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# Computer Aided Evaluation of Solvent Extraction for Light Hydrocarbon Using Carbon Dioxide

# Evaluación Asistida por Computadora de la Extracción de Solventes para Hidrocarburos Livianos usando Dióxido de Carbono

# Avaliação Auxiliada por Computador da Extração por Solvente para Hidrocarboneto Leve usando Dióxido de Carbono

**Viktor Oswaldo Cárdenas Concha[1*]; Luz Sheyla Cárdenas Concha[2]; Leandro Lodi[3]; Juliana Otavia Bahú[4]; Diana Paola Figueroa Hernández[5]; Willian Amaro-Marchioli[1]; Nelson Antonio Moreno Monsalve[5]; Jeffrey Leon-Pulido[5*]**

[1]Chemical Engineering Department, Federal University of São Paulo (UNIFESP),
Diadema, São Paulo, Brazil, 09913-030
[2]Universidad Nacional de Trujillo, Avenida Juan Pablo II, Trujillo, Perú, 13011
[3]Institute of Science and Technology, Federal University of Alfenas (UNIFAL), Poços de Caldas 37715-400
[4]School of Chemical Engineering, University of Campinas (UNICAMP),
Campinas, São Paulo, Brazil, 13083-852
[5]Faculty of Engineering, Chemical Engineering Program, EAN University, St. 79 #11-45, El Nogal, Bogotá,
Cundinamarca, Colombia, 110221

*viktor.cardenas@unifesp.br

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## Abstract

Different process of separation was used in the chemical industry, in particular, extraction is a process used to increase the quality of resins in oil removing impurities like organics solids and heavy metals. Supercritical carbon dioxide offers high selectivity at the end of the extraction process of light hydrocarbons from heavy oils mixture. A simulation technique in Aspen Plus ® software was used to develop the process and sensitivity analysis of the extraction configuration. The simulation of extraction process includes two output streams: the first one, a top stream (unpaved oil), and the second one a bottom stream (asphalt residue). A steady state methodology was implemented for process simulation. The sensitivity analysis was used to assess the influence of variables such as solvent flow rate, temperature and pressure. It was found a significant increase in the flow rate of unpaved oil when the solvent flow rate is increased. Optimal extraction values were selected depending on temperature and pressure effects over the process. An increase in temperature directly enhances the quality of API gravity. In certain occasions, an increase in pressure affects the light oils extraction because of product drag.

**Keywords:** Computer Aided-Design, extraction, hydrocarbon, carbon dioxide, simulation, process.

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## Resumen

En la industria química se utilizaron diferentes procesos de separación, en particular, la extracción es un proceso utilizado para aumentar la calidad de las resinas en el aceite eliminando impurezas como sólidos orgánicos y metales pesados. El dióxido de carbono supercrítico ofrece una alta selectividad al final del proceso de extracción de hidrocarburos ligeros a partir de una mezcla de aceites pesados. Se utilizó una técnica de simulación en el software Aspen Plus ® para desarrollar el proceso y el análisis de sensibilidad de la configuración de extracción. La simulación del proceso de extracción incluye dos flujos de salida: el primero, un flujo superior (petróleo sin pavimentar), y el segundo, un flujo inferior (residuo de asfalto). Se implementó una metodología de estado estacionario para la simulación de procesos. El análisis de sensibilidad se utilizó para evaluar la influencia de variables como el caudal de disolvente, la temperatura y la presión. Se encontró un aumento significativo en la tasa de flujo de petróleo cuando se incrementa la tasa de flujo de solvente. Los valores de extracción óptimos fueron seleccionados dependiendo de los efectos de temperatura y presión sobre el proceso. Un aumento en la temperatura mejora directamente la calidad de la gravedad API. En determinadas ocasiones, un aumento de presión afecta a la extracción de crudos ligeros por efecto del arrastre del producto.

