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Evaluation of various techniques for separation of non-polar modifier concentrates from petroleum waxy by-products

Fathi S. Soliman ^{a,*}, Magdy T. Zaky ^a, Amal S. Farag ^a, Nermen H. Mohamed ^a, Lamia S. Mohamed ^a, S. Hanafi ^b

^a Egyptian Petroleum Research Institute, Nasr City, 11727 Cairo, Egypt

^b Physical Chemistry Department, Faculty of Science, Ain Shams University, Egypt

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KEYWORDS

Slack wax; Multistage fractional crystallization; Liquid–solid chromatography; Urea adduction technique; Non-polar modifiers **Abstract** Two petroleum waxy by-products (light and middle slack wax crudes) were evaluated for separation of non-polar modifiers by using different techniques. The results showed that, the light slack wax is selected as a suitable wax for separation of n-alkanes with even number of carbon atoms ranging from C_{20} to C_{26} for their high n-paraffin contents and can be used as non-polar structural modifiers. Different separation techniques; multistage fractional crystallization and liquid–solid chromatography; followed by the urea adduction technique have been used to separate non-polar modifier concentrates from the light slack wax crude. The light slack wax, its saturate components, the hard wax fractions isolated from light slack wax by the multistage fractional crystallization echnique and their adducts were analyzed by GC to characterize and compare the produced components. The resulting data reveal that, the adducts of light slack wax and its saturate components; can be used as non-polar modifier concentrates of low carbon atoms ($C_{20} + C_{22}$). From an economic point of view, the light slack wax adduct is selected as a non-polar modifier concentrate whereas, the separation step can be neglected to save energy. Meanwhile, the adduct of the hard wax isolated at 30 °C can be used as the preferable non-polar modifier concentrate of the high carbon number atoms ($C_{24} + C_{26}$).

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1. Introduction

Commercial processes for dewaxing and deoiling of residual feedstocks are complex and laborious. The greatest difficulties

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are related to the stage of filtering slurries of solid hydrocarbons that tend to form an inter-crystalline structure. Improving the filtration rate for obtaining solid hydrocarbons, use was made of various additives that have a modifying effect on the crystal structure of solid hydrocarbons. The modifiers offer a means for a considerable improvement in the basic indices of the process and in the quality of the end-product without any additional costs, using existing equipment. The effect of a

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^{*} Corresponding author. Tel.: +20 1119177153.

E-mail address: fathi_online@hotmail.com (F.S. Soliman).

modifier on the crystallization of solid hydrocarbons is usually rated on the basis of the melting point of the microcrystalline wax, the oil content in the microcrystalline wax, and the slurry filtration rate such as ionic and non ionic modifiers, surfactants and additive [1].

Zolotarev and Nigmatullin, studied the purification and deoiling of slack wax with aluminum chloride complex. It can be used as a means for obtaining paraffin waxes with quality at the export-grade level, and for increasing the paraffin yield by 2–4% [2]. Nigmatullin et al. investigated the use of ionic modifiers; aqueous sodium chloride and aqueous iron sulfate; for deoiling petrolatum and slack wax, respectively. They concluded that the aqueous sodium chloride solution increases the selectivity of the highest-melting hydrocarbons from the petrolatum. But modifier did not affect the filtration rate and the modification function of iron sulfate was related to co-crystallization because crystal lattices of iron and solid paraffin waxes were similar [3,4].

Trends in improving production of oils and solid hydrocarbons were examined by optimization of crystallization of solid hydrocarbons by using surfactants and ultrasound. Surfactant structural modifiers significantly affect crystallization of solid hydrocarbons. Concentrated on the phase interface, they form very thin layers that change the molecular nature and properties of the surface. Primarily resins are adsorbed on an energetically inhomogeneous surface of arising crystallization centers consisting of high melting paraffins and naphthenes due to the strongly developed hydrocarbon part of their molecules. The metal containing additives and fractions of solid hydrocarbons are used as structure modifiers. Incorporation of the modifier in the concentration of 0.001-0.1 wt.% increases the yield of oil by 2-4 wt.% and the filtration rate and reduces the oil content in the solid phase by 2-3 times. To enhance the deoiling process, the slack wax (melting point of 54 °C, oil content of 5.5 wt.%) was treated with ultrasound. Exposure to ultrasound before deoiling stage I accelerated filtration in the following stages as well. In deoiling of slack wax in two stages with ultrasound treatment before the first stage for 10 min, the wax product contains a 0.43 wt.% oil content and has a melting point of 58 °C [5].

