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RESEARCH ARTICLE

INVESTIGATING THE EFFECT OF NMP/PHENOL SOLVENT BLEND ON LUBRICATING OIL PROPERTIES WITHOUT/WITH USING SURFACTANT.

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Abstract

In this study, solvent extraction of lubricating oil to improve its viscosity index is investigated. A blend of phenol/NMP co-solvent was tested at different feed to solvent ratio 1:1, 1:1.5 and 1:2. Different percent of phenol (25%, 50% and 75%) in the co-solvent were studied. The conradson carbon residue, refractive index, viscosity at 40 ^oC and 100 ^oC, viscosity index and %yield of raffinate were measured. It was found that at constant extraction temperature of 65 ^oC and at constant mixing time of 15 minutes, best solvent blend was 25% phenol-75% NMP.
Moreover, the effect of extraction temperature on the quality of raffinate was investigated using 25% phenol-75% NMP solvent blend and feed to solvent ratio of 1:2. Extraction temperatures up to 112 ^oC were tested. An optimum temperature of 103 ^oC was reached after which a deterioration in the quality of the raffinate is observed. Using surfactant as an additive in small quantities (0.01, 0.02, 0.05 and 0.1 wt.%) in the presence of 25% phenol-75%NMP as a solvent was studied. Temperature was held constant at 103 ^oC and feed to solvent ratio of 1:2 was used. An improvement in the properties of the raffinate was observed

Introduction:-

The market size for lubricating oil in continuously expanding, in 2018 it reached 128.51 billion US Dollars [1]. Increasing demand on lubricating oil has reached 36.1 million metric tons in 2017 [2]. This increase in demand has driven more expansion in production and new technology adaptation to meet such demand while maintaining economic value. Lubricating oil is hydrocarbon-based oil consisting of paraffins, iso-parrafins, naphthenes and aromatics. Naphthenic based oil has low viscosity index and pour point while paraffinic based oil has high viscosity index and high pour point. After fractional distillation of crude oil, the resulting long residue is sent to vacuum distillation, where lubricating oil is separated into four main fractions; spindle distillate (SPD), medium and heavy neutral distillate (MND and HND) and bright stock (BS). The four fractions are different in their boiling temperature range and viscosity.

Following vacuum distillation, the lubricating oil is deasphalted to remove asphaltenes and resins, then solvent extraction is conducted to reduce the viscosity index, reduce the aromatic content and improve lubricating oil quality. Dewaxing of lubricating oil either by using solvent or through catalytic dewaxing is carried to remove the wax content in the lubricating oil. Normal paraffins have high viscosity index, in the same time, their presence in

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lubricating oil increases its pour point, hence, they have to be removed. The final step is to adjust the color and stability of lubricating oil by clay treatment or hydro-finishing [3].

In the late 1920s solvent extraction was introduced as a way to treat lubricating oil. Through this process undesired compounds in the lubricating oil is removed using solvents that have certain characteristics. Nowadays also, solvent extraction of lubricating oil is employed on a wide commercial scale. Tailored solvent that can remove all the undesired product has not been found yet, hence, analysis and studies are conducted to find the most suitable solvent or combination of solvent to reach the required lubricating oil specifications. Major solvent that are currently being used is NMP (N-methyl-2-pyrrolidone), furfural and phenol [3-5].

By using solvent extraction, the viscosity index of the lubricating oil will improve in addition to the oxidation resistance and color. Heavy aromatic compounds have low viscosity index, low lubricating ability and low oxidative and thermal stability. This means that lubricating oil containing high molecular weight aromatic compounds, will has less stable viscosity that will deteriorate with the change in temperature, hence, it will not fulfill its job as a lubricant. By extracting aromatic compounds from lubricating oil, the viscosity index will improve and the overall lubricating oil quality will be enhanced. Other properties will be affected by solvent extraction such as viscosity, refractive index, color and conradson carbon residue.

A good solvent will be highly selective to aromatic compounds and will possess good solvent power. In addition, it will have good stability and be easily recoverable. Different solvents and co-solvents where investigated in the literature. The effect of furfural as a solvent on a mixed base oil was investigated by [6-8]. Different extraction temperature and solvent to feed ratio were studied. An improvement in the oil viscosity index was observed with the increase in extraction temperature and solvent to oil ratio. A viscosity index of 115.6 was reached at 110 ^{o}C extraction temperature and at 4:1 solvent to oil ratio.

