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Treatment of Spindle Distillate with Furfural and n-Heptane for the Production of Light Lubricating Oils

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Abstract

The production of light lubricating oils from the spindle distillate of Arzew refinery involves a furfural treatment step. The present study was conducted to investigate the effect of using n-heptane as a co-solvent during this treatment step. The experiments were carried out at 50°C with a furfural to feed ratio of 1 and n-heptane to feed ratios ranging from 0 to 0.5. The characteristics of the recovered oils, namely color, density, refractive index, molecular weight, sulfur content, carbon distribution, ring content, viscosity index and pour point were determined using ASTM standard methods. It was found that the addition of n-heptane increases the yield of refined oil and improves its quality, except its pour point which will be lowered during the subsequent solvent dewaxing step. These results were mainly attributed to the high extraction of aromatic hydrocarbons contained in the spindle distillate due to the presence of n-heptane. The oil refined at an n-heptane to feed ratio of 0.2 is of better quality, it was recovered with a yield of 84.4% and its viscosity index 109 is the highest.

Keywords: *Solvent extraction; Co-solvent; n-d-M method; Viscosity index; Pour point.*

1. Introduction

Heavy residues from the atmospheric distillation of crude oils are used as raw materials for the manufacture of lubricating base oils. The process starts with the vacuum distillation unit where the atmospheric residue is separated into several cuts having different boiling ranges and viscosities. The vacuum residue is then deasphalted (usually with propane) to recover a very viscous oil, commonly called Bright Stock. Vacuum distillates and Bright Stock fraction undergo further treatments such as solvent extraction, dewaxing and hydrofinishing to become high-quality lubricant bases. Solvent extraction is used to improve some of their characteristics, namely color, oxidative stability and viscosity index, by removing aromatic compounds and heteroatoms such as nitrogen and sulfur [1]. The most widely used solvents are furfural and N-methyl-2-pyrrolidone (NMP), with phenol and liquid sulfur dioxide used to a lesser extent [2].

The lightest and least viscous lubricating cuts from the process of vacuum distillation of atmospheric residues are called spindle distillates. After treatment, they will form the basis of light lubricants used for domestic purposes (e.g., for sewing machines and bicycles); some of the heavier fractions will also be used as blend stocks for light motor oils [3]. It is important to mention that some commercial spindle type oils are employed for lubrication of machine tools [4]. The treatment of spindle distillates by liquid-liquid extraction with furfural has been the subject of numerous publications [5-13]. Coto *et al.* [5] developed a method based on the properties of pseudo-components to calculate the physical properties of the hydrocarbon mixtures involved (spindle distillate, raffinates and extracts); the extraction operation was simulated using a model based on a reduced number of pseudo-components and the NRTL model. The investigation conducted by Mohammed *et al.* [9] showed that the viscosity index of the spindle fraction (40 stock) increases with increasing extraction temperature and increasing furfural to oil ratio, a maximum value of 115.6 was reached at 110°C with a solvent to oil ratio of 4:1 (wt/wt). Shakir and Qasim [10] concluded in their study that the use of formamide or NMP as a co-solvent can improve the solvent power and selectivity of furfural toward aromatic compounds. Izza and Korichi [13] reported that the addition of the ethoxylated anionic surfactant marketed as Galaxy LES 70 to furfural increases the raffinate yield.

The use of a non-polar solvent, in addition to the polar solvent, during the extractive purification process of petroleum fractions (kerosene, gas oil and vacuum distillates) improves the selectivity of the process toward undesirable compounds and increases the raffinate yield. The non-polar solvents tested are generally low boiling normal alkanes such as pentane [14-16], hexane [17-18] and heptane [16,19]. An experimental study on the extraction of aromatic hydrocarbons with furfural and n-hexane was performed by Hoseini *et al.* [18]. The aromatics were extracted from a viscous lubricating oil at different temperatures with a furfural to oil ratio of 1:1 (vol/vol). The best results were obtained at 333.15 K in the presence of 11.11% by volume of n-hexane. The same optimal conditions were recorded in another study where 2,2,4-trimethylpentane was used as a co-solvent instead of n-hexane [20]. Gaile *et al.* [16] showed that extractive refining at 50°C of a light vacuum gas oil with dimethylacetamide and n-heptane containing 1% toluene provides a high-quality raw material for hydrocracking and catalytic cracking processes. This solvent system allowed to extract 61.9% of the aromatic hydrocarbons and 62.8% of the sulfur compounds contained in a heavy vacuum gas oil; substitution of dimethylacetamide with NMP gave better results in this case [19].

