00	80.00	ND	—
Sulfur (S)	%	—	—	0.40	0.30	0.40	<0.01	—
Nitrogen (N)	ppm	—	—	0.3	0.3	0.3	0.3	—
Carbon (C)	ppm	—	—	74	71	73	72	—
Hydrogen (H)	ppm	—	—	15.6	15.5	15.4	15.5	—
Composition								
Paraffinic fraction	wt %	IR spectra	—	76	78	78	74	>60
Naphthenic fraction	wt %	IR spectra	—	23	22	22	23	>30
Aromatic fraction	wt %	IR spectra	—	1	0	0	3	<10

Notes: a) WLO acceptance specifications correspond to Portuguese regulatory criteria for waste oils eligible for regeneration operations (Agência Portuguesa do Ambiente, 2021), b) from reference (Pinheiro et al., 2017b); c) from reference (Pinheiro et al., 2017a); d) the regenerated extract was obtained at the optimal operating conditions of 140 bar of pressure, flow rate CO_2 of 14 mL/min and temperature of 40 °C, according to the optimization study; e) Base oil specifications correspond to quality ranges for regenerated base oils (SN-100 and SN-150 categories) as defined by Portuguese technical standards (Agência Portuguesa do Ambiente, 2021); ND = not detected, "—" indicates not specified in the relevant regulation or not measured for that sample.

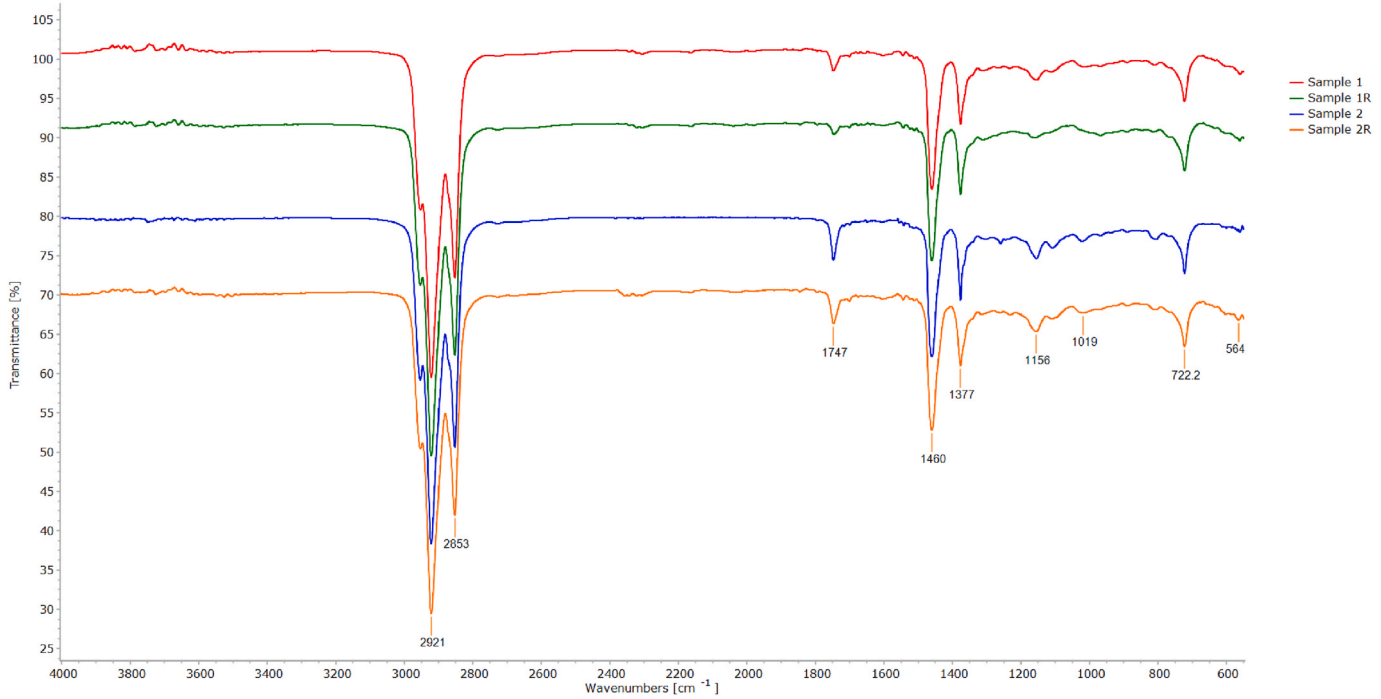


Fig. 2. FTIR spectra of raw waste lubricant oil (Sample 1, in red), coagulated waste (Sample 2 in dark blue), and regenerated base oil (sample 1R in green and 2R in yellow). Spectra are presented in transmittance mode and vertically offset for improved visualization of functional group transformations.

interactive terms, respectively.

The optimal extraction conditions for different WLO classes were determined using regression models (Eq. (1)) and a prediction profiler generated with STATISTICA7 software (Inc. STATSOFT). The prediction profiler visually represents the response surface, showing how changes in factors like pressure and flow rate affect yield (response). Desirability functions, used to optimize multiple response processes, helped identify the best operating conditions by transforming each measured response into a dimensionless scale using the fitted model.

