DEWAXING OF DISTILLATE OIL FRACTION (400- 500 °C) USING UREA

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De-waxing of lubricating oil distillate (400-500 °C) by using urea was investigated in the present study. Lubricating oil distillate produced by vacuum distillation and refined by furfural extraction was taken from Al-Daura refinery. This oil distillate has a pour point of 34 °C. Two solvents were used to dilute the oil distillate, these are methyl isobutyl ketone and methylene chloride. The operating conditions of the urea adduct formation with n-paraffins in the presence of methyl isobutyl ketone were studied in details, these are solvent to oil volume ratio within the range of 0 to 2, mixer speed 0 to 2000 rpm, urea to wax weight ratio 0 to 6.3, time of adduction 0 to 71 min and temperature 30-70 °C). Pour point of de-waxed oil and yield of wax produced were determined to show the effect of these operating conditions. The most favorable operating conditions were solvent to oil volume ratio of 1, mixer speed of 1500 rpm, urea to wax weight ratio of 5, time of adduction of 13 min and temperature of 30-52 °C.

KEYWORDS
 dewaxing; urea dewaxing; lubricating oil production; wax; paraffin wax; n-paraffin production; extractive crystallization.

An important operation in the production of lubricating oils is the de-waxing of the corresponding petroleum fractions and residues, since it is only possible to manufacture lubricating and industrial oils with low pour points and viscosities, suitable for use at low temperatures, from cruds containing paraffin (1,2,3). Several processes used in the refinery fall under the classification of de-waxing processes; however, such processes must also be classified as wax production processes (4).

There are three types of processes in use for de-waxing. The first process is called solvent de-waxing, where the oil portion diluted by organic solvents. After refrigeration of the oil-solvent mixture, the solvent dissolves paraffin waxes only to a very slight extent, while they are good for dissolving the other components of lubricating oils. Wax is then separated by filtration and solvent is removed by distillation from de-waxed oil (1, 2, 3, 5, 6, 7, 8, 9, 10).

The second process is called catalytic de-waxing. This process is a selective hydrocracking process to crack the wax molecules to light hydrocarbons (3, 5, 11, 12, 13, 14, 15).

Third de-waxing process is called urea de-waxing. In this process, various straight-chain organic compounds and also slightly branched compounds are capable of forming complexes with urea, called adducts, which are crystalline at room temperature. These adduct crystals can be separated

easily by filtration. By raising the temperature and/or diluting with a suitable solvent, which may even be the purified oil, it is possible to resolve the adduct into its constituents (1, 2, 5, 8).

The discovery of adduct formation with urea is linked with Bengen's name (16, 17), who, after his first results, systematically studied numerous alcohols, aldehydies, ketones, acids and esters with respect to their capacity for adduct formation. He found that in addition to normal paraffinic hydrocarbons, these compounds also form urea adducts, if their carbon chain is not branched, but a straight alkyl chain (methyl groups at one end of the chain also undergo this formation of adducts).

There is a minimum chain length required for adduct formation. For example, in the n-alkane series, n-hexane forms an adduct, but n-pentane not, at room temperature (16). Pentane forms a complex at lower temperatures in liquid SO_2 as solvent (18). In the ketone series, acetone is already the first member of the series that capable of adduct formation (19). Although there appears to be no theoretical upper limit to the length of paraffin chain which will react, an apparent decline in activity has been observed for n-paraffin above n-C_{50} (16, 18).

Thiourea forms similar complexes with branched-chain and certain ring type compounds; however, thiourea is not so specific as urea in its action (17,18, 20, 21). The complexes of urea and thiourea have been termed adducts, and their formation, adduction. Bailey (20) adopted the term "extractive crystallization" to describe new crystallization separations based upon adduct formation (18, 20, 21).

Extractive crystallization, based on the selective reaction of urea with straight-chain hydrocarbons, results in the separation of n-paraffins or olefins from petroleum fractions ranging from gasoline to heavy lubricating oil. Potential applications of this process include pour point reduction of oils and fuels, production of pure n-paraffins, recovery of straight-chain olefins from cracked stocks, and octane number improvement. High purity n-alkanes is produced from this process (more than 95%)(16, 18, 22, 23).

Before adduct formation, the waxy oil is usually diluted with a suitable solvent in order to reduce its viscosity. Wetting agents (activators) are also used for adduct formation in order to eliminate the effect of the impurities interfering with adduct formation. Some solvents are used as diluents and also as activator in the same time. Urea reacted with n-paraffins is added either as solid or as solution. Water and alcohols of low carbon atoms "1 to 3", such as methanol and iso-propanol, are suitable solvents for urea (16, 24, 25, 26, 27, 28, 29, 30).

EXPERIMENTAL

Materials.

- **Feed Stock.**

The lubricating oil fraction used as feed stock in this investigation is the raffinate of furfural extraction of vacuum distillate produced in Al-Daura refinery. Boiling range of 400-500 °C and pour point of 34 °C.

- **Urea.**

High quality industrial solid urea (NH_2-CO-NH_2) with spherical shape contained 46% of nitrogen produced by General Company for Production of chemical Fertilizers was used for de-waxing the lubricating oil feed stock.

- **Deionized Water.**

Deionized water supplied from Al-Mansour plant was used as solvent for the urea.

- **Solvents.**

➢ **Methyl Iso-butyl Ketone (MIBK).**

Technical grade methyl iso-butyl ketone ($C_6H_{12}O$) with purity of 99% was used as solvent for the feed stock. Molecular weight of the solvent is 100.16 g/g mol. It has boiling point of 115-117 °C, refractive index (n_D^{20}) of 1.396, and density of 0.8 g/ml.

➢ **Methylene Chloride (Di-chloro methane).**

Methylene chloride (CH_2Cl_2) produced by AJAX chemicals was used as another solvent for the feed stock. It has molecular weight of 84.93 g/g mol, boiling point 39.8 °C ± 0.1 °C, refractive index (n_D^{20}) 1.424, and density 1.315 to 1.321 g/ml at 20 °C.

