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Extraction of Aromatic Hydrocarbons from Lube Oil Using Different Co-Solvent

Ibtehal k. Shakir and Muslim A. Qasim
Chemical Engineering Department, College of Engineering, Baghdad University

Abstract

An investigation was conducted effect of addition co- solvent on solvent extraction process for two types of a lubricating oil fraction (spindle) and (SAE-30) obtained from vacuum distillation unit of lube oil plant of Daura Refinery. In this study two types of co-solvents (formamide and N-methyl, 2, pyrrolidone) were blended with furfural to extract aromatic hydrocarbons which are the undesirable materials in raw lubricating oil, in order to improve the viscosity index, viscosity and yield of produced lubricating oil. The studied operating condition are extraction temperature range from 70 to 110 °C for formamide and 80 to 120 °C for N-methyl, 2, pyrrolidone, solvent to oil ratio range from 1:1 to 2:1 (wt./wt.) for furfural with formamide extraction and 1:1 to 3:1 (wt./wt.) for furfural with NMP extraction. The results of the investigation show that the viscosity index of lubricating oil fraction increases while viscosity and percentage yield of raffinate decreases with increasing extraction temperature, the solvent to oil ratio and co-solvent to furfural ratio. For formamide the best temperature were 90 °C, furfural to co-solvent ratio (60:40) and solvent to lube oil ratio (1.5:1) to get best value of viscosity index 102, viscosity 3.01 cst and 69.23 % yield. While for NMP co-solvent 110 °C extraction temperature, (2:1) solvent to lube oil ratio and (60:40) furfural to co-solvent ratio, to produce lube oil with 96 viscosity index, 9.10 cst viscosity and 68.50 yield.

Key Words: Co-solvent, extraction of aromatic, lubricating oil, viscosity index, viscosity, yield

Introduction

Refining of crude oil to produce lubricating oil is one of the oldest refinery arts. Suitable crudes are fractional to isolate a suitable boiling range material, usually in the 316 to 593 °C range, to produce a distilled oil fraction. Various solvent purification steps are then used to reject components not suitable for lubricating stock. Aromatics are too unstable, and refiners resort to various means to remove aromatics from potential lube fraction. While many solvents were proposed for aromatics extraction, furfural has been a preferred solvent since about 1933 when the first commercial furfural extraction units were built. Since the furfural unit is often a bottleneck in the lube refining process, improvement in the capacity of furfural without loss of selectivity would be of value to the lube refining industry [1].

The most important properties of lubricating oil which solvent extraction is meant to improve are viscosity–temperature characteristics, stability toward oxidation, and carbon residue. The improvement of these properties is accomplished almost entirely by the extraction of aromatics [2].
Since the feed stock contains aromatic material usually ranging from 30 wt. % to 60 wt. %, the feed stock is initially subjected to an extraction step. Removal of aromatic material improves the viscosity index, color, and oxidation stability and inhibition response of the base oil [3].

A strong temptation to produce high viscosity index lubes has led to a prolific growth of solvent extraction. Solvents widely used for this affair are furfural, phenol, mixture of cresols and propane. Many other solvent's like aniline, sulfur dioxide are enlisted; but without much use [4].

Furfural is the most widely used solvent because its selectivity toward aromatic compounds is high enough [5]. Solvent and lubricating oil are especially complex systems because of the high number of components in the system [6]. Using a second solvent in liquid-liquid extraction is a common task in extraction. Second solvent could increase the yield of extraction by forming extract-solvent complex [7].

Specifications required achieved by Niigata Engineering co. are leading companies in Iraq to produce lubricating bas oil from lube-oil cut in furfural extraction unit [8]. The most important specifications and operation condition worked out by this company are listed in table (1).

Table (1): important specifications and operation condition present from Niigata Engineering Co. [8].

Type stock	Stock 40 (spindle)	Stock 60 (SAE-30)
Viscosity at 100 °C, cst	2.9-3.2	9.0-11.0
Viscosity index 80°C	97-105	92-97
Temperature (top) treating	90	121
Yield Vol. %	70	60
Furfural:lube oil	1.8-2:1	2.2-2.5:1

The improvement in the properties of an oil gained by solvent refining is somewhat dependent on the properties of the original stock as well as the type of solvent employed, and since base oils differ widely in molecular composition and physical properties due to the crude source, hence each refinery must satisfy himself by laboratory tests and studying in detail the selected fraction of lubricating oil [9].