3. Results and discussion

3.1. Physicochemical and compositional characterization of WLO before and after regeneration

The physicochemical properties and molecular composition of the analyzed waste lubricant oil feedstocks (Sample 1 and Sample 2) and the corresponding regenerated oils obtained under optimized supercritical CO_2 extraction conditions (Sample 1R and Sample 2R) were determined according to standard ASTM methods, FTIR spectroscopy, and elemental analysis. The consolidated results are presented in Table 2, together with the regulatory acceptance criteria for WLO regeneration and quality specifications for regenerated base oils defined by Portuguese standards (Agência Portuguesa do Ambiente, 2021).

The two WLO feedstocks, Sample 1 and Sample 2, exhibited comparable density and viscosity ranges, representative of multisource collected waste oils, with Sample 2 showing a positive coagulation response and a significantly higher saponification number, consistent with the presence of ester-based components and fatty acid derivatives associated with KOH-induced saponification phenomena. Both samples presented elevated total acid numbers (TAN) and measurable water contents (0.41 wt% for Sample 1 and 0.30 wt% for Sample 2), together with heteroatomic contaminants such as chlorine and sulfur, indicative of oxidative degradation and additive accumulation during service life. The higher TAN and lower viscosity index in Sample 2 are pointers of a possible increased degradation of the oil.

Following supercritical CO_2 extraction, at conditions of 140 bar of pressure, 14 mL/min flow rate of CO_2 and temperature of 40 °C (see section 3.4 for Optimization), marked improvements in oil quality were observed for both regenerated samples. The regenerated oils displayed substantial reductions in water content, total acid number and saponification number. Significant decreases in viscosity and viscosity index were also recorded. These changes reflect the selective removal of oxidation products, metallic soaps, and additive-derived compounds (Rincón et al., 2005). Elemental analysis confirmed the effective reduction of chlorine and sulfur contents. A detailed analysis of FTIR spectra for the waste lubricant oil samples (Fig. 2) reveals a hydrocarbon matrix predominantly composed of paraffinic and naphthenic fractions, evidenced by the multiple bands in the 2954−2856 cm^{-1} range, the strong band at 1460 cm^{-1}, and a weaker band around 1377 cm^{-1} and at 722 cm^{-1}. These are indicators of the presence of a mixture of hydrocarbon compounds with short carbon chain lengths and C−H branching vibrations associated with −CH groups, and = CH characteristics of paraffinic compounds, either from mineral or synthetic origin (Kupareva et al., 2013b). On the other side, the spectra exhibited a very week signal, at 1600 cm^{-1}, caused by the aromatic ring stretching vibrations, revealing a minimal aromatic contribution.

FTIR spectral analysis provided, also, direct insight into the chemical transformations associated with lubricant ageing, allowing the identification of additives and oxidation products originated from contamination or after lubricant oil use (Al-Ghouti and Al-Atoum, 2009), as well as subsequent regeneration (Fig. 3).

In the carbonyl region (1800–1600 cm^{-1}), the used oils exhibited an intense absorption band centered at approximately 1747 cm^{-1}, attributed to ester and carbonyl functional groups formed during oxidative degradation of the base oil. In Sample 2 (and corresponding regenerated oil Sample 2R), the higher intensity of this band can additionally be associated with a larger contribution of synthetic ester-type components in the original lubricant mixture. Additional lower-intensity bands observed between 1700 and 1650 cm^{-1} are associated with conjugated carbonyl species and secondary oxidation products. Following supercritical CO_2 extraction, a pronounced decrease in the intensity of these oxidation-related bands was observed for both regenerated samples, demonstrating the effective removal of degradation compounds.

