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FULL LENGTH ARTICLE

Solvent refining of heavy wax distillate for the removal of carcinogenic compounds

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KEYWORDS

Extraction; Wax distillate; Carcinogenic compounds; Ethylacetoacetate; Mixed solvents **Abstract** Neat ethylacetoacetate (EAA) and its mixtures with a co-solvent and an anti-solvent have been studied for refining of heavy wax distillate fraction to produce substantially non-carcinogenic base oil. The co-solvent and anti-solvent used are dipropylene glycol (DPG) and ethylene glycol (EG) respectively. The solubility characteristics of the main solvent and its mixed solvent systems were studied. Selection of the optimum solvent mixture and extraction variables has been studied. The effect of co-solvent and anti-solvent addition on the carcinogenic potential and raffinate quality has been determined under clearly comparable conditions.

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1. Introduction

The base mineral oils are manufactured from crude of vacuum distillation to produce several distillates and a residual oil. In relation to the health hazards, all crude oils contain some polycyclic aromatic compounds (PACs) some of which are known to be carcinogenic, particularly the four or more condensed ring compounds [1–4]. The content of polycyclic aromatic compounds in base oil must be greatly reduced usually to

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the extent that the refined product is no longer carcinogenic by the refining processes and the level or severity of treatment.

Furfural, phenol and N-methyl-2-pyrrolidone have been used for industrial scale lube oil refining till now. All have advantages and disadvantages from a technical and economical stand–point [5–8].

The European commission has adopted chemical test method IP 346 as the sole criterion for classifying base oil carcinogenicity, in line with its policy to minimize the regulatory requirements for animal testing .The adoption of IP 346 was based on its ability to predict the threshold of carcinogenicity as indicated by animal test data [1]. In Europe, suppliers of lubricant base oils must classify their products against European Union (EU) criteria, which say that "lubricant base oils must be classified as carcinogenic unless they can be demonstrated to contain less than 3% (w/w) DMSO extract by IP 346" [1,2,9].

The present work aims to study the effect of solvent refining at severe treatment conditions on the carcinogenic potential of

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the heavy wax distillate and to produce substantially non-carcinogenic base oil. Thus, ethylacetoacetate EAA was used as a basic solvent for refining the local heavy wax distillate fraction, as it has good selectivity for aromatics; low toxicity and it is non-corrosive and non-carcinogenic [10,11].

2. Experimental

Feedstock:

Heavy wax distillate fraction (b.r. 450-510 °C) was offered by the Suez Oil Processing Company.

Solvents:

Ethylacetoacetate (EAA) as the basic solvent. Dipropylene glycol (DPG) as a co-solvent. Ethylene glycol (EG) as an antisolvent.

2.1. Solvent extraction

All the solvents were of normal laboratory reagent grade. These solvents are considered not to be toxicant or mutagenic or carcinogenic to humans

[11–14].

The heavy wax distillate fraction was subjected to bench scale extraction with the basic solvent and its mixtures with the co-solvent and the antisolvent. A jacketed mixer settler apparatus was used. The extraction variables were studied as follows:

2.1.1. EAA

Extraction temperatures (60–90 °C) and solvent oil ratios (ranging from 2:1 to 7:1).

2.1.2. EAA + DPG mixture

Solvent composition (the percentage of DPG from 0–20 wt%), solvent oil ratios (ranging from 2:1 to 5:1) and at two extraction temperatures of 60 and 70 °C.

2.1.3. EAA + EG mixture

Solvent Composition (the percentage of EG from 0-15 wt%), solvent oil ratios (ranging of 2:1–5:1) and at two extraction temperatures of 80 and 90 °C.

2.2. Solvent dewaxing

Dewaxing process was carried out for the raffinate obtained under the most suitable extraction conditions with the solvent mixture of EAA + 15% EG, solvent oil ratio of 3:1 and extraction temperature of 80 °C. Methyl isobutyle ketone (MIBK) was used as dewaxing solvent, solvent feed ratio of 3:1 and 1:1 for dilution and washing respectively at dewaxing temperature of -15 °C [15].

2.3. Finishing

The finishing process was considered the final step in refining processes. The dewaxed oil was treated with adsorption via percolation technique using bentonite as adsorbent at 75 °C and under elution of N₂ gas carrier at 10 psi. The bentonite was firstly activated at 120 °C for 2 h. Percolation technique was carried out via a continuous process, where the dewaxed oil was passed through a static bed of bentonite in a double jacket long glass column to purify and decolorize and finish the oil.