In this work, the spindle distillate from Arzew oil refinery was treated with furfural using n-heptane as co-solvent. The experiments were carried out at 50°C with co-solvent to feed ratios ranging from 0 to 0.5 by weight. The main characteristics of the obtained oils were determined in order to find the optimal amount of n-heptane to be used.

2. Materials and methods

2.1. Materials

The spindle distillate used in this study was provided by the Arzew refinery located in northwest Algeria. This oil refinery is supplied with light crude from the Hassi Messaoud region (in Algeria) and receives reduced crude from abroad. The processes of purification of spindle distillate at the refinery level are presented in Fig. 1 and its characteristics are listed in Table 1.

Table 1. Characteristics of spindle distillate.

Properties	ASTM methods	Values	Properties	ASTM methods	Values
Density (d_4^{20})	D4052	0.8751	Carbon distribution	D3238	
Refractive index (n_D^{20})	D1218	1.4925	% C_A		20.7
Viscosity (mm^2/s)	D445		% C_N		15.7
100°F		12.87	% C_P		63.6
210°F		2.90			
40°C		11.74	Ring content	D3238	
100°C		2.82	R_A		0.77
Molecular weight (g/mol)	D2502	300	R_N		0.71
Sulfur content (wt %)	D4294	0.2456	Viscosity index	D2270	75
ASTM Color	D1500	< 2	Pour point (°C)	D97	12

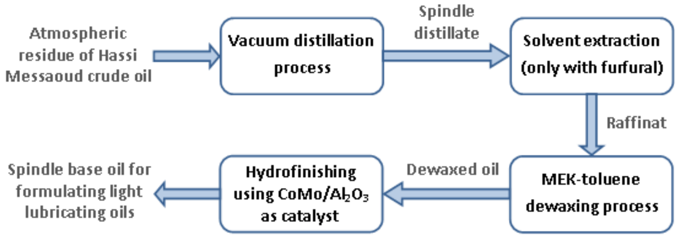

Fig. 1. Production steps of spindle base oil at the Arzew refinery.

Furfural, also provided by the Arzew refinery, was distilled before use to eliminate oxidation products formed due to air contact. n-Heptane with purity greater than 99% was purchased from Sigma-Aldrich.

2.2. Apparatus and experimental procedure

The extraction operations were performed in a jacketed glass mixer-settler equipped with a mechanical stirrer. The temperature was maintained constant within ±0.1°C by circulating water from a thermostatic bath.

Figure 2 shows a schematic diagram of the experimental setup used. The experimental conditions are given in Table 2. Each experiment required 1 hour of stirring at 450 rpm and 1 hour of settling. The furfural-poor phase (raffinate phase) and the furfural-rich phase (extract phase) were separated by difference in density. The raffinate phase mainly contains the less polar hydrocarbons, while the extract phase contains the more polar compounds (sulfur, nitrogen and aromatic compounds). Furfural and n-heptane were removed from the raffinate phases by vacuum distillation. The recovered oils called raffinates were weighed and analyzed in order to evaluate the performance of the extraction process.

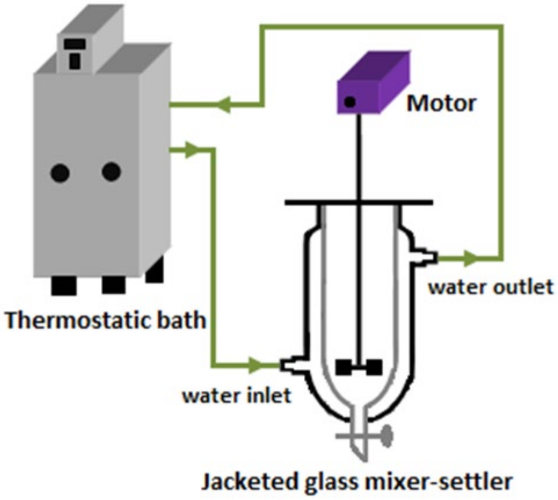

Fig. 2. Schematic diagram of the experimental setup.

Table 2. Experimental conditions used with a constant furfural to feed ratio.