**Palabras clave:** Diseño asistido por computadora, extracción, hidrocarburo, dióxido de carbono, simulación, proceso.

## Resumo

Diferentes processos de separação são usados na indústria química, em particular, a extração é um processo usado para aumentar a qualidade das resinas no óleo removendo impurezas como sólidos orgânicos e metais pesados. O dióxido de carbono supercrítico oferece alta seletividade ao final do processo de extração de hidrocarbonetos leves da mistura de óleos pesados. Uma técnica de simulação no software Aspen Plus ® foi utilizada para desenvolver o processo e a análise de sensibilidade da configuração da extração. A simulação do processo de extração inclui dois fluxos de saída: o primeiro, um fluxo superior (óleo não pavimentado) e o segundo um fluxo inferior (resíduos asfálticos). Uma metodologia de estado estacionário foi implementada para simulação de processos. A análise de sensibilidade foi utilizada para avaliar a influência de variáveis como: vazão do solvente, temperatura e pressão. Foi constatado um aumento significativo na vazão de óleo quando a vazão de solvente é aumentada. Os valores ótimos de extração foram selecionados dependendo dos efeitos da temperatura e pressão sobre o processo. Um aumento na temperatura aumenta diretamente a qualidade da gravidade API. Em certas ocasiões, um aumento de pressão afeta a extração de óleos leves devido ao arraste do produto.

**Palavras-chave:** Desenho assistido por computador, extração, hidrocarboneto, dióxido de carbono, simulação, processo.

## 1. Introduction

The production of lubricants has been of great importance for the petroleum industry through solvent route, which implies the identification of new loads to feed the existing units, as well as, to study several solvents to be used in the extraction process. During the last two decades, the average quality of oil derived from refineries has decreased by approximately 2 points in the American Petroleum Institute gravity scale - API scale, along with an increase in inorganic solids, heavy metals and heteroatoms [1,2]. Petroleum consists of colloidal systems containing hydrocarbons which can be classified into alkanes, naphthalene, and aromatics, the energy consumption and exergy of process describes the thermodynamics relation and equilibrium for process simulation [18, 19, 20]. There are two types of particles: dispersed in the colloidal solution (asphaltenes) and petroleum resins; asphaltenes are hydrocarbons with high molar weight and contain mainly oxygen, sulfur, organic particles, inorganic salts, among other components. One of the most used processes to "purify" resins is the extraction [3].

Extraction is a process for reducing the asphaltenes and metals of desired component through the solubility of the appropriate solvent (e.g. propane, pentane, heptane or carbon dioxide) resulting in its final flocculation [4]. Wilson, Keith, & Haylett [5] developed a separation process based on phase equilibrium, which became the basis for the deasphalting process to propane still in use during refining of lubricating oils. Such process makes use of the change in the solvency power of a liquid near its critical point. Several systematic studies of phase equilibrium of carbon dioxide determined a characteristic point for each substance along the liquid-vapor equilibrium zone and with the gradual increase of pressure and temperature, that is, the critical point, located in Figure 1 [6, 7]. CO2 is a solvent capable of extracting light hydrocarbons from a mixture of heavy oils. It is used as solvent for supercritical extraction due to its high selectivity at the end of the process [8].