Procedure.

Fig. 1 shows a schematic diagram of Adduct formation unit used for de-waxing of furfural refined-lubricating oil feed stock by adduct formation of n-paraffin components with urea.
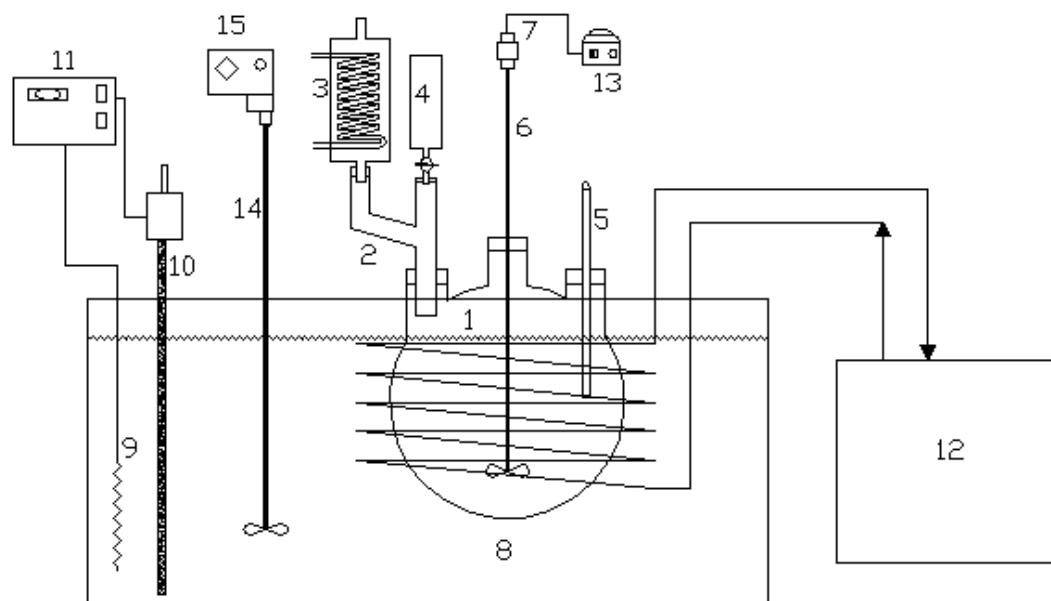
1. Reaction flask	6. Marine propeller	11. Relay
2. Adaptor	7. Electrical motor	12. Chiller
3. Condenser	8. Water bath	13. Voltage regulator
4. Dropping funnel	9. Electrical heater	14. Four flat blade pitched paddle
5. Thermometer	10. Thermocouple	15. Various speed electric motor

Fig1. Adduct formation unit.

- **De-waxing in the Presence of Methyl Iso-butyl Ketone Solvent.**

The required quantity of water is added into the reaction flask and is heated to the temperature of the water bath which is adjusted at a temperature higher than the initial reaction temperature (*e.g.* bath temperature of 60 °C and initial reaction temperature of 52 °C). Urea is then added to the flask

gradually under mixing to make an aqueous solution by dissolving the urea in the water. The quantity of urea added is equal to the saturation concentration of urea in water at an initial reaction temperature (*e.g.* 52 °C). Dissolving of urea in water is endothermic, so heat is needed to accomplish the dissolving, this heat is supplied from the water bath.

When the temperature of the aqueous solution reached 60 °C, the required amount of the feed stock and methyl isobutyl ketone solvent was added to the flask after being heated to a temperature of 60 °C.

Chilled water is pumped from the chiller into the cooling coil in order to reduce the temperature of the flask mixture from initial reaction temperature (*e.g.* 52 °C) to the final reaction temperature (*e.g.* 30 °C) at the desired time and mixer speed. Adduct crystals begin to appear at temperature of slightly below 52 °C, and proceed with temperature decreasing. When the mixture temperature reach the final reaction temperature (30 °C), the mixture is filtered off by using vacuum filtration to separate the adduct crystals. Then these crystals are washed twice by using for each wash 100-150 ml of cold methyl iso-butyl ketone solvent in 1 liter beaker and then re-filtered. This washing process removes the oil phase adhered at the surface of the crystals. Cold washing solvent (15 °C) is used in order to prevent adduct decomposition.

The filtrate from the adduct separation step and from the adduct washing step is mixed together and then placed into a separating funnel to separate the organic phase (oil and solvent) from the aqueous phase (urea solution). Organic phase is then washed with water to remove traces of aqueous solution which may contaminates the organic phase, and then separated by using separating funnel. Then the organic phase is introduced to the simple distillation to recover methyl iso-butyl ketone solvent from the de-waxed oil, which is then go to a pour point measurement after it is cooled and weighed.

The washed adduct crystals is then decomposed in 1 liter beaker by using water at a temperature of 70 °C. The quantity of water required for decomposition is equal to or higher than the quantity required for dissolving the urea presents in the adduct crystals. 300 ml of water was used in this study. The adduct is decomposed as the temperature is increased, and when the temperature reached 70 °C then all of the adduct crystals will be decomposed, where the urea dissolved in water while the wax is librated as a melt above the water layer. Wax is separated whilst hot from water by using a Pyrex separating funnel. Then the wax is weighed and stored in bottles.

- **De-waxing in the Presence of Methylene Chloride Solvent.**

The procedure for using methylene chloride as solvent is similar to that for methyl isobutyl ketone except in the method of solvent addition into the flask, and in the reaction temperature.