For improving the crystallization of solid hydrocarbons; during the deoiling of petrolatum to produce microcrystalline wax; a non-polar modifier (pure n-alkane from C₂₀-C₂₄) was added to the wax deoiling solvent mixtures. Kazakova et al. showed that non-polar modifiers, especially individual nalkanes with an even number of carbon atoms in the molecule $(C_{20}-C_{24})$ are very important when added in amounts highly effective for crystallization of petroleum solid hydrocarbons. A portion of the modifier; C20-C24 n-alkanes; participated in the creation of the crystal nuclei, and the remainder built in the surface of the growing crystals. This not only altered the surface but also enhanced interaction contact with other crystals that include long alkyl radicals. During this interaction, microcrystalline wax crystals and the modifier formed densely packed coagulated structures; the result was the squeezing of substantial amounts of oil and lower molecular weight components to the solvent layer. The filtration rate was greatly increased and the resulting microcrystalline wax contained 0.5 wt.% oil [1,6,7].

No literature was found about separation of non-polar modifiers from petroleum products for intensification of deoiling residual stocks. Thus, the present work deals with the use of the light slack wax; which is a dewaxing by-product obtained from dewaxing of the light wax distillate fraction through the manufacture of lubricating oil; for separation of non-polar modifier concentrates having the carbon numbers of C_{20} – C_{26} by applying two different techniques: multistage fractional crystallization and liquid–solid chromatography, followed by the urea adduction technique.

2. Experimental

2.1. Material

Two slack wax crudes (petroleum by-products) were obtained from light and middle wax distillate fractions from El-Ameria Refining Company and Butyle acetate.

2.2. Methods

2.2.1. Separation of non-polar modifier concentrates

Two different techniques: multistage fractional crystallization and liquid–solid chromatography, followed by the urea adduction technique were used to separate non polar modifier concentrates containing high contents of C_{20} – C_{26} n-alkanes.

2.2.2. Multistage fractional crystallization technique

Light slack wax was subjected practically to multistage stage fractional crystallization process by using butyl acetate solvent [8] at different fractionating temperatures under fixed dilution and washing solvent ratios (S/F) of 6:1 and 2:1 by weight, respectively. At each fractionating temperature the high melting point components of the slack wax were precipitated, while the low melting ones remained in the solution. The process of solvent fractionating temperatures ranging from 40 to 20 °C at intervals of 5 °C.

2.2.3. Liquid-solid chromatographic technique

Light slack was subjected to liquid–solid column chromatography to separate its total saturate components [9].

2.2.4. Urea adduction technique

The light slack wax, its saturate components and the hard wax fractions isolated from the light slack wax were subjected to the urea adduction technique [10] to separate pure non-polar modifier concentrates containing high contents of C_{20} – C_{26} n-paraffins.

2.3. Methods of analysis

The light and middle slack waxes, the light slack wax saturate and the isolated hard waxes from light slack wax and their adducts were physically characterized according to American Society for Testing and Materials (ASTM) standard methods [11]. The aromatics and n-paraffin contents were determined by using the liquid–solid column chromatographic technique [9] and by using the GC technique, respectively. The GC apparatus used was of model Perkin Elmer, Clarus 500, England, equipped with a hydrogen flame ionization detector and fused silica capillary column (30 cm \times 0.25 mm i.d.), packed with poly (dimethyl siloxane) HP-1 (non-polar packing) of $0.5 \,\mu\text{m}$ film thickness. The apparatus was also equipped with an integrated data handling system for computing the peak area and concentration. In the chromatograph, the injector was heated at 350 °C. The column temperature was programmed from 80 to 300 °C at a fixed rate of 3 °C/min., and nitrogen (oxygen-free) was used as a carrier gas with flow rate of 2 ml/min. The detector was heated at 350 °C, and operated with a hydrogen flow rate adjusted to optimize the detector sensitivity. The sample was melted and 0.1 μ l of it was introduced into the injector.