Raman and Devotta [3], in 2003, discovered an improved furfural extraction process for lube oil base stock containing aromatic type material by the addition of a co-solvent preferably an aliphatic amide or mixture of amides to furfural to facilitate phase separation, selectivity, and increase the raffinate yield while maintaining the same raffinate measured by raffinate refractive index. The results show that for extraction conducted at solvent to feed treat ratio of 1.5, the co-solvent –furfural blends are more effective than furfural alone, resulting in more than 3 vol % improvement in raffinate yield at same raffinate refractive index, and an increase of more than 5 vol. % of raffinate yield at same refractive index at solvent to feed treat ratio of 1.8.

Abdul-Halim and amal [10], in 2008, improved the viscosity index of a lubricating oil fraction (SAE – 30). By extracting the undesirable materials which reduce the viscosity index of raw lubricating oil fraction, the first solvent was furfural and the second was NMP (N-methyl, 2, pyrrolidone). Where the extraction temperature was range from 70 to 110 °C for furfural and NMP, solvent to oil ratio range from 1:1 to 5:1 (wt. /wt.) for furfural and from 0.5:1 to 2:1 (wt. /wt.) for NMP. The results show that the viscosity index of lubricating oil fraction increases with increasing extraction temperature and increasing the solvent to oil ratio and reaches 83 for NMP extraction at extraction temperature 110 °C and solvent to oil ratio 2:1, while the viscosity index reaches to 80 for furfural extraction at the same extraction temperature and solvent oil ratio. Higher viscosity index of lubricating oil fraction is obtained by using NMP instead of furfural under the same operating variables.

Hatampipour M. S. Etal [11], in 2009, used 2, 2, 4-tri methyl pentane as co-solvent with furfural for extraction of aromatic hydrocarbons from lube oil cut. General binary interaction parameters are computed and reported for estimating the liquid- liquid equilibrium products between 50 and 70 °C. Also, a generalized model is presented for calculation of the refractive index and specific gravity of lube oil fraction.

Raman et al.[3], in 2010, suggested to use N-dimethyl amide as a co-solvent with NMP to increase extraction yield by more than 6 wt. % than raffinate obtained by using NMP alone. This co-solvent was selected from a group consisting of formamide, N-methyl formamide, acetamide, propionamide.

Alibrahemi and Sharif [12], in 2010, study the extraction system under atmospheric pressure and different temperatures to improve the properties of lubricating oil produced from a vacuum distillation in the Al_Daura refinery, to increase the productivity of raffinate; the top product of extraction column, and to improve the process by using two different types of solvents (furfural and normal NMP). The best results achieved for specification and productivity blending (80% furfural with 20% NMP and up to 60% furfural with 40% NMP), where viscosity index increased to (114) and refractive index decreased with increasing production rate.

The aim of this study is to improve the percent raffinate yield and viscosity index of different lubricating base oil stock 40 (spindle) and stock 60 (SAE-30) obtained from vacuum distillation unit of Dura Refinery by using a new solvent mixture (furfural + a co-solvent). The investigation includes also a detail study of the effect of extraction temperature and the type of co-solvent choosing from two co-solvent (formamide and N-methyl, 2-pyrrolidone) which was blended to furfural, solvent to co-solvent ratio, and mixed solvent to lube oil ratio on viscosity, viscosity index and the percentage yield of the produced lubricating oil.

EXPERIMENTAL

Feedstock

Two feed stocks were used in this work, the distillate lube oil fraction Stock 40 (spindle) and Stock 60 (SAE-30) obtained from vacuum distillation unit of lube oil plant of Daura Refinery. The feedstock for vacuum distillation unit was atmospheric residue produced from mixed Iraqi crude oils (60 % of Basrah, 30 % of Kirkuk and 10 % of Sharki-

Baghdad).Table (2) shows the determined properties of the lube oil fraction [9].

