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EXTRACTION OF AROMATIC HYDROCARBONS WITH MIXED N -FORMYLMORPHOLINE-ETHYLENE GLYCOL MONOETHERS

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ABSTRACT

The purpose of the present work was intended to determine the effect of addition of Ethylene glycol mono-methylether (EGMME) and Ethyleneglycol mono-ethylether (EGMEE) on the N-formylmorpholine (5 vol% water) in scope of improving its selectivity using hydrocarbon-mixed solvent and condensate-mixed solvent systems. It has been found that aqueous N-formylmorpholine – 6%Ethyleneglycol monomethylether mixture was the preferred mixture for the extraction of aromatic from condensate at 20°C, and solvent to feed ratio 3/1 by volume.

Keywords: Aromatic hydrocarbons . N-Formylmorpholine , Ethylene glycol

INTRODUCTION

Petroleum naphtha is by far the most popular feedstock for aromatics production [Saeid Mokhatab, (2010)] and [Broughton et al.,(1967)] Reformed naphtha, or reformate, accounts for 70 percent of total world BTX supply. Other feedstock sources, including condensate or natural gasoline, are attractive reforming feeds [Kumar et al., (2010)] and [Gibbs and Wochner,(2010)].

Condensate is a large source of potential feedstock for aromatics production. Gas condensate has a specific gravity ranging from 0.5 to 0.8 and may contain hydrogen sulfide, mercaptant, carbon dioxide, alkanes, naphthenes and aromatics [Robert, (2003) and [Krup Unde, (1993)].

Separation of aromatic hydrocarbons (benzene, toluene and xylene) from C_4 to C_{10} aliphatic hydrocarbons is an important step in the production of the fuels, as well as in the production of the basic chemical feedstock [Alan Kramer, (2011)]. Aromatics are premium blending stocks for motor fuels, because the high octane-number

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hydrocarbons in the gasoline boiling range, are primarily aromatic hydrocarbons, which come from catalytic reformates. Moreover, some petroleum processes, such as naphtha steam cracking, need the removal of aromatic hydrocarbons to purify products and lower operation costs [Ali, et al (2003)] and [Preusser and Dipl, (1987)].

The recovery of aromatic hydrocarbons from mixtures by liquid-liquid extraction with selective solvents was practiced on a commercial scale since several decades. However, new solvents are introduced and patented for this purpose. Thus it can be concluded that the ideal solvent has yet not been found [Mohamed et al., (1995)] and [Hartman et al., (2001)]. In addition to pure solvents, many organic solvent mixtures were proposed as selective solvents [Ragozkin et al., (1981)].

EXPERIMENTAL

The experimental work is directed to test and evaluate some blending agents using aqueous N-formylmorpholine as a basic solvent for the extraction of aromatics from aromatics—non aromatics mixture. Individual model hydrocarbon components system (toluene n-heptane) and stabilized condensate with physical properties, given in Table (1) were chosen for this study.

Properties	Stabilized - Condensate	Properties	Stabilized - Condensate
Operating pressure	10 bar	Density of liquid @ 60 F	0.7200g/ml
Operating temperature	155 °C	API	64.83
Flow rate	200 m ³ /h	Volume of liquid	610.781 cc
Phase	Gas dissolved + condensate	GOR	0.787m3/m3
Weight of gas dissolved	480.382 cc	Concentration of aromatics	16.86 vol.%
Weight of liquid	439.762 g	Mol Wt	95.926
Water content ppm	42.50	Mol Wt (measured)	98.021

Table (1): Physical properties of stabilized condensate.

Also the composition of stabilized condensate which given in Table (2) was chosen for this study.

Table (2) : Composition of Stabilized Condensate.

