Fuel Processing Technology 92 (2011) 447–451

Contents lists available at ScienceDirect

Fuel Processing Technology

journal homepage: www.elsevier.com/locate/fuproc

Production of lubricating base oil from slop wax by different subsequent refining techniques

Magdy T. Zaky *, Sahar M. Tawfik

Petroleum Refining Division, Egyptian Petroleum Research Institute (EPRI), Nasr City, P. O. Box: 11727, Cairo, Egypt

ARTICLE INFO

Article history:
Received 28 April 2010
Received in revised form 18 September 2010
Accepted 21 October 2010
Available online 13 November 2010

Keywords:
Subsequent refining techniques
Solvent dewaxing
Solvent extraction
Slop wax waste by-product
Lubricating base oil

ABSTRACT

Different subsequent refining techniques including solvent dewaxing and solvent extraction have been used to produce lubricating base oil from slop wax waste by-product. The solvent dewaxing technique was performed using two different solvent mixtures of methyl ethyl ketone and toluene with and without benzene at different dilution solvent ratios and at different dewaxing temperatures. The solvent extraction technique was carried out using N-methyl-2-pyrrolidone solvent at 90 °C and at constant dilution solvent ratio of 3:1 by weight. The resulting data revealed that the highest yield of lubricating base oil having the lowest pour point (−6 °C) and the highest iso- and cyclo-paraffins to n-paraffins ratio (5.11) is obtained by solvent dewaxing process only. While the lowest yield of lubricating base oil having the highest pour point (−1 °C) and the lowest iso- and cyclo-paraffins to n-paraffins ratio (4.08) is obtained using solvent dewaxing followed by solvent extraction process. These lubricating base oil products, especially the one that having the lowest pour point (−6 °C) matches the principal characteristics of AX 973853 type of premium low viscosity textile machinery oils obtained by Mobile Velocite Oil Company.

1. Introduction

Slop wax is a waste by-product of slack wax deoiling. The slack wax is also a by-product of dewaxing process obtained through the manufacture of lubricating oils. Generally, slop wax does not find any high quality applications but is ordinarily added to boiler fuel and burned. Slop wax contains low melting point isoparaffins and naphthenes, both types of hydrocarbons having high viscosity indices. Better utilization of slop wax product will raise the yield of lube components from vacuum distillates and hence increase the production volume of high viscosity index lubes [1,2].

Ivanova et al. used slop wax as a raw material for wax production. Their studies have shown that the slop wax from the combination of dewaxing–deoiling process, containing up to 40% wax, can be used as a component of the feed in wax production [3].

Men'shchikov et al. suggested that slop wax can be used as a pyrolysis feedstock with virtually no pretreatment, since it formed from distillation and hence does not contain any non vaporizable components. They found that high yields of olefins would be obtained from the slop wax, as based on the structure of the average molecule; also, their results have demonstrated the feasibility of using slop wax as a partial replacement for naphtha pyrolysis feedstocks [1].

Esipko et al. investigated the possibility of using slop wax from a deoiling unit as a raw material for the production of commercial lube components by catalytic hydrodewaxing. They found that the hydrodewaxed product from this fraction matches industrial oils of the I-12A type in terms of the principal physicochemical characteristics [4].

In lube oil processing, the solvent dewaxing process is considered as an important technique to be used for removal of the wax from the feedstock in the temperature range of the desired pour point. Also, the solvent extraction process is used for the removal of undesirable components such as aromatics and other low viscosity index materials. The characteristics of ideal dewaxing solvent include the following: low solvent power of wax, high solvent power for oil, low freeze point, low viscosity, less in cost, non-toxic and have chemical and thermal stabilities. Also, ideal extraction solvent must have the following characteristics: high solvent power, low selectivity, easy recovery, low vapor pressure, high specific gravity for rapid separation of the oil and solvent phases, high thermal and chemical stabilities, non-corrosive and non-toxic for the environmental safe, adaptability to a wide range of feedstocks and availability at a reasonable cost [5,6].

Thus, the present work deals with the use of slop wax; which is a waste by-product obtained through the lube oil manufacture; for production of useful lubricating base oil which can be used in an

* Corresponding author. Tel.: +20 2 22745902; fax: +20 2 22747433.
E-mail address: magdytadrous@hotmail.com (M.T. Zaky).

0378-3820/$ – see front matter © 2010 Elsevier B.V. All rights reserved.
doi:10.1016/j.fuproc.2010.10.008

industrial application by using different sequence of refining techniques.

2. Experimental

2.1. Materials

One appropriate slop wax crude; separated from slack wax mixture (light, middle and heavy slack waxes) obtained from El-Ameria Refining Company is used in this study for production of useful lubricating base oil.

2.2. Production of lubricating base oil

Three sequences of refining techniques were used to refine slop wax crude which are as follow: solvent dewaxing, solvent dewaxing followed by solvent extraction and solvent extraction followed by solvent dewaxing.

2.2.1. Solvent dewaxing technique

Two solvent mixtures of methyl ethyl ketone (MEK) containing 30 wt.% toluene (T) and 30 wt.% benzene (B) (MEK:T:B = 40:30:30) and methyl ethyl ketone containing 60 wt.% toluene (MEK:T = 40:60 by weight) were used for dewaxing the feed at different solvent feed ratios of dilution (S/F, by weight) ranging from 3:1 to 7:1 at fixed washing solvent ratio of 1:1 and at different dewaxing temperatures ranging from −15 to −25 °C.