The feedstock, raffinats, dewaxed oil and base oil were subjected to the following analysis:

- The physico-chemical characteristics according to the standard methods [16].
- Polycyclic aromatics content (PCAs) by IP 346 method as an inspection test of carcinogenicity [17].
- The hydrocarbon component analysis by using liquid solid column chromatography technique [18].
- The structural group analysis based on physical constants by n-d-m method [16,19].

3. Results and discussion

The physical characteristics, hydrocarbon component analysis and structural group analysis of the feedstock are presented in Table 1.

| Table 1 | Physical | characteris | tics, hyd | lrocarbon | component |
|-------------|------------|-------------|------------|-----------|-----------|
| analysis an | nd structu | ral group a | nalysis of | the feeds | tock. |

| Characteristics | Feed |
|----------------------------------|--------|
| Refractive index, 70 °C | 1.4918 |
| Density (gm/cc) 70 °C | 0.8839 |
| Mean molecular weight | 415 |
| Pour point (°C) | 47 |
| Sulfur content (wt%) | 1.80 |
| Kinematic viscosity 40 °C, cSt | 99 |
| Kinematic viscosity, 100 °C, cSt | 9.8 |
| Viscosity index | 70 |
| Conradson carbon residue (wt%) | 0.630 |
| PCAs (IP346) (wt%)* | 9.28 |
| Component analysis | |
| Total saturates (wt%) | 47.53 |
| Total aromatics (wt%) | 52.47 |
| Mono aromatics (wt%) | 16.42 |
| Di aromatics (wt%) | 26.01 |
| Poly aromatics (wt%) | 10.05 |
| Structural group analysis | |
| Carbon distribution | |
| %C _A | 17.47 |
| %C _N | 20.81 |
| %C _R | 38.28 |
| %C _P | 61.72 |
| Ring content analysis | |
| R _A | 0.89 |
| R _T | 2.25 |
| R _N | 1.36 |

* PCAs = Polycyclic aromatics content; C_A = Aromatic carbon; C_N = Naphthenic carbon; $C_R = C_P + C_A$; C_P = Paraffinic carbon; R_A = Aromatic ring; R_N = Naphthenic ring; R_T = $R_A + R_N$.



Figure 1 Effect of extraction temperature on the yield, physical characteristics and polycyclic aromatics content of the raffinates.

3.1. Extraction with pure EAA solvent

Extraction was carried out for the feedstock at different temperatures with pure EAA solvent. Data are presented graphically in Figs. 1 and 2. Extraction temperature is considered as one of the main factors of extraction variables. The extraction should comprise between selectivity and solubility.

The yield and the sulfur content of the raffinates decrease by increasing the extraction temperature, the decrease may be attributed to the removal of the more polar aromatic components by extraction (Fig. 1).

The spectrum carbon distribution and ring content analysis are parallel with the above finding, whereas the percentage of aromatic carbon (% C_A) and aromatic ring (R_A) per molecule decrease with increasing the extraction temperature (Fig. 2).

Fig. 1 shows that, improvement in raffinate quality on raising extraction temperature in terms of increasing the viscosity index and mean molecular weight of the raffinates.

Polycyclic aromatics (PCA_S) content of the raffinates; which is a measure of carcinogenic potential; decreases with the increase of extraction temperature (Fig. 1). But their values are still higher than 3 wt%, thus the raffinates are classified as carcinogenic [2].

To increase the solvent power of EAA toward the polycyclic aromatics, solvent extractions were carried out for the feedstock with pure EAA solvent at various solvent feed ratios ranging from 2:1 to 7:1 at two extraction temperatures of 80 and 90 °C. Data are presented graphically in Figs. 3 and 4.



Figure 2 Effect of extraction temperature on carbon distribution and ring. Content of the raffinates.

Figs. 3 and 4 indicate that the increase of solvent feed ratio lowers the raffinate yield and improves its quality in terms of



Figure 3 Effect of solvent feed ratio on the yield, physical characteristics and polycyclic aromatics content of the raffinates.



Figure 4 Effect of solvent feed ratio on carbon distribution and ring content of the raffinates, at 80 °C (a) and 90 °C (b).



