

Comparison of Dimethylformamide with Dimethylsulfoxide for Quality Improvement of Distillate Recovered from Waste Plastic Pyrolysis Oil

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Abstract: As a part of improving the quality of the distillate (distilling temperature 120–350 °C) recovered from waste plastic pyrolysis oil (WPPO) by simple distillation, the enrichment of paraffin components present in the distillate was compared by the equilibrium extraction of dimethylformamide (DMF) and dimethylsulfoxide (DMSO). Regardless of the solvent used, the concentration increase rate of the paraffin component in the raffinate relative to the raw material was reduced by increasing the mass fraction of water in the solvent in an initial state. On the other hand, it increased by increasing the mass ratio of the solvent to the raw material in an initial state. The enrichment performance of paraffin component in raffinate recovered by DMF was higher than that by DMSO under the same experimental conditions. Furthermore, the two solvents were compared by adding color and the waxing phenomena of recovered raffinate to assess the enrichment performance of paraffin components.

Keywords: waste plastic pyrolysis oil; paraffin enrichment; DMF equilibrium extraction; DMSO equilibrium extraction

1. Introduction

The effective treatment of plastic waste products is extremely valuable in that it can solve the problems of serious environmental pollution and resource depletion occurring all over the world. Currently, the most common method of treating waste plastics is landfilling and incineration, but it is recognized that treatment through landfill and incineration is not the ultimate method for treating waste plastics because it causes various environmental problems [1]. Therefore, in recent years, research on the high-temperature thermal decomposition method as one of the efficient treatment of waste plastic products has been actively conducted from the point of view that it is eco-friendly and, at the same time, can be used in the recycling of resources [2].

A large amount of paraffin, olefin, aromatic and acid components are contained in mixed waste plastic pyrolysis oil (WPPO) such as polyethylene (PE), polypropylene (PP), polyvinyl chloride (PVC) and polyethylene terephthalate (PET). Until now, many studies, such as reaction by pyrolysis [2–8] only and pyrolysis reactions [1,9–13] using a catalyst to produce high quality WPPO for use as fuel, have been reported. Moreover, many studies have been reported that examine the possibility of using WPPO as a renewable energy by comparing with commercial fuels [14–16], but few studies have been conducted to improve the quality of the distillate recovered through a distillation operation for low-grade WPPO.

To investigate the quality improvement of the distillate recovered from WPPO for the purpose of producing products similar to commercial light oil, as shown in Figure 1a, it is necessary to examine the separation between paraffin components C₇–C₂₄ contained in the distillate and other components using methods such as solvent extraction [17–23] and emulsion liquid [24–27], which are widely used for the dearomatizing of petroleum and coal tar systems.

This study investigates the quality improvement of distillate (distilling temperature 120–350 °C) recovered by the simple distillation of WPPO produced from four kinds of mixed waste plastics (PE, PP, PVC, PET). Using an aqueous solution of dimethylformamide (DMF) and dimethylsulfoxide (DMSO) as a solvent, the effects of liquid–liquid contacting time (t), the mass fraction of water in solvent of an initial state ($y_{w,0}$) and the mass ratio of solvent to the raw material of an initial state $(S/F)_0$ on the enrichment of paraffin components contained in the raw material (WPPO distillate) were examined. The comparison of the two solvents took into account the ease of phase separation, the enrichment performance of the paraffin components, their color, and the waxing phenomena of the recovered raffinate. Based on these results, I examined whether the final product (solvent-free raffinate) recovered in this study could be applied as commercial light oil.