In addition, NMP and furfural were studied in [9] as a single solvent to extract the polyaromatics compounds from lubricating oil, up to 4:1 solvent to feed ratio was tested. A 107.82 viscosity index was obtained using NMP at 110 ^{o}C, while a viscosity index of 101 was reached when using furfural at the same temperature. This confirms the higher selectivity of NMP compared to furfural. In [10] the extraction of aromatic compounds from lubricating oil using co-solvents formamide/furfural and NMP/furfural were investigated. It was found that viscosity index value of 96 and 68.5% yield was achieved using 1:2 feed to NMP/furfural co-solvents ratio at 110 ^{o}C. While lower viscosity index of 102 and higher yield of 69.23% were obtained when using 1: 1.5 feed to formamide/furfural co-solvents ratio at extraction temperature of 90 ^{o}C.

According to [11-14] using surfactant in the solvent extraction process will increase the mass transfer coefficient while simultaneously decrease the interfacial area. Surfactant as an additive in solvent extraction of lubricating oil was studied in [15]. It was found that the addition of surfactant enhanced the extraction of polyaromatic compounds and increased solvent selectivity [16-17]. Furfural performance was improved by the addition of 0.01 to 0.1 wt.% sodium lauryl ether sulfate (SLES) at optimum extraction temperature of 70 ^{o}C and an increase in the yield was observed [16-17].

It is clear from the literature that NMP is highly selective but in the same time its operating cost is high compared to that of phenol. On the other hand, one of the drawbacks of using phenol as single solvent is its toxicity. Using phenol as co-solvent with NMP will reduce the toxicity of phenol in the solvent mixture, this has not been investigated in the literature before.

In this work a co-solvent containing different ratios of phenol/NMP are tested, in addition the feed to solvent ratio was investigated. The effect of such variation on refractive index, viscosity, viscosity index, conradson carbon and yield were studied. In addition, the optimum blend and the optimum extraction temperature were identified. Moreover, the effect of using surfactant, sodium lauryl sulfate (SLS) as an additive to NMP/Phenol solvent mixture was studied.

Materials and Methodology:-

Materials:

Lubricating oil sample of specification listed in **Table 1** was used for our study. Egyptian standards for lubricating oil were listed in **Table 1**, and checked to identify the off specifications lubricating oil properties. NMP of purity of

99% (Sigma-Aldrich) and Phenol of 99% purity (Sigma-Aldrich) were used. The specifications of NMP and Phenol are listed in **Table 2**. Sodium laureth sulfate (SLS) of purity 99% was also used.

Table 1: Specifications of untreated lubricating oil sample versus Egyptian lubricating oil standards.

Specifications	Units	Method	Egyptian Standard Limit	Lubricating Oil Sample
ASTM Color		D1500	**1.5 (Max.)**	**3.5**
Specific gravity @ 60/60 °C	g/cm^3	D4052	-	0.887
Viscosity @ 100 °C	cSt (mm^2/s)	D 445	4	5.2
Viscosity @ 40 °C		D 445	20	34.8
Viscosity index		D 2270	**95 (Min.)**	**67**
Boiling Point	°C	-	-	350 - 400
Flash point	°C	D 93	**149 (Min.)**	**155**
Pour point	°C	D 97	**-6 (Min.)**	**-6**
Refractive Index @ 20 °C	-	D1218	**-**	**1.4700**
Conradson Carbon	wt.%	D189	**0.015 (Max.)**	**0.038**
Ash content	wt.%	D 482	**0.01 (Max.)**	**0.01**
Acidity	mgKOH / g	D664	**0.05 (Max.)**	**0.04**
Sulfur content	wt.%	-	**0.068 (Max.)**	**0.062**

Table 2: Specifications of Phenol and NMP used in our experiments.

Specifications	Units	Phenol	NMP
Purity	%	99	99
ASTM Color	-	25	40
Specific gravity @ 15 °C	g/cm^3	1.08	1.027
Viscosity @ 25 °C	cP	1.58	1.65
Saybolt Viscosity @ 140 °C	cPoise	2.58	1.02
Refractive Index @20 °C	-	1.5425	1.4690
Boiling Point	°C	182	202
Flash Point	°C	79	95
Crystallization point	°C	40.6	
Freezing point	°C	40.9	-25
Water content	%	0.1	0.04
Critical temperature @ 1:1 °C		66.8[18]	232

Methodology:-

200 gm of lubricating oil sample is placed in a 500 ml round flask with condenser. The round flask was then placed into a water bath with a magnetic stirring system (ADVANTEC, USA). The prepared solvent mixture was added to the flask according to the specified feed to solvent ratio. Different percent of phenol (25%, 50% and 75%) in the solvent mixture was tested. Mixture was agitated using magnetic stirrer for 15 minutes, at the end of the experiments, mixture was placed in a separation funnel for 1 hr. Two layers were separated; the top raffinate layer and the bottom extract layer. The extract contains solvent with the dissolved aromatic compounds, while the raffinate contains lubricating oil after the extraction of aromatic compounds. The raffinate was separated and analyzed.