The pure	hydrocar	bon syste	m (toluene- n-hepta	ne) was	extracted	
Components	Mol%	Wt%	Components	Mol%	Wt%	
Ethane	0.082	0.026	Hexadecanes	0.194	0.457	
Propane	0.572	0.263	Heptadecanes	0.102	0.256	
i-Butane	0.972	0.589	Octadecanes	0.053	0.140	
n-Butane	6.752	4.091	Nonadecanes	0.039	0.111	
i-Pentane	8.315	6.254	Eicosanes	0.030	0.088	
n-Pentane	9.420	7.085	Eneicosanes	0.023	0.072	
Hexanes	18.155	16.312	Dodeicosanes	0.018	0.059	
Benzene	4.014	3.268	Tricosanes	0.012	0.041	
Heptanes	13.293	13.886	Tetracdsane	0.010	0.035	
Toluene	6.823	6.554	Pentacosanes	0.008	0.028	
Octanes	13.352	15.900	Hexacosanes	0.004	0.015	
Ethyl-benzene	0.592	0.655	Heptacosanes	0.003	0.011	
P,m-xylene	4.630	5.125	Octacosanes	0.002	0.009	
o-xylene	0.802	885.0	Nonacosanes	0.001	0.006	
Nonanes	4.571	6.111	Triacontanes	0.001	0.003	
Decanes	3.772	5.594	Entriacontanes	0.000	0.000	
Undecanes	1.528	2.490	Dotriacontanes	0.000	0.000	
dodecanes	0.709	1.259	Tritriacontanes	0.000	0.000	
Tridecanes	0.574	1.102	Tetratriacontanes	0.000	0.000	
Tetradecanes	0.338	0.699	Pentatriacontanes	0.000	0.000	
Pentadecanes	0.234	0.518	Hexatricontanes	0.000	0.000	
Total	100.00	100.00	Total	100.00	100.00	

The pure hydrocarbon system (toluene- n-heptane) was extracted with

commercial grade products of ethylene glycol mono-methyl ether (EGMME) and/or ethylene glycol mono-ethyl ether (EGMEE) in a mixture with aqueous Nformylmorpholine (NFM 5 vol.% water). The percentages of pure N-formylmorpholine and their mixtures are given in Table (3).

Percentage (vol %)
100%
96% + 4%
94% + 6%
92% + 8%
90% +10%
96% + 4%
94% + 6%
92% + 8%
90% +10%

Table (3): The Percentages of Pure Solvents and Solvents Modifiers Mixtures.

Extraction Techniques

A batch extractor of 250 ml capacity was used. The desired temperature was adjusted by circulation into the jacket water whose temperature is controlled with the help of a precised thermostatic bath. Predetermined quantities of the feed mixture were taken into the batch extractor and stirred thoroughly for 60 minutes which is a sufficient time, as determined in trial experiments, for establishment of equilibrium between phases, then allowed to settle for 60 minutes for complete separation. The two phases were taken out separately and measured.

The solvents were present only in small quantities in the raffinate phase, and so the raffinate phase was made solvent free by washing with distillate water (4-5 times) in a separating funnel. The hydrocarbons free solvent was then dried with anhydrous calcium chloride.

The quantitative removal of hydrocarbons from the extract phase was done using distillation under stripping with nitrogen.

The free solvent hydrocarbons (P_R and P_E) were analyzed by the fluorescent indicator adsorption (FIA) method (ASTM D1319-88), The components necessary for tracing the binodal curves and tie-lines to calculate the solvent power (K_A) and the selectivity (β) were determined by the commonly used formula [Alders, (1955)].

Aromatic recovery and raffinate yield were calculated considering material balance as follows:

Raffinate yield =
$$\frac{X_{PE} - X_F}{X_{PE} - X_{PR}}$$

Aromatic recovery = $= \frac{X_{PE} (X_F - X_{PR})}{X_F (X_{PE} - X_{PR})}$

Where,

 X_F = aromatic concentration in the feed,

 X_{PE} = aromatic concentration in the feed of solvent extraction and

 X_{PR} = aromatic concentration of raffinate free of solvent.