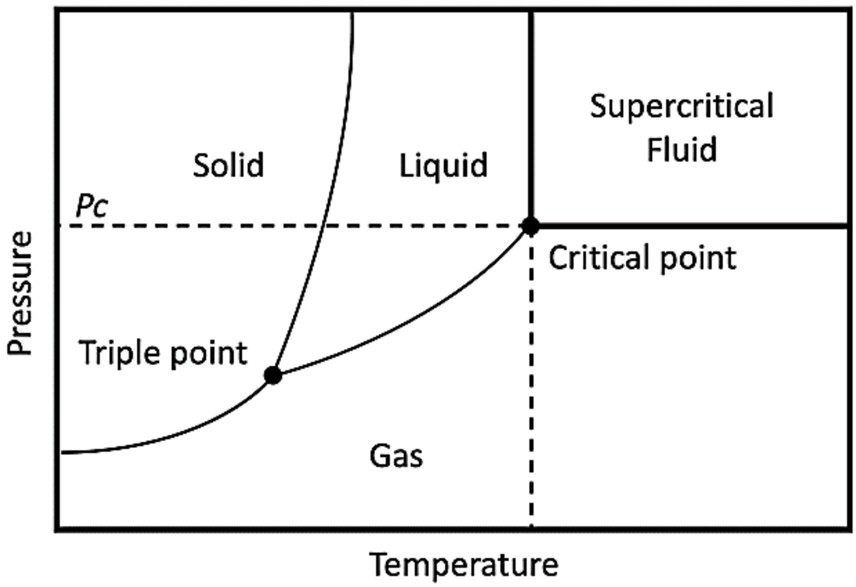


**Figure 1.** Pressure x Temperature diagram of a substance in its supercritical state. Source: Adapted from [6].

### 1.1. Thermodynamic modeling

To perform extraction simulation, it is necessary to define the best thermodynamic model that can explain appropriately solvent extraction. Based on current contributions, it was selected the thermodynamic model Predictive Soave-Redlich Kwong (PSRK), showed in Equation 1 [1, 7, 9]. The PSRK template is based on the Soave-Redlich-Kwong- Equation of State (SRK-EOS), with modifications suggested by Mathias & Coperman [10], and it makes use of the Universal Functional-group Activity Coefficients (UNIFAC) Original method to calculate the parameters of the mixture, with the first order modified by Huron & Vidal mixing rule (MHV1). However, PSRK uses a value of q1 different from that suggested by Michelsen in Equation 1, which leads to better results at high pressure. Where $G^E$ is the Gibbs free energy in excess and is related intrinsically with the pressure of the half together with the α property defining the mixed pattern according to the UNIFAC properties.

$$\alpha_{mix} = \frac{1}{q_1}\left[\frac{G^E}{RT} + \sum_{i=1}^{C} x_i \ln\left(\frac{b}{b_1}\right)\right] + \sum_{i=1}^{C} x_i \alpha_i \quad (1)$$

At high pressures, PSRK predicts liquid-vapor equilibrium with greater accuracy than Modified Huron-Vidal seconder-order model (MHV2); however, the quality of their predictions deteriorates when the mixing components differ in molecular sizes. PSRK also presents all the advantages of a state equation since it can be applied to supercritical component systems and allows the calculation of densities, enthalpies, and other properties, even in systems with polar components [11, 12]. Asymmetric systems are commonly found in processes with supercritical fluids, where the solutes are larger in size than the solvent, usually $CO_2$ or paraffin of low molar mass, which is why the state equations are modified, often empirically, to achieve a better representation of the equilibrium of these complex systems.

## 2. Methods and materials

### 2.1. Simulation of the extraction process

In the assembly of a process to be simulated, it is necessary the characterization of raw material. For this study, petroleum with characteristics of heavy oil (low-grade API = 7.1) was used. Tables 1 and 2 show the physical-chemical characterization, the PEV curve or true boiling point of the oil, as well as the API gravity for each distillate percentage. These data were fed to the simulation software in order to generate the pseudo-components, which will be used in the simulation.