Run No.	Furfural/Feed (wt/wt)	Heptane/Feed (wt/wt)	Temperature (°C)
1	1.0	0.0	50
2	1.0	0.1	50
3	1.0	0.2	50
4	1.0	0.3	50
5	1.0	0.4	50
6	1.0	0.5	50

2.3. Determination of refined oil yield

The yield of refined oil was calculated for each experiment as follows:

$$Yield\ (\text{wt}\,\%) = \frac{R}{F} \times 100 \qquad (1)$$

where R is the mass of the raffinate (free of solvents) and F is the mass of the feed (spindle distillate).

2.4. Test methods

The properties of the spindle distillate and raffinates were determined using standard methods of the American Society for Testing and Materials (ASTM). These methods are briefly described in this section.

2.4.1. ASTM Color

The color of the samples was measured by the ASTM UNION colorimeter according to the ASTM D1500 standard. A light source allows a visual comparison between the liquid sample and colored glass disks. The color scale ranges from 0.5 for the lightest glass to 8.0 for the darkest glass. When an exact match is not found and the color of the sample is intermediate between those of two standard glasses, the value assigned to the darker glass is reported under the notation "L value" or "< value" [21].

2.4.2. n-d-M method

The ASTM D3238 standard test method [22] was used for the calculation of carbon distribution and ring content of the considered oils from measurements of refractive index, density and molecular weight (n-d-M). This method is based on the determination of the factors v and w using the following equations:

$$v = 2.51(n_D^{20} - 1.4750) - (d_4^{20} - 0.8510) \tag{2}$$

$$w = (d_4^{20} - 0.8510) - 1.11(n_D^{20} - 1.4750) \tag{3}$$

where n_D^{20} and d_4^{20} are respectively the refractive index and the density of the oil at 20°C.

The refractive index was measured with an Abbe-type refractometer according to the ASTM D1218 method. The density was determined using the DMA 48 digital density meter (Anton Paar) according to the ASTM D4052 standard.

The carbon distribution ($\%\,C_A$, $\%\,C_N$ and $\%\,C_P$) was calculated as follows:

$$\%\,C_A = 430\,v + \frac{3660}{M} \quad \text{for positive } v \tag{4}$$

$$\%\,C_A = 670\,v + \frac{3660}{M} \quad \text{for negative } v \tag{5}$$

$$\%\,C_R = 820\,w - 3\,S + \frac{10000}{M} \quad \text{for positive } w \tag{6}$$

$$\%\,C_R = 1440\,w - 3\,S + \frac{10600}{M} \quad \text{for negative } w \tag{7}$$

$$\%\,C_N = \%\,C_R - \%\,C_A \tag{8}$$

$$\%\,C_P = 100 - \%\,C_R \tag{9}$$

where $\%\,C_A$ is the percentage of aromatic carbon; $\%\,C_R$ is the percentage of carbon in ring structures (aromatic and naphthenic); $\%\,C_N$ is the percentage of naphthenic carbon; $\%\,C_P$ is the percentage of paraffinic carbon; M is the molecular weight of the oil (in g/mol) and S its sulfur content (in wt %).

The molecular weight was estimated from kinematic viscosity measurements at 100 and 210°F according to the ASTM D2502 method. These measurements were made according to the ASTM D445 standard using an Ubbelohde viscometer. The sulfur content was determined by energy dispersive X-ray fluorescence spectrometry according to the ASTM D4294 method using a Horiba analyzer.

The ring content (R_A and R_N) was calculated by the following equations:

$$R_A = 0.44 + 0.055\,M\,v \quad \text{for positive } v \tag{10}$$

$$R_A = 0.44 + 0.080\,M\,v \quad \text{for negative } v \tag{11}$$

$$R_T = 1.33 + 0.146\,M\,(w - 0.005\,S) \quad \text{for positive } w \tag{12}$$

$$R_T = 1.33 + 0.180\,M\,(w - 0.005\,S) \quad \text{for negative } w \tag{13}$$

$$R_N = R_T - R_A \tag{14}$$

where R_A is the average number of aromatic rings per molecule; R_T is the average total number of rings per molecule and R_N is the average number of naphthenic rings per molecule.