3. Results and discussion

3.1. Characterization of slack wax crudes

The physical characteristics and the molecular type composition for the two slack wax crudes are presented in Table 1. Data indicate that the refractive index, density, kinematic viscosity and congealing point increase with increasing boiling point range and/or mean molecular weight of the slack wax fraction. Thus, the light slack wax shows lower refractive index, density, kinematic viscosity and congealing point than those of the middle one.

Molecular type composition data revealed that, the middle slack wax has somewhat higher total aromatic content (3.03 wt.%) as compared to that of the light slack wax (2.26 wt.%). These aromatic constituents are totally monoaromatic ones only. Also, it is clear from the data that, the light slack wax is characterized by its high n-paraffin content (88.42 wt.%) while the middle slack wax is characterized by its high iso- and cyclo-paraffin content (32.11 wt.%) than that of the light one (Table 1).

Table 1	Physical characteristics and molecular type co	ompo-
sition of	he two slack waxes.	

Characteristics	Test method	Slack wa	Slack waxes		
		Light	Middle		
Congealing point, °C	ASTM D-938	46	59		
Kinematic viscosity,	ASTM D-445	2.83	4.30		
98.9 °C					
Refractive index, 98.9 °C	ASTM D-1747	1.4214	1.4270		
Density, 70 °C	ASTM D-1418	0.7910	0.8035		
Mean molecular weight	ASTM D-2502	376	446		
Oil content, wt.%	ASTM D-721	4.25	6.23		
Needle penetration, 25 °C	ASTM D-1321	67	40		
Sulfur content, wt.%	ASTM D-4294	0.08	0.10		
Color	ASTM D-1500	0.5	1.5		
Molecular type composition					
Total saturates, wt.%		97.74	96.97		
N-paraffin content, wt.%		88.42	64.86		
$\% (C_{20} + C_{22})$		21.73	2.17		
$\% (C_{24} + C_{26})$		18.11	13.95		
Iso- & cyclo-paraffin		9.39	32.11		
content, wt.%					
Total aromatics, wt.%		2.26	3.03		
Mono-aromatics, wt.%		2.26	3.03		
Di- aromatics, wt.%		-	_		
Poly-aromatics, wt.%		_	_		

Individual n-paraffin contents and carbon number distribution data of the two slack waxes are shown graphically in Fig. 1. It can be observed that, the carbon atoms per molecule ranged from C₁₉ to C₃₄ and from C₁₉ to C₃₈ for light and middle slack waxes, respectively. This means that the middle slack wax has higher carbon numbers than the light one. On the other hand, the light slack wax shows higher n-paraffin contents of the even carbon atoms (C₂₀ + C₂₂) and (C₂₄ + C₂₆) (21.73 and 18.11 wt.%, respectively) than those of the middle one (2.17 and 13.95 wt.%, respectively) (Table 1 and Fig. 1).

Thus, it can be deduced that, the light slack wax is more suitable wax for separation of n-alkanes with an even number of carbon atoms ranging from C_{20} to C_{26} for their high n-paraffin contents and can be used as non-polar structural modifiers.

3.2. Separation of non-polar modifier concentrates

The light slack wax has been used to separate non-polar modifier concentrates having the carbon numbers of C_{20} - C_{26} n-alkanes by using two different techniques: multistage fractional crystallization and liquid–solid chromatography, followed by the urea adduction technique.