The temperature of the water bath is held at 40 °C. Lubricating oil feed stock is added to the flask at 40 °C. Then methylene chloride solvent is added to the flask at laboratory temperature by using the dropping funnel. Chilled water is passed through the condenser in order to reflux any solvent evaporated from the flask where the boiling point of the solvent is equal to about 40 °C. While the flask content is held at 40 °C, aqueous solution of urea heated to about 70 °C and has a saturation concentration at 62 °C is added to the flask. The temperature of mixture after this addition will reach about 50 °C and then reduced rapidly to 40 °C by the action of solvent reflux. The temperature of the bath is adjusted at 40 °C by using chilled water pass through the cooling coil and a heater immersed in the bath. The reaction is continued at temperature of 40 °C for 30 minutes, after that adduct is filtered, washed and decomposed. Oil phase separated from the aqueous phase is then washed with water, separated from water, and then supplied to simple distillation to remove solvent.

RESULTS & DISCUSSION.

- **De-waxing in the Presence of Methyl Iso-butyl ketone Solvent.**

➢ **Effect of Solvent to Oil Ratio.**

The effect of solvent to oil volume ratio upon the pour point of de-waxed oil and the yield of n-paraffin wax extracted from the feed stock was investigated as shown in **Fig. 2** and **Fig. 3** respectively.
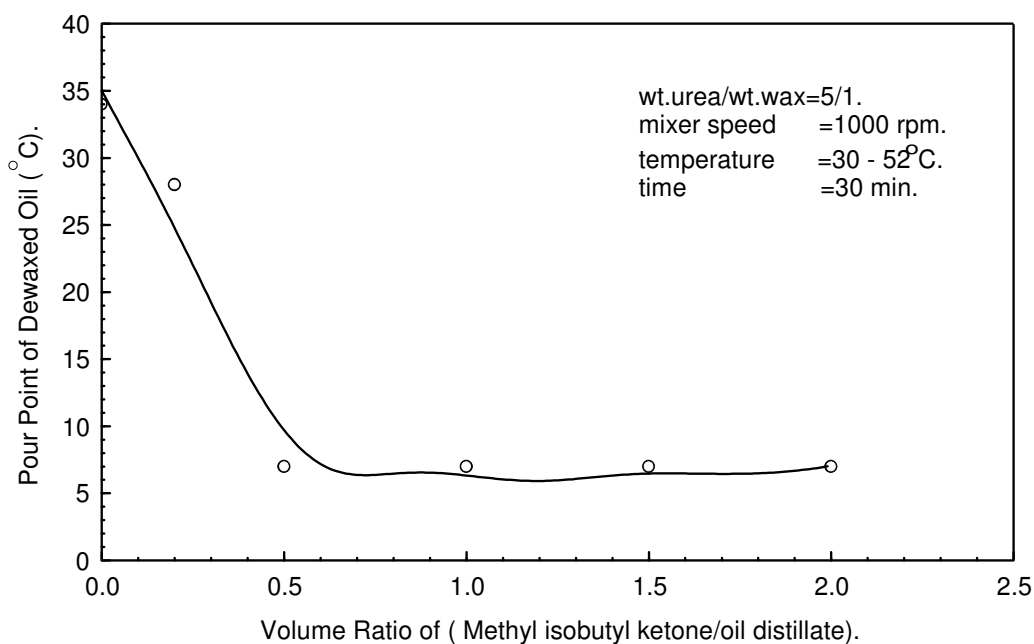

Fig 2. Effect of volume ratio of MIBK solvent to oil distillate on pour point of de-waxed oil.
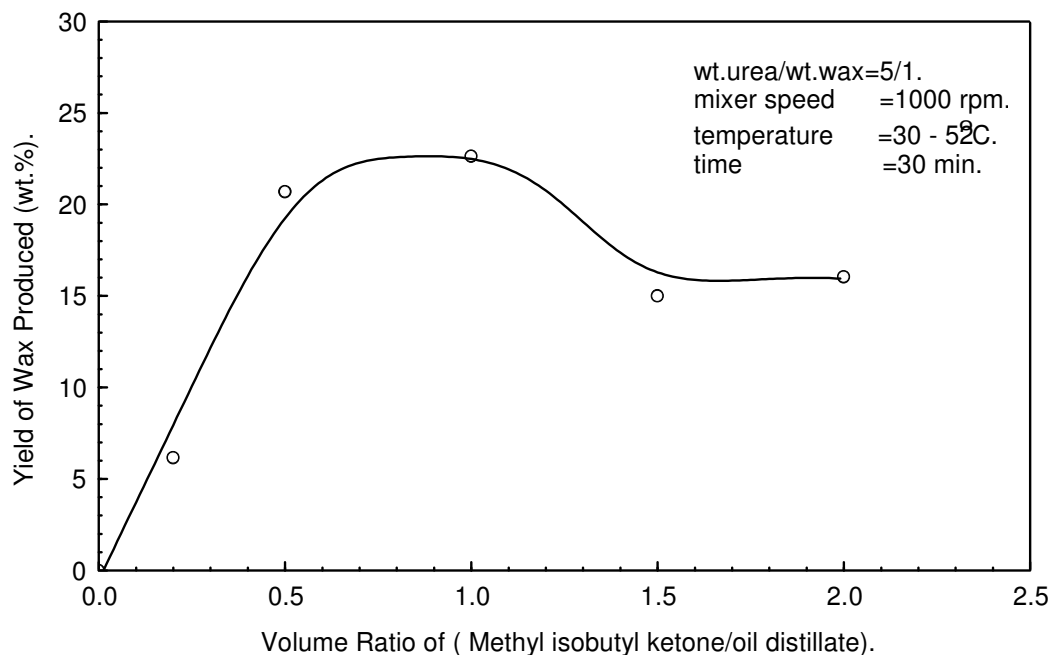
Fig 3. Effect of volume ratio of MIBK solvent to oil distillate on yield of wax produced.

Fig. 2 shows that the pour point of de-waxed oil decreases from 34 °C to 7 °C when the volume ratio of solvent to oil increases from 0 to 0.5 and higher. When the adduction reaction was done in the absence of solvent, the pour point of de-waxed oil remains unchanged (34 °C) and no wax was extracted. For volume ratio higher than 0.5, the pour point of de-waxed oil was being stable at 7 °C.