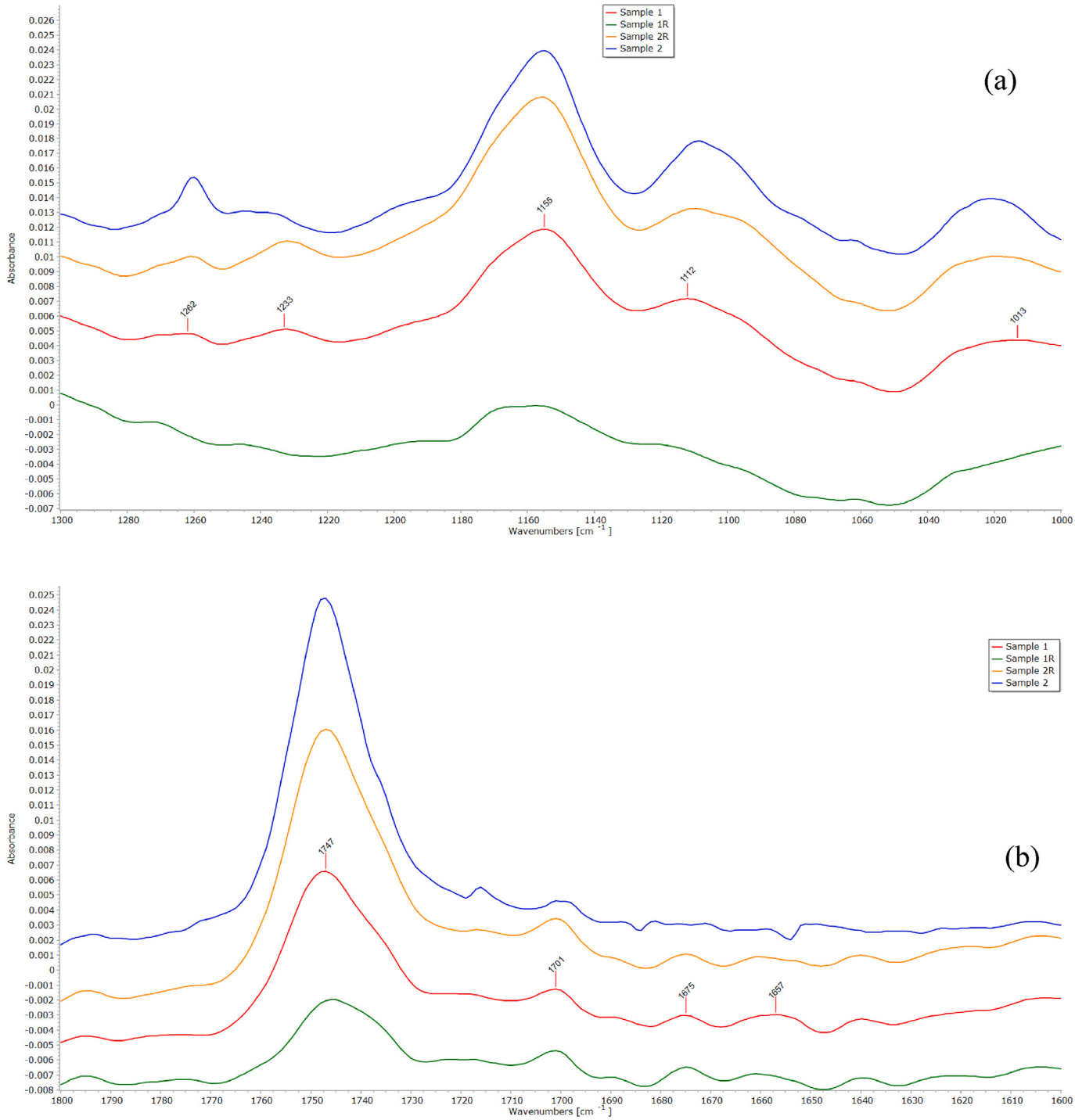


Fig. 3. FTIR spectra for waste and regenerated oil samples in the regions (a) 1300-1000 cm^{-1} and (b) 1800–1600 cm^{-1}.

In the 1300–1000 cm^{-1} spectral region, the used oils displayed multiple absorption bands at approximately 1262, 1233, 1155, 1112, and 1013 cm^{-1}, corresponding to C–O and S-O vibrations, characteristic of ester-based polymethacrylate viscosity modifiers and pour point depressant additives, and ZDDP antioxidant (Wooton, 2001), commonly present in formulated lubricants (Rudnick, 2018). These bands were strongly attenuated in the regenerated oils, indicating substantial removal of additive-derived polymeric species during extraction. No absorption features associated with ethylene glycol or ether-type antifreeze contaminants were detected in the 1040–1130 cm^{-1} range, confirming that coolant contamination was not present in the analyzed waste oils (Agência Portuguesa do Ambiente, 2021).

Importantly, with the exception of color (Fig. 6), the regenerated oils complied with the physicochemical specifications for base oils obtained from regeneration operations, demonstrating the capacity of supercritical CO_2 extraction to remove degradation products and contaminants. The regenerated extracts obtained under the optimal operating conditions of 140 bar, 14 mL/min CO_2 flow rate and a temperature of 40 °C appeared as brownish-red translucent liquids with significantly lower viscosity compared to the original waste oil feedstocks. Indeed, viscosity and viscosity index (VI) were reduced towards typical base oil values. This change in rheological behavior and appearance reflects the

Table 3
Preliminary study of the influence of SFE parameters in extraction yield for Sample 1 and 2.

Run	Temperature/ °C	Pressure/ bar	CO_2 density/ kg/m^3	Flow rate CO_2/ mL/ min	Residence time/min	Yield/ %
1	40	82	303.05	1	30	2.3
2	40	83	317.90	3	30	10.4
3	40	82	303.05	3	194	14.3
4	40	104	654.66	3	203	23.8
5	40	123	726.04	3	212	28.5
6	40	123	726.04	5	242	23.4
7	40	122	723.36	3	355	30.5
8	40	123	726.04	3	184	19.5
9	50	123	602.44	3	180	21.0
10	40	122	726.04	10	180	61.4
11	40	122	723.36	12	180	68.3
12*	40	122	723.36	12	181	61.1

Notes: Runs 1 to 11 were performed with Sample 1, Run 12 was performed with Sample 2.

Fig. 4. Waste lubricant oil (a) and regenerated oil (b) for Sample 1.

Table 4
Experimental points of the Box Behnken design and the experimental yield for Sample 1.

Run	Flow rate X_1 /mL/min	Pressure X_2 /bar	Yield / %
1	6	80	28.4
2	6	110	42.0
3	6	140	47.1
4	10	80	41.0
5	10	110	57.0
6	10	140	65.1
7	14	80	48.9
8	14	110	66.4
9	14	140	73.2

effective removal of high molecular weight degradation products, additive residues, and contaminants, as well as the original additive package (e.g., VI improvers, anti-wear agents). The resulting effluent is therefore a purified base oil rather than a finished lubricant. To meet industrial performance standards (e.g., API groups), the regenerated oil must be re-blended with a fresh additive package tailored to its intended application (engine oil, hydraulic fluid, etc.).