Experiments were conducted in 6 sets of experiments. In the first three sets of experiments, **Table 3,** extraction temperature of 65 °C, feed to solvent ratio of 1:1, 1:1.5 and 1:2 were used. The amount of phenol in the solvent blend was varied (0%, 25%, 50%, 75% and 100%). All other variables are kept constant. In the fourth and fifth set of experiments, see **Table 4,** percent of phenol in solvent was kept constant at 25% and 75% respectively. The feed to solvent ratio was kept at 1: 2 while the extraction temperature changed from 65 °C to 112°C. The effect of solvent ratio variation on 5 specifications; viscosity, viscosity index, conradson carbon residue, color, refractive index and yield were studies.

Refractive index was conducted according to ASTM D-1218 using Abbe Refractometer, VEE GEE Model C10 (Thomas scientific). Also, Kinematic viscosity was measured according to ASTM D445 at 100 ^{o}C and at 40 ^{o}C. Color was measured according to ASTM D-1500. Conradson carbon residue apparatus, Model 80030 (Koehler, USA) confirming with ASTM D-189 was used.

After identifing the optimum: feed to solvent ratio, percent of phenol in solvent and extraction temperature, different concentrations of sodium lauryl ether sulfate (0.01 , 0.02, 0.05 and 0.1 wt.%) were added to the mixture, see **Table 5** to study the effect of surfactant on the performance of the co-solvent under study.

Table 3: List of experiments variables studied (set 1, 2 and 3 of experiments) at constant extraction temperature of 65 ^{o}C and mixing time of 15 minutes.

Set 1 of Experiments					
Experiment No.	**# 1**	**# 2**	**# 3**	**# 4**	**# 5**
Feed to Solvent ratio	**1 : 1**				
% Phenol in solvent	0	25%	50%	75%	100%
% NMP in solvent	100%	75%	50%	25%	0
Set 2 of Experiments					
Experiment No.	**# 6**	**# 7**	**# 8**	**# 9**	**# 10**
Feed to Solvent ratio	**1 : 1.5**				
% Phenol in solvent	0	25%	50%	75%	100%
% NMP in solvent	100%	75%	50%	25%	0
Set 3 of Experiments					
Experiment No.	**# 11**	**# 12**	**# 13**	**# 14**	**# 15**
Feed to Solvent ratio	**1 : 2**				
% Phenol in solvent	0	25%	50%	75%	100%
% NMP in solvent	100%	75%	50%	25%	0

Table 4: List of experiments variables studied (set 4 and 5 of experiments) at constant feed to solvent ratio of 1:2 and constant mixing time of 15 minutes.

Set 4 of Experiments						
Experiment No.	**# 12**	**# 16**	**# 17**	**# 18**	**# 19**	**# 20**
% Phenol in solvent	25%					
% NMP in solvent	75%					
Temperature, ^{o}C	65	90	97	103	108	112
Set 5 of Experiments						
Experiment No.	**# 14**	**# 21**	**# 22**	**# 23**	**# 24**	**# 25**
% Phenol in solvent	75%					
% NMP in solvent	25%					
Temperature, ^{o}C	65	90	97	103	108	112

Table 5: List of experiments variables studied (set 6 of experiments) at different concentrations of surfactant SLS, constant feed to solvent ratio of 1:2, 103 ^{o}C extraction temperature and constant mixing time of 15 minutes.

Set 6 of Experiments					
Experiment No.	**# 18**	**# 26**	**# 27**	**# 28**	**# 29**
wt.% surfactant (SLS)	0	0.01	0.02	0.05	0.1
% Phenol in solvent blend	25%	25%	25%	25%	25%
% NMP in solvent blend	75%	75%	75%	75%	75%

Results and Discussion:-

Different experiments were conducted to test the effect of NMP/Phenol co-solvent without and with the present of SLS as an additive to the mixture. Results are listed in **Table 6**, a graphical representation of the data are shown in **Figure 1, 2** and **3** for comparison.

Conradson carbon residue is a measure of the amount of carbon deposits that can form during the combustion of lubricating oil. A higher value of conradson carbon residue indicate a low-quality lubricating oil. The Egyptian standards limit for Conradson carbon residue is 0.015 wt.%. According to **Table 6** and **Figure 1-a**, the amount of carbon residue in the raffinate decreases with the increase in the % phenol present in the NMP/phenol solvent mixture up to a value of 50% phenol. Any further increase in the amount of phenol present in the solvent mixture causes the conradson carbon residue of the raffinate to increase. The performance of the 25% phenol-75% NMP solvent mixture was even better than that of NMP alone, specially at high feed to solvent ratio of 1:2. The highest carbon residue value of 0.018 wt.% was obtained at 100% phenol while a low value of 0.009 wt.% of carbon residue was reached when using 25% pheno-75% NMP solvent mixture.