Figure 5 Effect of co-solvent type and its concentration on the critical. Solution temperature of the feed solvent systems.

increasing viscosity index and mean molecular weight and lowering sulfur and polycyclic aromatic contents, the percentage of aromatic carbon (% C_A) and aromatic ring (R_A) per molecule. Although the polycyclic aromatic content is reduced to 3.26 and 3 wt% by extraction of the feedstock at 80° and 90 °C respectively with EAA at the highest solvent feed ratio of 7:1,however, these severe extraction conditions are not enough to reduce the carcinogenic potential of the raffinate.

To increase the severity of the extraction process, the basic solvent EAA is mixed with a co-solvent to increase its solvent power toward the polycyclic aromatics, or mixed with an antisolvent to increase its selectivity.

3.2. Critical solution temperature (CST)

Solvent feed miscibility temperature data are the prime requirements for fixing solvent extraction operation [20,21]. Phenyl, ethanol, ethylene glycol monophenylether, dipropylene glycol and cyclohexane were tested as co-solvents. Therefore the CST for the feedstock with different concentrations of each solvent in EAA was determined as shown in Fig. 5.

As may be expected, increasing the concentration of each in EAA decreases the critical solution temperature of the system and consequently increases the solvent power and decreases its selectivity.

Fig. 6 demonstrates that increasing the solvent feed ratio from 1:1 to 2:1 decreases the CST of the system. The maximum



Figure 6 Effect of solvent composition and solvent feed ratio on the critical. Solution temperature (CSt) of the feed solvent systems.



Figure 7 Effect of solvent composition on the critical solution temperature of the feed solvent systems.

Table 2 Effect of solvent composition on physical characteristics and structural group analysis of the raffinates. solvent: EAA + *DPG. Extraction temperature = 70 °C. Solvent feed ratio = 2:1.

| DPG in EAA | 0% | 5% | 10% |
|--|--------|--------|--------|
| Characteristics | | | |
| Yield (wt%) | 78 | 77.12 | 76.84 |
| Refractive index (70 °C) | 1.4776 | 1.4775 | 1.4774 |
| Density, gm 1 cc 70 °C | 0.8658 | 0.8646 | 0.8643 |
| Mean molecular weight | 446 | 449 | 451 |
| Pour point (°C) | 49 | 50 | 51 |
| Sulfur content (wt%) | 1.51 | 1.50 | 1.48 |
| Kinematic viscosity, cSt, 40 °C | 79 | 78.5 | 78 |
| Kinematic viscosity, cSt, 100 °C | 8.75 | 8.7 | 8.7 |
| Viscosity index | 78 | 78 | 79 |
| Refining effectiveness number (REN _{VI}) | 2.7500 | 2.8600 | 2.5733 |
| PCAs (IP346) (wt%) | 6.66 | 4.18 | 4.15 |
| Structural group analysis | | | |
| Carbon distribution | | | |
| %C _A | 10.50 | 10.50 | 10.49 |
| %C _N | 24.70 | 23.91 | 23.73 |
| %C _R | 35.20 | 34.42 | 34.22 |
| %C _P | 64.80 | 65.58 | 65.78 |
| Ring content analysis | | | |
| R _A | 0.55 | 0.55 | 0.55 |
| R _T | 2.23 | 2.18 | 2.18 |
| R _N | 1.68 | 1.63 | 1.63 |

* DPG = dipropylene glycol.

extraction temperature must be 20–25 °C below the CST of the solvent feedstock system. Thus the most suitable co-solvent is dipropylene glycol (DPG) as it gives moderate CST for the system.

Also the CST for the feedstock with different concentrations of water or ethylene glycol (EG) as antisolvent in EAA was determined. Data are presented graphically in Fig. 7. It can be noticed from the graph that increasing the concentration of H_2O or EG in EAA elevates the critical solution temperature of the system and consequently decreases the solvent power and increases its selectivity.

3.3. Extraction with EAA and a Co-solvent

According to CST data in Fig. 6, dipropylene glycol (DPG) was chosen as a blending solvent (co-solvent) mixed with EAA and the extraction temperature was predicated for each DPG concentration in EAA. Extractions were carried out at 70 °C with EAA containing 5% and 10% DPG at solvent feed ratio of 2:1 and at 60° with EAA containing 15% and 20% DPG at solvent feed ratios ranging from 2:1 to 5:1. Data are represented in Tables 2–4.