However, the regenerated oils exhibited ASTM D1500 color values in the range of 3 to 4, indicating the persistence of chromophoric species commonly associated with oxidation by-products and trace aromatic compounds. A gradual darkening of the extracts was also observed with increasing operating pressure, likely due to enhanced solubilization of heavier colored components at higher CO_2 densities. In conventional re-refining processes, such coloration is typically corrected through a final finishing step, such as adsorption using activated clay or bleaching earths, which selectively remove residual polar and colored species. Accordingly, the incorporation of a mild post-treatment polishing step would enable full compliance with base oil color specifications while preserving the environmentally friendly nature of the supercritical CO_2 regeneration process.

The combined physicochemical, elemental, and FTIR functional group analyses provide strong evidence of effective contaminant, additive residue, and degradation product removal during regeneration, supporting the suitability of supercritical CO_2 extraction as a standalone purification route. Following validation of regeneration performance, process parameters governing extraction efficiency were systematically investigated and optimized, as detailed in the subsequent sections.

3.2. *Preliminary screening of experimental conditions*

Regeneration of waste lubricant oils by supercritical CO_2 is governed by selective solvation and phase separation phenomena rather than by chemical transformation reactions (Brunner, 2004; McHugh and Krukonis, 2013). Under supercritical conditions, CO_2 penetrates the viscous oil matrix and is expected to preferentially dissolve low-polarity hydrocarbon fractions constituting the base oil, whereas more polar species such as oxidation products, metallic soaps, and additive-derived compounds are anticipated to exhibit limited solubility. Increasing pressure enhances CO_2 density and solvent strength, thereby extending solubilization toward heavier hydrocarbon components, while higher CO_2 flow rates are expected to promote solvent renewal and improved mass transfer. Upon depressurization, the reduction in solvent power should induce phase separation and recovery of regenerated oil, with poorly soluble contaminants remaining in the extraction vessel.

Although numerous factors can influence the SFE process, these mechanistic considerations indicate that pressure and CO_2 flow rate are expected to be the dominant variables controlling extraction performance. Nevertheless, temperature and extraction time were also included in the preliminary experimental design to comprehensively evaluate their potential contribution. Accordingly, four parameters, temperature, pressure, CO_2 flow rate, and extraction time, were investigated to identify the most influential factors governing SFE yield, and the preliminary results are presented in Table 3.

In supercritical fluid extraction (SFE), changes in pressure and temperature directly affect the solvent properties, causing variations in its density. Specifically, as pressure increases, the density of CO_2 rises, enhancing its dissolution capacity to behave more like a liquid (Brunner, 2004; McHugh and Krukonis, 2013). This improves its ability to dissolve the solute because higher CO_2 density leads to stronger van der Waals interactions between the CO_2 and the solute, facilitating better solubility. These interactions are crucial for the solubilization of compounds, especially those nonpolar, with higher molecular mass, like the ones present in base oils, or those that form stronger bonds with the solvent.

Another important parameter is the CO_2 flow rate. In run 1, for example, the flow rate is set at 1 mL/min, meaning there is little CO_2 circulating within the system. As a result, the amount of regenerated oil is relatively low, yielding a very low efficiency of only 2%. If the flow rate is insufficient, the CO_2 may become saturated with base oil before exiting, leading to an inefficient extraction. On the other hand, when the flow rate is increased, a greater amount of CO_2 is available, making mass transfer more efficient. This allows the CO_2 to transport a significantly larger quantity of base oil to the separator. The reason for this lies in the fact that a higher CO_2 flow rate results in a faster renewal of the solvent in contact with the sample in the reactor, thereby facilitating the transfer of base oil from the used oil sample to the supercritical CO_2. This effect is evident in the yields obtained: as the flow rate increases, the yield