2.4.3. Viscosity index

The ASTM D2270 standard practice [23] was applied to determine the viscosity index of oils from their kinematic viscosities. The viscosity index (VI) was calculated as follows:

$$VI = \frac{(L - U)}{(L - H)} \times 100 \quad \text{for } U > H \tag{15}$$

$$VI = 100 \quad \text{for } U = H \tag{16}$$

$$VI = \frac{(anti\log N) - 1}{0.00715} + 100 \quad \text{for } U < H \tag{17}$$

$$N = \frac{\log H - \log U}{\log Y} \tag{18}$$

where L is the kinematic viscosity at 40°C (in mm^2/s) of an oil of 0 viscosity index having the same kinematic viscosity at 100°C as the oil whose viscosity index is to be calculated; H is the kinematic viscosity at 40°C (in mm^2/s) of an oil of 100 viscosity index having the same kinematic viscosity at 100°C as the oil whose viscosity index is to be calculated; U and Y are respectively the kinematic viscosities at 40 and 100°C (in mm^2/s) of the oil whose viscosity index is to be calculated. The values of L and H were taken from the tables provided by the ASTM D2270 standard. The kinematic viscosities of the oils (U and Y) were measured with an Ubbelohde viscometer according to the ASTM D445 method.

2.4.4. Pour point

The ASTM D97 standard test method [24] was followed to determine the pour point of the oils. After preheating, the sample is gradually cooled and examined at intervals of 3°C. The lowest temperature at which movement of the sample is observed is recorded as its pour point.

3. Results and discussion

The determination of raffinate yields allows a quantitative evaluation of the extraction operations. Figure 3 shows that the raffinate yield increases with increasing n-heptane to feed ratio. A maximum yield of 86.5% was obtained with an n-heptane to feed ratio of 0.5, while the minimum yield (77.2%) was recorded for run No. 1 carried out without n-heptane.

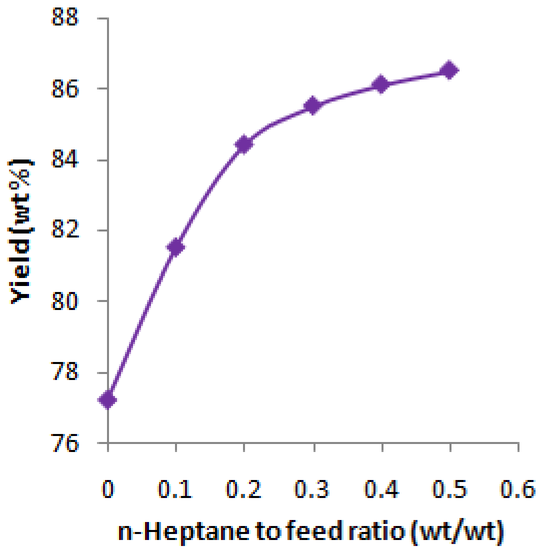

Fig. 3. Effect of co-solvent to feed ratio on raffinate yield.

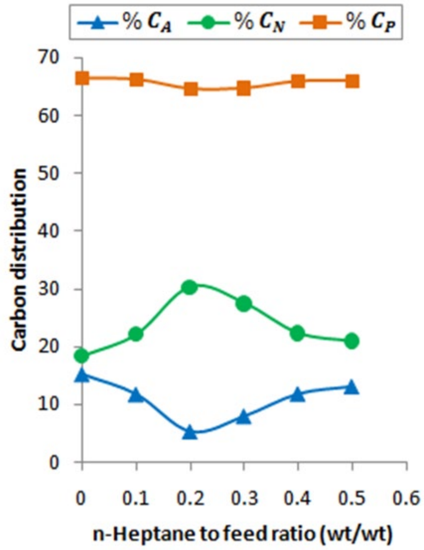

Fig. 4. Carbon distribution of refined oil at different co-solvent to feed ratios.

Table 3. ASTM Color of recovered raffinates.

Run No.	ASTM Color
1	< 1.5
2	< 1.5
3	< 1
4	< 1
5	< 1.5
6	< 1.5

The determination of the color of oils during the production steps of lubricating base oils is mainly used for manufacturing control purposes. Indeed, color is an important quality characteristic that can indicate the degree of refinement of the oil. As a general rule, light oils are more severely refined than dark oils of the same viscosity [25]. The color values of the raffinates recovered from the extraction operations are given in Table 3. As can be seen, runs No. 3 and No. 4 provide the lightest raffinates with an ASTM Color < 1. These results show that the experiments performed with n-heptane to feed ratios of 0.2 and 0.3 probably allow a better purification of the spindle distillate.