Same trend was observed with the refractive index measurement, see **Table 6** and **Figure 1-b**, a decrease in the refractive index value was observed with the increase in the feed to solvent ratio. Refractive index is an indication of the aromatic content in the raffinate, a higher refractive index value indicates high aromatic content and vice versa. The lowest refractive index value of 1.4540 was reached when using 25% phenol – 75% NMP solvent mixture and when using 100% NMP at a feed to solvent ratio of 1:2. In addition, an increase in viscosity measured at both 40^{o}C and 100^{o}C was observed with the increase in the % phenol present in solvent, see **Table 6**, **Figure 1-c** and **Figure 1-d**. Improvement in viscosity index was observed at low % of phenol, see **Table 6** and **Figure 1-e**. The highest viscosity index value of 104 and 102 was reached when using 100% NMP and 25% phenol-75% NMP solvent respectively at 1:2 feed to solvent ratio. Analysis of **Figure 1-a** to **Figure 1-e** confirm that 25%phenol-75%NMP has high selectivity to aromatic compounds comparable to that of 100% NMP solvent and better than that of other phenol solvent mixtures.

Highest raffinate yield of 90.8% was obtained by using 25% phenol-75% NMP solvent mixture at 1:1 feed to solvent ratio, while the lowest raffinate yield of 67% was obtained at 100% phenol with 1:2 feed to solvent ratio, see **Table 6** and **Figure 1-f**. Yield is affected by the feed to solvent ratio, the higher the feed to solvent ratio the lower the raffinate yield. It can be inferred from **Figure 1-f** that 25%phenol-75% NMP has moderate solvency power and high selectivity. In addition, the cost of this solvent mixture is lower than that of pure NMP solvent, while the phenol toxicity is diluted by the NMP solvent. Hence this mixture is considered an optimum mixture and will be further investigated at different extraction temperature.

Different extraction temperature was investigated using 25% phenol-75% NMP solvent mixture as an optimum solvent blend. In this study 75% phenol-25% NMP solvent was used for comparison. According to **Table 6** and **Figure 2-a**, it was found that carbon residue decreases with the increase in extraction temperature, up to 103 ^{o}C where a value of 0.004 wt.% of residue carbon is obtained. Any further increase in temperature above the 103^{o}C, causes the carbon residue in raffinate to increases. Same behavior was observed for raffinate refractive index measurements, with the increase in extraction temperature, see **Table 6** and **Figure 2-b**. A minimum value of 1.4538 for refractive index was obtained at extraction temperature of 103 ^{o}C by using 25% phenol-25% NMP solvent mixture. This trend in the refractive index and conradson carbon residue was confirmed by the viscosity and viscosity index measurements, **Table 6**, **Figure 2-c**, **Figure 2-d** and **Figure 2**. This shows an increase in selectivity till the optimum extraction temperature (near the critical temperature) after which loss of selectivity is observed.

Table 6: Properties of the raffinate analyzed after solvent extraction; Experiments set 1 to 6.

Results for Set 1 of Experiments						Lubricating Oil sample before treatment
Experiment No.	**# 1**	**# 2**	**# 3**	**# 4**	**# 5**	
Viscosity @ 100 ^{o}C	3.19	4.11	4.55	4.8	3.5	**5.2**
Viscosity @ 40 ^{o}C	13.80	21.05	25.50	28.30	16.58	**34.8**
Viscosity Index	90	90	85	83	79	**67**
Conradson Carbon	0.016	0.014	0.017	0.018	0.018	**0.038**
Color	2.5	2.5	2.5	3	3	**3.5**
Refractive Index	1.4590	1.4589	1.4604	1.4610	1.4600	**1.4700**
Yield %	90	90.8	87.5	85	77	**-**
Results for Set 2 of Experiments						**Lubricating Oil sample before treatment**
Experiment No.	**# 6**	**# 7**	**# 8**	**# 9**	**# 10**	
Viscosity @ 100 ^{o}C	3.22	3.69	3.66	3.71	3.28	**5.2**
Viscosity @ 40 ^{o}C	13.65	17.20	17.16	17.65	14.42	**34.8**

Viscosity Index	100	98	94	92	91	**67**
Conradson Carbon	0.013	0.009	0.0011	0.016	0.015	**0.038**
Color	2.0	2.0	2.0	2.5	2.5	**3.5**
Refractive Index	1.4565	1.4565	1.4570	1.4575	1.4570	**1.4700**
Yield %	85	85.8	83.8	82.5	75	**-**

Results for Set 3 of Experiments						**Lubricating Oil sample before treatment**
Experiment No.	**# 11**	**# 12**	**# 13**	**# 14**	**# 15**	
Viscosity @ 100 ^{o}C	3	3.4	3.45	3.44	3.05	**5.2**
Viscosity @ 40 ^{o}C	12.00	14.80	15.34	15.39	12.66	**34.8**
Viscosity Index	104	102	99	96	95	**67**
Conradson Carbon	0.010	0.009	0.009	0.013	0.012	**0.038**
Color	1.5	1.5	2.0	2.0	2.0	**3.5**
Refractive Index	1.4540	1.4540	1.4548	1.4547	1.4545	**1.4700**
Yield %	81	80	79	76	73	**-**