**Table 1.** Pseudo-Components of raw material

| Pseudo-components | Temperature (°C) | °API | Specific gravity | MW | Critical Temperature (°C) | Critical Pressure (bar) |
|---|---|---|---|---|---|---|
| PC312C | 311,96 | 14,48 | 0,97 | 223,21 | 528,11 | 20,52 |
| PC323C | 322,60 | 13,84 | 0,97 | 232,22 | 537,93 | 19,88 |
| PC336C | 336,48 | 13,03 | 0,98 | 244,32 | 550,65 | 19,10 |
| PC350C | 350,35 | 12,25 | 0,98 | 256,80 | 563,26 | 18,36 |
| PC364C | 364,21 | 11,50 | 0,99 | 269,66 | 575,74 | 17,66 |
| PC378C | 378,06 | 10,78 | 0,99 | 282,90 | 588,12 | 17,00 |
| PC392C | 391,91 | 10,08 | 1,00 | 296,51 | 600,40 | 16,38 |
| PC406C | 405,88 | 9,39 | 1,00 | 310,62 | 612,69 | 15,79 |
| PC420C | 419,71 | 8,75 | 1,01 | 324,99 | 624,75 | 15,23 |
| PC441C | 440,85 | 7,75 | 1,02 | 347,53 | 643,11 | 14,44 |
| PC467C | 467,42 | 6,65 | 1,02 | 377,26 | 665,70 | 13,52 |
| PC497C | 497,03 | 5,93 | 1,03 | 413,49 | 689,43 | 12,50 |
| PC519C | 518,99 | 5,19 | 1,04 | 440,66 | 707,39 | 11,86 |
| PC552C | 551,59 | 2,95 | 1,05 | 476,69 | 736,91 | 11,23 |
| PC580C | 580,36 | 1,16 | 1,07 | 508,98 | 762,49 | 10,70 |
| PC608C | 608,04 | -0,50 | 1,08 | 539,75 | 786,90 | 10,23 |
| PC637C | 636,69 | -2,24 | 1,09 | 570,36 | 812,25 | 9,80 |
| PC683C | 682,82 | -5,47 | 1,12 | 612,21 | 854,55 | 9,27 |
| PC720C | 720,17 | -7,40 | 1,14 | 647,37 | 886,66 | 8,79 |

**Table 2.** Main simulation equipment and their process variables

| TAG | Equipments | Temperature (°C) | Pressure (bar) |
|---|---|---|---|
| **MIXER** | **Mixer** | - | - |
| TC1 | Exchange | 150 | 300 |
| EXT | Extractor | 150 | 300 |
| TC2 | Exchange | 150 | 150 |
| TC3 | Exchange | 150 | 150 |
| SEP1 | Separator 1 | 150 | 150 |
| SEP2 | Separator 2 | 150 | 150 |

The most assertive method for determining the fractions of charge is through experimental measurements. However, this method is not valid for all petroleum mixtures; for these cases statistical and simulated methods are employed [13, 21]. A pseudo-component represents each temperature range. It is understood that the normal boiling temperature of it corresponds to the average temperature of the interval [14]. Through the data of Table 1 generated, it is possible to cross the information and characterize the pseudo-components generated as a function of the molar mass and its API gravity. Figure 2 shows the correlation between both and again we note the inverse proportion of each characteristic.