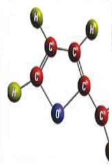
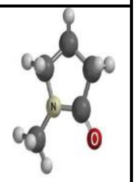
Table (2), Properties of lube oil distillation fraction

No.	Specification	Feedstock 40 (Spendile)	Feedstock 60 (SAE-40)
1	Specific Gravity @ 60/60 °F	0.896	0.932
2	Viscosity, cSt, @ 40°C	21	151.48
3	Viscosity, cSt, @ 100°C	3.9	11.68
4	Viscosity index	59	47
5	Pour point, °C	22.3	38.34
6	COC flash point, °C	204.45	260
7	Sulfur content, % Wt.	1.95	2.76
8	Color, ASTM-D1500, at 25°C	2.5	4.5

Solvents

Three solvents were used in this work. These solvents are furfural (Liaosin private limited company, China), N-Methyl-2-pyrrolidinone (Fluka chemicals AG, Germany) and formamide (Exact Chemical Co., China) Table (3) shows the determined properties of these solvent.

Table (3), Properties of furfural, formamide and NMP

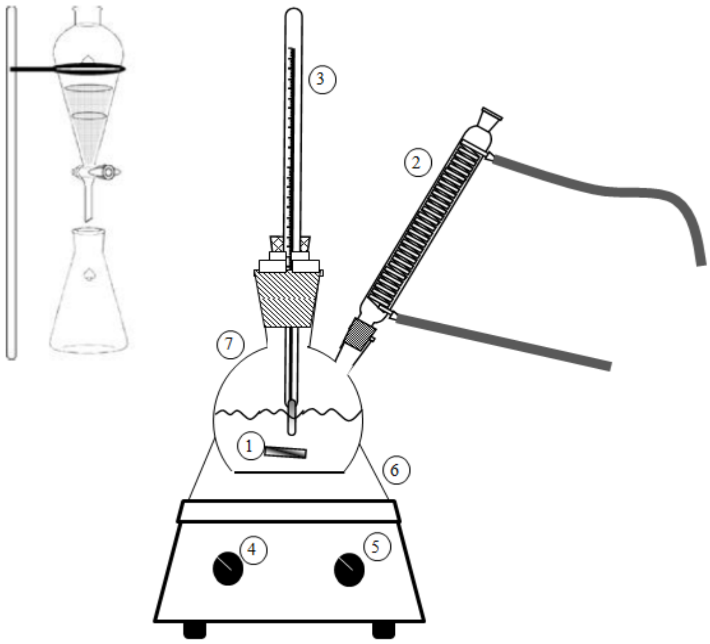
	furfural	formamide	NMP
Structure			
Density (d_4^{25}), g/cm^3	1.1563	1.134	1.0270
Boiling point, °C	161	210	202
Refractive Index (n_D^{25})	1.5235	1.447	1.4690
COC Flash Point, °C	68	175	95
Melting Point, °C	-36.5	2.5	-24

Extraction Experiment

Laboratory batch extraction unit was used in the present work. Fig.(1) shows the details of the laboratory extraction unit. This unit consists of a Heat Source Stuart Magnetic Stirrer Device, a bench scale 1-liter and a 250ml, of 2-necks Pyrex flask extraction apparatus. The middle flask neck was connected with thermometer and the second neck was connected with recycle condenser.

The magnetic stirrer with 1.5"length magnetic stirrer bars was used to mix the feedstock with the extraction solvent inside the flash extractor.

Agitation speed and flask temperature were kept constant using regulator attached with electrical magnetic stirrer. The lube oil fraction was mixed with the extraction solvent in the extractor flask at controlled specified temperature.



1	Mixer(bars)
2	Condenser
3	Thermometer
4	Heat
5	stir
6	magnetic stirrer
7	2 necks Pyrex Flask

Fig.1, Laboratory extraction unit

The operating condition of total solvent to feedstock ratio, treating temperature and solvent to co-solvent ratio were specified. Mixing the three materials at the specified temperature was continued for the best period of 60 minutes then the mixture was left at the specified temperature for 60 minutes to be separated in to two phases by using a separating funnel. Top of the liquid phases are light raffinate solution and the bottom liquid phase is heavier extract solution. The raffinate phase contained about (70-80) % of lube oil without aromatics substances and (30-20) % solvent, the extract phase contained about (70-80) % 0f solvent with (30-20) % aromatics and naphthenic substances. The interface level formed after steady state to be constant. The two solutions were weighted to ensure material balance closure [9].