3.2.1. Evaluation of the hard waxes separated by multistage fractional crystallization technique

The yield, the physical characteristics and n-paraffin evaluation of the hard waxes separated from the light slack wax by multistage fractional crystallization are presented in Table 2. It is of interest to note that by using butyl acetate solvent to fractionate the light slack wax at fractionating temperature of 40 °C, there is no wax yield separated. This may be attributed to the high solubility power of the solvent at this fractionating temperature which makes it dissolve all the components present in the slack wax.

Also, it can be observed that, the hard waxes isolated at the other fractionating temperatures differ considerably in their yield on decreasing the fractionating temperatures from 35 to 20 °C. This may be attributed to the subsequent precipitation of the different constituents of the wax according to their melting points.

The effect of decreasing the fractional crystallization temperature upon the isolated wax quality is reflected by a slight decrease in refractive index, kinematic viscosity, congealing point and mean molecular weight and considerable increase in oil content and needle penetration. This may be related to the increase of n-paraffins of lower melting points (Table 2).

N-Paraffin content and carbon number distribution data of hard waxes isolated from the light slack wax at fractionating temperatures of 35, 30, 25 and 20 °C are represented in Table 2 and shown graphically in Fig. 2.

It can be observed from the data that, the total n-paraffin content of the hard wax isolated from the light slack wax at fractionating temperature of 30 °C is the highest content (96.50 wt.%) as a result of the highest precipitation of the even carbon atoms $C_{24} + C_{26}$ (33.36 wt.%) at this temperature (Table 2). The disappearance of the carbon atom C_{19} in all the isolated hard wax is due to the higher solvent power of butyl acetate toward the lowest melting point carbon atom (C_{19}) in the light slack wax (Fig. 2).

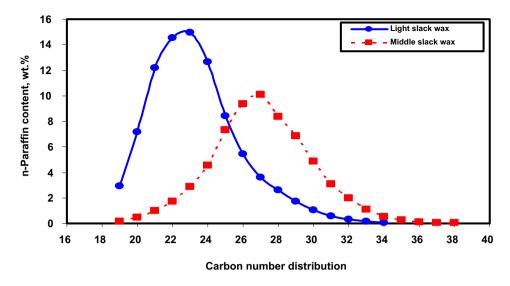


Figure 1 Carbon number distribution versus n-paraffin contents of light and middle slack waxes.

Table 2 Physical characteristics and n-paraffin evaluation of hard waxes separated from the light slack wax by multistage fractional crystallization at different fractionating temperatures under fixed dilution and wash solvent ratios of 6:1 and 2:1 by weight, respectively.

Characteristics	Light slack wax	Hard waxes separated by multistage deoiling at different temperatures				
		40 °C	35 °C	30 °C	25 °C	20 °C
Yield on crude, wt.%	100	_	8.48	13.21	7.57	7.55
Congealing point, °C	46	_	54.5	54	53.5	51.5
Kinematic viscosity at 98.9 °C, mm ² /s	2.83	_	3.12	3.00	2.87	2.78
Mean molecular weight	376	_	401	395	390	385
Needle penetration, 25 °C	67	_	15	17	18	22
Oil content, wt.%	4.25	_	0.20	0.63	0.87	1.12
Refractive index, 98.9 °C	1.4214	_	1.4130	1.4125	1.4114	1.4107
Total n-paraffin content, wt.%	88.42	_	93.00	96.50	95.72	94.55
$\% (C_{20} + C_{22})$	21.73	_	5.64	8.33	12.48	17.03
$\% (C_{24} + C_{26})$	18.11	_	28.40	33.36	30.95	27.15
% $(C_{20} + C_{22})/\%$ $(C_{24} + C_{26})$ Ratio	1.20	-	5.04	4.00	2.48	1.59

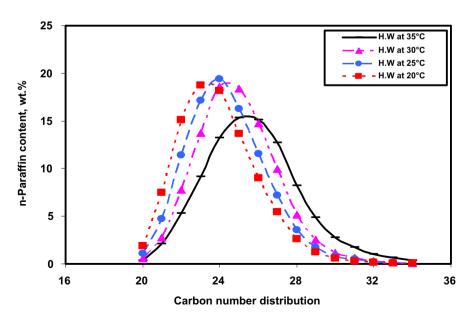


Figure 2 Carbon number distribution versus n-paraffin contents of the light slack wax and the hard waxes isolated at 35, 30, 25 and 20 °C.