Results for Set 4 of Experiments							**Lubricating Oil sample before treatment**
Experiment No.	**# 12**	**# 16**	**# 17**	**# 18**	**# 19**	**# 20**	
Viscosity @ 100 ^{o}C	3.4	3.24	3.15	3.03	3.23	3.52	**5.2**
Viscosity @ 40 ^{o}C	14.8	13.58	12.85	11.98	13.38	15.63	**34.8**
Viscosity Index	102	104	107	109	107	103	**67**
Conradson Carbon	0.009	0.008	0.005	0.004	0.008	0.011	**0.038**
Refractive Index	1.4540	1.4538	1.4536	1.4535	1.4539	1.4546	**1.4700**
Yield %	80	73	71	69	65	62	**-**

Results for Set 5 of Experiments							**Lubricating Oil sample before treatment**
Experiment No.	**# 14**	**# 21**	**# 22**	**# 23**	**# 24**	**# 25**	
Viscosity @ 100 ^{o}C	3.44	3.32	3.10	2.91	3.04	3.19	**5.2**
Viscosity @ 40 ^{o}C	15.39	14.43	12.82	11.45	12.45	13.70	**34.8**
Viscosity Index	96	98	100	103	99	93	**67**
Conradson Carbon	0.013	0.01	0.009	0.008	0.011	0.015	**0.038**
Refractive Index	1.4547	1.4548	1.4544	1.4542	1.4546	1.4553	**1.4700**
Yield %	76	71	70	70	68	67	**-**

Results for Set 6 of Experiments						**Lubricating Oil sample before treatment**
Experiment No.	**# 18**	**# 26**	**# 27**	**# 28**	**# 29**	
Viscosity Index	109	111	113	113	114	**67**
Refractive index	1.4535	1.4533	1.4529	1.4527	1.4525	**1.4700**
Yield, %	69	72	74	77	80	**-**