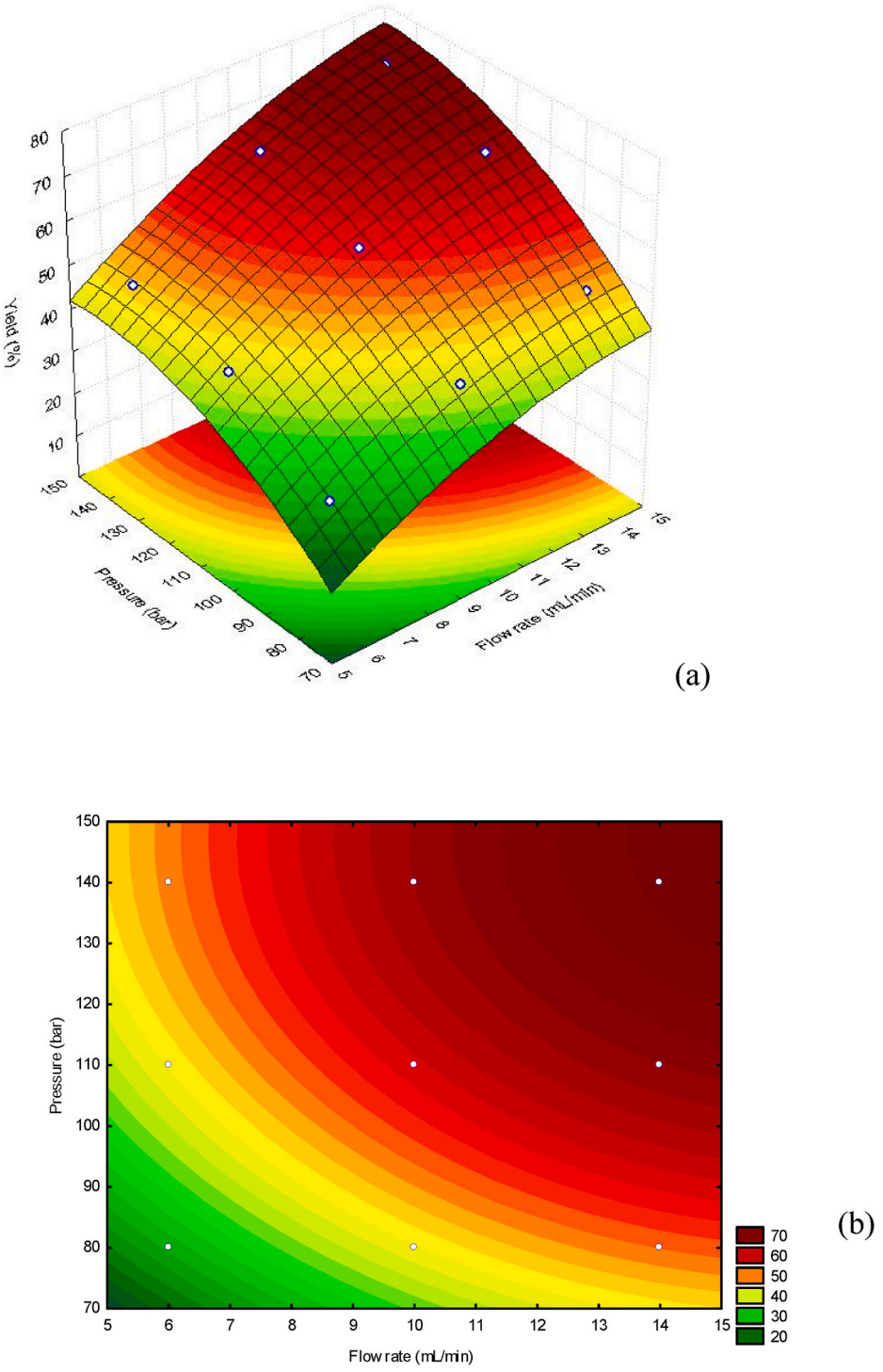


Fig. 5. Response surface plots for WLO regeneration yield (a) and 2D contour (b).

increases from 2 up to almost 70%. The slightly lower yield observed in Run 7 may be attributed to reduced solvent density at lower pressure, limiting solubilization capacity despite increased flow rate, as well as possible mass transfer constraints within the packed extraction vessel.

Fluid density decreases when the temperature increases. However, the increase in temperature results in accelerated mass transfer and is expected to improve the extraction yield but, in this case, temperature increase by 10 °C seemed to have a negligible effect on extraction yield. Considering that the implicit goal is to have the most sustainable experimental conditions, the 40 °C, just above the critical point, seem like and adequate temperature. Short residence times appear to negatively affect the yield extraction, for this particular case. Run number 12, aimed at testing the usefulness of the method for samples that test positive for coagulation, had a smaller but still significant yield of extraction.

Beyond influencing extraction yield, the operating conditions of supercritical CO_2 strongly affect regeneration selectivity and, consequently, the physicochemical quality of the purified oils. As pressure increases, the density of CO_2 rises markedly (Table 3), enhancing its solvating power toward higher molecular weight hydrocarbons. This behavior explains not only the increase in extraction yield observed at higher pressures but also the pronounced removal of polar degradation