Solvent Recovery

The solvent was recovery from the raffinate solution by distillation under vacuum about (0.1 bar) to reduce flash point of distillate, prevent furfural decomposition and avoid oxidation of furfural (because the furfural will be converting to coal at higher temperature when contact with air). The unit consists of a 250ml, tow neck Pyrex flask, the middle flask neck was connected with thermometer (to indicate vapor temperature) and the second neck connected with condenser. At the end of condenser there is a receiver flask which connected with a trap and vacuum pump with a vacuum gage pressure. The raffinate solution was heated in the flask using heating mantle with a regulator to control heat supply. The stripped raffinate was weighted and the raffinate yield was obtained.

Result and Discussion

Effect of Extraction Temperature and Furfural to Co-Solvent (Formamide) Ratio For Stock 40 (Spindle) On Raffinate Viscosity

Fig. (2), shows the effect of the extraction temperature, and furfural to co-solvent (formamide) ratio on raffinate viscosity which calculated in laboratory at 100°C. The viscosity of raffinate product from solvent extraction process increase with decrease the extraction temperature or decreasing the solvent to lube oil ratio [6].

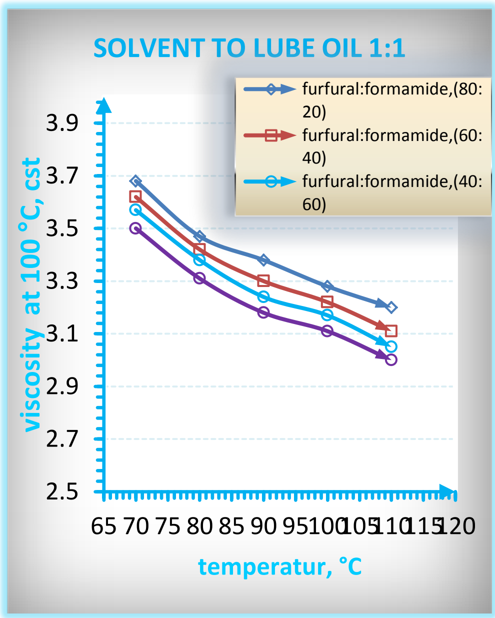


Fig. 2, Effect of extraction temperature on raffinate viscosity at various solvent to co-solvent ratio (at solvent to lube oil ratio 1:1)

Effect of extraction temperature and furfural to co-solvent (formamide) ratio for stock 40 (spindle) on Raffinate Viscosity Index

Figures (3) to (5) show the effect of the extraction temperature and solvent (furfural) to co-solvent (formamide) ratio on raffinate viscosity index at various mixed-solvent(furfural and formamide) to lube oil ratio. By analyzing the graph, the increase in extraction temperature will increase the viscosity index in raffinate phase for 1:1 mixed-solvent to oil ratio at different furfural to co-solvent (formamide) ratio.

Fig. (3), clearly indicates that furfural to formamide ratio slightly effect on the viscosity index for raffinate produced from extraction compared with extraction temperature for a given solvent to oil ratio, due to that the power solvent and selectivity for formamide and furfural solvents were nearly identical. The increase of extraction temperature by 10°C will increase the viscosity index one point for a given solvent to lube oil ratio at the different ratio from furfural and formamide solvent. The increase in extraction temperature will increase the solubility of undesirable materials especially polycondensed aromatics in solvent, the aromatics materials which reduce the viscosity index of lubricating oil [13].

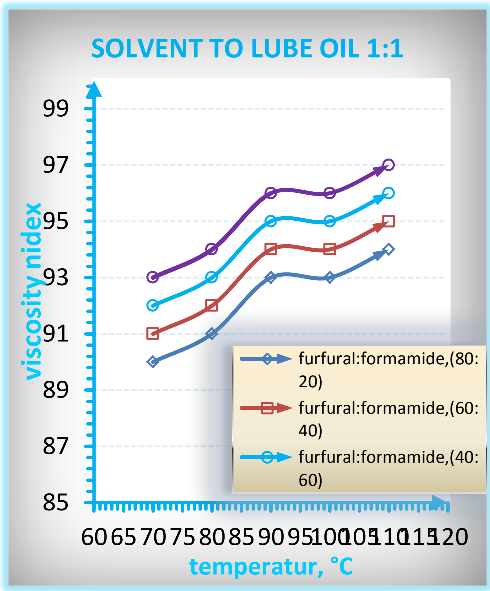


Fig.3, Effect of extraction temperature on raffinate viscosity index at various solvent to co-solvent ratio (at solvent to lube oil ratio 1:1)

Figures (4) and (5) indicate that the increase in solvent to lube oil ratio has a higher effect on increasing the viscosity index of lubricating oil fraction compared with extraction temperature and furfural to formamid ratio in extraction process. The increase of solvent to oil ratio (1.5:1) in Fig. (5) will increase viscosity index above five points nearly for a given extraction temperature and furfural to formamid ratio as extraction solvents.