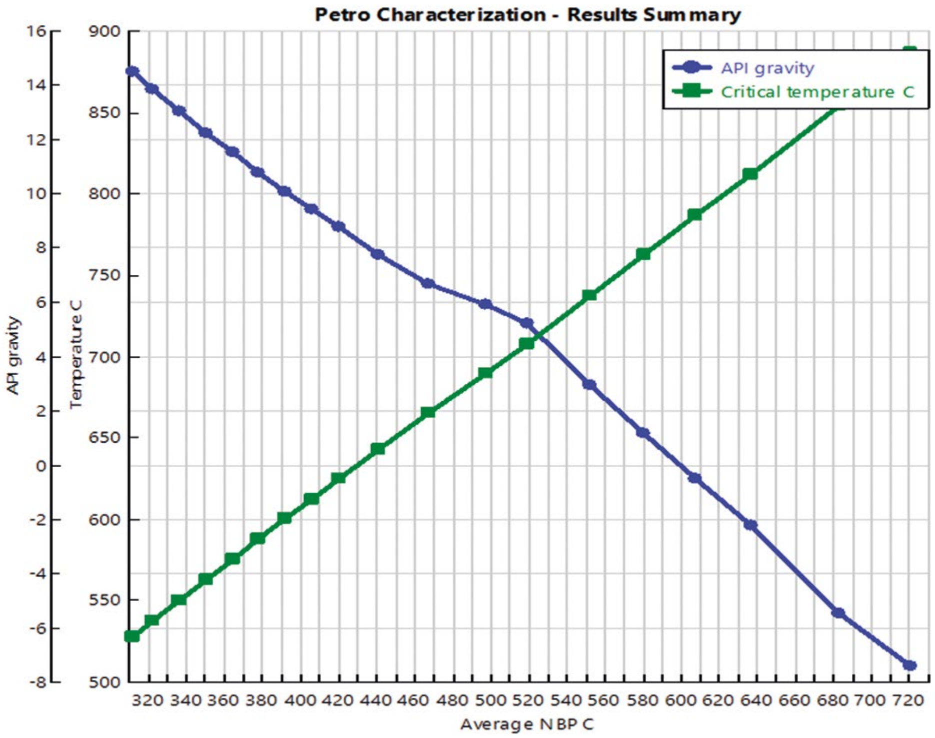


**Figure 2.** Characteristics of Pseudo-Components

The virtual $CO_2$ extraction plant was built in the Aspen Plus ® commercial simulator, where the main equipment and process streams were added and their operating parameters were identified in Figure 3 and Tables 3, 4 and 5.

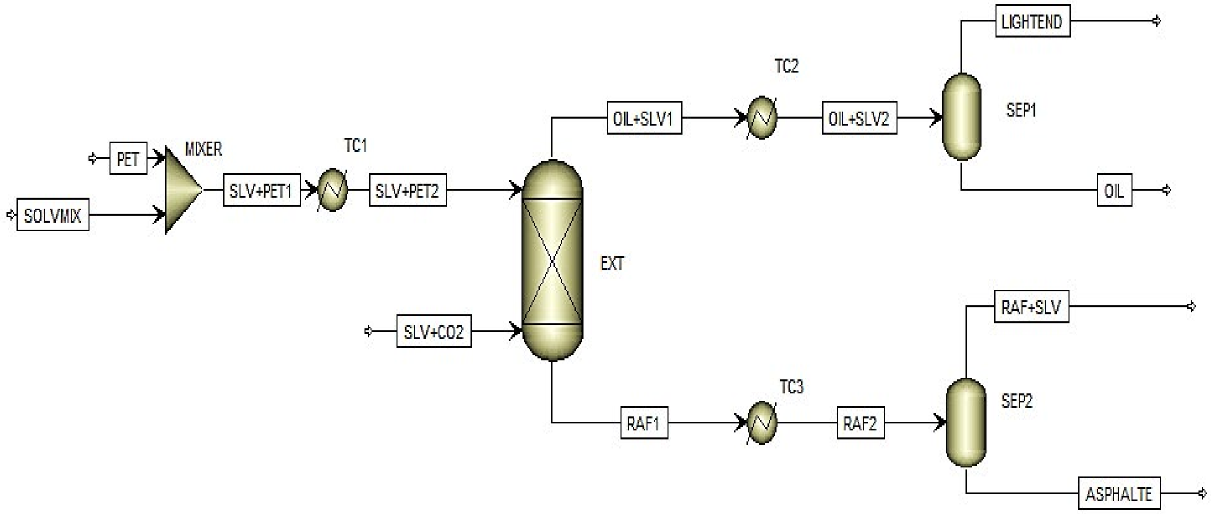


**Figure 3.** Flowchart of the deasphalting process.

**Table 3.** Characterization of the main streams of the process

| | Flow IN | Flow OUT | |
|---|---|---|---|
| | PET | ODES | RASF |
| Temperature (°C) | 135,00 | 150,00 | 150,00 |
| Pressure (bar) | 300,00 | 150,00 | 150,00 |
| Flow rate (kg/hr) | 200,00 | 9,10 | 182,20 |
| Vapor Fraction | 0,00 | 0,00 | 0,00 |
| Average MM (g/mol) | 318,10 | 179,50 | 350,50 |
| °API | 7,10 | 43,90 | 11,90 |

Next, Tables 4 and 5 present the main operating parameters used in the process equipment (DAO separation vessels).