Fig. 3 shows that the yield of paraffin wax increases to 20.7 wt.% with increase in solvent to oil ratio up to 0.5, after which further additions of solvent up to a ratio of 1 increases the yield slightly to 22.63 wt.%. Further increase in volume ratio over 1 decreases the yield of paraffin wax to 15 wt.% at ratio of 1.5 and 16.04 wt.% at ratio of 2.

In the absence of methyl iso-butyl ketone no wax was extracted and the pour point was still at 34 °C because of the high viscosity of the oil, where good contact between oil and urea was not achieved. Using solvent dilution improves the rate of diffusion through heavy oil films. Also, a very slow rate of reaction is observed unless promoter solvents are used which these promoter solvents weaken the surface mono-layer between aqueous urea solution and oil interphase (18, 27). On the other hand, the rate of reaction of solid waxes may depend upon their rate of solution in a solvent, so an increase in solvent dilution improves this rate (18). Methyl iso-butyl ketone is a good diluent (solvent) for oil and a good promoter (activator) for adduct formation. The pour point of the de-waxed oil and yield of n-paraffin extracted were enhanced by using this solvent.

Methyl iso-butyl ketone and water are only slightly soluble in each other and that urea is soluble in water but only slightly so in methyl iso-butyl ketone. When hydrocarbons are added, methyl iso-butyl ketone is partitioned largely into the hydrocarbon phase; it serves as a diluent for the hydrocarbon and operates to reduce the reaction rate barrier which exists between the aqueous urea solution and the oil phase; the urea remains largely in the aqueous phase. Thus a rapid reaction can be effected by bringing together a saturated aqueous solution of urea and a solution of the hydrocarbon feed in methyl iso-butyl ketone (20).

The pour point of oil is unchanged (7 ℃) although the yield of paraffin wax extracted reduces from 22.63 wt.% at ratio of 1 to yield of 15-16 wt.% at ratios of 1.5-2.0. This is because the equilibrium of straight-chain hydrocarbon and urea with adduct is, of course, influenced by the presence of the ketone solvent, as also stated by Bailey and co-workers (20). Dilution of the adducting mixture with inert solvents has the effect of displacing equation 1 to the left and thus lowering the adduct yield (17, 21):

$$Urea(dissolved) + n - Paraffin(dissolved) \leftrightarrows Adduct(dissolved) \ \ ......(1)$$

n-Paraffins that diluted become under saturation by using high solvent ratios were those which have a relatively low chain length and with melting point around 7 ℃ (17), and would not be adductable with urea, while n-paraffins of higher chain length satisfies equation 1 and the reaction was displaced to the right towards adduct formation and not affected by dilution, so the yield of wax was reduced while the pour point of oil was not changed.

> **Effect of Mixer Speed.**

Fig. 4 and **Fig. 5** shows, respectively, the effect of mixer speed on the pour point of de-waxed oil and on the yield of n-paraffin wax produced.

The pour point of de-waxed oil reduces as the mixer speed increases up to 1000 rpm, after which the pour point remains unchanged (7 ℃). For example, the pour point of de-waxed oil was 31 ℃ for mixer speed of 300 rpm and 17 ℃ for mixer speed of 600 rpm. The yield of paraffin wax increases with increase in mixer speed up to 1000 rpm, after which the yield of wax is approximately unchanged.

The results of the effect of mixer speed on the pour point of de-waxed oil and on the yield of n-paraffin wax obtained are not surprising. Where, rapid stirring improves the rate of diffusion of aqueous urea through oil films and also improves mass transfer rates through aqueous urea-oil interphase (18).
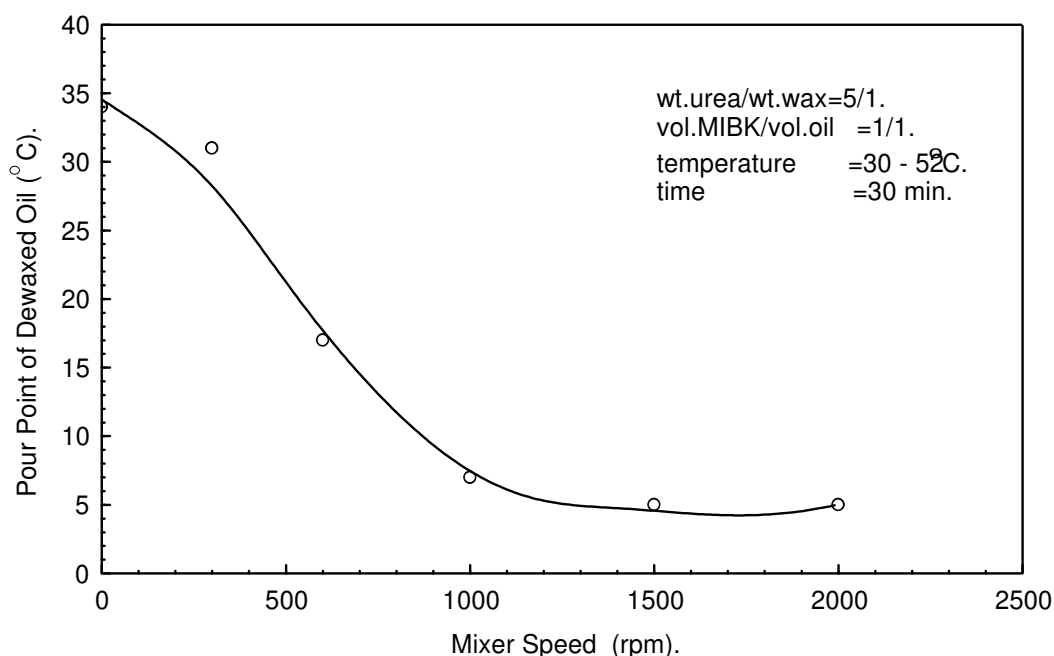