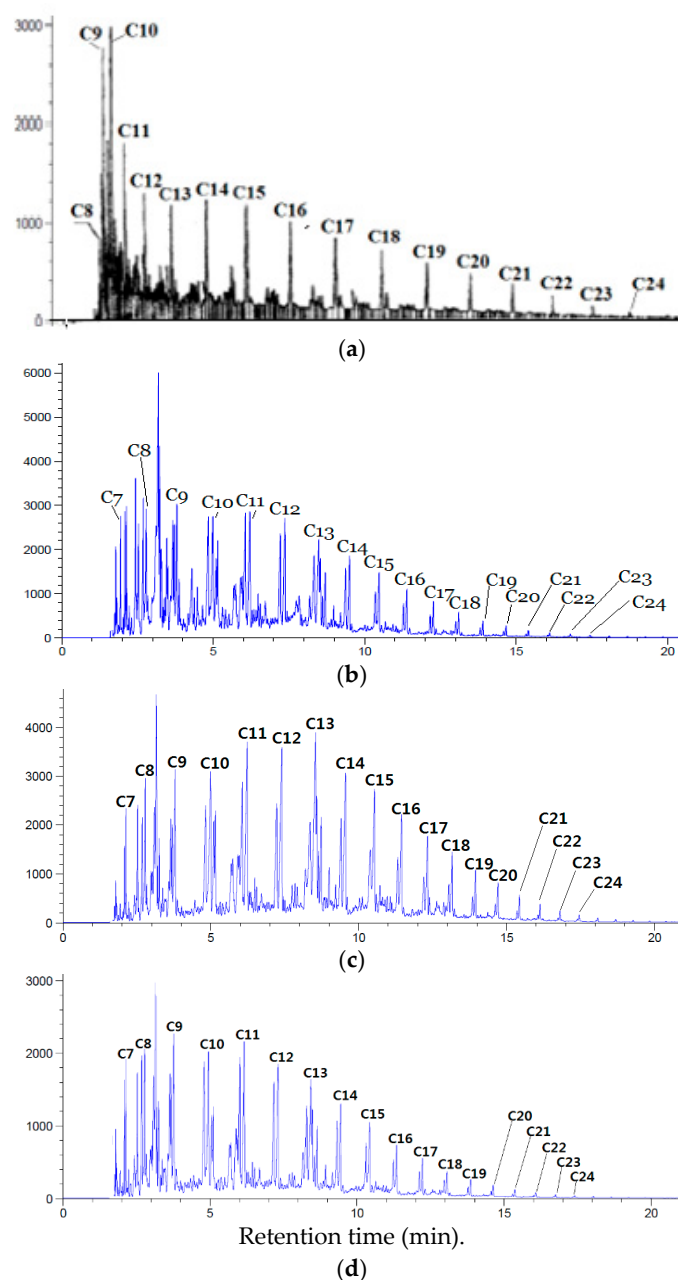


Figure 1. Gas chromatogram of (a) commercial light oil, (b) raw material (distillate recovered from WPPO through simple distillation), (c) raffinate (solvent-free) recovered using dimethylformamide (DMF), and (d) raffinate (solvent-free) recovered using dimethylsulfoxide (DMSO). Experimental conditions of (c) and (d): $t = 72$ h, $y_{w,0} = 0.03$, $(S/F)_0 = 10$, $T_0 = 28$ °C.

2. Material and Methods

2.1. Material

WPPO (pyrolysis raw material: mixed waste plastic of PE, PP, PVC and PET, reactor: batch pyrolysis furnace, reaction temperature: 430–550 °C) was supplied by Omega Energy Environmental Technology Co., Ltd. (Incheon, Korea). DMF (Anhydrous, ≥99.8% purity), paraffin component C₈ (octane, ≥99.7% purity), C₉ (*n*-nonane, ≥99% purity), C₁₀ (*n*-decane, ≥99.8% purity), C₁₂ (*n*-dodecane, ≥99.8% purity), C₁₄ (*n*-tetradecane, ≥99.5% purity) and C₁₆ (*n*-hexadecane, ≥99.8% purity) were purchased from Sigma-Aldrich Korea (Seoul, Korea).