Also, it can be noticed from the data that, the n-paraffin content of the even carbon atoms ($C_{20} + C_{22}$) increases gradually in the hard wax on decreasing the fractionating temperature from 35 to 20 °C and shows the highest value of 17.03 wt.% at 20 °C. Moreover, the total n-paraffin content of the even carbon atoms ($C_{24} + C_{26}$) increases by decreasing the fractionating temperature from 35 to 30 °C. The highest value (33.36 wt.%) is achieved at the fractionating temperature of 30 °C. On further decrease in the fractionating temperature, the total n-paraffin content of ($C_{24} + C_{26}$) decreases (Table 2).

3.2.2. Evaluation of the saturate separated by liquid–solid chromatography

The saturate of the light slack wax was separated by the liquid–solid column chromatographic technique and has been analyzed by the gas chromatographic technique to determine its n-paraffin content and carbon number distribution. Data are presented in Table 3 and shown graphically in Fig. 3.

It can be observed that, the individual n-paraffin contents for the saturate components of the light slack wax are nearly the same as those of the light slack wax and the individual n-paraffin contents increase by increasing the carbon number up to C_{23} and then decrease till C_{34} (Fig. 3).

Also, it can be noticed that, the light slack wax and its saturates have the highest total n-paraffin contents of the even carbon atoms ($C_{20} + C_{22}$) (21.73 and 22.03 wt.%, respectively) than those of the carbon atoms ($C_{24} + C_{26}$) (18.11 and 18.39 wt.%, respectively) and their ratios of ($C_{20} + C_{22}$)

 Table 3
 Carbon number distribution and n-paraffin evaluation of the light slack wax and its saturate.

Characteristics	Light slack wax	Light slack wax saturate
Carbon number distribution	C ₁₉ –C ₃₄	C ₁₉ -C ₃₄
Total n-paraffin content, wt.%	88.42	89.73
$\% (C_{20} + C_{22})$	21.73	22.03
$\% (C_{24} + C_{26})$	18.11	18.39
$\frac{1}{2} (C_{20} + C_{22})/\frac{1}{2}$	1.20	1.20
$(C_{24} + C_{26})$ Ratio		

to $(C_{24} + C_{26})$ are the same (1.20) due to the identical carbon number distribution and n-paraffin contents for both the light slack wax and its saturates (Table 3).

3.2.3. Evaluation of the products of urea adduction technique

Urea adduction technique was used in this study for separation of pure n-paraffin components from the light slack wax, light slack wax saturates and the hard waxes isolated from the light slack wax by the multistage fractional technique at 35, 30 and 20 °C. Individual and n-paraffin contents and their carbon number distribution of the adducts have been determined by the gas chromatographic technique.

The yield, the physical characteristics, n- paraffin evaluations of the adducts of light slack wax, light slack wax saturates and the hard waxes isolated from the light slack wax are represented in Table 4.

Data indicate that, the adduct yield of the light slack wax (83 wt.%) is higher than that of its saturates (80.75 wt.%). This may be attributed to the presence of small amounts of iso-paraffins and mono-aromatics in the light slack wax (Table 1) possessing only a slight branching as compared to the length of the carbon chains that can form adducts with urea. The lower congealing point and the higher refractive index of the light slack wax adduct than those of its saturate adduct (Table 4) confirm the above findings.

The refractive indices and congealing points of the adducts of the separated hard waxes decrease with decreasing the fractionating temperature from 35 to 20 °C due to the precipitation of the high melting point n-paraffins possessing high carbon numbers at first followed by the lower melting point ones (Table 4).