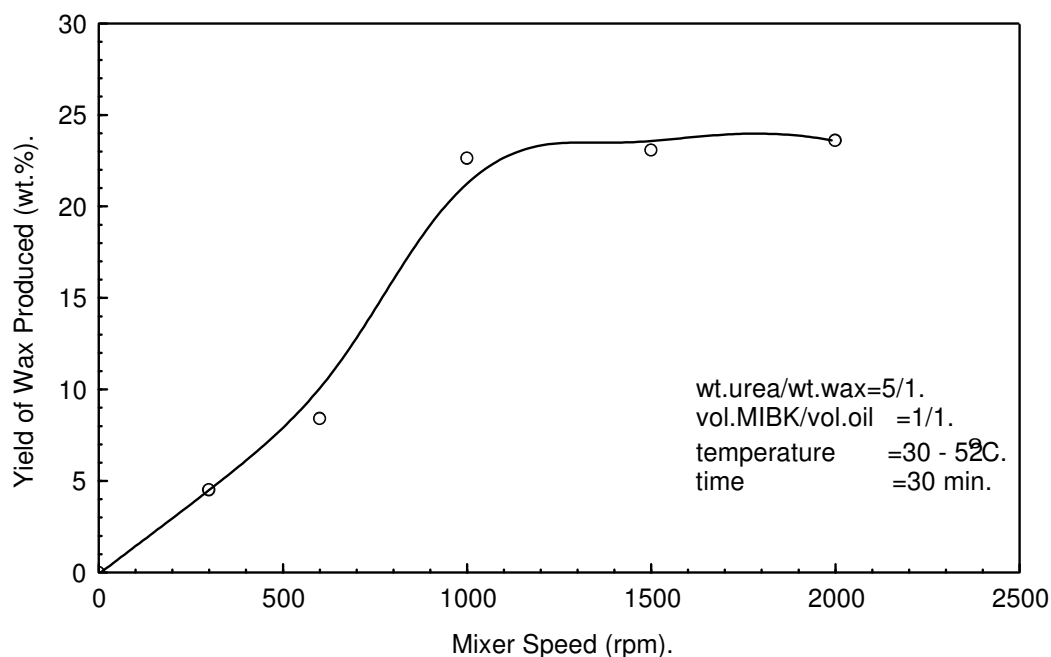**Fig 4**. Effect of mixer speed on pour point of de-waxed oil.

Fig 5. Effect of mixer speed on yield of wax produced.

> ### Effect of Quantity of Urea.

Fig. 6 shows the effect of the weight ratio of urea to wax on the pour point of de-waxed oil, while **Fig. 7** shows the effect of the urea to wax weight ratio on the yield of n-paraffin wax.

The pour point of de-waxed oil was reduced from 34 °C to 7 °C when the urea to wax weight ratio was increased from 0 to 1.27. Pour points higher than 7 °C were obtained for ratios lower than 1.27, while the pour points of de-waxed oil were unchanged (7 °C) for ratios higher than 1.27.

The yield of n-paraffin wax increases with increase in the weight ratio of urea to wax, and reaches to a maximum yield, around 23.3 wt.%, for weight ratio of 3.5. Using weight ratio higher than 3.5 remains the yield at its maximum value. This ratio (3.5) is the stoichiometric amount of urea required to form an adduct with n-paraffin and it is in agreement with the stoichiometric amount cited in the literature (21, 27, 25, 26).