RESULTS AND DISCUSSION

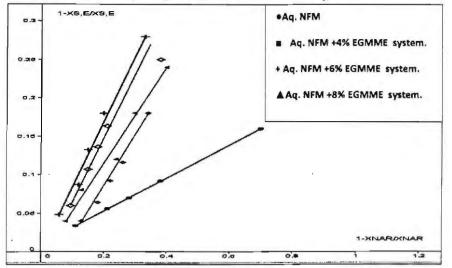
The purpose of the present work was intended to determine the effect of addition of Ethylene glycol mono-methylether (EGMME) and Ethyleneglycol mono-ethylether (EGMEE) on the N-formylmorpholine (+5 vol% water) in scope of improving its selectivity using hydrocarbon-mixed solvent and condensate-mixed solvent systems.

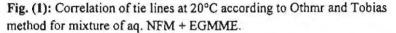
The consistency of the data obtained is tested according to the method of Tobias and Othmer The linear relationship plots related $\{(1-Xs,E)/Xs,E VS.(1-XNA,R) / XNA,R\}$ on log-log coordinates paper were presented for mixtures of aqueous N-formImorpholine (5 vol.% water)+ ethylene glycol monomethyl ether Fig.(1) and for mixtures of aqueous N-formyImorpholine (5 vol.% water)+ ethylene glycol monoethyl ether Fig. (2).

In an attempt to determine the influence of the modifier proportion on the the solubility characteristics of the basic solvent, we added ethylene glycol monomethyl ether or ethylene glycol monoethyl ether from 4 to 10 vol.% to aq. N-formyl morpholine (5 vol.% water) as given in Table (3). The variation in the values of the different solubility parameter as aresult of the combination of the principal solvent and the modifiers leads to a remarkable modification in the reciprocal solubility between the components of the systems (basic solvent, modifier, aromatic and non-aromatic). It is

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of interest, therefore, to study the selectivity, the distribution coefficient and the aromatic recovery for the pure hydrocarbons- mixed solvents systems.





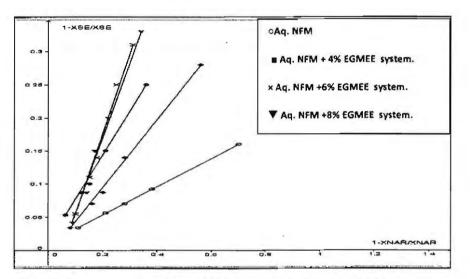


Fig. (2): Correlation of tie lines at 20°C according to Othmr and Tobias method for mixture of ag. NFM + EGMEE.

Taking into consideration the contradictory variations of the selectivity and the distributions coefficient along the heterogeneous regions of the systems, and the distribution coefficient is more important than the selectivity from the economic point of view so the selection of the optimum modifier according to these values is more difficult. Therefore, it should be much more beneficial to use the product of KA β values for such selection.

Results data as shown in Fig. (3 & 4), indicate that EGMME and EGMEE had the maximum value of the product of KA β , for the modifiers from (4-10 vol.%).

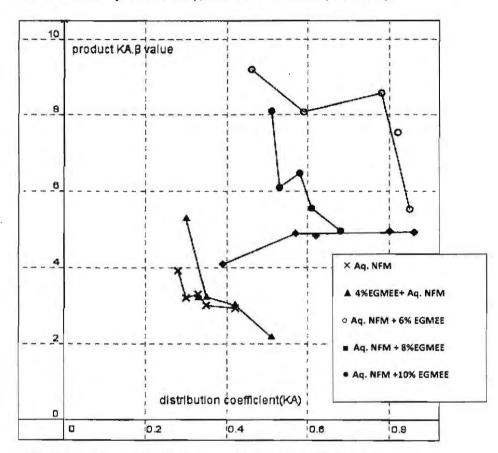


Fig. (3): variation of the distribution coefficient (KA) with the product (KA. β) value for toluene –n-heptane – aq.NFM + EGMEE mixtures at various composition.

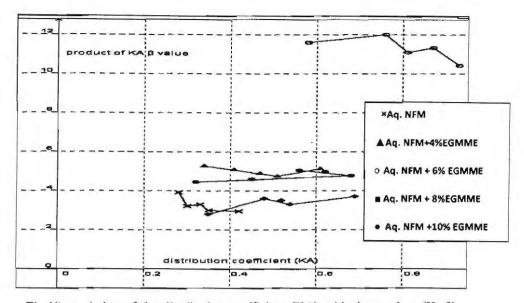


Fig.(4): variation of the distribution coefficient (KA) with the product (K_A,β) value for toluene -n-heptane -aq. NFM+EGMME mixtures at various compositions.