**Table 4.** Characterization of streams: Input and output of the separator 1

| | **Flow IN** | **Flow OUT** | |
|---|---|---|---|
| SEP1 | **OIL+SLV2** | **LIGHTEND** | **OIL** |
| Temperature (°C) | 150,00 | 150,00 | 150,00 |
| Pressure (bar) | 150,00 | 150,00 | 150,00 |
| Flow rate (kg/hr) | 1062,10 | 1053,00 | 9,10 |
| Enthalpy (Gcal/hr) | -2,20 | -2,20 | 0,00 |
| Vapor Fracion | 1,00 | 1,00 | 0,00 |
| Average MM (g/mol) | 44,60 | 44,30 | 179,50 |

**Table 5.** Characterization of the streams: Input and output of the separator 2

| | Flow IN | Flow OUT | |
|---|---|---|---|
| SEP2 | **RAF2** | **RAF+SLV** | **ASPHALTE** |
| Temperature (°C) | 150,00 | 150,00 | 150,00 |
| Pressure (bar) | 150,00 | 150,00 | 150,00 |
| Flow rate (kg/hr) | 187,90 | 5,80 | 182,20 |
| Enthalpy (Gcal/hr) | -0,10 | 0,00 | -0,10 |
| Vapor Fracion | 0,20 | 1,00 | 0,00 |
| Average MM (g/mol) | 288,80 | 44,00 | 350,50 |

**Table 6.** Final yields of the process.

| | |
|---|---|
| Deasphalted Oil Yield | 4,38% |
| Asphalt Yield | 93,60% |
| Solvent Recovery | 99,27% |

As can be seen, the yield is low, however, Liu et al.[15] describes that $CO_2$ extractions present low yields and high selectivity for light components (hydrocarbons). This phenomenon of low $CO_2$ extraction yields is due to the solvent's own characteristics. In normal conditions the $CO_2$ is apolar, but as it happens changes in the temperature and pressure of the system the solvent happens to present/display polarity quadripolar, that is, that defines four moments dipole, bringing it to an opposite plateau of the compounds to be extracted. Another point to be evaluated is that the carbon dioxide, when worked at temperatures above 66 ° C, shows a decrease in density, leading to a sharp fall in the extraction, around 20% to 30%, thus suggesting the follow- solvents. These two factors, together, justify the low extraction of deasphalted oils using $CO_2$ a critical solvent[7]. In this way, once the characterization of the residue was carried out and the process flowchart was assembled, the tests were carried out in the simulator.

## 3. Results and discussion

The following are results obtained in the simulation of the extraction process, as well as the sensitivity analysis performed for the process. Figure 6 clearly synthesizes the incoming stream distillation curves compared to the ODES (deasphalted oil) and RASF (asphaltic residue) generated at the end of the process.

