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ANALYSIS OF PARAMETERS INFLUENCING THE PROPERTIES AND YIELD OF LUBE-OIL EXTRACTION PRODUCTS

P. KALAICHELVI

(Department of Chemical Engineering, Regional Engineering College, Trichy – 620 015, INDIA)

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An analysis of the influence of various parameters (mainly the compositional effect of the lube feed stock and the extraction temperature) on the percentage yield and properties of the lube oil is presented. The experiment is conducted at different temperatures in a single-stage extractor using furfural as the solvent. Based on the analysis, a correlation has been developed and verified with the experimental results.

Keywords: extraction, lube oil

Introduction

Crude oil is distilled in atmospheric distillation column to get various useful distillates and the bottom product (residue) is taken to vacuum distillation column from which the lube oil feed stocks are obtained as distillates. The feed stocks contain saturates, olefins, napthenes, aromatics and asphaltic components [1]. The asphaltic compenents are removed by deasphalting. Aromatic components are prone to oxidative and thermal degradation and also have poorest viscometric properties. Removal of these aromatics is needed and is done by solvent extraction.

Various solvents suitable for lube oil extraction are sulphur dioxide, phenol, furfural, N-methyl pyrrolidone(NMP), duo -sol and propane. Comparison of physical properties of the above mentioned solvents shows that NMP is an attractive solvent due to its high solvent power, low toxicity, good selectivity and adaptability [2]. However, due to the availability, low cost and high selectivity furfural is widely used for this purpose [3].

Mixer-settler, Rotating Disc Contactors, Pulsed columns and Centrifugal extractors are used for solvent extraction. Rotating Disc Contactors(RDC) shows better performance when compared with other extraction columns. However, modifying, specifying or designing as extractors from basic principles alone is unsound. Effect of various parameters including system behavior can only be studied through small scale equipment like single stage extractor [4].

Lube oil is obtained only from imported crudes. It is also well known that crude composition will change not only with respect to source but also with respect to time. Based on this, the compositions of lube feed stocks will also vary. Analysis of the above mentioned constrains revealed that there is a need to study the effect of various parameters mainly the effect of composition of lube base stocks on extraction yield and properties of the finished product in single stage extractor using furfural as a solvent [5]

Experiments

A single stage equilibrium set-up as shown in *Fig.1* is used for the present study. This is a cylindrical vessel with outer jacket both made of glass. This is closed at the top with a cork having provisions for thermometer, stirrer inserting and feed solvent charging. Hot oil is circulated through the outer jacket from a hot oil bath to heat the content of the vessel at constant temperature. A stirrer driven by an electric variable motor is provided for constant mixing. The bottom tapering end is provided with a stop cork for separating the two phases (bottom phase - extract and top phase – raffinate) after extraction.

The circulating bath is switched on and the temperature is set at a desired value. The lube oil feed stock and the solvent are charged in a ratio of 1:1.4 with the help of a funnel. Then the content of the inner cylinder is heated to a required temperature and the stirrer is switched on and kept at a desired speed. Mixing is done for one hour. After mixing it is allowed to settle at the same temperature for one and half hour. Mixing and settling times are fixed based on the earlier

Contact information: E-mail: kalai@rect.ernet.in

Table 1 Properties of various lube oil feed mixtures

Properties	Feed	Feed Mixtures #1	#2	#3	#4
Density, 15 °C (gm/ml)	0.9254	0.8979	0.8822	0.9536	0.9314
Pour Point (°C)	+42	+42	+45	+39	+39
Kinematic Viscosity (CST)					
(i) 100 °C	10.37	9.1	8.3	12.58	10.49
(ii) 75 °C	NA	18.44	16.42	29.90	23.25
Refractive Index, 75 °C	1.49669	1.47948	1.46888	1.51370	1.50140
Wt % Saturates	41	54.2	61.2	28.5	38.2
Wt % Aromatics	59	45.8	38.8	71.5	61.8

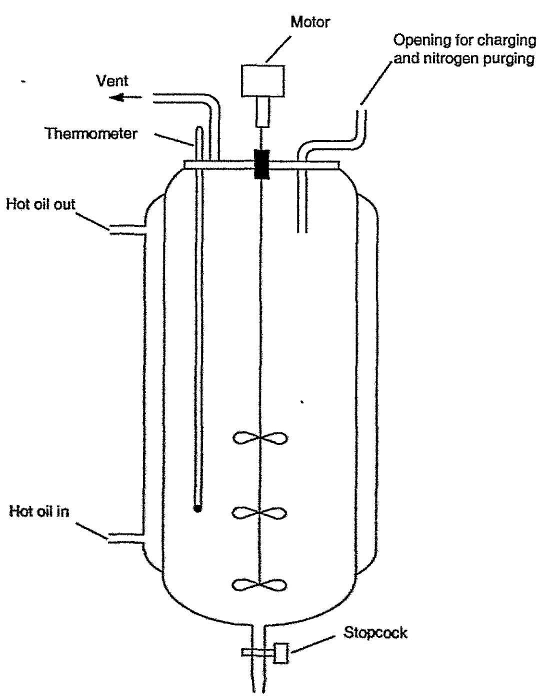

Fig.1 Single stage equilibrium setup

studies in the same set-up. Then the two phases namely raffinate and extract are separately collected with the help of stop cork. Solvents present in two phases viz., raffinate and extract are recovered by simple distillation with nitrogen purging. Final traces are removed by evaporation with nitrogen purging. Properties of the feed stocks and solvent free raffinates (before and after dewaxing) and extracts are estimated based on the procedures given in ASTM standard books [6,7].