For the modifiers of ethylene glycol mono-methyl ether (EGMME), the values of the of KA β product decrease in the following manner:

aq.NFM+6%EGMME>aq.NFM+4%EGMME>aq.NFM+10%EGMME>aq.NFM+ 8%EGME > aq.NFM.

While for EGMEE modifiers the product of KA β value decreases in the following order:

aq.NFM+6%EGMEE>aq.NFM+10%EGMEE>aq.NFM+8%EGMEE>aq.NFM+

4%EG-MEE > aq.NFM.

It is clear that the mixture of aq.NFM +6% EGMME is more efficient in accordance of the product of KA β value than the mixture of aq.NFM+6%EGMEE.

In order to illustrate the results of the addition of the different types and percentages of the modifiers on the solubility characteristics of the principal solvent (aq. NFM) we used constant concentration of the aromatic in the feed 31 vol.% as shown in **Table (4)** and **Fig.(5)**.

Table (4): Equilibrium Composition of Toluene- n-heptane Solvent Systems at Temp.20°C, S/F=3/1 and XF=31 vol.%

Modifier Coac <u>n</u> . vol%	Aromatic Concentration			Raffinate Phase, vol.%			Extract Phase, vol.%			Aromatic Recovery	Yield vol.%		Distribution coefficient		Selectivity	Кар
	XF	XPR.	XPE	XS,R	XAR	XNA,R	XS,E	XAE	XNA E	RA	ημε	η _{PR}	KA	KNA	β	
Aq.NFM	31	17.5	64.4	12.2	15.4	72.4	91.6	5,4	3	59.8	28.7	71.3	0.35	0,041	8.5	3
Aq.NFM+ 4%EGMME	31	12.5	69	9	11.8	79.6	89.6	6	4.4	72.9	32.7	67.3	0.51	0.055	93	4,74
Aq.NFM+ 6%EGMME	31	11.5	62.5	6.2	10.8	83	85	9.4	5.6	77.1	38.2	61.8	0.87	0.067	13	11.3
Ag_NFM+ 8%EGMME	31	13.3	48.57	4.8	12.7	82.5	86	6.8	72	78.6	50.2	49.8	0.54	0.087	6.2	3.3
Aq.NFM+ 10%EGMME	31	14.5	58	10	13	77	86	8.1	5.9	70.97	38	62	0.62	0.077	8	4.96
Aq.NFM+ 4%EGMEE	31	18.6	62.8	4	17.9	78.1	88	7.5	4.5	56.9	28.2	71.8	0.42	0.058	7.2	3.02
Aq_NFM+ 6%EGMEE	31	12	56	6.5	11.22	82.28	83.5	9.2	7.3	78	43.2	56.8	0.82	0.089	9.2	7.54
Aq.NFM+ 8%EGMEE	31	13	48	8	11.96	80.04	86	9.6	10.4	79.6	51.4	48.6	0.80	0.13	6.2	4.96

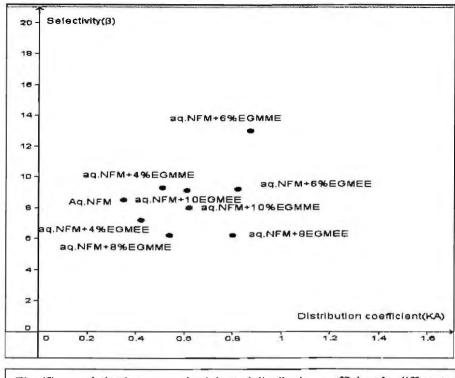


Fig. (5): correlation between selectivity and distribution coefficient for different solvent composition at XF=31 VOL%.

Results obtained as given in Table (4) show that:

-As the proportion of EGMME and EGMEE in aq.NFM increases, the distribution coefficient and the selectivity increase.Generally it has been observed that aq.NFM +6% EGMME mixtures indicate the maximum β , KA, KA β values compared with aq.NFM and aq.NFM +6% EGMEE.