Irrespective of temperature, the DPG concentration of 5%, 10% and 15% in solvent mixture and at solvent feed ratio of 2:1 give raffinates having more or less the same physical characteristics, carbon distribution, ring content per molecule and PCAs content.

Data in Tables 3 and 4 indicate that, increasing the solvent ratio lowers the yield, sulfur, and PCAs content, percentage of aromatic carbon (% C_A) and aromatic ring (R_A) per molecule accompanied with improvement in raffinate quality in terms of

Table 3 Effect of solvent feed ratio for ethylacetoacetate containing 15% dipropylene glycol on the physical characteristics, hydrocarbon component analysis and structural group analysis of the raffinates. Extraction temperature = $60 \,^{\circ}$ C.

| Solvent feed ratio, by wt. | 2/1 | 3/1 | 5/1 |
|--|--------|--------|--------|
| Characteristics | | | |
| Yield (wt%) | 79.25 | 75.50 | 70.45 |
| Refractive index, 70 °C | 1.4779 | 1.4747 | 1.4707 |
| Density, gm 1 cc, 70 °C | 0.8651 | 0.8602 | 0.8544 |
| Mean molecular weight | 457 | 474 | 490 |
| Pour Point (°C) | 51 | 52 | 53 |
| Sulfur content (wt%) | 1.54 | 1.42 | 1.27 |
| Kinematic viscosity, cSt, 40 °C | 78 | 73 | 64 |
| Kinematic viscosity, cSt, 100 °C | 8.75 | 8.65 | 8.50 |
| Viscosity index | 80 | 88 | 103 |
| Refining effectiveness number (REN _{VI}) | 2.0750 | 1.3611 | 0.8955 |
| PCAs (IP346) (wt%) | 4.10 | 3.71 | 2.75 |
| Refining effectiveness number $(REN_{IP})^*$ | 4.0058 | 4.3986 | 4.5253 |
| Component analysis | | | |
| Total saturates (wt%) | _ | _ | 65.20 |
| Total aromatics (wt%) | _ | _ | 34.80 |
| Mono aromatics (wt%) | - | - | 11.53 |
| Di aromatics (wt%) | _ | _ | 19.76 |
| Poly aromatics (wt%) | - | - | 3.51 |
| Structural group analysis | | | |
| Carbon distribution | | | |
| %C _A | 10.55 | 9.10 | 7.11 |
| %C _N | 23.34 | 23.21 | 23.81 |
| %C _R | 33.89 | 32.31 | 30.91 |
| %C _P | 66.10 | 67.69 | 69.09 |
| Ring content analysis | | | |
| R _A | 0.57 | 0.50 | 0.39 |
| R _T | 2.19 | 2.16 | 2.13 |
| R _N | 1.62 | 1.66 | 1.74 |

* Refining effectiveness number for PCAs content determined by IP346.

elevating viscosity index and mean molecular weight due to the increase of solvent power for the two solvent mixtures (EAA + 15% and 20% DPG). Also, it is clear from component data that the solvent power increases by the increase of DPG concentration from 15% to 20% in EAA, toward aromatics.

The data of PCAs content reveal that extraction of the feed with solvent mixtures containing 15% and 20% DPG in EAA, using solvent ratio of 5:1 and at extraction temperature of 60 °C gives non-carcinogenic oils.

3.4. Extraction with EAA with an antisolvent

Ethylene glycol (EG) was chosen as a blending solvent mixed with EAA. The effects of EG concentration in the solvent mixture on the yield, physical characteristics and structural group analysis of the raffinates have been studied at two temperatures of 80 and 90 °C and at solvent feed ratio of 3:1. Data are represented in Tables 5 and 6.

The increase in EG concentration gives high increase in raffinate yield accompanied with the decrease of PCAs content and improvement in its quality in terms of mean molecular weight and viscosity index.

