



Selective separation of solvent from deasphalted oil using CO₂ for heavy oil upgrading process based on solvent deasphalting

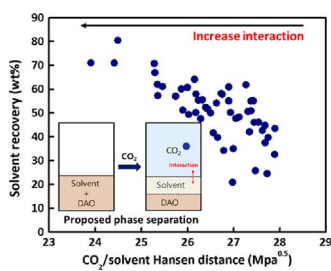


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GRAPHICAL ABSTRACT



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ABSTRACT

The solvent deasphalting (SDA) process is a heavy oil upgrading process in which deasphalted oil (DAO) is extracted from heavy oil feedstock by precipitating asphaltene using an excess amount of alkane solvent (C3–C6). After the extraction, solvent recovery should be carried out for separating the solvent from the DAO in order to recycle the expensive solvent. In the conventional solvent recovery method, the mixture of solvent and DAO is heated to evaporate the solvent, which requires massive heat energy, resulting in reduced process efficiency. In this study, CO₂ is applied for the first time to selectively separate solvent from DAO at a relatively low temperature. The experimental results in a batch separator indicate that the temperature required for high solvent recovery of over 80% decreases from 200 °C to 40 °C when using CO₂ compared to the conventional method. The theoretical approach using Hansen distance calculation based on the Hansen solubility parameter (HSP) was used to verify the mechanism of solvent separation using CO₂. The results suggest that the increase in the interaction between CO₂ and solvent causes the separation of solvent from DAO, leading to an increase in solvent recovery. Also, numerical simulation results show the possibility of continuous operation for solvent recovery using CO₂.

1. Introduction

The global demand for petroleum (crude oil) has increased due to the steady increase in population and industrial development [1,2]. In order to meet the ever-increasing oil demand, unconventional oils such as heavy oil, extra heavy oil, oil sand bitumen, and residual oils should

be utilized. However, these unconventional and residual oils are limited for application to conventional refinery processes as feedstocks because of their high viscosity and high content of heteroatoms such as heavy metal (Ni, V), nitrogen, and sulfur. For these reasons, several processes to convert heavy oils into light oils have been developed, including carbon rejection (thermal cracking, catalytic cracking, and solvent

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extraction) and hydrogen addition (catalytic and non-catalytic hydrogenation) methods [3,4]. Since most of these processes require high energy consumption with severe temperature or pressure conditions [5], the development of energy-efficient processes is imperative to save operating cost.

The solvent deasphalting (SDA) process based on liquid-liquid extraction is a promising option for reducing the energy requirement in heavy oil upgrading because the SDA process is operated at a mild temperature (100–150 °C) compared with thermal cracking processes (> 400 °C). In the SDA process, vacuum residue (VR), the residue from vacuum distillation of crude oil, is normally used as heavy oil feedstock. The feed is treated based on extraction using light alkane solvents (C3–C6) to produce asphaltene-removed light components, called deasphalted oil (DAO); the remaining VR precipitates in the form of asphaltene at the bottom of the extractor. DAO yield and quality are dependent on the type of process scheme, the properties of the heavy oil feedstock, and the solvent used [6–10]. Normally, the amount of solvent consumed in the extractor is four to ten times the amount of heavy oil feed on a weight basis. The solvent used in DAO extraction is expensive, therefore the solvent recovery is as important as DAO extraction for the development of economic SDA process.

In the 1970s, Kerr-McGee's residual oil supercritical extraction (ROSE) was developed as a commercial SDA process [11–13]. In this process, solvent is recovered by heating the mixture of solvent and DAO to a supercritical condition in order to generate two different phases. However, this method requires additional heat energy to separate the solvent and DAO phases, leading to an increase in the operating cost of the SDA process. Several studies have focused on reducing the amount of energy spent in the solvent recovery step by enhancing the process scheme [14], generating energy from the expanding high pressure stream [15], and treating the solvent with sonication below the supercritical condition [16]. Studies have also been carried out to investigate the effect of operating parameters on the efficiency of the solvent recovery step [17,18]. However, none of these studies has contributed to reducing the severity of the operating temperature in the solvent recovery step.

In this study, a novel scheme of solvent recovery using CO₂ is proposed to selectively separate solvent from DAO. While studies have been performed to separate ionic liquid and solvent using supercritical CO₂ [19,20], to the best of our knowledge, the application of CO₂ to the solvent recovery step in the SDA process has not been reported. Therefore, in this study, experiments were performed in a batch separator to confirm the possibility of CO₂ usage in solvent recovery and to study the effect of temperature and pressure on solvent recovery using CO₂. In order to investigate the mechanism of solvent and DAO separation by introducing CO₂, theoretical studies based on the Hansen solubility parameter were carried out. In addition, numerical simulation was adopted to demonstrate the feasibility of the continuous operation of the solvent recovery step using CO₂.