Also, it is clear from the data that, n-paraffin content and carbon number distribution of the adducts of the light slack wax and its saturates (Table 4) have the same trend like those of the light slack wax and its saturates (Table 3) as discussed before (Section 3.2.2).

Meanwhile, the n-paraffin contents of the even carbon atoms ($C_{20} + C_{22}$) and ($C_{24} + C_{26}$) for the adducts of light slack wax and its saturates (23.55 & 20.72 and 23.74 & 20.73 wt.%, respectively) are higher than those of the light slack wax and its saturates (21.73 & 18.11 and 22.03

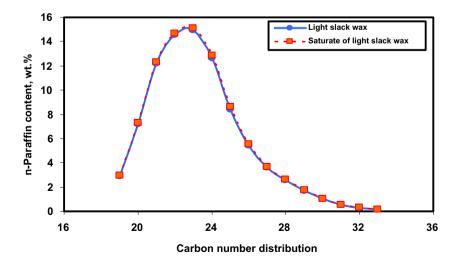


Figure 3 Carbon number distribution versus n-paraffin contents of the light slack wax and its saturate.

Characteristics	Adducts of light slack wax		Adducts of h	ıt	
	Itself	Its saturate	35 °C	30 °C	20 °C
Yield of adducts, wt.%	83	80.75	75.23	80.19	82.87
Yield of adducts on crude, wt.%	83	78.93	6.38	10.59	6.26
Congealing point, °C	51	53	55.5	55	52.5
Refractive index, 98.9 °C	1.4126	1.4110	1.4133	1.4125	1.4109
Total n-paraffin content, wt.%	93.44	94.51	97.12	97.61	96.64
$\% (C_{20} + C_{22})$	23.55	23.74	5.82	9.20	15.80
$\% (C_{24} + C_{26})$	20.72	20.73	33.62	32.96	28.50
$\frac{1}{2} (C_{20} + C_{22}) / \frac{1}{2} (C_{24} + C_{26})$ Ratio	1.14	1.15	5.78	3.58	1.80

 Table 4
 Physical characteristics and n-paraffin evaluation of the adducts of light slack wax, its saturate and the hard waxes separated at different temperatures.

&18.39 wt.%, respectively) (Compare Tables 3 and 4). This is attributed to the fact that the urea adduction technique purifies and concentrates n-paraffins.

Moreover, the adducts of the light slack wax and its saturates have n-paraffin contents of the even carbon atoms $(C_{20} + C_{22})$ (23.55 and 23.74 wt.%, respectively) somewhat larger than those of carbon atoms $(C_{24} + C_{26})$ (20.72 and 20.73 wt.%, respectively) and their ratios are similar (1.14:1). Thus, these adducts; light slack wax and its saturate components; can be used as non-polar modifier concentrates of low carbon atoms $(C_{20} + C_{22})$. From an economic point of view, it is preferable to select the light slack wax adduct as a nonpolar modifier concentrate whereas, the separation step can be neglected to save energy and its yield calculated on crude basis is larger than that of the adduct of its saturate (83 and 78.93 wt.%, respectively) (Table 4).

The hard wax fractions isolated from the light slack wax by multistage fractional crystallization at temperatures of 35, 30 and 20 °C were subjected to urea adduction. Normal paraffin content and carbon number distribution of the adducts are represented in Table 4 and shown graphically in Fig. 4.

Data indicate that the total n-paraffin contents for the adducts of the hard wax fractions isolated at the fractionated temperatures of 35, 30 and 20 °C are more or less the same (97.12, 97.61 and 96.64 wt.%, respectively). Meanwhile, on decreasing the fractionating temperature from 35 to 20 °C, the n-paraffin contents for the carbon atoms ($C_{20} + C_{22}$) increase and the n-paraffin contents for the carbon atoms

 $(C_{24} + C_{26})$ decrease. Consequently, the ratios of n-paraffin contents for the carbon atoms $(C_{24} + C_{26})$ to those of carbon atoms $(C_{20} + C_{22})$; the higher melting n-paraffins to the lower ones; decrease with the decrease of the fractionating temperature due to the higher n-paraffin contents of the carbon atoms $(C_{24} + C_{26})$ than those of carbon atoms $(C_{20} + C_{22})$ (Table 4).