2.2. Method

A certain concentration of solvent was prepared by the addition of distilled water. A 500 mL Erlenmeyer flask containing a certain amount of the raw material and solvent was placed in a shaking water bath maintained at an experimental temperature and vibrated for 12–72 h to reach liquid–liquid equilibrium. After the equilibrium was reached, the vibration was stopped and settled. The raffinate phase and the extract phase were separated using a 500 mL separatory funnel, and the mass of each phase was measured. The two phases were analyzed by adding acetone, and their compositions were determined. An analysis of two phases recovered was performed using a gas chromatograph (GC, Agilent Technologies Korea Co. (Seoul, Korea), 6890N) equipped with a flame ionization detector (FID) and column (J & W Co., DB-1HT (length: 30 m, inner diameter: 0.32 mm, film: 0.1 µm)). The analysis conditions of samples were as follows: carrier gas, N₂; flow rate, 2.2 mL·min^{−1}; injection port temperature, 300 °C; sample size, 2 µL; splitting ratio, 20:1; initial temperature, 40 °C; retention time, 1 min; rising temperature speed, 12 °C·min^{−1} and final temperature, 350 °C.

2.3. Material System and Conditions

Table 1 shows the material system and experimental conditions used in this study. Low-grade distillate (distilling temperature: 120–350 °C), which recovered by simple distillation of WPPO supplied from the company mentioned above, was used as the raw material. Aqueous solutions of DMF and DMSO were used as the solvents. The operating temperature of an initial state (T_0) was fixed constantly and t , $y_{w,0}$ and $(S/F)_0$ were changed. In this study, commercial reagent grade DMF, DMSO, *n*-octane, *n*-nonane, *n*-decane, *n*-dodecane, *n*-tetradecane, and *n*-hexadecane were used without further purification.

Table 1. Material system and experimental conditions.

System	
Raw material	Distillate ^a of Waste Plastic Pyrolysis Oil (WPPO)
Solvent	(1) aqueous solution of dimethylformamide (DMF) (2) aqueous solution of dimethylsulfoxide (DMSO)
Experimental Conditions	
Liquid-liquid contacting time, t (h)	12–72
Mass fraction of water in solvent of initial state, $y_{w,0}$ (–)	0–0.06
Mass ratio of solvent to raw material of initial state, $(S/F)_0$ (–)	1–10
Operating temperature of initial state, T_0 (°C)	28

^a disatillate (distilling temperature: 120–350 °C) recovered by simple distillation of waste plastic pyrolysis oil (WPPO).

3. Results and Discussion

3.1. Gas Chromatogram of Raw Material (WPPO Distillate)

The raw material, which is composed of more than 100 components, contains paraffin, olefin, aromatic and acid components. Figure 1b shows a gas chromatogram of the raw material analyzed using capillary column DB-1HT and the carbon number of the 18 kinds of paraffin components (C₇–C₂₄) identified. The above-mentioned 18 kinds of paraffin components corresponded with peak

patterns shown in the gas chromatogram, the result of GC mass analysis of the raw material and result analyzed by adding a small amount of the standard reagents of 6 kinds of paraffin components (C_8 , C_9 , C_{10} , C_{12} , C_{14} , C_{16}) purchased from Sigma-Aldrich Korea (Seoul, Korea).

The concentration of C_7 – C_{20} paraffin components present in the raw material were in the range of about 0.04–2.77%, except for the concentration for the paraffin components of C_{21} – C_{24} , which were very small.

In this study, it was difficult to accurately quantify each paraffin component contained in the raw material, so the area % of the peak in the gas chromatogram was treated as the concentration of each paraffin component [28]. As shown in Figure 1a, the paraffin components (C_7 – C_{24}) contained in the raw material of this study were almost identical to those contained in the commercial light oil (C_8 – C_{24}).