The maximum purity that can be achieved from the extract and raffinate products in limited by the characteristics of the applied solvent and the feed.

The separation of the aromatic compounds from mixtures of aromatic and non aromatic compounds must be effected in such a manner so that the recovered aromatic compounds possess a high degree of purity. Thus the aromatic compounds showed their efficiency as intermediate in the preparation of various compounds.

On the other hand, a correlation between the raffinate yield and the aromatic recovery is shown in Fig. (6 & 7).

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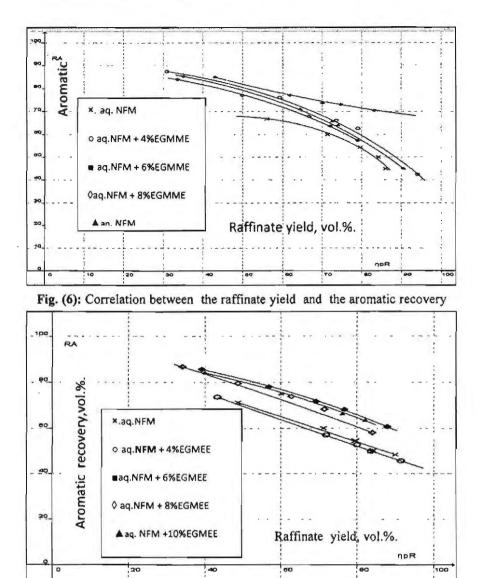
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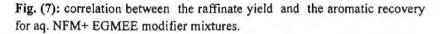
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It has been observed that for the same raffinate yield, the efficiency of the solvent for the aromatic recovery decreases in the following manner:

aq.NFM+6%EGMME>aq.NFM+4%EGMME>aq.NFM+10%EGMME>aq.NFM+8%E GME> aq.NFM, at the ethylene glycol mono-methyl ether concentrations (4-10 vol.%).

Also,aq.NFM+6%EGMEE>aq.NFM+10%EGMEE>aq.NFM+8%EGMEE>aq.NFM>aq .NFM+4% EGMEE, at the ethylene glycol mono-ethyl ether concentrations (4-10 vol.%).

Results as given in Fig. (8), which represent the relation between raffinate yield and solvent feed ratio indicate that (EGMME) has the maximum raffinate yield while EGMEE shows the minimum raffinate yield.

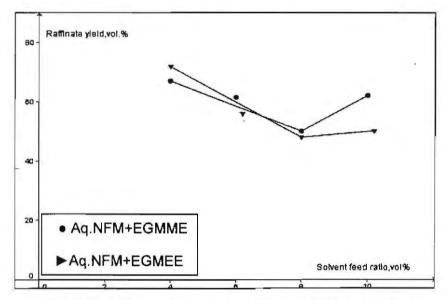


Fig. (8): Effect of the modifier content on Raffinate yield in extract for aq. NFM+EGMME and aq. NFM+EGMEE at XF=31vol.%.

It is clear that the aq.NFM +0%EGMME and aq.NFM+0%EGMEE show the maximum aromatic recovery. Accordingly it could be deduced that aq. NFM + 6% EGMME mixtures are the selected mixtures for the extraction of the aromatic hydrocarbons from the pure components(toluene-n- heptane) at the same set of conditions.

Condensate - mixed solvent systems

By using the mentioned selected mixtures from the pure hydrocarbon components treatment data, we carried out the extraction experiments with the following systems:

- Mixture of aq. NFM +6% EGMME - Condensate system

- Mixture of aq. NFM +6% EGMEE – Condensate system, In addition to the pure aq. NFM – Condensate system for comparison.

All the experiments are carried out at different solvent to feed stock ratios 1:1, 2:1, 3:1 and 5:1 and at temperature 20°C in order to determine the influence of solvent to feed stock ratios on the basic parameters of the aromatic hydrocarbons extraction with mixed solvents. The extraction results are presented in Tables (5-7).