Also, data reveal that, the adduct of the hard wax isolated at 20 °C has the highest n-paraffin content of the carbon number atoms ($C_{20} + C_{22}$) (15.80 wt.%), as compared to the adducts of hard waxes isolated at 30 and 35 °C, but it is the lowest n-paraffin content as compared with the adducts of light slack wax and its saturates (23.55 and 23.74 wt.%, respectively) (Table 4).

Moreover, it is clear from the data that, the adducts of hard waxes isolated at 35 and 30 °C have nearly the same n-paraffin contents of the carbon number atoms ($C_{24} + C_{26}$) (33.62 and 32.96 wt.%, respectively) and smaller n-paraffin contents of the carbon number atoms ($C_{20} + C_{22}$) (5.82 and 9.20 wt.%, respectively). Consequently, the ratio of n-paraffin contents of the carbon number atoms ($C_{24} + C_{26}$) to those of the carbon number atoms ($C_{24} + C_{26}$) to those of the carbon number atoms ($C_{24} + C_{26}$) to those of the carbon number atoms ($C_{20} + C_{22}$) is larger for the adduct of hard wax separated at 35 °C than that at 30 °C (5.78 and 3.58 wt.%, respectively) (Table 4). Thus, the adducts of hard wax separated at 35 and 30 °C can be used as non-polar modifier concentrates of high carbon atoms ($C_{24} + C_{26}$). From an economic point of view, it is preferable to select the adduct of the hard wax isolated at 30 °C as its yield calculated on crude basis (light slack wax) is larger than that of the adduct

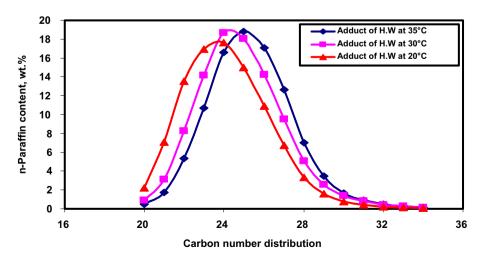


Figure 4 Carbon number distribution versus n-paraffin contents of the adducts of hard waxes isolated at 35, 30 and 20 °C.

isolated at 35 °C (10.59 and 6.38 wt.% at 30 and 35 °C, respectively) (Table 4).

From the above results, it can be concluded that, the light slack wax adduct is the most suitable non-polar modifier concentrate of the low carbon number atoms ($C_{20} + C_{22}$), as its concentration to the high carbon number atoms ($C_{24} + C_{26}$) is 23.55–20.72 wt.%, respectively. Meanwhile, the adduct of the hard wax isolated at 30 °C can be chosen as the preferable non-polar modifier concentrate of the high carbon number atoms ($C_{24} + C_{26}$), as its concentration to the low carbon number atoms ($C_{24} + C_{26}$), as its concentration to the low carbon number atoms ($C_{24} + C_{26}$), as its concentration to the low carbon number atoms ($C_{20} + C_{22}$) is 32.96–9.20 wt.%, respectively.

4. Conclusions

The study shows that, the light slack wax is selected as a suitable crude wax for separation of n-alkanes with an even number of carbon atoms ranging from C_{20} to C_{26} for their high n-paraffin contents and can be used as non-polar structural modifiers. The adduct of light slack wax is more preferable than the adduct of its saturates to be used as non-polar modifier concentrates of low carbon atoms ($C_{20} + C_{22}$) whereas, the separation step can be neglected to save energy. Meanwhile, the adduct of the hard wax isolated at 30 °C can be used as the preferable non-polar modifier concentrate of the high carbon number atoms ($C_{24} + C_{26}$).

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