Table 4 Effect of solvent feed ratio for ethylacetoacetate containing 20% dipropylene glycol on the physical characteristics, hydrocarbon component analysis and structural group analysis of the raffinates. Extraction temperature = 60 °C.

| Solvent feed ratio, by wt. | 2/1 | 3/1 | 5/1 |
|--|--------|--------|--------|
| Characteristics | | | |
| Yield (wt%) | 76.52 | 73.75 | 68.29 |
| Refractive index, 70 °C | 1.4777 | 1.4748 | 1.4707 |
| Density, gm 1 cc, 70 °C | 0.8649 | 0.8604 | 0.8544 |
| Mean molecular weight | 469 | 477 | 494 |
| Pour point (°C) | 51 | 52 | 54 |
| Sulfur content (wt%) | 1.50 | 1.40 | 1.25 |
| Kinematic viscosity, cSt, 40 °C | 72 | 68.5 | 62 |
| Kinematic viscosity, cSt, 100 °C | 8.60 | 8.55 | 8.45 |
| Viscosity index | 88 | 94 | 107 |
| Refining effectiveness number (REN _{VI}) | 1.3044 | 1.0938 | 0.8570 |
| PCAs (IP346) (wt%) | 4.00 | 3.68 | 1.97 |
| Refining effectiveness number (REN _{IP}) | 4.4470 | 4.6875 | 4.3379 |
| Component analysis | | | |
| Total saturates (wt%) | - | 62.17 | 68.06 |
| Total aromatics (wt%) | _ | 37.83 | 31.94 |
| Mono aromatics (wt%) | - | 13.03 | 11.02 |
| Di aromatics (wt%) | - | 20.56 | 18.26 |
| Poly aromatics (wt%) | _ | 4.24 | 2.66 |
| Structural group analysis | | | |
| Carbon distribution | | | |
| %C _A | 10.24 | 9.07 | 7.04 |
| %C _N | 23.15 | 23.21 | 23.74 |
| %C _R | 33.39 | 32.29 | 30.78 |
| %CP | 66.61 | 67.71 | 69.22 |
| Ring content analysis | | | |
| R _A | 0.56 | 0.50 | 0.39 |
| R _T | 2.22 | 2.17 | 2.15 |
| R _N | 1.65 | 1.67 | 1.76 |

Correlating the data of PCAs content, aromatics content, percentage of aromatic carbon (${}^{\wedge}C_A$) and aromatic ring (R_A) per molecule with the data of mean molecular weight and viscosity index of the raffinates)Table 5(reveal that in spite of decreasing the PCAs content, there is an increase in the percentage of aromatic carbon (${}^{\wedge}C_A$), aromatic ring (R_A) per molecule, the percentage of paraffinic carbon (${}^{\wedge}C_P$) and the mono-aromatics content increase with the increase of EG concentration in EAA. This can be explained that these aromatic components may contain few number of long paraffinic side chain attached to the aromatic ring; especially the mono-aromatic ones; which affect the mean molecular weight of the raffinates and increase their viscosity indices. These data indicate the advantage of EG addition to EAA solvent as it increases the selectivity of the solvent mixture as discussed before.

Data in Table 7 reveal that extraction of the feedstock with solvent mixture EAA containing 15% EG, using solvent feed ratio of 5:1 and at extraction temperature of 80 °C gives raffinate of the lowest PCAs content (1.75 wt%) with the highest mean molecular weight (520) and viscosity index (134) due to the high selectivity and high solvent power for the solvent mixture.

According to the European union for lubricant oils carcinogenicity classification, using the standard method IP346, the obtained raffinates (Table 8) are classified as non-carcinogenic lubricating oils as their PCAs contents are less than 3%.

Table 5 Effect of solvent composition on physical character-
istics, hydrocarbon component analysis and structural group
analysis of the raffinates. Solvent: EAA + *EG. Extraction
temperature = 80° C. Solvent feed ratio = 3:1.