3.2. Equilibrium Extraction

To confirm the time to reach equilibrium, the raffinate phases and the extract phases obtained through contacting (liquid–liquid contacting time: 12, 24, 48, 72 h) the solvent and the raw material under the same conditions [$y_{w,0} = 0.03$, $(S/F)_0 = 3$, $T_0 = 28$ °C] were analyzed. The compositions of the raffinate phases and the extract phases obtained by contact for 48 and 72 h were identical to one another regardless of the contact time. The equilibrium arrival time was found to be less than 48 h. Therefore, equilibrium extraction was performed under the fixed condition of $t = 72$ h.

Figure 2a–d, respectively, show the effect of $y_{w,0}$ on the recovery rate of paraffin component i ($Y_{i,P}$, $i = C_{12}$, C_{16}), the recovery rate of the raffinate (Y_R), the concentration increase rate of the paraffin component i ($x_{i,P}$) and the densities (ρ) of the raffinate and extract phase when using DMF and DMSO. $Y_{i,P}$, Y_R and $x_{i,P}$, respectively, are defined as:

$$Y_{i,P} = \frac{R \times x_i}{R_0 \times x_{i,0}} \times 100\% \quad (1)$$

$$Y_R = \frac{R}{R_0} \times 100\% \quad (2)$$

$$x_{i,P} = \frac{x_i}{x_{i,0}} \times 100\% \quad (3)$$

where R and R_0 , respectively, represent the mass of the extract phase after the liquid–liquid contact run and that of the raw material. $x_{i,0}$ and x_i denote the mass fraction of the paraffin component i in the raw material and that in raffinate recovered after a liquid–liquid contact run, respectively.

Regardless of the solvent used, an increase in $y_{w,0}$ was found to increase $Y_{i,P}$, as shown in Figure 2a. It is believed that this is the result of the increase in the extract phase polarity as $y_{w,0}$ increases. At the same $y_{w,0}$, the larger the carbon number of the paraffin component, the lower the solubility in the solvent, so the $Y_{i,P}$ increases. In addition, $Y_{i,P}$ in DMSO extraction showed a much higher value than that of DMF extraction in the entire range of $y_{w,0}$ in this study. The results showed that the DMF extraction method had better a solubility in paraffin components than the DMSO extraction method. In the case of DMF at $y_{w,0} = 0$, the $Y_{i,P}$ values of paraffin components C_{12} and C_{16} were about 42.7% and 60.5%, respectively, whereas DMSO showed about 79% and 93.2%, respectively. In the case of DMSO, considering that the $Y_{i,P}$ of the C_{16} paraffin component is 97.8% at $y_{w,0} = 0.06$, it is found that the paraffin components greater than the C_{16} paraffin component contained in the raw material remain at almost 100% in the raffinate recovered at $y_{w,0} = 0.06$.

Figure 2b shows that Y_R increases as $y_{w,0}$ increases, regardless of the solvent used. It is considered that this is caused by decreasing the solubility of all the components such as paraffin, aromatic components, etc., contained in the raw material. In the entire range of $y_{w,0}$ in this study, DMSO extraction showed a much higher Y_R than DMF extraction. From this, it is considered that DMF extraction is superior to DMSO extraction in the solubility of paraffin components. For DMF, the Y_R values at $y_{w,0} = 0$ and 0.06 were approximately 38% and 72.2%, respectively. However, DMSO was

about 81% and 86.5%, much larger than DHF. From this tendency, it was predicted that the polarity of the solvent changed much more with the increase in $y_{w,0}$ in the case of DMF than DMSO.

Figure 1c,d, respectively, show the results of analysis of the raffinate recovered under constant conditions ($y_{w,0} = 0.03$, $(S/F)_0 = 10$, $T_0 = 28^\circ\text{C}$) using DMF and DMSO. The gas chromatogram of raffinate is very different from that of the raw material (Figure 1b). It was found that the components, other than the paraffin components in the raw material, were extracted in large amounts by the extraction operation, and the peak of the paraffin component was remarkably increased, so that the concentration of the paraffin component in the raffinate increased. The concentrations of C_{12} and C_{16} paraffin components in the raffinate from $x_{i,p}$ of $y_{w,0} = 0$ shown in Figure 2c increased approximately 1.12 and 1.56 times for DMF and about 0.98 and 1.15 times for DMSO, respectively, compared to their concentration in the raw material.