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S/F ratio		Aromation	1939	Raffin	ate Phas	e, vol.%	Extra	et Phase	e, vol.%	Aromatic recovery vol.%	Raffinate yield yol.%	and the second sec	ibution ficient	Selectivity	КАВ	REN
X _F	X _{PR}	XPE	X _{S,R}	X _{A,R}	X _{NA,R}	X _{S,E}	X'AE	X _{NA,E}	RA	ղ _{PR}	KA	K _{NA}	β			
1:1	15.2	12.47	30.7	3.8	12	84.2	86	4.3	9.7	30.2	85	0.36	0.12	3	1.08	5.49
2:1	15.2	10.88	29.3	3.5	10.5	86	87.7	3.7	8.8	45.2	76.5	0.35	0.10	3.5	1.23	5.44
3:1	15.2	9.45	28	3	9.2	87.8	90	2.8	7.2	57.1	69	0.30	0.081	3.66	1.10	5.39
5:1	15.2	7.7	26.5	2.6	7.5	89.9	92	2.1	5.9	69.6	60.1	0.28	0.066	4.24	1.19	5.32

Table (5): Equilibrium Composition of Condensate + Aqueous (5% water) NFM (pure) at 20°C.

S/F ratio	I Ratinate Pr		ate Phas	e, vol.%	Ex	tract Phase, vol.%		Aromatic recovery vol.%	Raffinate yield voL%	Distribution coefficient		Selectivity	Кар	REN		
	X _F X _{PR} X _{PE} X _{S,R} X _{A,R} X _{NA,R} X _{S,E}	X _{a,e}	X _{NA,E}	RA	η _{PR}	KA	K _{NA}	β								
1:1	15.2	9	46	6.1	8.5	85.4	85	6.9	8.1	50.7	83.2	0.81	0.096	8.5	6.9	2.71
2:1	15.2	7.7	45.2	5.7	7.3	87	88	5.4	6.6	59.5	80	0.74	0.076	9.7	7.2	2.67
3:1	15.2	6.1	44.5	5	5.8	89.2	92	3.6	4.4	69.4	76.3	0.62	0.049	12.7	7.9	2.60
5:1	15.2	3.8	43	4.3	3.6	92.1	95	2.1	2.9	82.3	71	0,58	0.031	18.7	10.8	2.54

Table (6): Equilibrium Composition of Condensate - Aqueous (5% water) NFM (pure) + EGMME (94:6) system at 20 °C.

S/F ratio	Aromatic Concentration			Raffinate Phase, vol.%			Extract Phase, vol.%			Aroma tic recove ry vol.%	Raffin ate yield vol.%	ate Distribution yield coefficient			К _л β	REN
	X _F	X _{PR}	X _{PE}	X _{S,} R	X _{A,R}	X _{NA,R}	X _{S,E}	X _{A,E}	X _{NA}	R _A	η _{PR}	KA	KNA	β		
1:1	15.2	11.6	47.6	5.8	10.9	83.3	88	5.7	6.3	31.3	90	0.52	0.076	6.8	3.5	2.78
2:1	15.2	9.7	46.4	5.2	9.2	85.6	91	4.2	4.8	45.7	85	0.46	0.056	8.2	3.8	2.73
3:1	15.2	8.1	45.5	4.7 5	7.72	87.53	93	3.2	3.8	56.8	81	0.41	0.043	9.5	3.9	2.68
5:1	15.2	6.5	44.3	4.3	6.2	89.5	96	1.8	2.2	67.1	77	0.29	0.025	11.6	3.4	2.64

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The consistency of the data presented is tested by Othmr and Tobias method. The linear relationship of Othmr and Tobjas plots relate l-XS,E/XS,E Vs. l-XNA,R/XNA,R on log-log coordinates paper was obtained as shown in Fig. (9).

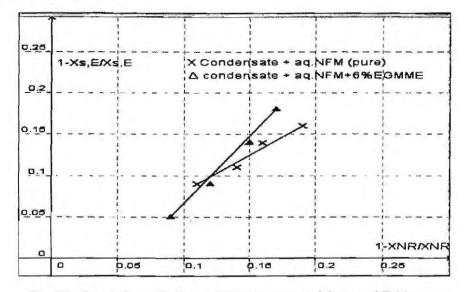


Fig. (9): Correlation of the lines at 20°C according to Othmar and Tobias method.