While when increasing the extraction temperature above 100 °C the viscosity index has affixed value , due to that formamid solvent at high temperature will be has a low selectivity and high power solvent where it dissolves aromatic and paraffinic compound, together in extraction phase [14].

Since poly-aromatics and hetero compounds are components with a low viscosity index, poor oxidation and color stability, their removal is required in solvent extraction. Together with the poly-aromatics and the hetero compounds, most of the di-aromatics are extracted with only a part of the mono-aromatics [13].The selectivity of furfural is very higher than other solvents at low temperature, while the solvent power to furfural increasing at high temperature.

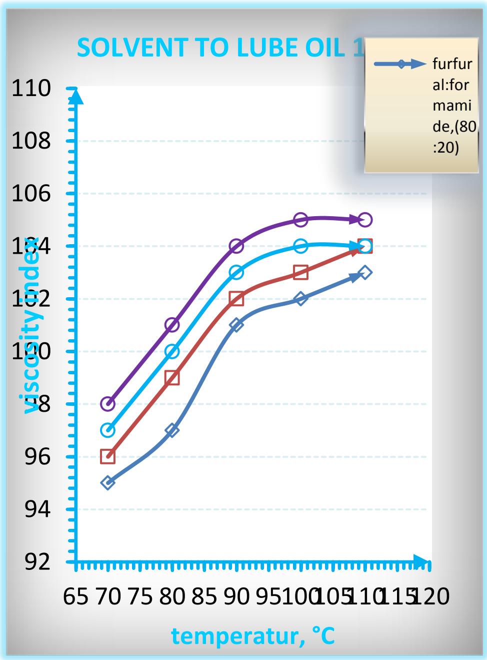


Fig.4, Effect of extraction temperature on raffinate viscosity index at various solvent to co-solvent ratio (at solvent to lube oil ratio 1.5:1)

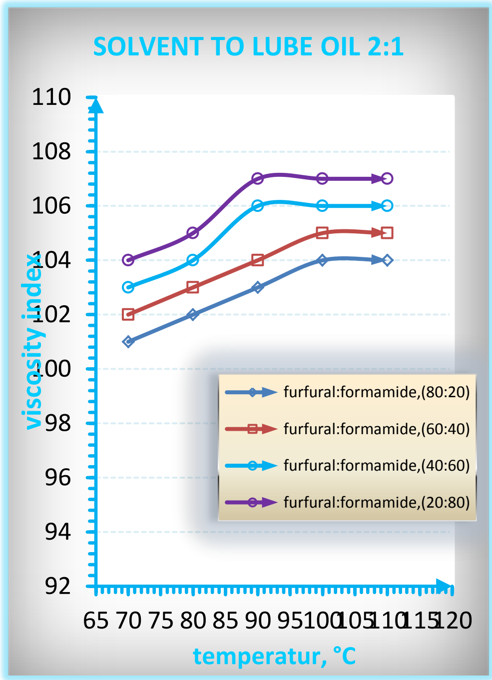


Fig. 5, Effect of extraction temperature on raffinate viscosity index at various solvent to co-solvent ratio (at solvent to lube oil ratio 2:1)

Effect of extraction temperature and furfural to co-solvent (formamide) ratio for stock 40 (spindle) on Raffinate Yield

Fig. (6) shows the effect of the extraction temperature, mixed-solvent (furfural and formamide) to lube oil ratio and solvent (furfural) to co-solvent (formamide) ratio on raffinate yield. By analyzing data in the graph, the furfural to formamide ratio slightly effected on the yield of raffinate produced from extraction compared with extraction temperature for a given solvent to oil ratio, due to the power solvent and selectivity for formamide and furfural solvents were the same specification also this feedstock of lube oil has few aromatic substance[1].