On the other hand, when deficient urea was used for adduct formation (ratio of urea to wax of 1.27) the pour point did not changed from it's minimum value (7 °C), which obtained when stoichiometric amount(ratio of 3.5) of urea to wax was used, although the amount of n-paraffin wax crystallized was reduced. This is because of the selectivity of deficient urea to extract heaviest normal paraffins as mentioned by Marechal and Radzitzky (27). These heavy normal paraffins have a large effect on the pour point of de-waxed oil, because these paraffins have a high melting (freezing) points (17). So, n-paraffins that was not extracted when deficient urea was used, had a relatively small chain length with a melting points around 7 °C (17), and this was not effected on the pour point of the de-waxed oil and remained at 7 °C.
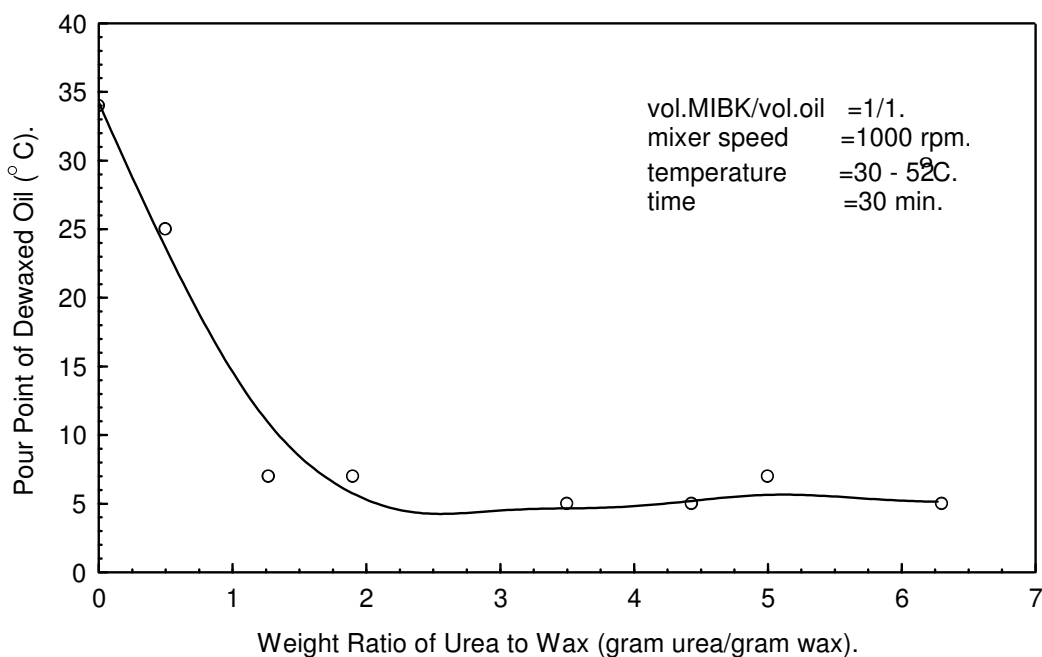
Fig6. Effect of weight ratio of urea to wax on pour point of de-waxed oil.
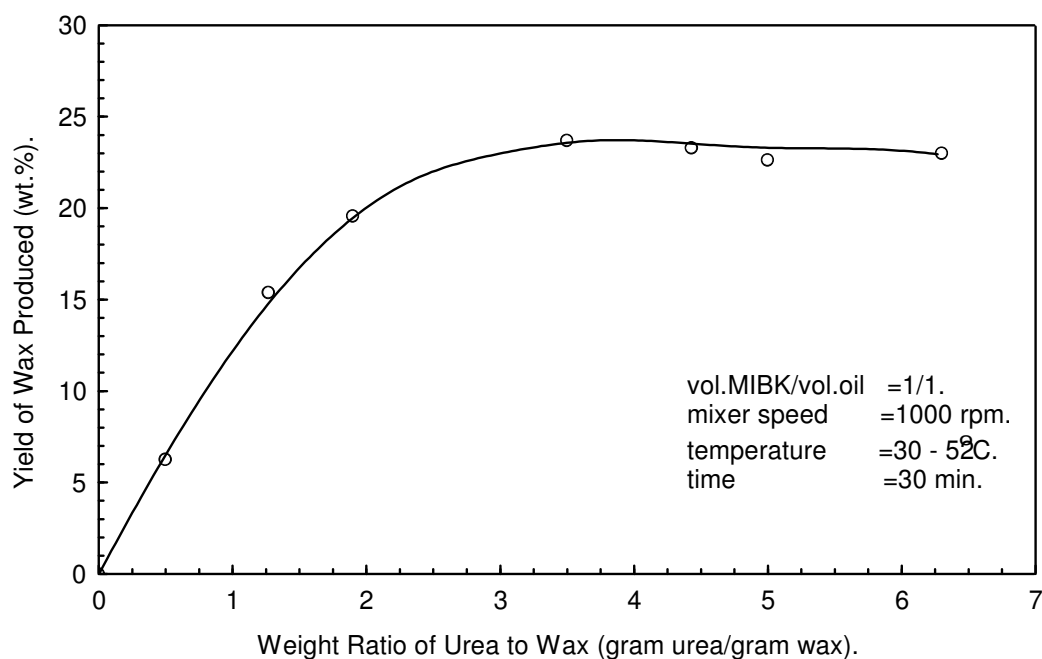

Fig 7. Effect of weight ratio of urea to wax on yield of wax produced.

➢ **Effect of Time of Reaction.**

The influence of time on the pour point of de-waxed oil and on the yield of n-paraffin wax are shown in **Fig. 8** and **Fig. 9** respectively, using solvent (MIBK) to oil volume ratio of 1, urea to wax weight ratio of 5, and temperature of adduction of 30-52 ºC.

Fig. 8 shows that for mixer speed of 300 rpm, there is only slightly effect of adduction time on the pour point. The pour point reduced from 34 ºC to 31 ºC after 30 min and remained at 31 ºC although the adduction time was increased to 71 min. For mixer speed of 1000 rpm, the pour point of de-waxed oil reduced from 34 ºC to 7 ºC when the time of adduction was held at 30 min, and the change of time from 30 to 60 min had no effect on the pour point. Using lower time than 30 min increases the pour point.

For mixer speed of 1500 rpm, the pour point of de-waxed oil reduces from 34 ºC to 5 ºC at time of 13 min only, and it is unchanged (5 ºC) for time higher than 13 min.It is also clearly shown from **Fig. 8** that the time required for adduct formation reduces as the speed of mixer increases, for obtaining the same pour point.

The effect of time on the yield of n-paraffin wax is shown in **Fig. 9**. Maximum yield around 4.5 wt.% of n-paraffin wax was obtained for adduction time equals to or higher than 30 min when 300 rpm of mixer speed was used. For mixer speed 1000 rpm, the time required to reach maximum yield (more than 22%) was 30 min, while for mixer speed 1500 rpm the time reduces to 13 min.
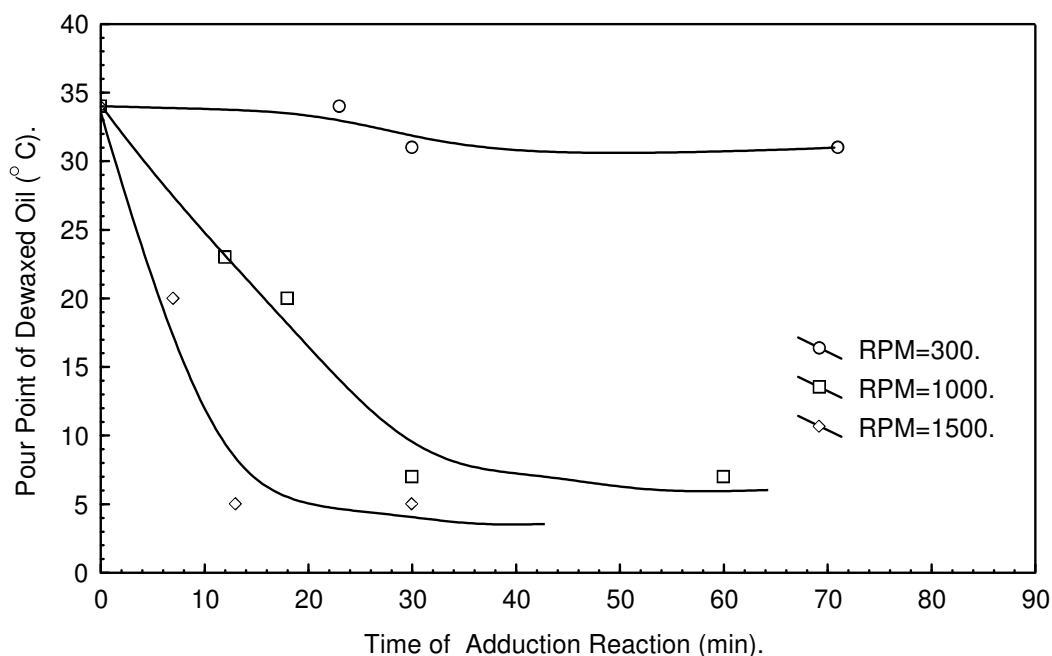