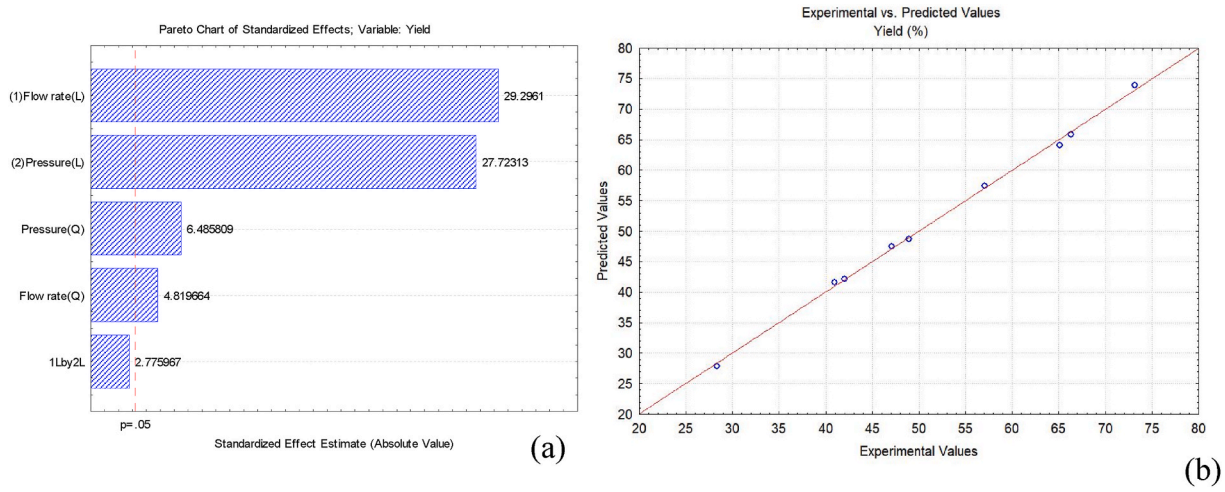


Fig. 6. Pareto chart of effects in variable Yield (a) and Experimental vs Predicted Values for Yield (b).

Table 5
Regression coefficients of the predictive model.

	Regression Coefficient	Std.Error	*t* Ratio	*p* Value
β_0	**−82.6746**	10.90526	−7.58116	0.004761
β_1	**5.9110**	0.98970	5.97254	0.009393
β_{11}	**−0.2106**	0.04370	−4.81966	0.017018
β_2	**1.3672**	0.17633	7.75384	0.004462
β_{22}	**−0.0050**	0.00078	−6.48581	0.007441
β_{12}	0.0114	0.00412	2.77597	0.069223

Note: coefficients in bold are statistically significant.

compounds evidenced by reductions in TAN, saponification number, water content, and oxidation-related FTIR bands in the regenerated samples. Higher CO_2 flow rates promote continuous renewal of the solvent phase within the extraction vessel, improving mass transfer and preventing early saturation of the supercritical fluid. This facilitates more efficient transport of solubilized base oil fractions while simultaneously enhancing the removal of residual contaminants and additive polymers, as reflected in the attenuation of ester- and sulfur-containing functional groups in FTIR spectra and the decrease in chlorine and sulfur concentrations. The progressive darkening of regenerated oils at higher pressures (Fig. 4) indicates increased extraction of heavier chromophoric compounds, highlighting a trade-off between regeneration completeness and color quality that can be readily addressed through conventional finishing steps. Overall, the combined effects of pressure-driven solvent density and flow-rate-controlled mass transfer govern not only the quantity of regenerated base oil recovered but also its chemical purity and physicochemical restoration. These findings demonstrate that optimization of supercritical CO_2 operating parameters is crucial for balancing high extraction yield with desirable oil quality.

3.3. Design of experiments for SFE

From the prior analysis, pressure (X_1) and flow rate (X_2) have been chosen as the main factors affecting yield. Within the possible regression models developed using a design of experiments (DoE) to link SFE output parameters with input factors (Kajdas, 2000a, 2000b) we have chosen Box–Behnken design (BBD), developed by Box and Behnken (1960) and frequently employed to determine the critical conditions for optimizing yield in extraction process (Nie et al., 2010; Turner et al., 2004). This model was selected due to its efficiency in requiring fewer experimental runs than central composite designs while providing reliable estimation of quadratic effects and avoiding extreme operating conditions, a characteristic very relevant in SFE systems, since excessive pressure or flow conditions may lead to operational constraints and compromise system stability. It consists of a full factorial design with three levels and an incomplete block design in such a way to present as a rotatable or nearly rotatable design and to avoid the extreme vertices. Input factors were chosen from the analysis of preliminary tests, and their levels are presented in Table 1.

The experimental results based on the full factorial design, for the set of 9 runs, done in duplicate, are shown in Table 4, determined by the standard least squares technique.

3.4. Optimization design

3.4.1. Response surface

The experimental data for the yield of the supercritical fluid extraction for Sample 1 is presented in Table 4. Statistical analysis and response surface regression procedure were applied. The effects of pressure and flow rate on the yield of extraction for WLO are shown in Fig. 5. In general, an increasing pressure and flow rate resulted in a higher extraction yield, thus attaining a higher amount of regenerated base oil.

The mathematical model representing the yield of the regeneration of WLO as a function of the independent variables within the region under investigation was expressed by the following second order polynomial Eq. (2):

