Production of Paraffinic Wax from Balayiem Land Crude

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With 3 figures

Summary

This work deals with the production of paraffinic waxes from the Balayiem land crude by solvent dewaxing and filter pressing. The diesel oil cut included 8.8 wt.% paraffin wax, spindle oil 16.6 wt.% and neutral oil I 16.3 wt.%. The deoiled waxes were pure white with solidification points at 41, 49 and 59 °C in the three oil fractions. The waxes included very small amounts of iso-paraffins, which was a good property for fatty acid and soap production.

Methods

1. Distillation

The wax distillate[1]) obtained from the Balayiem land crude was supplied from the asphalt vacuum unit of the Suez petroleum company.

59 kg wax distillate were distilled in a pilot unit at a vacuum of 1 to 2 Torr in the following three fractions,

a-Diesel oil	320—350 °C
b-Spindle oil	350—420 °C
c-Neutral oil I	420—480 °C

The three fractions were characterised by their physical and chemical constants, i. e. refractive index n_D^{70}, density d_{70}, kinematic viscosity, flash point, Conradson carbon residue, acid number, iodine number, paraffin content (at −30 °C)[2]) and n-paraffin content (urea addition). The results are given in Table 1.

2. Solvent dewaxing[2])

The three fractions obtained from the was distillate were dewaxed with an acetone, benzene, toluol (30:35:35) mixture in a ratio to the oil of 3:1

[1]) S. ELBADRAWY, Evaluation of the wax distillate in Balayiem land crude, The 5th. Arab. petroleum congress 1963.

[2]) S. GIPP, Dissertation Leipzig/G. D. R. (1961).

Table 1
Characterisation of the oil fractions

Fraction	wt. %	d_{70}	n_D^{70}	pour pt. °C	flash pt. °C	Conrad. wt. %	acid No.	iodine No.	n-paraf. wt. %	S_P °C n-paraf.	paraffin wt. % −30
Diesel oil	7.8	0.8466	1.4658	+2	147	0.06	0.2	30.7	17.2	24.2	8.8
Spindle oil	42.0	0.8724	1,4790	+22	196	0.1	0.16	30.0	22.7	40.2	16.6
Neutral oil	36.5	0.8966	1.4868	+37	228.5	0.4	0.09	21.5	17.8	55.0	16.3

The residue made 13.7 wt. % from the wax distillate and contained 8.3 wt. % n-paraffin with $S_P = 64.4$ °C.

in one experiment and in another one in the ratio of 2:1. Washing was carried out using 100 wt.% ABT mixture. Deoiling was performed with ABT in the ratio of ABT to the wax cake of 7:1. Dewaxing and deoiling were carried out at −10 °C. The results are given in table 2.

Table 2
Solvent dewaxing

Wax from	3:1 ABT:oil				2:1 ABT:oil			
	wt. %	oil content wt. %	S_P °C	n_D^{70}	wt. %	oil content wt. %	S_P °C	n_D^{70}
Before deoiling								
Spindle oil	12.2	0.5	47.6	1.4284	11.8	0.7	48.4	1.4291
Neutral oil I	14.3	2.7	57.4	1.4344	13.4	1.5	58.0	1.4340
After deoling								
Spindle oil	11.3	0.0	49.0	1.4280	11.5	0.1	49.2	1.4281
Neutral oil I	13.2	0.0	58.8	1.4328	13.0	0.0	58.6	1.4329

3. Filter pressing

The filtration was carried out in an apparatus as given in fig. 1. 250 g from the oil fractions were weighed in the molten state in 400 ml beakers. After cooling over night, the good crystallised masses were given to the filter press and cooled with a cooling thermostat to the working temperature. The

filtration process was represented in a filtrate amount — time curve (fig. 2 and 3). Spindle oil and neutral oil I were used in these experiments. Before pressing (using N_2-gas), the oil fractions were warmed to about 50 °C and