Table 4 lists the characteristics of the raffinates used for the calculation of carbon distributions, ring contents and viscosity indexes. It appears that the density, refractive index, kinematic viscosity and sulfur content decrease from run No. 1 to run No. 3 and then increase. Meanwhile, the molecular weight increases to 308 g/mol and then decreases. It is important to note that the minimum sulfur content (0.0952%) was recorded for run No. 3 carried out with an n-heptane to feed ratio of 0.2. In contrast, the raffinate with the highest sulfur content (0.1118%) was obtained from run No. 1 conducted in the absence of n-heptane. In refineries, the sulfur content of oils is reduced to lower levels during the finishing step, which consists of sulfuric acid treatment or contact with clay or catalytic hydrogenation [26].

Table 4. Properties of raffinates used for the calculation of carbon distributions, ring contents and viscosity indexes.

Run No.	Density (d_4^{20})	Refractive index (n_D^{20})	Viscosity at 100°F (mm^2/s)	Viscosity at 210°F (mm^2/s)	Viscosity at 40°C (mm^2/s)	Viscosity at 100°C (mm^2/s)	Molecular weight (g/mol)	Sulfur content (wt %)
1	0.8593	1.4812	12.18	2.83	11.07	2.75	304	0.1118
2	0.8541	1.4761	11.58	2.80	10.46	2.72	306	0.1076
3	0.8504	1.4708	11.04	2.77	9.93	2.69	308	0.0952
4	0.8532	1.4735	11.29	2.78	10.33	2.71	307	0.0982
5	0.8548	1.4764	11.61	2.81	10.51	2.72	306	0.1081
6	0.8565	1.4782	11.74	2.82	10.62	2.73	305	0.1096

Figure 4 shows the effect of increasing the n-heptane to feed ratio on the carbon distribution of the refined oil. The percentage of aromatic carbon ($\% \, C_A$) decreases and then increases; the maximum (15.2) and minimum (5.2) values correspond to the ratios 0 and 0.2, respectively. Hoseini *et al.* [18] observed the same phenomenon using n-hexane as a co-solvent. It was attributed to the formation of a co-solvent–aromatic complex from a certain amount of n-hexane, due to the solubility of aromatics in n-hexane. The transfer of the complex to the raffinate phase resulted in an increase in the aromatic content. Figure 4 indicate that the percentage of naphthenic carbon ($\% \, C_N$) changes in the opposite direction; it increases from 18.5 to 30.3 and then decreases to 21.0. The maximum value is reached when the n-heptane to feed ratio is equal to 0.2, while the minimum value corresponds to a ratio of 0. It can also be seen that the percentage of paraffinic carbon ($\% \, C_P$) does not change significantly, it varies between 64.5 for a ratio of 0.2 and 66.3 for a ratio of 0. These results prove that the extraction process in the presence of n-heptane leads to a greater removal of aromatic compounds compared to naphthenic and paraffinic compounds, especially at an n-heptane to feed ratio of 0.2.

Figure 5 shows the variation of the ring content of the refined oil with increasing n-heptane to feed ratio. As can be seen, the average number of aromatic rings per molecule (R_A) and the average number of naphthenic rings per molecule (R_N) change in opposite directions. Indeed, R_A decreases from a maximum value of 0.56 to a minimum value of 0.20 and then increases to 0.48. The maximum and minimum values of R_A correspond to ratios of 0 and 0.2, respectively. At the same time, R_N increases from a minimum value of 0.81 to a maximum value of 1.29 and then decreases to 0.91. The minimum and maximum values of R_N were reached at ratios of 0 and 0.2, respectively. These observations are consistent with the results presented previously in Fig. 4. It should be mentioned here that the R_A and R_N values of the spindle distillate are 0.77 and 0.71, respectively (see Table 1).

One of the main characteristics of a lubricating oil is its ability to maintain its viscosity at engine operating temperatures [27]. The viscosity index is a dimensionless number used to measure this characteristic; it quantifies the variation in the kinematic viscosity of the oil due to temperature changes between 40 and 100°C. The higher the viscosity index, the less the viscosity varies with temperature [27-28]. Figure 6 shows the effect of increasing the n-heptane to feed ratio on the viscosity index of the refined oil. It is clear that the viscosity index increases and then decreases. A maximum value of 109 was recorded at a ratio of 0.2, while the minimum value (82) was recorded at a ratio of 0. On the other hand, the respective ratios 0.2 and 0 also correspond to the minimum and maximum values of the percentage of aromatic carbon in the oil (see Fig. 4). It is known that aromatic hydrocarbons contained in lubricating fractions have a low viscosity index [20,27,29]. Thus, their extraction leads to an increase in the viscosity index of the fraction. The results obtained are therefore consistent with the literature. The viscosity index of base oils can also be improved by using additives such as terpolymers [30-31].