2. Experiments

2.1. Materials and characterization

HPLC grade *n*-pentane (> 99.0 wt% purity, Sigma Aldrich) and CO₂ (in the form of pelletized dry ice) were used in the solvent recovery experiments. *n*-pentane was selected as the solvent for experimental tests because it exists as a liquid at room temperature, providing easier handling than *n*-propane and *n*-butane which exist as vapor at room temperature.

The DAO used in the experiment was obtained from a commercial SDA process (SK Innovation, Ulsan, Korea). The specific gravity of DAO was measured at 60 °F using a density meter (Anton Paar, DMA 4500) and the American Petroleum Institute (API) gravity was calculated based on the specific gravity (ASTM D287 and ASTM D1298 E2). Kinematic viscosity was determined using a rheometer (AR-2000, TA

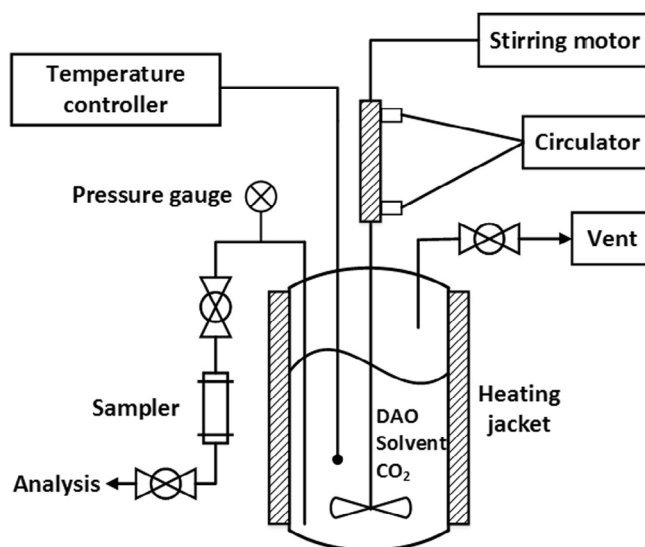


Fig. 1. Schematic of experimental apparatus for solvent recovery experiments.

Instruments). The analysis of the saturate, aromatic, resin, and asphaltene (SARA) fraction of DAO was carried out using a thin layer chromatography-flame ionization detector (TLC-FID, IATROSCAN MK-6s) with a hydrogen flow of 160 ml/min and an air flow of 2000 ml/min. SARA analysis was repeated three times for a sample and the average value was taken for the SARA fraction. Simulated distillation (SIMDIS, ASTM D7169 method) was carried out to obtain the boiling point distribution of DAO using a gas chromatography analysis system (HP/AC SIMDIS Alliance Product G1540A).

2.2. Experimental procedure for solvent recovery

Experiments of solvent recovery were carried out in a lab-scale batch separator covered with an electric heating jacket (Fig. 1). The separator was first filled with DAO, solvent (*n*-pentane), and dry ice at a specific ratio. The separator temperature was controlled by supplying an electric current to the heating jacket and measuring the temperature difference between the inside and outside of the separator. After the separator was heated to a desired temperature (40, 60, 80, and 100 °C), the mixture was constantly stirred at 100 rpm by a magnetic drive stirrer for 2 h, while maintaining the temperature and pressure. When no further pressure change was detected, stirring was stopped when the mixture reached a phase equilibrium. A sample was then collected in a vial through a sampling port from the bottom of the separator.

After the sample was collected, it was heated at 120 °C for 1 h to evaporate the residual solvent and to estimate the amount of solvent left in the sample [17]. The boiling point distribution of DAO shown in Fig. 2 indicates that DAO weight loss occurs at over 200 °C; it is therefore expected that only solvent is removed by evaporation at 120 °C. From the solvent evaporation in the sample, the weights of the DAO (W_1) and solvent (W_2) were obtained from the change in the weight of the sample before and after the solvent evaporation. Solvent recovery was then calculated as follows:

$$\text{Solvent recovery (wt\%)} = \left(1 - \frac{W_2}{W_1 R}\right) \times 100 \quad (1)$$

where R is the solvent/DAO ratio in the feed. A sample containing a small amount of solvent means that high solvent recovery is achieved in the DAO/solvent separator.

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