In the gas chromatogram of the raw material (distillate of WPPO) and the raffinate, each olefin component peak was positioned immediately before each paraffin component. It was expected that it would be difficult to separate the paraffin–olefin components by equilibrium extraction by comparing the gas chromatogram of the raw material (distillate of WPPO) in Figure 1b and the raffinate of Figure 1c.

Although this study only mentions the separation of paraffin components among raw materials by equilibrium extraction, we plan to conduct a detailed review of the separation of olefin components among raw materials by equilibrium extraction in the future.

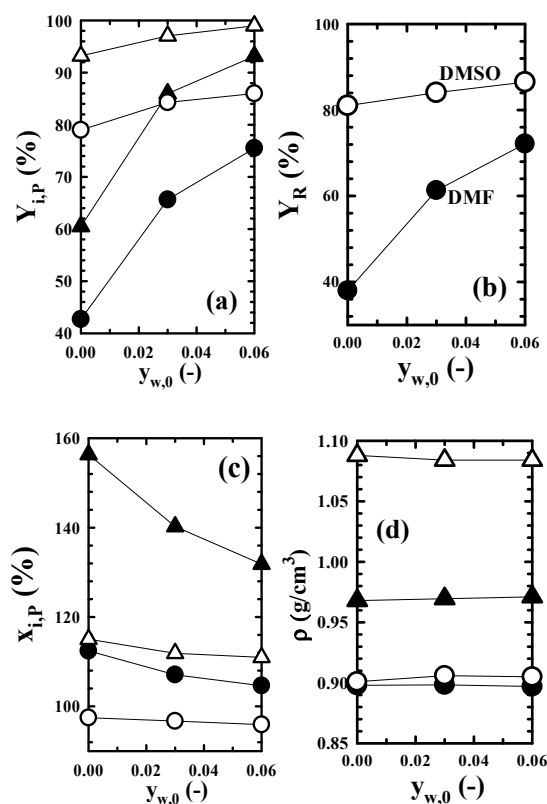


Figure 2. Comparison of solvent for (a) recovery rate of paraffin component i ($Y_{i,p}$) present in raffinate, (b) recovery rate of raffinate (Y_R), (c) the concentration increase rate of the paraffin component i ($x_{i,p}$) present in raffinate to that present in raw material (WPPO distillate), and (d) density (ρ) of raffinate and extract phase according to mass fraction of water in solvent of initial state ($y_{w,0}$). Experimental conditions: $t = 72$ h, $(S/F)_0 = 3$, $T_0 = 28^\circ\text{C}$. Keys of (a) and (c): \bullet C_{12} (DMF), \blacktriangle C_{16} (DMF), \circ C_{12} (DMSO), \triangle C_{16} (DMSO). Keys of (d): \bullet raffinate phase (DMF), \circ raffinate phase (DMSO), \blacktriangle extract phase (DMF), \triangle extract phase (DMSO).

In the extraction operation, the difference in density between two phases (raffinate phase, extract phase) is an important factor that determines the extraction processing speed. Figure 2d shows the density (ρ) of the two phases recovered after the extraction operation using DMF and DMSO. The effect of $y_{w,0}$ on the density of the raffinate and extraction phases was not recognized regardless of the solvent. The difference in the density of the two phases due to DMSO extraction was greater than that of DMF extraction. From this result, it was found that DMSO is more advantageous than DMF in terms of extraction processing speed.