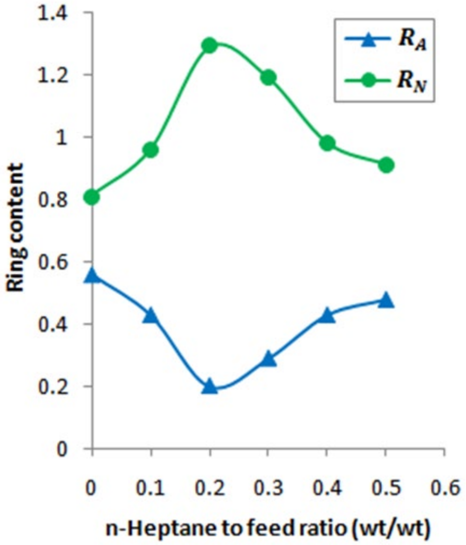

Fig. 5. Ring content of refined oil at different co-solvent to feed ratios.

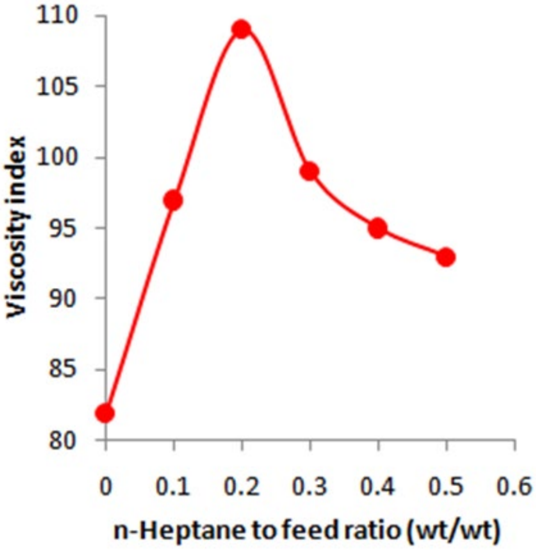

Fig. 6. Effect of co-solvent to feed ratio on raffinate viscosity index.

Table 5. Pour points of recovered raffinates.

Run No.	Pour point (°C)
1	15
2	15
3	18
4	18
5	15
6	15

The pour point is the lowest temperature at which an oil can still flow under standard cooling conditions [32-33]. It is essential that a lubricating oil does not solidify when used at very low temperatures, so its pour point (a few degrees above the solidification temperature) must be low [29]. The pour points of the raffinates recovered from the extraction operations are given in Table 5.

It appears that the raffinates obtained from runs No. 3 and No. 4 have the highest pour point (18°C). These two raffinates also present the lowest average numbers of aromatic rings per molecule (see Fig. 5). The aromatic compounds contained in the lubricating cuts generally have a low pour point [27]. Their high removal during runs No. 3 and No. 4 led to the production of raffinates with a high pour point. It is important to mention that runs No. 3 and No. 4 were carried out with n-heptane to feed ratios of 0.2 and 0.3, respectively. In all cases, furfural treatment of the spindle distillate with or without n-heptane resulted in an increase in its pour point, which was 12°C (see Table 1). In refineries, the pour point of oils is lowered during the subsequent solvent dewaxing step.

4. Conclusions

The use of n-heptane as a co-solvent in the treatment of spindle distillate with furfural increases the yield of refined oil and improves its quality in terms of color, sulfur content and viscosity index. The addition of n-heptane increases the solvent power and selectivity of furfural towards aromatic compounds contained in the spindle distillate. The optimum value of the n-heptane to feed ratio is 0.2. The oil refined at this ratio is of better quality. It was recovered with a very good yield of 84.4% and its viscosity index of 109 is the highest. The treatment of spindle distillate with furfural leads to an increase in its pour point, especially at n-heptane to feed ratios of 0.2 and 0.3.

The addition of n-heptane during furfural treatment of spindle distillate is a promising practice to produce larger quantities of higher-quality light base oil. However, this treatment must be followed by solvent dewaxing in order to improve the cold properties of the oil.

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