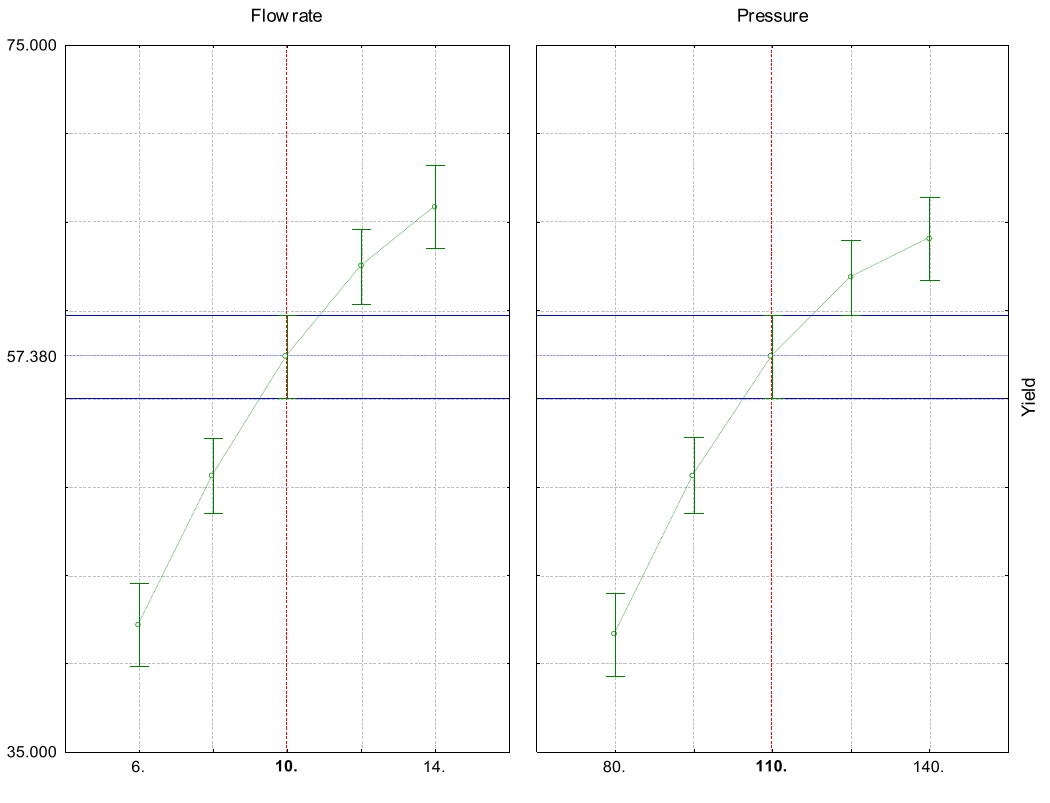
$$Y = -82.6746 + 5.9110X_1 - 0.2106X_1^2 + 1.3672X_2 - 0.005X_2^2 + 0.0114\,X_1X_2 \quad (2)$$

where Y is the yield of base oils regenerated, and X_1, X_2 are the variables for pressure and flow rate, respectively. Regression coefficients were determined by the standard least squares technique.

The effect of treatment variables as the linear, quadratic and interaction terms were tested for adequacy and fitness by analysis of variance (ANOVA). The statistical analysis, based on a 95% confidence level, indicated that the linear and quadratic effects of the two factors were statistically significant (p-values <0.05) (Table 5). In contrast, interactions between the flow rate (X_1) and pressure (X_2) did not produce a significant effect on this response ($p > 0.05$). Pareto chart of effects (Fig. 6) also reinforces the observations that interactions between variables do not impact in yield. Still, there is a very good agreement between experimental and predicted values for yield with this model.

3.4.2. Sensitivity analysis and desirability optimization

The influence of individual factors on the extraction yield was further evaluated through a perturbation diagram, which allows for a