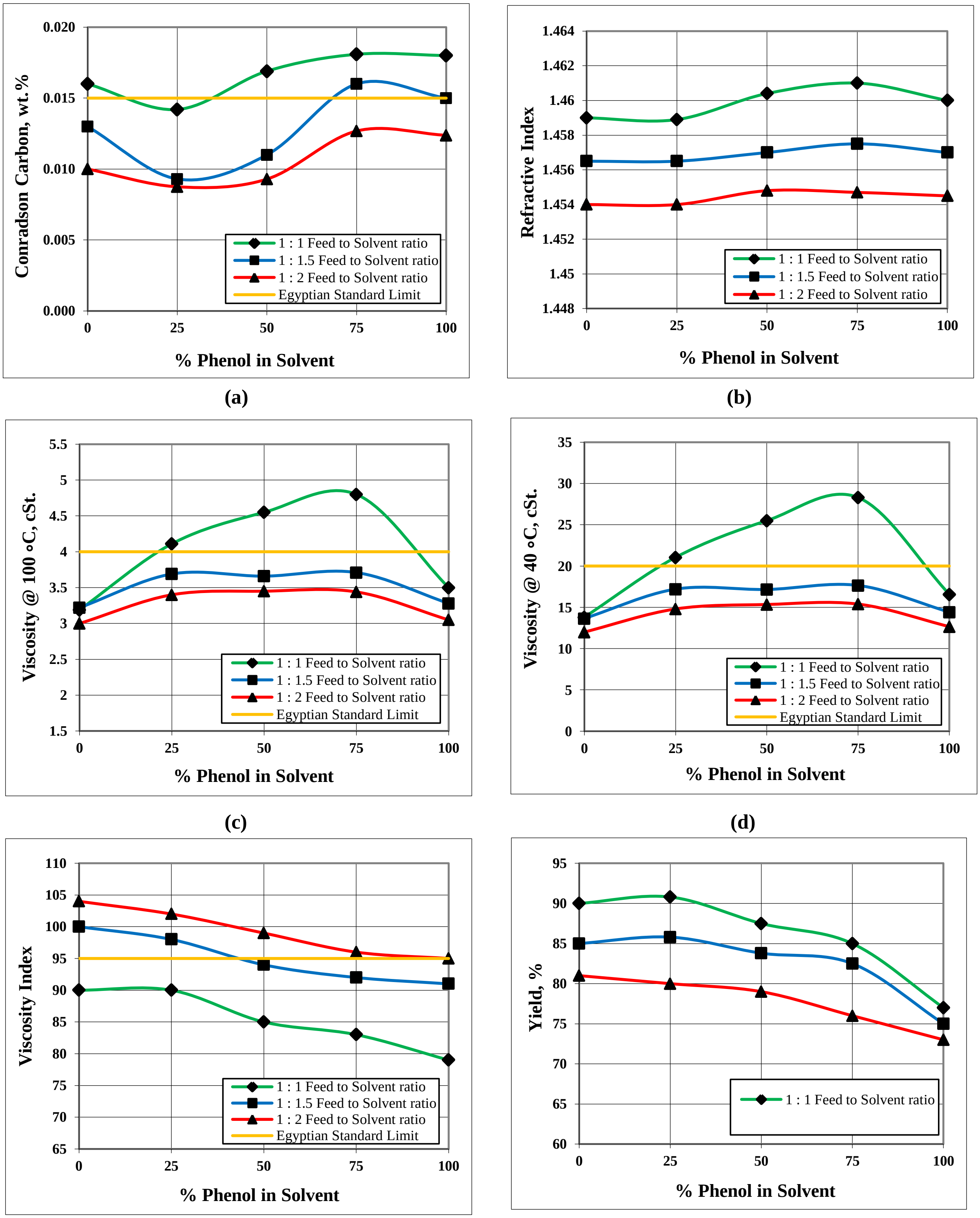
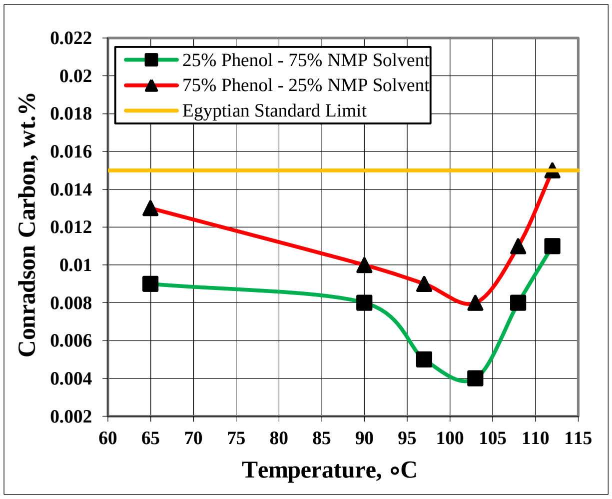
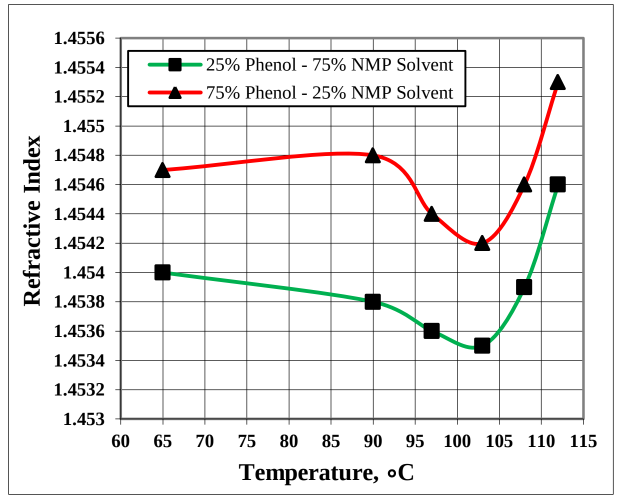


Figure 1: Properties of the raffinate after solvent extraction; experiments set 1 -3, solvent to feed ratio and % of phenol in solvent mixture were changed at constant extraction temperature of 65 °C and mixing time of 15 minutes; **(a)** Conradson Carbon Residue, **(b)** Refractive Index, **(c)** Viscosity measured at 100°C, **(d)** Viscosity measured at 40°C, **(e)** Viscosity Index and **(f)** % Yield.