Using a second solvent in liquid–liquid extraction is a common task in extraction. The co-solvent could increase the yield of extraction in two ways, first is acting in parallel with main solvent, when the second solvent is selective to extract and increases the yield of extraction by forming extract–solvent complex. And the second is acting against main solvent when the second solvent is selective to raffinate. In this case second solvent increases the yield of extraction by forming raffinate–solvent complex, and the mixture reaches to equilibrium state in a shorter time. However, by use of the co-solvent the purity of extract may be increased. For this reason second solvent should have higher solubility in one of the extract or raffinate phases [5]. In this work formamide has a higher solubility in extract phase.

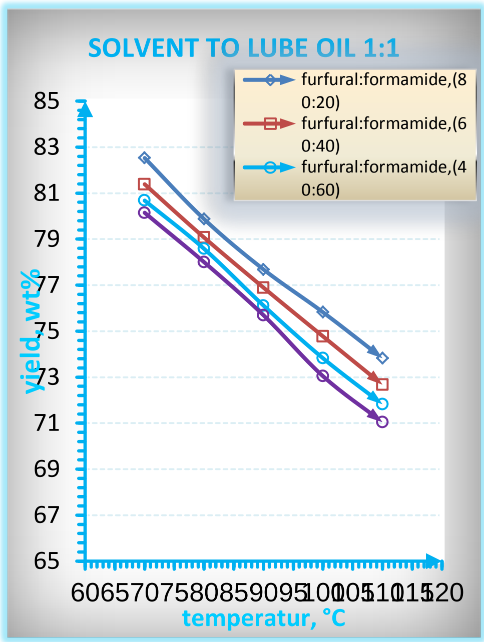


Fig.6 Effect of extraction temperature on raffinate yield at various solvent to co-solvent ratio (at solvent to lube oil ratio 1:1)

Effect of extraction temperature and furfural to co-solvent (NMP) ratio for stock 60 (SEA-30) on Raffinate Viscosity

Fig. (7) shows the effect of the extraction temperature, mixed-solvent (furfural and NMP) to lube oil ratio and solvent (furfural) to co-solvent (NMP) ratio on raffinate viscosity which calculated in laboratory at 100°C. The figure clearly indicated that the effect of the ratio of two solvents are higher than the effect of the extraction temperature on the viscosity of raffinate phase produced from solvent extraction for a given solvent to lube oil ratio. The gradual addition of co-solvent (NMP) decreases the amount of furfural for a given extraction temperature and solvent to lube oil ratio respectively which will have a higher effect in decreasing the raffinate viscosity, this caused by of the higher activity and solvent power of NMP in compare with furfural solvent[14].

The high molecular aromatics have the higher viscosity among the hydrocarbons in raw lubricating oils and the extraction of these materials decrease its content in the produced raffinate and increase the paraffin content which have a viscosity relatively lower than that of aromatics as mentioned by Kosters [15].

In general, the extraction temperature is responsible upon the relation of immiscibility between solvent and lube base oil, and a significant impact to extraction process. Its effect on selectivity of the solvent and solvent power, the solvent power increase with increase extraction temperature to become solvent and lube oil are mixing completely, while selectivity increase with decrease extraction temperature. The extraction temperature must install for each type of lubricating oil for any extraction process, so get the equilibrium between selectivity and power solvent to give best results qualitative and quantitative [16].

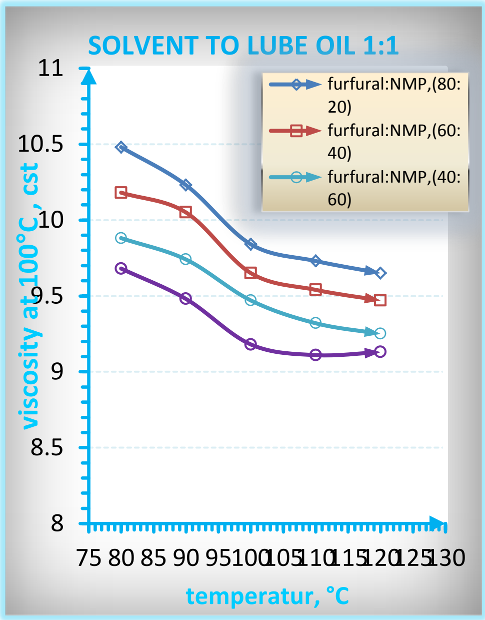


Fig. 7 Effect of extraction temperature on raffinate viscosity at various solvent to co-solvent ratio (at solvent to lube oil ratio 1:1)