| EG in EAA | 0% | 5% | 10% | 15% |
|---|--------|--------|--------|--------|
| Characteristics | | | | |
| Yield (wt%) | 69.19 | 72.85 | 77.81 | 81.8 |
| Refractive index, 70 °C | 1.4734 | 1.4748 | 1.4761 | 1.4775 |
| Density, gm 1 cc, 70 °C | 0.8605 | 0.8619 | 0.8634 | 0.8649 |
| Mean molecular weight | 473 | 482 | 491 | 499 |
| Pour point (°C) | 50 | 51 | 52 | 53 |
| Sulfur content (wt%) | 1.28 | 1.38 | 1.44 | 1.45 |
| Kinematic viscosity, cSt, 40 °C | 70.0 | 68.38 | 68.63 | 64.88 |
| Kinematic viscosity, cSt, 100 °C | 8.7 | 8.87 | 8.94 | 9.10 |
| Viscosity index | 95 | 103 | 104 | 119 |
| Refining effectiveness number (RENy) | 1.2844 | 0.8227 | 0.6526 | 0.3714 |
| PCAs (IP346) (wt%) | 6.16 | 3.14 | 2.96 | 1.95 |
| Refining effectiveness number | 9.8750 | 4.4218 | 3.5111 | 2.4829 |
| (REN _{IP}) | | | | |
| Component analysis | | | | |
| Total saturates (wt%) | - | 59.02 | 59.36 | 60 |
| Total aromatics (wt%) | - | 40.98 | 40.64 | 40 |
| Mono aromatics (wt%) | _ | 15.00 | 16.72 | 19.05 |
| Di aromatics (wt%) | _ | 22.20 | 20.52 | 18.52 |
| Poly aromatics (wt%) | _ | 3.78 | 3.40 | 2.43 |
| Structural group analysis | | | | |
| Carbon distribution | | | | |
| %C _A | 7.69 | 8.38 | 8.91 | 9.57 |
| %C _N | 26.44 | 24.88 | 23.78 | 22.67 |
| %C _R | 34.13 | 33.26 | 32.69 | 32.24 |
| %C _P | 65.87 | 66.74 | 67.31 | 67.76 |
| Ring content analysis | | | | |
| R _A | 0.41 | 0.46 | 0.51 | 0.56 |
| R _T | 2.33 | 2.29 | 2.29 | 2.29 |
| R _N | 1.92 | 1.83 | 1.78 | 1.73 |

* EG = Ethylene glycol.

For all solvent mixtures studied for the removal of carcinogenic compounds, solvent efficiency was measured by the refining effectiveness number (REN) [22]. It is considered to be a good measure of attainable refining effect, i.e. the lower REN, the better is the solvent efficiency. Taking PCAs content and the viscosity index changes as two refining parameters and applying this to the present investigation, (Table 8) it is evident that the solvent mixture EAA + 15% EG is the best solvent mixture used for refining the feedstock with solvent feed ratio of 3:1 at an extraction temperature of 80 °C to produce substantially non-carcinogenic lube oil with best quality.

3.5. Solvent dewaxing

It can be noticed that, the extraction of the feedstock under the most suitable solvent composition of EAA + 15% EG for the removal of polycyclic aromatics, the paraffinicity nature of the raffinates increases in terms of elevating the pour point, mean molecular weight, viscosity index, the percentage of paraffinic carbon (%C_P), and total saturates content (compare Table 1 with Table 5). The high pour point of the raffinate is related to the high wax content. Hence, the raffinate must be subjected

Table 6 Effect of solvent composition on physical characteristics, hydrocarbon component analysis and structural group analysis of the raffinates. Solvent: EAA + EG. Extraction temperature = 90 °C. Solvent feed ratio = 3:1.

| EG in EAA | 0% | 3% | 5% | 10% |
|----------------------------------|---------|--------|--------|--------|
| Characteristics | | | | |
| Yield (wt%) | 61.98 | 71.59 | 72.77 | 76.36 |
| Refractive index, 70 °C | 1.4725 | 1.4730 | 1.4734 | 1.4753 |
| Density, gm 1 cc, 70 °C | 0.8593 | 0.8595 | 0.8600 | 0.8620 |
| Mean molecular weight | 476 | 489 | 491 | 493 |
| Pour point, °C | 50 | 51 | 51 | 52 |
| Sulfur content (wt%) | 1.27 | 1.37 | 1.40 | 1.47 |
| Kinematic viscosity, cSt, 40 °C | 67 | 67.73 | 64.37 | 64.34 |
| Kinematic viscosity, cSt, 100 °C | 8.6 | 8.72 | 8.73 | 8.74 |
| Viscosity index | 99 | 100 | 108 | 109 |
| Refining effectiveness number | 1.2690 | 0.9470 | 0.7166 | 0.6062 |
| (REN _{VI}) | | | | |
| *PCAs (IP346) (wt%) | 5.50 | 3.22 | 3.00 | 2.93 |
| Refining effectiveness number | 10.0580 | 4.6881 | 4.3360 | 3.7228 |
| (REN _{IP}) | | | | |
| Component analysis | | | | |
| Total saturates (wt%) | _ | - | 61.10 | 61.74 |
| Total aromatics (wt%) | - | - | 38.90 | 38.26 |
| Mono aromatics (wt%) | _ | _ | 14.01 | 16.11 |
| Di aromatics (wt%) | _ | - | 21.40 | 19.13 |
| Poly aromatics (wt%) | - | - | 3.44 | 3.02 |
| Structural group analysis | | | | |
| Carbon distribution | | | | |
| %C _A | 6.93 | 7.46 | 7.63 | 8.66 |
| %C _N | 26.92 | 25.18 | 24.86 | 23.44 |
| %C _R | 33.85 | 32.64 | 32.49 | 32.10 |
| $%C_{P}$ | 66.15 | 67.36 | 67.51 | 67.90 |
| Ring content analysis | | | | |
| R _A | 0.37 | 0.41 | 0.42 | 0.49 |
| R _T | 2.32 | 2.28 | 2.28 | 2.25 |
| R _N | 1.95 | 1.87 | 1.86 | 1.76 |