In this present work solvent/feed (*S/F*) ratio (by volume) is kept at 1.4 for all the cases for comparison purpose. Extraction studies are carried out for lube oil feed stock whose properties are given in *Table 1*. To have various feed compositions, different combinations of feed + (raffinate/extract) on volume basis are mixed and properties of these mixed feed stocks are also reported along with the original feed stock [5] in *Table 1*. Extraction temperatures are fixed based on miscibility temperatures of various combinations of feed mixtures [2,5].

Table 2 Extraction results for extraction temperatures of 65 °C and *S/F* ratio of 1.4

Properties	Feed	Feed Mixtures #1	#2	#3	#4
RAFFINATE					
Yield (Wt %)	80	90	94	70	77
Density, 15 °C (gm/ml)	0.9079	0.8856	0.8777	NA	NA
Pour Point (°C)	+45	+42	+45	+39	+39
Kinematic Viscosity (CST)					
(i) 100 °C	9.35	8.32	8.13	10.22	9.38
(ii) 75 °C	19.31	17.40	15.82	22.33	19.50
Refractive Index, 75 °C	1.48307	1.47081	1.46475	1.49342	1.48471
Wt % Saturates	NA	NA	NA	38.2	146.2
Wt % Aromatics	NA	NA	NA	61.8	53.8
EXTRACT					
Yield (Wt %)	20	10	6	30	23
Kinematic Viscosity (CST)					
(i) 100 °C	26.40	NA	NA	27.63	27.39
(ii) 75 °C	85.73	NA	NA	98.94	94.28
Refractive Index, 75 °C	NA	1.54395	1.54334	NA	1.56569

Results

Results of single stage extraction of all the feed mixtures at three different temperatures are obtained in the present study. Yields and properties of raffinates and extracts at an extraction temperature viz., 65 °C, 90 °C and 110 °C are estimated and sample data are presented in *Table* 2 for 65 °C. Extraction results at other temperatures (90 & 110 °C) are available elsewhere [5]. Effect of extraction temperature and feed composition on raffinate yield and quality are discussed based on the results obtained.

Discussion

Increase in saturates content in the feed has increased the raffinate yield and quality at all extraction temperatures and decreased the extract yield (shown in *Fig.*2). Extractions were not carried out for the feed mixture-3 at 110 °C, since the miscibility temperatures

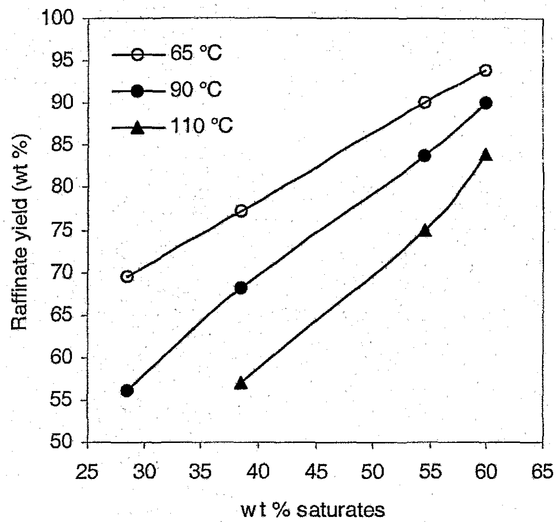

Fig.2 Plot of wt % saturates in feed versus Raffinate yield ($S/F = 1.4$)

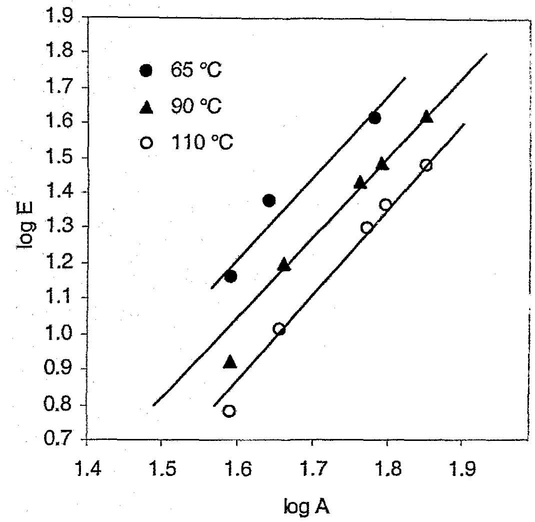

Fig.4 Logarithmic plot of Aromatic content versus Extract yield ($S/F = 1.4$)