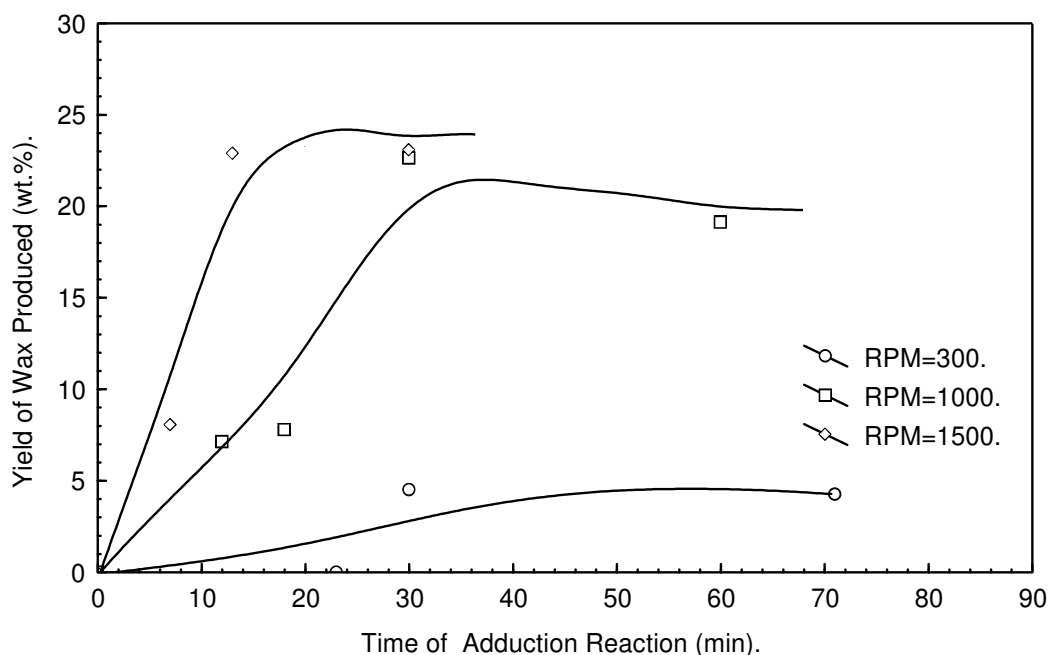Fig 8. Effect of time of adduct formation on pour point of de-waxed oil.

Fig9. Effect of time of adduct formation on yield of wax produced.

➤ **Effect of Adduction Temperature.**

Four experiments were conducted to study the effect of cooling temperature on adduct formation in the adduction reaction. The results are shown in **Table 1**.

Table 1. Effect of cooling temperature on adduct formation.
Wt. ratio (urea/wax) = 5; vol. ratio (MIBK/oil) = 1; mixer speed = 1500 rpm.

Exp. No.	Cooling temp., ℃	Time, min	Yield, wt.%	Pour point, ℃
1	52-30	13-30	22.90	5
2	55-40	20	23.86	4
3	62-40	20	21.50	7
4	70-40	20	16.58	7

This table shows that the yield of n-paraffin wax reduces as the temperature of the reaction increases, while the pour point of the de-waxed oil is approximately the same. This is because the effect of temperature on the equilibrium of urea and n-paraffin with adduct, where at high temperatures the equilibrium is less favorable and decomposition of adduct crystals may occur (16, 19, 28). At high temperatures, the long chain n-paraffins (which have relatively high melting points) formulate more stable adducts than those formulated from short chain n-paraffins (which have relatively low melting points). So, short chain n-paraffins will not adductable with urea at higher temperatures reducing the yield of n-paraffin wax, while the pour point will approximately the same because of low melting point of these n-paraffins (17).