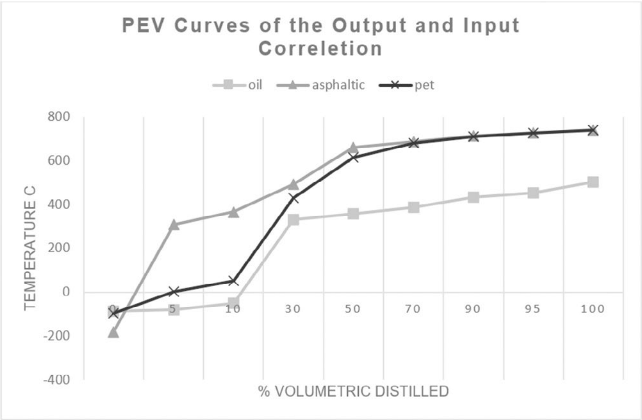
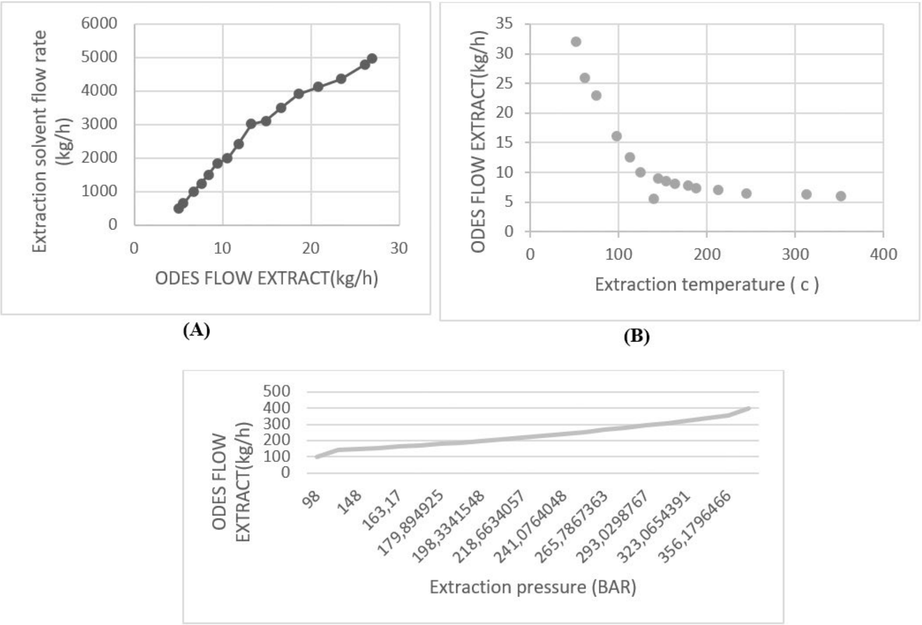


**Figure 4.** PEV curves of the products and the sample fed

In Figure 4, it can be observed that the profiles of the PEV curves generated for the process output streams (extract and raffinate) are different between them. It can be noticed that the PET of the main operating parameters used in the process equipment (DAO) is in the inferior part as expected because it presents components of low molar mass, consequently, the lower boiling point. In contrast, it is observed that the stream corresponding to the asphalt (raffinate) residue is in the upper part, showing that the composition of the same is mostly composed of heavy compounds, thus raising the boiling temperature thereof, and as already expected, the oil fed is in the middle of the two curves mentioned above.

### 3.1. Sensitivity analysis

Figure 5 (A), (B) and (C) show the behavior of deasphalted oil (ODES) extraction flow in relation to $CO_2$ flow, extraction temperature, and extraction pressure.