to solvent dewaxing to remove the high melting waxes to overcome their bad effect on the low temperature characteristics and to study the effect of wax removal on the carcinogenic potential of the dewaxed oil.

The yield of dewaxed oil is greatly affected due to the separation of a great amount of wax as well as the oil inherent to such wax (Table 9). The decrease in pour point, viscosity index and mean molecular weight is due to the decrease of the saturated components and consequently the corresponding increase in the aromatic components and PCAs and sulfur contents of the dewaxed oil.

Carbon distribution spectrum and ring content analysis are parallel to the above findings.

The polycyclic aromatics content of the dewaxed oil is increased from 1.95 to 2.5 wt% and can be classified as non carcinogenic oil.

3.6. Finishing

The dewaxed oil must be subjected to finishing process to improve the color, color stability and oxidation resistance. It was treated with adsorption technique via percolation using activated bentonite to reduce the level of trace contaminated constituents.

Table 7 Effect of solvent feed ratio for ethylacetoacetate containing 15% ethylene glycol on the physical characteristics, hydrocarbon component analysis and structural group analysis of the raffinates. Extraction temperature = $80 \,^{\circ}$ C.

| Solvent feed ratio, by wt. | 2/1 | 3/1 | 5/1 |
|---|--------|--------|--------|
| Characteristics | | | |
| Yield (wt%) | 86.47 | 81.8 | 76.32 |
| Refractive index, 70 °C | 1.4802 | 1.4775 | 1.4744 |
| Density, gm 1 cc, 70 °C | 0.8698 | 0.8649 | 0.8600 |
| Mean molecular weight | 466 | 499 | 520 |
| Pour point (°C) | 41 | 53 | 54 |
| Sulfur content (wt%) | 1.60 | 1.45 | 1.34 |
| Kinematic viscosity, cSt, 40 °C | 77.36 | 64.88 | 57.46 |
| Kinematic viscosity, cSt, 100 °C | 9.12 | 9.10 | 8.96 |
| Viscosity index | 91 | 119 | 134 |
| Refining effectiveness number | 0.6443 | 0.3714 | 0.3700 |
| (REN _{VI}) | | | |
| PCAs (IP346) (wt%) | 3.95 | 1.95 | 1.75 |
| Refining effectiveness number (REN _{IP}) | 2.5385 | 2.4829 | 3.1448 |
| Component analysis | | | |
| Total saturates (wt%) | _ | 60 | 63.55 |
| Total aromatics (wt%) | _ | 40 | 36.45 |
| Mono aromatics (wt%) | — | 19.05 | 18.51 |
| Di aromatics (wt%) | _ | 18.52 | 15.62 |
| Poly aromatics (wt%) | - | 2.43 | 2.32 |
| Structural group analysis | | | |
| Carbon distribution | | | |
| $%C_{A}$ | 10.75 | 9.57 | 8.21 |
| $%C_{N}$ | 24.15 | 22.67 | 22.30 |
| %C _R | 34.90 | 32.24 | 30.51 |
| %C _P | 65.10 | 67.76 | 69.49 |
| Ring content analysis | | | |
| R _A | 0.59 | 0.56 | 0.49 |
| R _T | 2.32 | 2.29 | 2.26 |
| R _N | 1.73 | 1.73 | 1.77 |

From data in Table 9, it is clear that refractive index, density and viscosity values are slightly decreased.