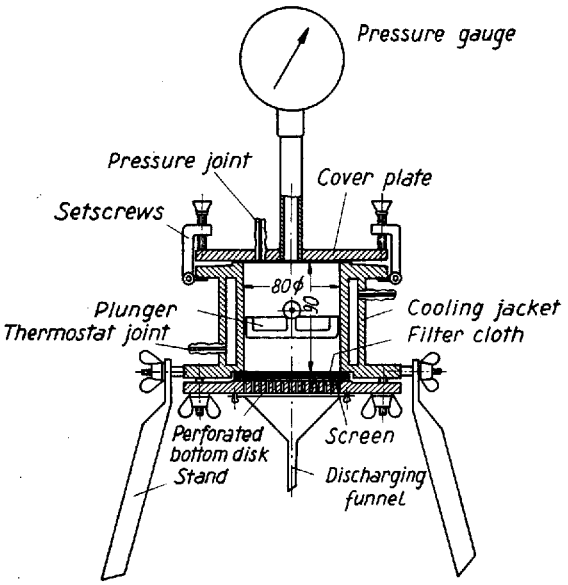

Fig. 1. Apparatus for filtration

then slowly cooled. The spindle oil was cooled in the different experiments (see table 3) to −5, 0, +5 and +10 °C. The nitrogen over pressure was in every case 6 kp/cm². The filtration time in min, the solidification point S_P [3])

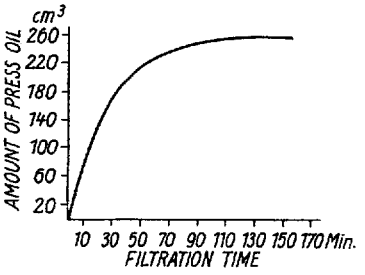

Fig. 2. Dependence of the filtrate amount from the filtration time. Pressure 6 kg/cm². Temp. + 10 °C

and the refractive index n_D^{70} of the filter cake were determined as given in table 3. The neutral oil was pressed at the given temperatures for spindle oil and also at +20 °C. In this case very small crystalls were formed in the neutral oil, the wax cake could not be filtered and passed through the filter

[3]) C. Zerbe, Mineralöle und verwandte Produkte, Berlin (1952), 381.

cloth. Therefore the neutral oil was mixed with amounts of ABT mixture in the ratio to the oil of 1:1, 2:3, 1:3, 1:4, 1:6, 1:8, and 1:10. Pressing was performed at the temperatures from 0 °C till +20 °C. In the case of the

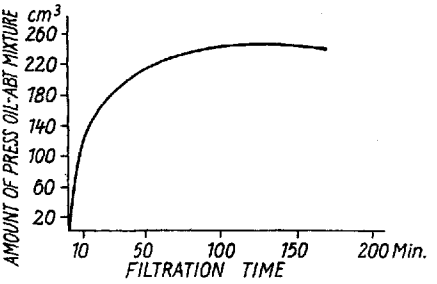

Fig. 3. Dependence of filtrate amount from the filtration time. Pressure 1—6 kg/cm². Temp. +20 °C. ABT:Oil = 1:6

neutral oil the pressure must be increased gradually from 1 to 6 kp/cm², otherwise the filter cake also passed through the filter cloth (results are given in table 3). The wax cakes were deoiled with ABT mixture in the ratio to the wax of 7:1 at the temperature 0 °C and washed with 100 wt.% ABT. The deoiling results are given in table 4.

Table 3
Filter pressing

Press temp. °C	Pressing time Min.	cake wt. %	loss	S_P °C	n_D^{70}	Press oil wt. %
			1. Spindle oil			
−5	250	28.0	2.0	38.2	1.4554	70.0
0	240	22.4	—	39.6	1.4505	78.0
+5	120	14.0	8.0	47.2	1.4452	84.0
+10	100	11.2	0.8	48.0	1.4352	88.0
			2. Neutraloil I			
ABT:oil						
1:1 at 0 °C	95	13.5	18.5	55.4	1.4422	68.0
2:3 at 0 °C	70	—	—	57.2	1.4384	—
1:3 at 0 °C	80	11.1	12.1	57.2	1.4380	76.8
1:4 at 0 °C	240	21.5	7.5	54.0	1.4494	71.0
1:4 + 5 °C	150	17.5	6.0	54.5	1.4471	76.3
1:6 +10 °C	220	15.7	2.4	55.6	1.4430	81.9
1:6 +15 °C	120	14.8	4.2	56.5	1.4406	81.0
1:6 +20 °C	120	13.8	2.9	57.5	1.4378	83.3
1:8 + 5 °C	The filter cake passed through the filter cloth					
1:10 + 5 °C	The filter cake passed through the filter cloth					

Table 4
Deoiling of the filter cake from the filter press

Press-temp. °C	before deoiling			after deoiling			
	wt. %	S_P °C	n_D^{70}	wt. %	S_P °C	oil wt. %	n_D^{70}
1. Spindle oil							
+5	14.0	47.2	1.4452	8.0	50.2	0.04	1.4291
+10	11.2	48.0	1.4352	6.8	50.4	0.0	1.4288
2. Neutral oil							
+15	14.8	56.5	1.4430	9.5	60.2	0.2	1.4332
+20	13.8	57.5	1.4406	9.0	60.5	0.2	1.4330

4. Characterisation of the deoiled paraffin waxes obtained by solvent dewaxing

The deoiled waxes obtained from spindle oil and neutral oil I were pure white. The characteristics of these waxes such as density at 70 °C, iodine No., acid No., saponification No., Sum value[4]), kinematic viscosity and soft paraffin content[5]) were determined (table 5).