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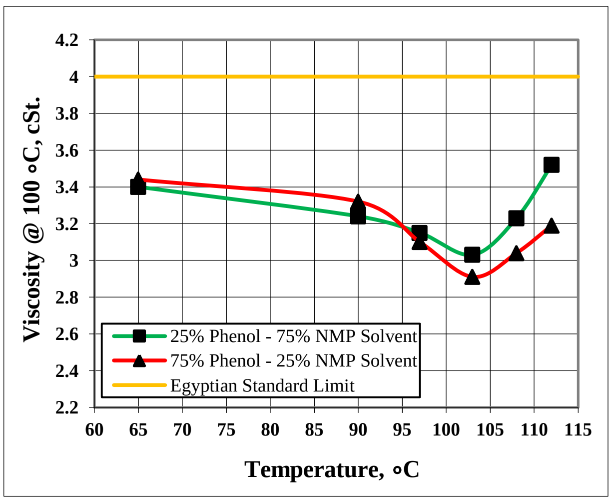
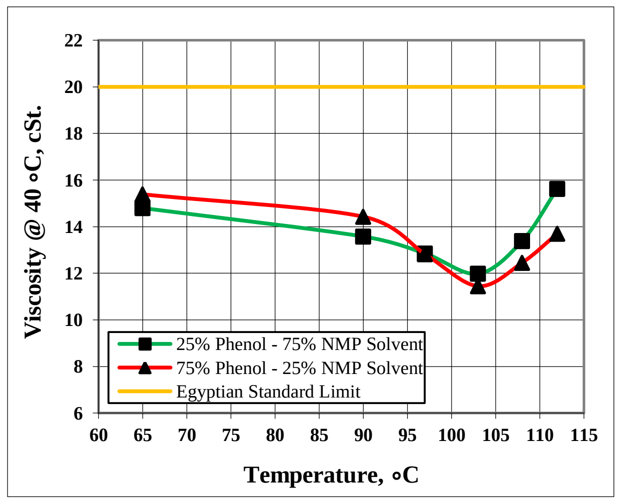
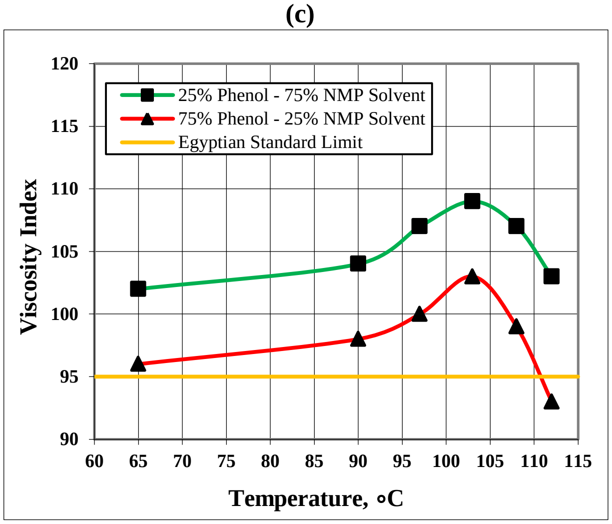
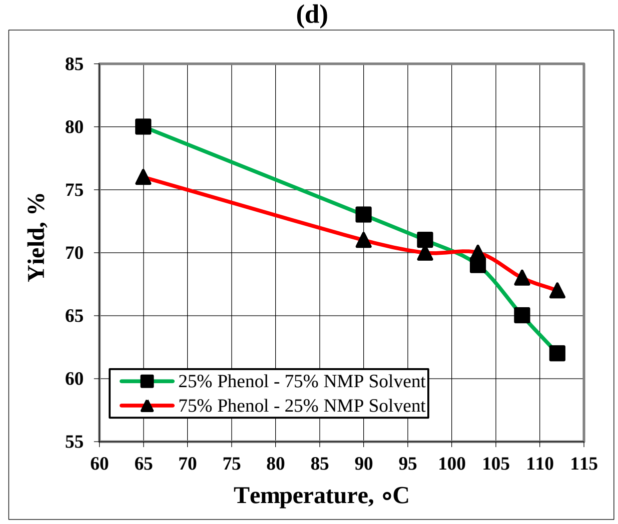




(b)

(c) (d)

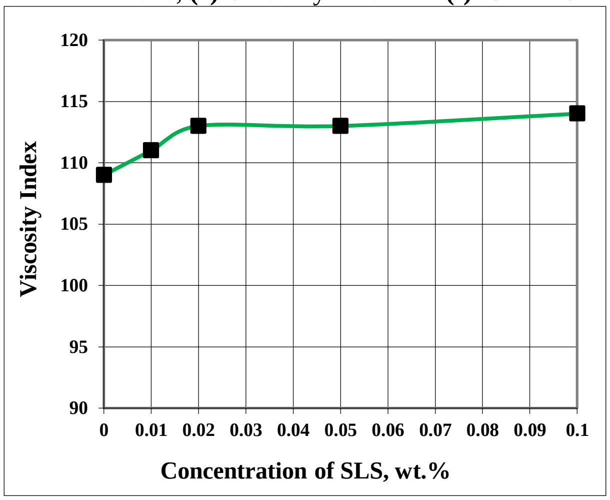




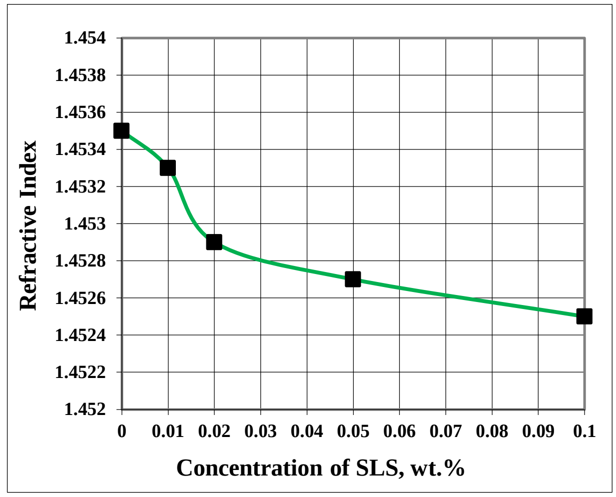
(e) (f)