- **De-waxing in the Presence of Methylene Chloride Solvent.**

The effect of volume ratio of methylene chloride to lubricating oil distillate on the pour point of de-waxed oil and on the yield of n-paraffin wax are shown in **Fig. 10** and **Fig. 11** respectively. Similar effect of volume ratio for this solvent to that for methyl iso-butyl ketone was gained from these figures.
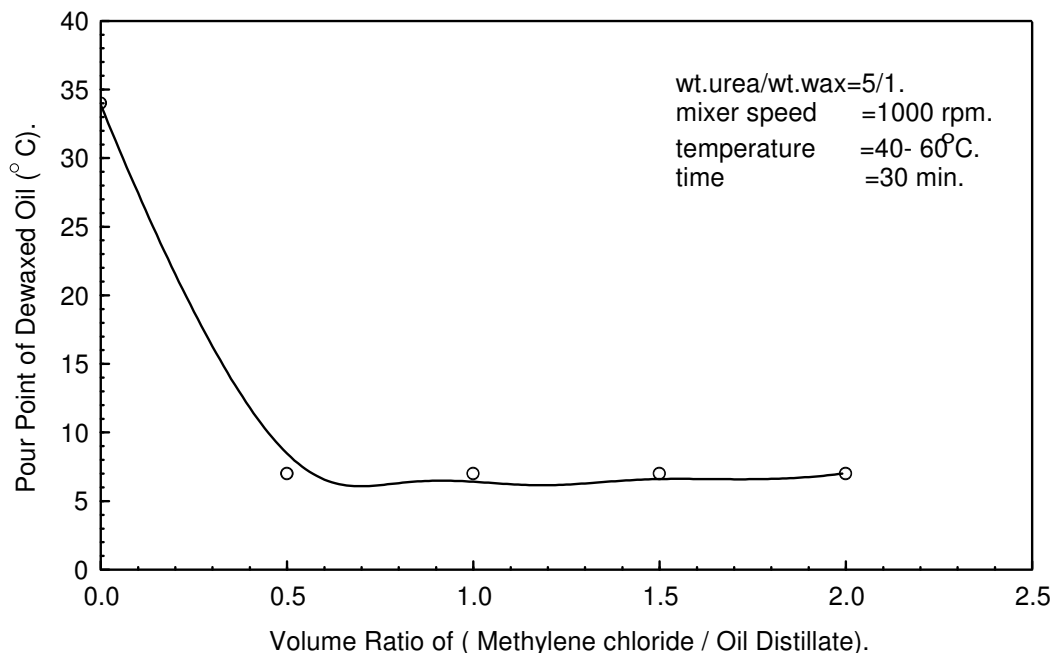
Fig 10. Effect of volume ratio of methylene chloride solvent to oil distillate on pour point of de-waxed oil.
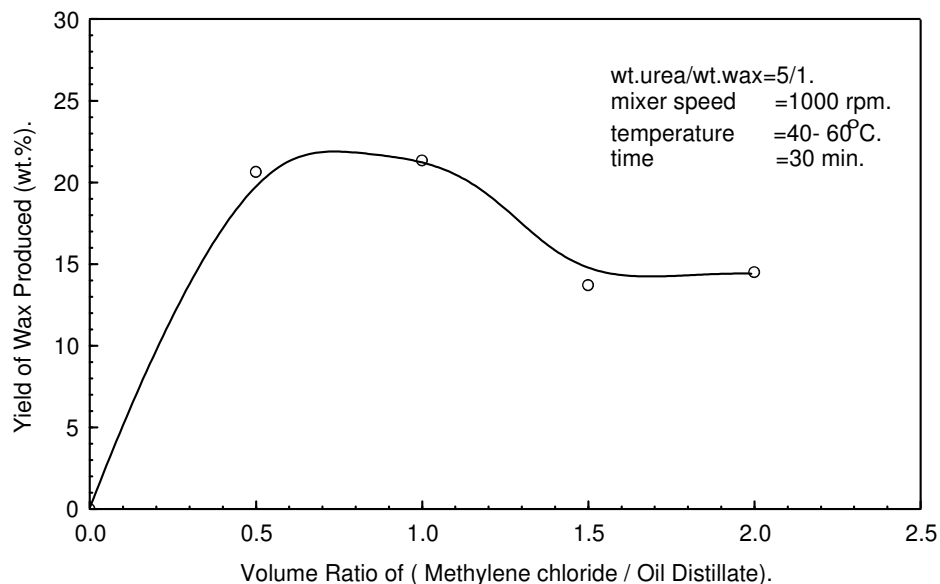
Fig 11. Effect of volume ratio of methylene chloride solvent to oil distillate on yield of wax produced.

CONCLUSIONS.

Urea de-waxing is an effective method for lowering the pour point of lubricating oil distillate refined by furfural and has a boiling point range of 400 to 500 ºC. The pour point of the feed stock was lowered from 34 ºC to about 5 ºC with a maximum yield of n-paraffin wax of about 22.9% when methyl iso-butyl ketone solvent was used. The most favorable operating conditions were solvent to oil volume ratio of 1, mixer speed of 1500 rpm, urea to wax weight ratio of 5, time of adduction of 13 min and temperature of 30-52 C. When methylene chloride is used instead of methyl iso-butyl ketone then pour point of de-waxed oil and the yield of wax are same as MIBK.

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الخلاصة

يتناول هذا البحث عملية إزالة الشمع من مقطر زيت التزييت باستخدام اليوريا. مقطر زيت التزييت الذي تم التعامل معه في هذا البحث مأخوذ من مصفى الدورة والمنتج بواسطة التقطير التجزيئي تحت الضــغط المخلخــل (vacuum distillation) والمعالج بطريقة الاستخلاص بالفورفورال(furfural extraction). هذا المقطر الزيتي له حدود درجة غليان بين 400– 500 ْم ونقطة انسكاب 34 ْم. تم استخدام مذيبين لغــرض تخفيــف مقطــر زيــت التزييــت همــا مثيــل ايزوبيوتيــل كيتــون (methyl isobutyl ketone) وميثيلين كلورايد (methylene chloride). الظروف التشغيلية لتفاعل اليوريــا مـــع الشـــمع البارافيني المتواجد في مقطر زيت التزييت بوجود مذيب مثيل ايزوبيوتيل كيتون (MIBK) تم دراستها بالتفصيل, وهــي نســبة حجم المذيب الى حجم الزيت ضمن حدود صفر– 2 ، سرعة الخلاط ضمن حدود صفر– 2000 دورة/دقيقة، نسبة وزن اليوريا إلى وزن الشمع الموجود في الزيت ضمن حدود صفر– 6.3 ، وزمن التبريد (التفاعل) ضمن حدود صفر– 71 دقيقة, ودرجـــة الحرارة ضمن حدود 30– 70 ْم. درجة الانسكاب للزيت (pour point) الناتج من عملية إزالة الشمع تم قياسها وكذلك النتــاج (yield) للشمع المستخلص تم حسابه لغرض بيان تأثير الظروف التشغيلية آنفة الذكر. أفضل ظروف تشغيلية تم التوصل إليهــا هي نسبة حجم المذيب الى حجم الزيت بمقدار 1, سرعة الخلاط بمقدار 1500 دورة/دقيقة , نسبة وزن اليوريا إلـــى وزن الشـــمع الموجود في الزيت بمقدار 5 , وزمن التبريد (التفاعل) بمقدار 13 دقيقة , ودرجة الحرارة بمقدار 30– 52 ْم .