Effect of extraction temperature and furfural to co-solvent (NMP) ratio for stock 60 (SEA-30) on Raffinate Viscosity Index

Figures (8) to (10) show the effect of the extraction temperature and solvent furfural to co-solvent (NMP) ratio on raffinate viscosity index at various mixed-solvent (furfural and NMP) to lube oil ratio. The increase in extraction temperature will encourage the solubility of undesirable materials such as aromatics and a small percentage of the paraffinic in extraction solvent. The graphic shows that the viscosity index increases with increase the extraction temperature. The increase of extraction temperature by 10°C will increase the viscosity index by approximately two points for a given solvent to lube oil ratio at the different ratio of furfural and NMP solvent.

The increases of the co-solvent NMP to furfural ratio will increase the viscosity index by approximately two point for a given solvent to lube oil ratio at the different extraction temperature, this caused by the high power solvent and selectivity of the co-solvent NMP in compared with furfural. The higher solvent power of co-solvent compared with furfural gave higher viscosity index for the same operating variables. These results are in agreement with those obtained by Sequeira and Sherman [14] which is stated that the solvent power is better (solvent to oil ratio is lower) for NMP than for furfural.

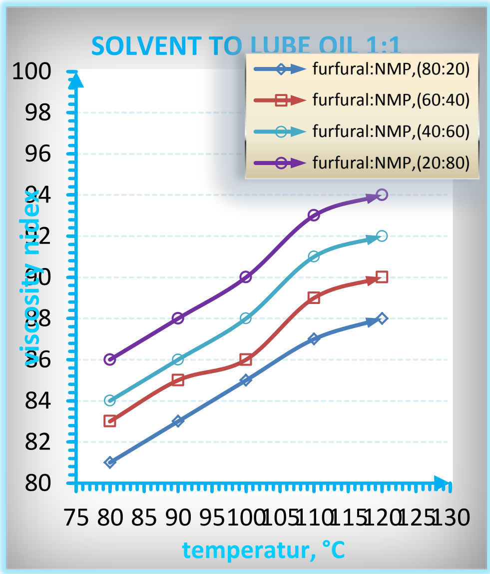


Fig.8 Effect of extraction temperature on raffinate viscosity index at various solvent to co-solvent ratio (at solvent to lube oil ratio 1:1)

Figures (9) and (10) show that the increase in solvent to lube oil ratio has a higher effect on increasing the viscosity index of lubricating oil fraction compared with extraction temperature and co-solvent to furfural ratio. In case of using furfural as extraction solvent in operating unit, the significant solvent to oil ratio should be higher than (1:1) ratio because of the constancy of the viscosity index on a fixed value using different extraction temperatures and that is due to the saturation of this amount of furfural with undesirable materials [16].

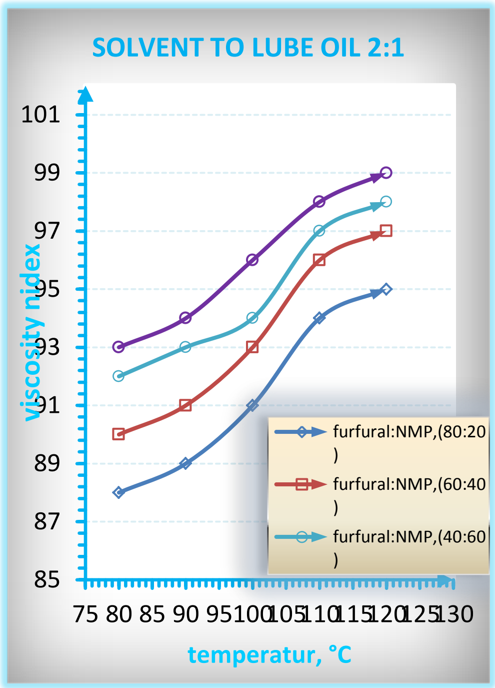


Fig. 9, Effect of extraction temperature on raffinate viscosity index at various solvent to co-solvent ratio (at solvent to lube oil ratio 2:1)