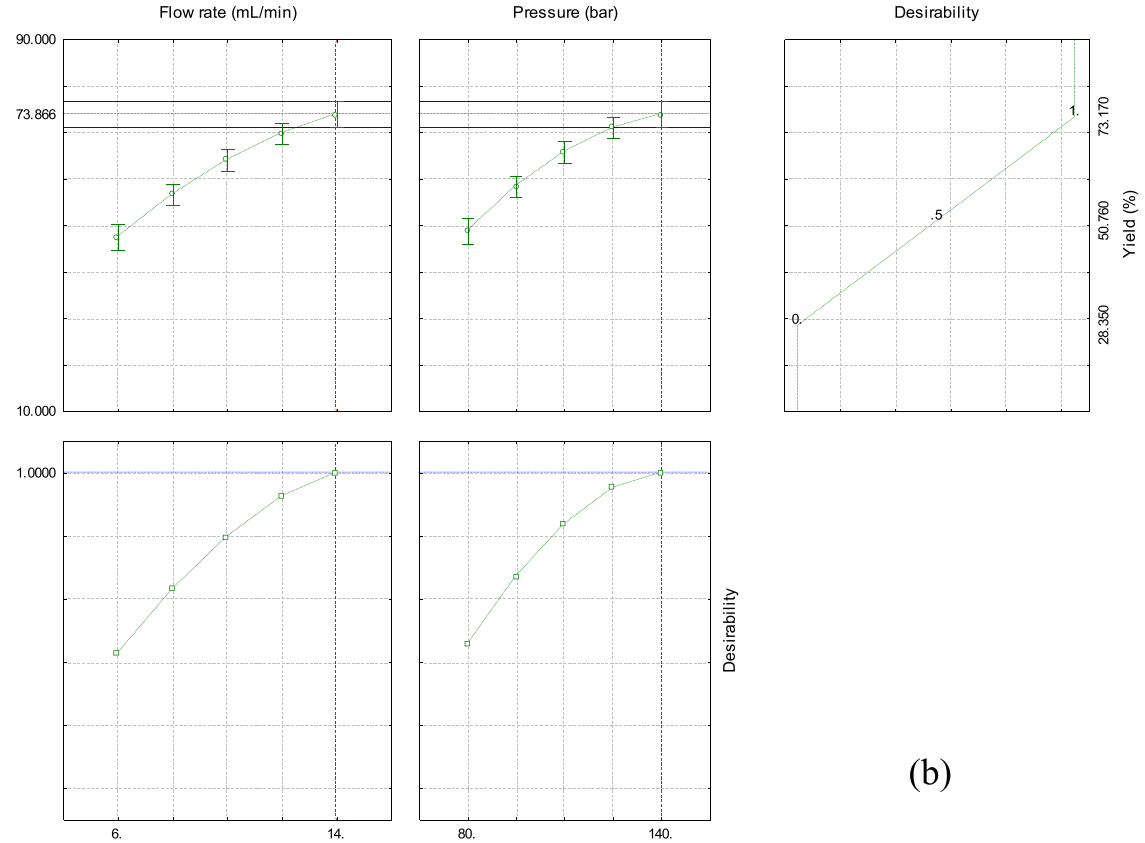


Fig. 7. Optimization and sensitivity analysis: (a) perturbation diagram showing the effect of CO_2 flow rate (X_1) and pressure (X_2) on the yield, (b) profile for predicted values and desirability for the optimized extraction.

Table 6
Experimental validation of predictive model.

Run	Pressure/ bar	Flow rate CO_2/mL/min	Predicted Yield/%	Experimental Yield/%	RE[a)] (%)
10	122	10	61.7	61.4	0.4
11	122	12	67.0	68.3	1.9
12*	122	12	67.0	61.1	9.7

Notes: [a)] Relative error, determined as $RE = \left|\frac{Experimental\ Yield - Predicted\ Yield}{Experimental\ Yield}\right| \times 100$

direct comparison of the sensitivity of the response to each variable while others are kept constant at their reference points, shown in Fig. 7a. As observed, the slope of the curve for pressure (X_2) is considerably steeper than that for the CO_2 flow rate (X_1), indicating that the process yield is more sensitive to changes in pressure. This behavior is consistent with the density-dependent nature of supercritical CO_2, where higher pressures significantly enhance the solvating power of the fluid. The distinct curvature in the traces reinforces the adequacy of the quadratic model in describing the non-linear behavior of the system near the optimal region.

The regression models were subsequently utilized to create a prediction profiler and optimize the overall desirability function, helping to identify the best extraction conditions for various types of WLO (Fig. 7). The 2D and 3D response surface plots previously shown in Fig. 5 indicated that optimal conditions are found near the maximum experimental limits, specifically 140 bar and 14 mL/min.

Fig. 7b illustrates the prediction profile and desirability function for the extraction of WLO Sample 1. The green dashed lines represent the predicted responses with 95% confidence intervals, while the desirability function, along with the chosen control points, is shown in the rightmost column. The bottom row highlights the desirability for each individual factor, confirming that the optimal conditions are achieved at the extremes of the evaluated parameters, where a maximum predicted yield of approximately 70% is attained with a total desirability of 1.0.

3.4.3. Validation of predictive models

To validate the accuracy of the predicted model and rule out any bias, experimental verification was conducted by reproducing certain conditions from the preliminary tests within the established experimental range. As it is observed (Table 6), model is able to predict yield quite accurately, with less than 2% deviation values. Additionally, it gives a reasonable prediction of the yield of extraction for Sample 2.

4. Conclusions

The results obtained with this study showed that supercritical extraction with CO_2 is an effective method for the regeneration of waste lubricant oils, including oils that would be segregated due to coagulation phenomena. Performance of supercritical extraction with CO_2 slightly varies depending on the oil sample, showing a superior yield for non-coagulating samples (above 70%), yet with a very reasonable yield for samples that exhibit coagulation (around 60%). Regeneration process has shown to be able to remove most oxidation products and additives from the samples, reducing viscosity and viscosity index to values consistent with pure base oils. The regenerated samples comply with the technical specifications for base oils (SN-100 or SN-150) resulting from the regeneration process, as defined by the Portuguese law in force, with color being the only parameter that requires and additional correcting step.

Process was modeled using response surface methodology and optimal extraction conditions were determined using a profiler and desirability approach. As a result, an operating pressure of 140 bar and a flow rate of at least 14 mL/min were chosen as the optimal conditions. Sensitivity analysis through perturbation diagrams further confirmed that operating pressure is the most influential parameter, directly governing the CO_2 solvating power and extraction efficiency.

To our knowledge, this study provides the first demonstration that $scCO_2$ extraction can regenerate both complex, multi-source WLO blends and KOH-coagulated oils—achieving extraction yields only ~10 % lower for coagulated samples (60 %) compared to non-coagulating ones (70 %). The resulting purified base oil, while requiring re-additivation to meet specific finished lubricant performance standards, represents a high-quality intermediate for the circular economy. By overcoming the coagulation barrier, this methodology significantly broadens the scope of green SFE technologies for hazardous WLO feedstocks previously considered unrecoverable.

CRediT authorship contribution statement

Cecília I.A.V. Santos: Writing – review & editing, Writing – original draft, Visualization, Validation, Supervision, Software, Resources, Project administration, Methodology, Investigation, Funding acquisition, Formal analysis, Data curation, Conceptualization. **Sofia B. Jerónimo:** Investigation, Data curation. **Licínio M. Gando-Ferreira:** Writing – review & editing, Supervision, Methodology, Funding acquisition, Formal analysis.

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.

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Data availability

Data will be made available on request.

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