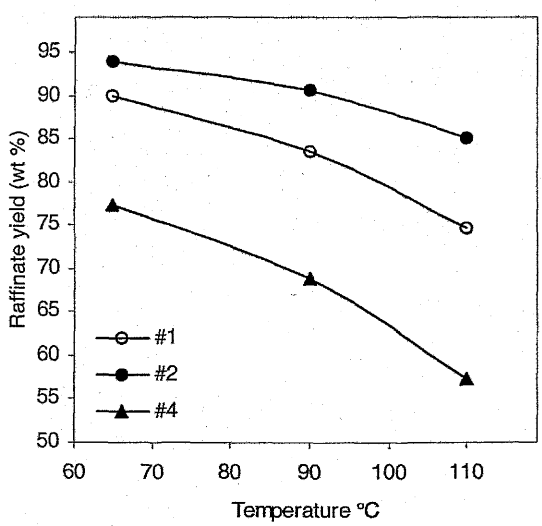

Fig.3 Plot of Temperature versus Raffinate yield for various feed mixtures: #1, #2, #4

of this feed mixture is below (110 °C). Effect of feed composition viz., weight % of saturates in feed on raffinate yield can be observed in *Fig.2*. Effect of other parameter viz., extraction temperature is shown in *Fig.3*. This shows that increase in extraction temperature will decrease the raffinate yield, whereas the quality of raffinate will increase due to the removal of aromatics since at higher temperatures the miscibility of aromatics will be higher. Also, since amount of aromatics present in the feed mixtures (1 and 2) is less than that of the original feedstock, it has increased the extraction efficiency , whereas it is reverse in the other two feed mixtures (3 and 4) due to high aromatic content present in them, which can be observed in *Fig.3*.

Correlation Development

Kalichevsky [8] has developed series of equations for expressing solubility characteristics of petroleum oils in various solvents and checked over relatively wide ranges of solvent feed ratios and temperatures. He has developed the following equation for calculating extract yield from solvent quantity and temperature based on the assumptions that extract yield linearly varies with solvent quantity and solubility is an exponential function of temperature.

$$\log(E) = (m + nT)\log(S) + p + qT \qquad (1)$$

In this present work, linear relationship is observed between extract yield and aromatic content in feed (*Fig.4*). In liquid-liquid extraction, the above said assumption on solubility is also applicable and hence the equation suggested by Kalichevsky is modified as

$$\log(E) = (m + nT)\log(A) + p + qT \qquad (2)$$

since the S/F ratio is maintained constant in the present work, where m, n, p & q are constants. These constants are estimated using the experimental results and the values are

$m = 4.079$
$n = -9.703 \times 10^{-3}$
$p = -6.588$ and
$q = 2.137 \times 10^{-3}$
A = Aromatic content in the feed
T = Extraction temperature
S = Solvent quantity
E = Extract yield

Using the above estimated values of constants, extract yields are estimated and compared with the experimental results and this comparison is shown in *Fig.5*. From this, it is inferred that percentage deviation of extract yield is only (+/-) 2 %.

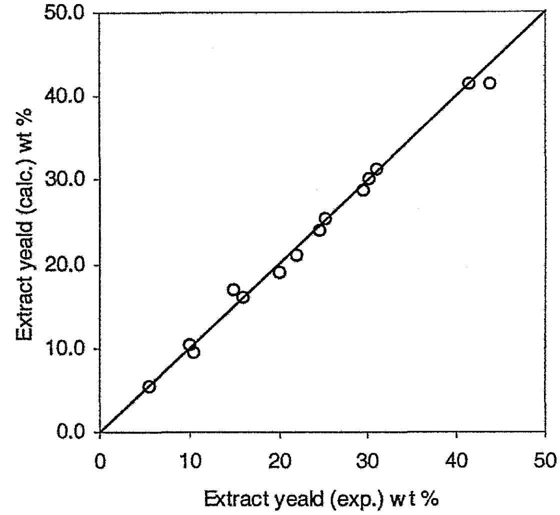

Fig.5 Plot of calculated extract yield versus experimental yield

Conclusion

Effect of temperature and feed composition on raffinate, extract yield and their qualities have been analysed for a single stage equilibrium set-up and based on this a correlation has been developed and verified with the experimental results. However, this correlation should be modified in such a way that it should include other operating parameters like *S/F* ratio, stirrer speed, geometry of the extraction column and physical properties of the feed stocks and solvents [8,9] for which data can be generated by taking all the above said parameters into account.

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SYMBOLS

E	extract yield
S	solvent quantity
T	extraction temperature
A	aromatic content in the feed
CST	critical solution temperature
NA	not available
NMP	n-methyl pyrrholidone
RDC	Rotating Disc Contactor
m	constant
n	constant
p	constant
q	constant

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