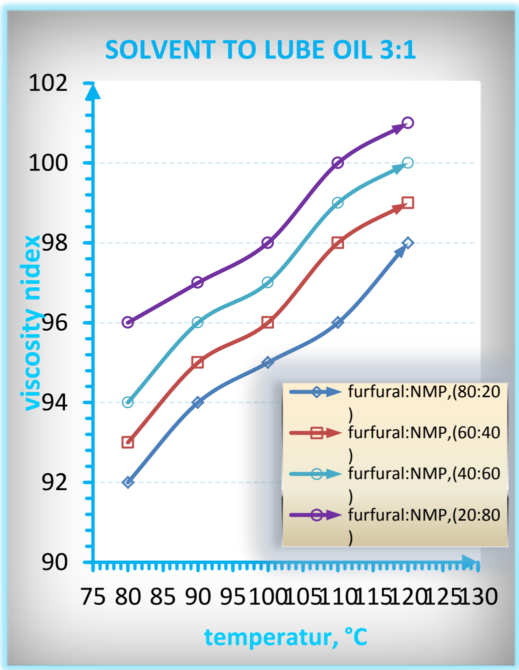


Fig. 10, Effect of extraction temperature on raffinate viscosity index at various solvent to co-solvent ratio (at solvent to lube oil ratio 3:1)

Effect of extraction temperature and furfural to co-solvent (NMP) ratio for stock 60 (SEA-30) on Raffinate Yield

Fig. (11) clearly indicate the effect of extraction temperature on raffinate yield at various solvent to oil ratio and different ratio of furfural and NMP solvent. This explains that yield percentage decreases as the extraction temperature increases. The increasing the value of co-solvent (NMP) to furfural, lead to decrease yield on raffinate phase, due to the aromatics substances dissolve in extraction phase. The effect of increasing solvent to oil ratio results in a deeper extraction this reduces the aromatics content of the raffinate and therefore decreases its raffinate yield. The overall effect of an increase in solvent to oil ratio will be a decrease in raffinate yield but also a decrease of the aromatic content of the raffinate and an improved raffinate quality in extraction process [17].

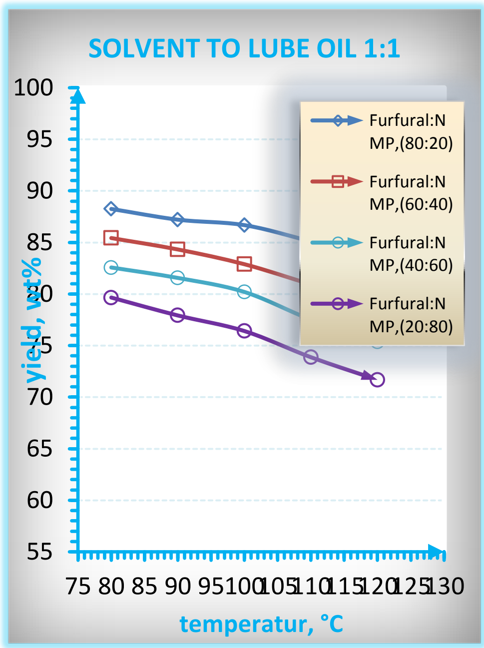


Fig. 11, Effect of extraction temperature on raffinate yield at various solvent to co-solvent ratio (at solvent to lube oil ratio 1:1)

Conclusions

1. Addition one of the two solvents (formamide and NMP) as a co-solvent can increase solvent power and selectivity of furfural toward aromatic compounds in extraction of aromatics from lube oil cut.
2. The effect of co-solvent to solvent ratio and the solvent to oil ratio is higher than the effect of the temperature on increasing the raffinate viscosity index and decreasing yield, viscosity, and refractive index.
3. Viscosity index of lube oil will increase, while viscosity and raffinate yield decreases when increasing extraction temperature, the solvent to oil ratio and co-solvent to furfural ratio.
4. From formamide co-solvent the best condition were extraction temperature at 90 °C, furfural to co-solvent ratio (60:40) and mixed-solvent to lube oil ratio (1.5:1) to get the best value of viscosity index 102, viscosity 3.01 sct and raffinate yield 69.23 wt. %.
5. The best condition for NMP co-solvent were 110 °C extraction temperature, (2:1) solvent to lube oil ratio and (60:40) furfural to co-solvent ratio, to produce lube oil with 96 viscosity index, 9.10 sct viscosity and 68.50 wt.% raffinate yield.

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