The refining effectiveness number (REN) which is considered to be a good measure of the attainable refining effect taking the change in the concentration of aromatics in the raffinate product as the refining performance parameter is given by:

100-1pr Loss in Yield Change in Property REN -XPR

The lower the value of REN, the higher is the efficiency of the solvent under the predetermined set of conditions. As listed in Tables(5-7) an aromatic extraction is directly related to the quantity of the solvent required for agiven feed stock. In recent years process improvements have ben aimed at reducing the solvent to feed ratio but the increase in the aromatic concentration in feed stock will tend to force the solvent to feed ratios back to higher values. The results given in Tables (5-7) and Fig. (10) indicate that the efficiency of aq.NFM + 6%EGMME mixture is more than the efficiency of aq. NFM + 6%EGMEE for the aromatic recovery of the condensate.

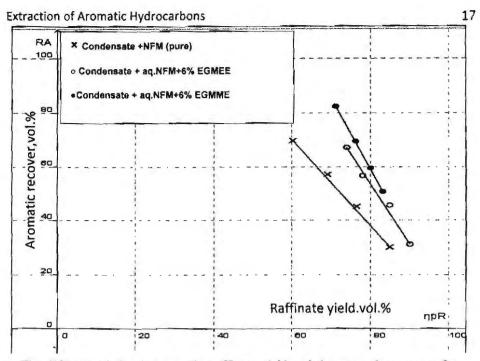


Fig. (10): correlation between the raffinate yield and the aromatic recovery for condensate + NMF (5% vol. water)pure and both condensate + NMF (5% vol. water)+6% EGMME and condensate + NMF (5% vol. water)+6% EGMEE solvent mixtures.

As a conclusion for the previously mentioned discussion the aq.NFM + 6%EGMME mixture was the preferred mixture for the extraction of aromatics from condensate at 20°C and a solvent to feed ratio 3/1 by volume.

CONCULSION

1. N-formylmorpholine (5 vol% water) + 6% ethylene glycol monomethyl ether(EGMME) and N-formylmorpholine (5 vol% water) + 6% Ethyleneglycol monoethylether(EGMEE) mixtures are more efficient than N-formylmorpholine (5 vol% water).

2. N-formylmorpholine (5 vol% water) + 6% ethylene glycol monomethyl ether mixture was the preferred mixture for the extraction of the aromatics from condensate at the predetermined set of conditions.

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الملقص العريى

استخلاص المواد الاروماتيه باستخدام خليط من مذيب الفورمايل مور فولين وايثيلين جليكول احادى الايثيل والميثيل إيثر

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يهدف هذا البحث الى تجربه وتقييم مذيبات ايثيلين جليكول الاحادى الميثيل إيثر وإيثيلين جليكول احادى الايثيل إيثر عند اضافتها الى المذيب الاساسى فورميل مورفولين المائى بهدف استخلاص المواد الاروماتيه من مخاليط اروماتيه وغير اروماتيه وذلك باستخدام الطولوين والهبتان والجازولين الناتج من تكثيف الغازات البتروليه.

وقد تم دراسه تأثير التركيزات المختلفة للمذيبات المستخدمة على درجة الانتقاء وقوه لذابة هذه المذيبات وقد وجد ان خليط المورفولين المانى مع ٦%ايثيلين جليكول احادى ميثيل إيثر وخليط الفورميل مورفولين المانى مع ٦%ايثيلين جليكول احادى إيثيل إيثر افضل من الفورميل مورفولين المانى وان افضل نسبة لاستخلاص المواد الارومانية من الجازولين الناتج من تكثيف الغازات البترولية هى ٦% ايثيلين جليكول احادى ميثيل إيثر مع الفورميل مورفولين المانى عند درجة حراره ٢٠ ونسبة ٢٢. المخلوط المذيبات الى الجازولين الناتج من تكثيف الغازات البترولية.