When referring to the change in the color of the raffinate according to $y_{w,0}$ and the solvent, as shown in Figure 3, the color of the raffinate became lighter as the $y_{w,0}$ decreased regardless of solvent, and changed to very light yellow at $y_{w,0} = 0$ (raw material: dark brown). At the same $y_{w,0}$, the color of the raffinate recovered using DMF was lighter than that of DMSO. In particular, the raffinate recovered at $y_{w,0} = 0$ using DMF showed a pale yellow color, almost similar to the color of commercial light oil. Considering that the paraffin component is almost colorless, it can be confirmed that the concentration of paraffin components in the raffinate increases with decreasing $y_{w,0}$ in the case of DMF rather than DMSO.

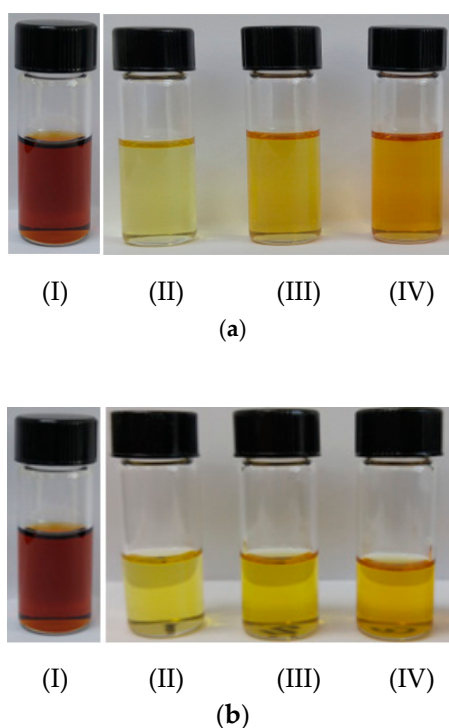


Figure 3. Comparison of solvent for color change in raffinate according to $y_{w,0}$. Solvent used: (a) DMF, (b) DMSO. Experimental condition: $t = 72$ h, $(S/F)_0 = 3$, $T_0 = 28$ °C. (I) raw material, (II) $y_{w,0} = 0$, (III) $y_{w,0} = 0.03$, and (IV) $y_{w,0} = 0.06$.

Furthermore, because the waxing phenomenon of commercial light oil can occur at a very low temperature, the raw material of this study and the raffinate recovered by DMF and DMSO extraction at $y_{w,0} = 0$ were left at -25 °C for 24 h. The occurrence of a waxing phenomenon was confirmed throughout this process. The waxing phenomenon was observed in the raw material and the raffinate recovered from DMSO, but not in the raffinate recovered from DMF.

Figure 4a shows that the extraction amount of paraffinic component i , which is extracted in the extraction phase, increased as $(S/F)_0$ increased regardless of the solvent used, while $Y_{i,P}$ decreased. In the case of $(S/F)_0 = 1$ for DMF, $Y_{i,P}$ of paraffin components C_{12} and C_{16} were about 84.9% and 100%, respectively, whereas those of DMSO were about 88.8% and 100%, respectively. Regardless of solvent, considering that the $Y_{i,P}$ of the C_{16} paraffin component is 100% at $(S/F)_0 = 1$, it was found that the

paraffin components with a carbon number higher than 16 contained in the raw material remained at 100% in the raffinate recovered from $(S/F)_0 = 1$.

The Y_R values at $(S/F)_0 = 1$ for DMF and DMSO were about 81% and 88.6%, respectively (Figure 4b). Y_R decreased sharply as $(S/F)_0$ increased due to an increase in the extraction amount of all the components other than the paraffin components and the paraffin components contained in the raw material. At $(S/F)_0 = 10$, the Y_R values of DMF and DMSO were about 23.8% and 76.2%, respectively.

Figure 4c shows that $x_{i,P}$ increases with increasing $(S/F)_0$, regardless of the solvent used. This is considered to be a phenomenon caused by an increase in the selectivity of all the paraffin components compared to all other components such as aromatic components contained in the raw material by the increase in $(S/F)_0$. The concentration of C_{12} and C_{16} paraffin components in raffinate recovered at $(S/F)_0 = 10$ increased by about 1.37 and 2.46 times for DMF, 1.05 and 1.26 times for DMSO, respectively, compared to the concentration in the raw material. It is predicted that when DMF is used rather than DMSO, the selectivity of all the paraffin components to all the components other than the paraffin components contained in the raw material is large.