A known weight of the feed (slop wax and its raffinate) was dissolved in the corresponding amount of solvent in a beaker and heated until the mixture becomes homogenous. Then the mixture was cooled gradually at room temperature. The beaker and the Buchner funnel were transferred to a controlled temperature unit and gradually cooled to the desired temperature. The beaker contents were transferred to the funnel and filtered through a Whatman filter paper No. 43 by using gentle suction. The wax cake was washed with additional solvent at the same temperature and added at small increments. Solvents were removed from the wax cake and oil filtrate by distillation.

2.2.2. Solvent extraction technique

The slop wax crude and its dewaxed oil were exposed to solvent extraction technique by using N-methyl-2-pyrollidone solvent at constant extraction temperature of 90 °C and at solvent to feed ratio of 3:1 [7]. A jacketed mixer settler apparatus was used. The extraction temperature was adjusted to an accuracy of ±1 °C by using an ultra thermostat. The feed and the solvent were kept in good contact by continuous agitation for 45 min. After agitation, the phases were left to settle for 45 min and then separated. The solvent was removed from the raffinate phase by washing six times with hot distilled water. The raffinate was then dried over anhydrous calcium chloride. The solvent was removed from the extract phase by distillation under reduced pressure.

2.3. Methods of analysis

The slop wax crude, its raffinate and the isolated base oils were physically characterized according to American Society for Testing and Materials (ASTM) standard methods [8]. The standard methods for analysis are, congealing point (ASTM D-938), pour point (ASTM D-97), flash point (ASTM D-92), kinematic viscosity (ASTM D-445), viscosity index (ASTM D-2270), refractive index (ASTM D-1747), density (ASTM D-1418), specific gravity (ASTM D-1298), mean molecular weight (ASTM D-2502), oil content (ASTM D-721), color (ASTM D-1500), n-d-M analysis (ASTM D-3238) and sulfur content by using X-ray fluorescence sulfur meter (ASTM D-4294).

The aromatic content of the slop wax crude, its raffinate and the isolated base oils was determined using the liquid–solid column chromatography technique. A 1.3 cm diameter and a height of 130 cm column packed with activated (60–200 mesh) silica gel was used [9]. The column was then moistened with 100 ml of petroleum ether 40–60 °C to dissipate the heat of adsorption. A 10 g sample of the soft wax or the oil dissolved in few milliliters of petroleum ether 40–60 °C was transferred to the column. The column was then eluted with 300 ml of petroleum ether 40–60 °C followed by 200 ml benzene and finally 100 ml of a 1:1 mixture of absolute methanol and benzene. Fractions of 25 ml were taken from the column, the solvent distilled off and the refractive index of each fraction was determined. According to the refractive index data at 20 °C, eluates were combined into saturates, mono-, di- and poly-aromatics. The saturate hydrocarbons have refractive indices not more than 1.48. The mono-cyclic, bi-cyclic and poly-cyclic aromatics have refractive indices from 1.48 to 1.53, 1.53 to 1.59 and higher than 1.59, respectively [10].

n-Paraffins content, iso- and cyclo-paraffins content and iso- and cyclo-paraffins to n-paraffins ratio were determined for the slop wax crude, its raffinate and the isolated base oils using GC technique. The GC apparatus used was Perkin Elemer (Clarus 500), equipped with a hydrogen flame ionization detector and fused silica capillary column (60 m length × 0.32 mm i.d), packed with poly (dimethyl siloxane) HP-1 (non-polar packing) of 0.5 μm film thickness. In the chromatograph, the injector was heated at 350 °C. The column temperature was programmed from 100 to 300 °C at a fixed rate of 3 °C/min, and nitrogen (oxygen-free) was used as a carrier gas with flow rate of 2 ml/min. The detector was heated at 350 °C, and operated with a hydrogen flow rate adjusted to optimize the detector sensitivity. The sample was melted and 0.1 μl of it was introduced into the injector. A mixture of pure n-paraffins was used as standard. The peak area of each resolved component (consisting of either n- and iso-paraffin) is determined individually. However, the unresolved complex mixtures (humps); composed of non n-paraffins presumably mainly cyclo-paraffins and aromatics with long side chains; were determined only as a total.

3. Results and discussion

3.1. Characterization of crude wax

The physical characteristics and the molecular type composition for the slop wax crude are presented in Table 1. Data indicate that the slop wax has high pour point and mean molecular weight and low refractive index due to its high saturates content; especially its n-

Table 1
The physical characteristics and molecular type composition of slop wax crude.

Characteristics	Slop wax
Congealing point, °C	39
Density at 70 °C	0.8069
Density at 20 °C	0.8452
Refractive index at 20 °C	1.4707
Kinematic viscosity at 100 °C, mm^2/s	4.24
Mean molecular weight	404
Oil content, wt.%	15.94
Sulfur content, wt.%	0.33
Color	4
Molecular type composition	
Saturates content, wt.%	84.55
n-Paraffin content, wt.%	34.79
Iso-& cyclo-paraffins content, wt.%	49.76
Iso-& cyclo-paraffins/n-paraffins ratio	1.43
Aromatics content, wt.%	15.45
Mono-aromatics, wt.%	13.49
Di-aromatics, wt.%	1.96