Table 5
Characterisation of the paraffin waxes obtained by solvent dewaxing

Wax from	d_{70}	S_P °C	n_D^{70}	Iodine No.	acid No.	Sapon. No.	V_K 70 °C cSt.	Soft paraff. wt. %
Spindle oil	0.7671	49.2	1.4281	1.1	0.0	0.0	4.7	1.5
Neutral oil I	0.7766	58.6	1.5329	1.1	0.0	0.0	6.77	0.0

Discussion

It was clear from the dewaxing experiments (table 2), that washing with ABT in a ratio of 3:1 decreased the oil content in the wax cake obtained from spindle oil from 0.5 wt.% to 0.0 wt.-% and of neutral oil wax from 2.7 to 0.0 wt.%. Also the use of ABT in the ratio to the oil of 2:1 was sufficient by the dewaxing processes to produce pure white paraffin wax of very low oil content. Using filter pressing, ist was clear, that spindle oil could be pressed without using a solvent, which was due to the formation of good plate-like wax crystalls. The suitable pressing temperature (table 3)

[4]) H. Gross u. K. Grodde, „Öl und Kohle" **38**, 419 (1942).
[5]) U. Schwarz u. V. Huber, Chem. Rev. über d. Fett- und Harzind. **20**, 242 (1913).

was found to be +10 °C, where a wax cake of solidification point 48.0 °C and refractive index n_D^{70} 1.4352 in a yield 11.2 wt.% was obtained. At lower pressing temperatures higher yields were obtained, for ex. at +5 °C 14.0 wt.%, at 0 °C 22.4 wt.% and at −5 °C 28.0 wt.%, at the same time the refractive index increased and the solidification temperature decreased. The increase of refraction was a result of the increase of the oil content in the filter cake with the lowering of the dewaxing temperature. The shortest pressing time needed (see fig. 2) was 100 min at +10 °C.

In the case of neutral oil I the solvent mixture ABT (30:35:35) must be mixed with the oil to obtain a good crystallised filter cake. The most suitable ratio of ABT to oil was found to be 1:6, while at a ratio of 1:8 and 1:10 the whole product passed through the filter cloth. The most suitable pressing temperature was found to be +20 °C, where a wax cake of solidification point 57.5 °C and refractive index n_D^{70} 1.4378 and yield of 13.8 wt.% was obtained. The pressing time amounted to 120 min (see fig. 3). At lower temperatures higher yields were obtained with longer pressing time, the refractive index increased and the solidification temperature decreased.

A comparison between solvent dewaxing and pressing shows, that by the pressing process lower solidification points and higher refractions of the filter cakes were obtained than by solvent dewaxing. This means, as expected, the obtained wax cake was richer in oil in the case of pressing than in the case of solvent dewaxing (see table 2 and 4). Pressing took place at higher temperatures than selective separation by solvent mixture, thereby a higher solubility of the wax in the oil is caused. The paraffinic components remaining in the dewaxed oil, passed through the filter cloth causing unsuitable pour points of the dewaxed oil. At low temperatures pressing needed a longer filteration time with corresponding higher cooling costs. Therefore solvent dewaxing is more suitable for the separation of waxes with low oil content than pressing. The waxes obtained by solvent dewaxing and deoiling (table 5) are acid free and have a very low olefin content (iodine No. 1.1). The neutral oil waxes had sum values (4) of 1.2, which means that thay contained small amounts of iso-paraffins, which caused the rise of the sum value from zero for n-paraffins to 1.2. Spindle oil wax had a sum value of 0.4. The soft paraffin content in the spindle oil wax was 1.5 wt.%, which was a cery low content. The neutral oil wax was free from soft paraffin.

Conclusion

Solvent dewaxing was more suitable than pressing to obtain waxes, which were nearly free of oil.

The waxes obtained have a very low isoparaffinic content and this means that these paraffinic waxes were suitable raw materiales for fatty acid and soap production.

Cairo (VAR), Petroleum Research Unit, National Research Centre.

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