Figure 4d shows the density (ρ) of two phases recovered after the extraction operation using DMF and DMSO. Regardless of the solvent used, the effects of $(S/F)_0$ on the densities of the raffinate phase and the extract phase were not acceptable.

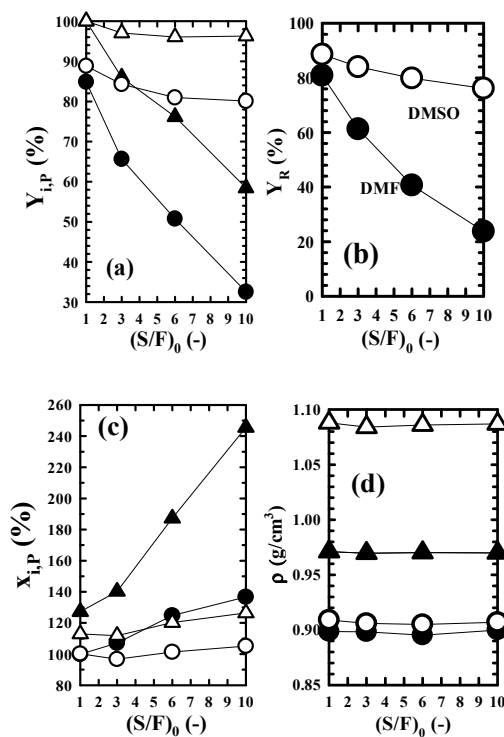


Figure 4. Comparison of solvent for (a) $Y_{i,P}$, (b) Y_R , (c) $x_{i,P}$, and (d) ρ according to mass ratio of solvent to raw material in initial state $(S/F)_0$. Experimental conditions: $t = 72$ h, $y_{w,0} = 0.03$, $T_0 = 28$ °C. Keys are shown in Figure 2.

Regardless of the solvent in Figure 5, it was found that the color of the raffinate becomes thinner as the $(S/F)_0$ increases, and becomes very pale yellow at $(S/F)_0 = 10$. DMF showed a color lighter than DMSO in the color of raffinate recovered at the same $(S/F)_0$ and, in particular, the raffinate recovered at $(S/F)_0 = 10$ using DMF showed a pale yellow color almost similar to the color of commercial light oil. As described above, when the raffinate obtained at $(S/F)_0 = 10$ was left at -25 °C for 24 h, a waxing phenomenon was observed in the raffinate recovered from DMSO, but not in that recovered from DMF.

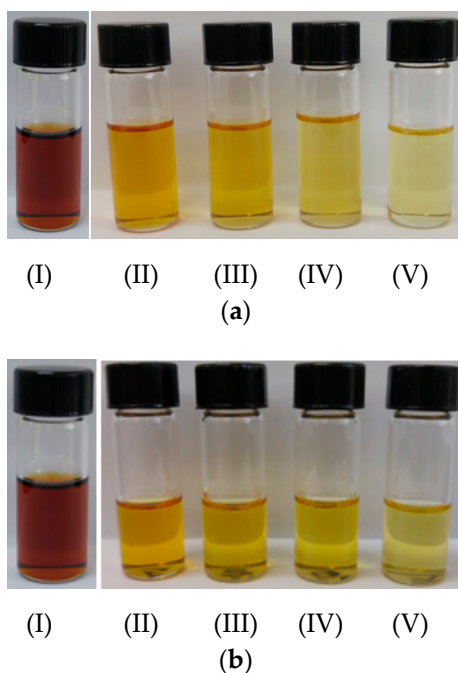


Figure 5. Comparison of solvent for color change in raffinate according to $(S/F)_0$. Solvent used: (a) DMF, (b) DMSO. Experimental conditions: $t = 72$ h, $y_{w,0} = 0.03$, $T_0 = 28$ °C. (I) raw material, (II) $(S/F)_0 = 1$, (III) $(S/F)_0 = 3$, (IV) $(S/F)_0 = 6$ and (V) $(S/F)_0 = 10$.