Figure 2: Properties of the raffinate after solvent extraction; experiments set 4 and 5, the extraction temperature and % of phenol in solvent was changed while Feed to Solvent ratio of 1:2 and mixing time of 15 minutes were held

constant; **(a)** Conradson Carbon Residue, **(b)** Refractive Index, **(c)** Viscosity measured at 100^{o}C, **(d)** Viscosity measured at 40^{o}C, **(e)** Viscosity Index and **(f)** % Yield.



(a)



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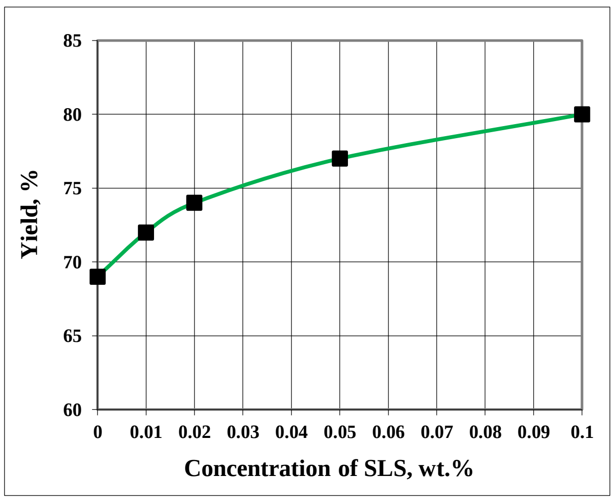

(c)

Figure 3: Properties of the raffinate after solvent extraction with 25% phenol-75% NMP and SLS as an additive; experiments set 6. The amount of SLS added to the solvent mixture was varied at constant extraction temperature of 103 ^{o}C, Feed to Solvent ratio of 1:2 and mixing time of 15 minutes; **(a)** Viscosity Index, **(b)** Refractive Index, **(c)** % Yield.

One the other hand, the amount of raffinate, % yield decreased continuously with the increase in extraction temperature, **Table 6** and **Figure 2-f**, a value to 69% was obtained at an optimum extraction temperature of 103 ^{o}C. This reflect an increase in the solvency power with the increase in temperature.

In all cases the 25% phenol-75% NMP solvent mixture performed better than the 75% phenol-25% NMP solvent mixture. By the analysis of the results, the optimum operating condition is at temperature of 103^{o}C, feed to solvent ratio of 1:2 and 25%phenol-75% NMP solvent mixture. This was used as a starting point for the investigation of the effect of surfactant as an additive. Different amount of SLS was used as listed in **Table 5**, the resulting raffinate was analyzed and data was listed in **Table 6**. As can be seen from **Figure 3,** the addition of surfactant to the solvent mixture increased the selectivity of solvent from aromatic compounds. This was confirmed from the increase in the viscosity index and the refractive index with the increase in the amount of SLS added, **Figure 3-a** and **Figure 3-b**. The highest increase was achieved by the addition of 0.02 wt.% of SLS surfactant. Further addition of surfactant increased the viscosity index and refractive index but to a lesser extent. Moreover, the raffinate yield increased with the continuous addition of SLS surfactant to the solvent, **Figure 3-c**. This means that the solvent power was reduced with the addition of surfactant which enhanced the phase separation while in the same time increased the solvent selectivity.

Conclusion:-

Solvent extraction of lubricating oil was investigated using NMP and Phenol solvent blend. The effect of surfactant as an additive to the mixture was also tested. The feed to solvent ratio, the percent of phenol in the solvent blend, the extraction temperature and the amount of SLS surfactant added was changed, the resulting raffinate was tested. It was found that:

- The higher the feed to solvent ratio the better the properties of the resulting raffinate.
- Increasing the percent of phenol in the solvent mixture at the same feed to solvent ratio and at constant temperature, decrease the quality of the lubricating oil.
- The best lubricating oil quality was obtained at 25% phenol-75% NMP even better than using 100% NMP solvent at 1:2 feed to solvent ratio.
- Increasing the extraction temperature improves the properties of lubricating oil till an optimum temperature of 103 ^{o}C is reached above which the quality of the oil become worse.
- The addition of surfactant in small quantities up to 0.02 wt.% improves the properties of the lubricating oil.
- The best combination of extraction condition to get high quality lubricating oil is to, use 25% phenol-75% NMP solvent blend in 1:2 feed to solvent ratio at 103 ^{o}C extraction temperature, with the option to add 0.02 wt.% of SLS surfactant.

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