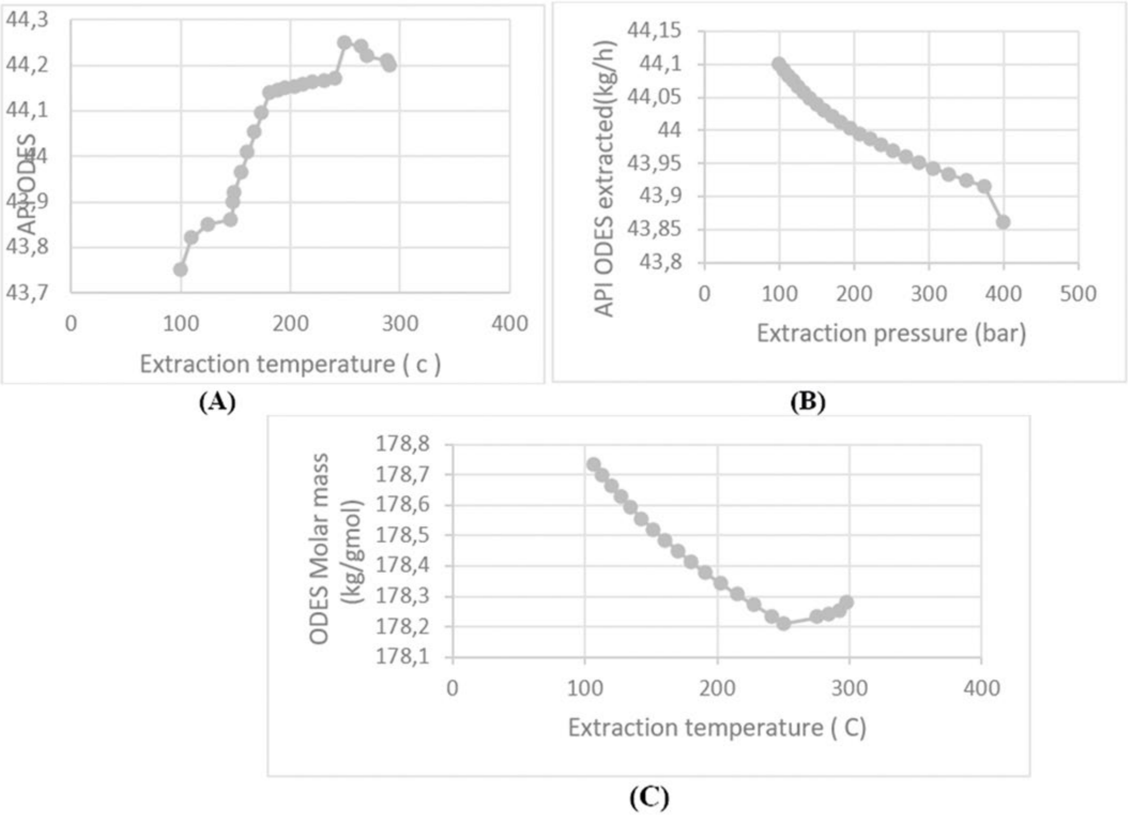
**(A)**

**(B)**

**(C)**

**Figure 5.** Effect of solvent flow rate, temperature and pressure on ODES flow

As expected, as the solvent flow increases the amount of extracted oils also increases ODES, which indicates the possibility of finding the ideal amount of $CO_2$ in the extraction process. Figure 5 (B) and (C) show that the temperature and pressure parameters directly influence the extraction flow, and to obtain the best yield the ideal is to work at temperatures below (100 ° C) and at higher pressures (350 to 400bar), supercritical $CO_2$ conditions [16, 17, 22, 23]. However, when evaluating the API gravity that is extracted under these conditions of temperature and pressure (100 ° C and 400bar), it is seen that there is some contradiction. Figures 6 (A) and (B) show that under these conditions API gravity are slightly lower than if working under reverse conditions. However, they would still be high-grade API oils, so there would be a high index extraction of light oils.



**(A)** **(B)**

**(C)**

**Figure 6.** Effect of solvent flow rate, temperature and pressure on ODES API gravity

Figure 6 (A) shows the behavior of the API gravity as a function of the extraction temperature. As it can be seen there is a tendency for the API gravity to increase with increasing temperature, which means a selectivity of the top product as a function of temperature. However, Figure 6 (B) shows that the pressure exerts a decrease of the API gravity, which implies in the drag of components of high molar mass to the top stream (extract), not being favorable for the extraction process of light oils. Figure 6 (C) corroborates the results obtained in Figure 6 (A), showing that the molar mass of the top stream decreases with increasing temperature, which means extraction of light (high-grade API) components.

## 4. Conclusions

The process of extraction of light hydrocarbons using $CO_2$ under supercritical conditions presented data consistent with those found in the literature. It can be observed that it had a low yield extraction as observed in Table 6 and high selectivity, which was contacted by the high API gravity of the extract stream. Two output streams (extract = DAO and raffinate = RASF) were obtained, which presented different PEV curves, showing the $CO_2$ efficiency as a solvent. In addition, the sensitivity analysis confirmed that temperature is a parameter that influences the yield and quality of the DAO stream and that the pressure has no significant influence on the process. However, it should be emphasized that the characterization of the raw material is an important factor in the extraction process.

## 5. Acknowledgments

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