To confirm the reproducibility of all the experimental data obtained using two solvents in this study, the experiments were conducted under the same experimental conditions two or three times. The range of the reproducibility for the measured experimental value was within $\pm 5\%$.

Considering the effects according to $y_{w,0}$ and $(S/F)_0$ for the enrichment performance of paraffin components present in the raffinate, the color of the raffinate compared to commercial light oil and the waxing phenomena of the raffinate, DMF extraction was thought to be more advantageous from the point of view of the quality improvement of the distillate of WPPO than DMSO extraction. Of course, it is believed that the final evaluation of the products (raffinate) obtained from this study should be carried out in more detail through a quality evaluation according to the Petroleum Quality Inspection Method, as well as a review of the possibility of the separation of the olefin components contained in the distillate recovered from WPPO by DMF equilibrium extraction, which will be performed later.

Figure 6 shows the enrichment process of paraffin components from the distillate of WPPO, which is considered to be possible using the equilibrium extraction data of DMF. The proposed process was composed of one extraction tower (tower 1) to recover the paraffin components in the distillate of WPPO, one stripping tower (tower 2) to recover the extract in the extract phase, one washing tower (tower 3) to recover DMF in the raffinate and one distillation tower (tower 4) to recover the extraction solvent (DMF). This process is considered to be a valuable process for the enrichment of paraffin components contained in the distillate of WPPO.

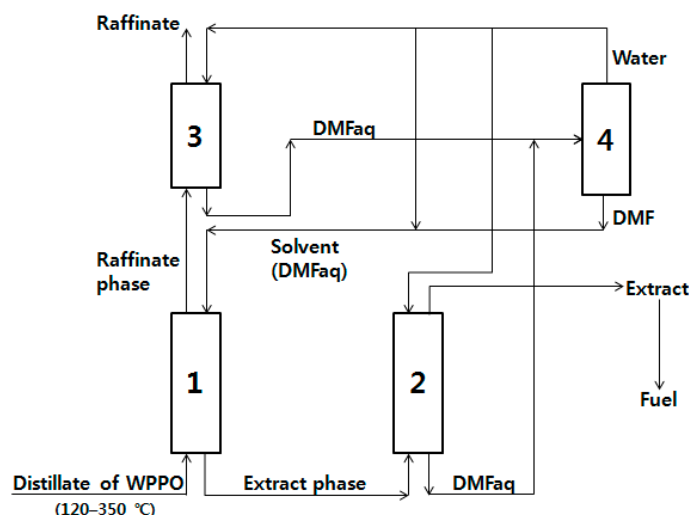


Figure 6. Enrichment process of paraffin components in distillate of WPPO. Tower no. 1: extraction tower, Tower no. 2: stripping tower, Tower no. 3: washing tower, Tower no. 4: distillation tower.

4. Conclusions

As part of the upgrading of the distillate (distilling temperature 120–350 °C.) of WPPO, this study examined the enrichment of paraffin components in the distillate by the equilibrium extraction of DMF and DMSO. Considering the effects according to $y_{w,0}$ and $(S/F)_0$ for the enrichment performance of paraffin components present in the raffinate, the color of the raffinate compared to commercial light oil and the waxing phenomena of the raffinate, DMF extraction was thought to be more advantageous from the point of view of the quality improvement of the distillate of WPPO than DMSO extraction. The process proposed using the results of DMF equilibrium extraction in this study, which comprised of one extraction tower, one stripping tower, one washing tower and one distillation tower, is a valuable process to enrich of the paraffin compounds contained in the distillate of WPPO.

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Conflicts of Interest: The author declare no conflict of interest.

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