It is obvious from carbon distribution spectrum and aromatic content that clay treatment is accompanied by a decrease in aromatic components, the percentage of aromatic (%C_A) and naphthenic (%C_N) carbons and consequently an increase in the percentage of paraffinic carbon (%C_P) and total saturated components. Accordingly, the pour point, viscosity and mean molecular weight of the base oil are slightly increased (Table 9).

Meanwhile, there is a slight improvement in ASTM color of the finished oil.

The PCAs content of the dewaxed oil is decreased from 2.5 to 2.26 wt% by clay percolation and the base oil is substantially non-carcinogenic.

4. Conclusion

The results in this study reveal the following:

• The addition of ethylene glycol as an antisolvent to ethylacetoacetate solvent improves its solvent efficiency than the addition of dipropylene glycol as a co-solvent in reducing the polycyclic aromatics content.

| Raffinate No. | Extraction condition | 8 | | PCAs content REN _{IP} | REN _{IP} | VI | RENVI |
|---------------|----------------------|-----------|--------------------|--------------------------------|-------------------|-----|--------|
| | Temperature (°C) | S/F ratio | % Blending solvent | | | | |
| 1 | 60 | 5:1 | 15% DPG | 2.75 | 4.5253 | 103 | 0.8955 |
| 2 | 60 | 5:1 | 20% DPG | 1.97 | 4.3379 | 107 | 0.8570 |
| 3 | 80 | 3:1 | 10% EG | 2.96 | 3.5111 | 104 | 0.6526 |
| 4 | 90 | 3:1 | 10% EG | 2.93 | 3.7228 | 109 | 0.6062 |
| 5 | 80 | 3:1 | 15% EG | 1.95 | 2.4829 | 119 | 0.3714 |
| 6 | 80 | 5:1 | 15% EG | 1.75 | 3.1448 | 134 | 0.3700 |

 Table 8
 Non-carcinogenic lubricating oils.

Table 9 Effect of the solvent dewaxing and clay finishing on the physical characteristics, hydrocarboncomponent analysis and structural group analysis of the raffinate.

| Characteristics | Raffinate | Dewaxed oil | Finished oil |
|----------------------------------|-----------|-------------|--------------|
| Yield on dewaxed oil (wt%) | _ | - | 78.9 |
| Yield on raffinate (wt%) | - | 52.99 | - |
| Yield on feedstock (wt%) | 81.8 | 43.35 | 34.20 |
| Refractive index 70 °C | 1.4775 | 1.4856 | 1.4815 |
| Density, gm 1 cc, 70 °C | 0.8649 | 0.8828 | 0.8736 |
| Mean molecular weight | 499 | 479 | 483 |
| Pour point, (°C) | 53 | -6 | -5 |
| Sulfur content (wt%) | 1.45 | 1.60 | 1.49 |
| Kinematic viscosity, cSt, 40 °C | 64.88 | 84.35 | 74.11 |
| Kinematic viscosity, cSt, 100 °C | 9.10 | 9.91 | 9.14 |
| Viscosity index | 119 | 96 | 97 |
| PCAs (IP346) (wt%) | 1.95 | 2.5 | 2.26 |
| Color | 8 | 8.5 | 7.5 |
| Component analysis | | | |
| Total saturates (wt%) | 60 | 51.5 | 54.2 |
| Total aromatics (wt%) | 40 | 48.5 | 45.76 |
| Mono aromatics (wt%) | 19.05 | 18 | 18.55 |
| Di aromatics (wt%) | 18.52 | 27.68 | 24.65 |
| Poly aromatics (wt%) | 2.43 | 2.82 | 2.56 |
| Structural group analysis | | | |
| Carbon distribution | | | |
| %C _A | 9.57 | 10.57 | 10.21 |
| %C _N | 22.67 | 29.08 | 25.97 |
| %C _R | 32.24 | 39.66 | 36.18 |
| %C _P | 67.76 | 60.34 | 63.82 |
| Ring content analysis | | | |
| R _A | 0.56 | 0.60 | 0.58 |
| R _T | 2.29 | 2.84 | 2.56 |
| R _N | 1.73 | 2.24 | 1.98 |

• Extraction of the feedstock with the solvent mixture of ethylacetoacetate containing 15% ethylene glycol, followed by solvent dewaxing and finishing process, gives substantially non-carcinogenic base oil with good quality.

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