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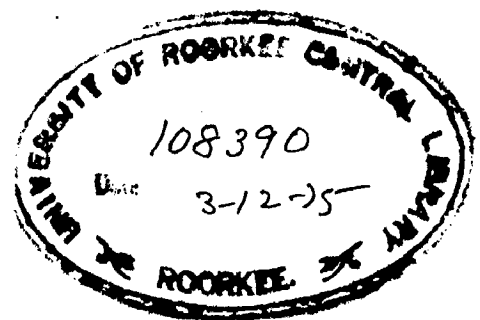
SOLVENT DEOILING OF BARAUNI SLACK WAX
USING
METHYL-ISOBUTYL-KETONE

A Dissertation
submitted in partial fulfilment of the
requirements for the award of the Degree
of
MASTER OF ENGINEERING
in
CHEMICAL ENGINEERING
(EQUIPMENT & PLANT DESIGN)

By

UMESH CHANDRA AGRAWAL

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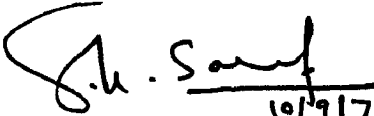
DEPARTMENT OF CHEMICAL ENGINEERING
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12

C E R T I F I C A T E

CERTIFIED that the thesis entitled 'SOLVENT DEOILING OF BARAUNI SLACK WAX USING METHYL ISOBUTYL KETONE' which is being submitted by Sri Umesh Chandra Agrawal in partial fulfilment of the requirements for the award of the Degree of MASTER OF ENGINEERING IN CHEMICAL ENGINEERING (Equipment and Plant Design) of the University of Roorkee, Roorkee is a record of candidate's own work carried out by him under the supervision and guidance of the undersigned. The matter embodied in this thesis has not been submitted for the award of any other degree or diploma.

This is further certified that he has worked for a period of about eight months for preparing this thesis at this university.


(S.K.SARAF) 10/9/74

Professor
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A C K N O W L E D G E M E N T

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TABLE I.1.1. EXPERIMENTAL DATA FOR FIRST STAGE DEOILING, SOLVENT-TO-SLACK WAX RATIO 2:1

DATA	RUN NO.	1.21	2.21	3.21	4.21
FIRST STAGE DEOILING					
Weight of slack wax, g		438.00	380.50	387.50	400.50
Weight of MIBK, g					
initially		438.00	400.00	387.50	400.00
1st increment		199.00	170.30	193.10	200.00
2nd increment		199.00	170.30	193.10	200.00
washing		298.60	255.50	289.70	300.00
Initial Temperature, °C					
slack wax		76.00	70.00	78.00	77.00
MIBK		45.00	45.00	45.00	45.00
Slack wax-MIBK solution		55.50	51.00	56.00	57.00
Final Deoiling Temperature, °C		19.50	20.00	19.00	19.00
Filtration/washing Temperature, °C		22.00	20.50	20.00	20.00
Average chilling Rate, °C/min					
from initial to 45°C		0.70	1.00	0.37	0.80
from 45°C to 30°C		0.60	0.94	0.21	0.43
from 30°C to 20°C		0.18	0.20	0.18	0.17
from 20°C to deoiling temp.		-	-	-	-
Vacuum attained after 90 sec., mmHg					
filtration		320.00	360.00	370.00	360.00
washing		320.00	360.00	370.00	370.00
Filtrate collected, g					
filtration					
washing		880.00	816.50	888.50	888.50
Cake obtained, g		622.00	488.50	585.50	585.50
Cake for recovery, g		622.00	488.50	554.50	554.50
Product wax, g		331.00	272.00	301.50	328.00
Foots oil, g		95.00	76.00	94.50	72.50
MIBK from filtrate, g		714.10	714.10	814.00	569.2
MIBK from wax-cake, g		145.5	186.2	232.80	396.00

Table continued

TABLE I.1.1 EXPERIMENTAL DATA FOR FIRST STAGE DECILLING, SOLVENT-TO-SLACK WAX RATIO 2:1 (Continued)

DATA	5.22	6.22	7.22	8.22	9.22	10.22	11.22	12.22	13.22
FIRST STAGE DECILLING									
Weight of slack wax, g	404.50	400.00	399.00	396.00	401.00	401.00	402.50	402.00	399.50
Weight of MIBK, g									
initially	400.00	400.00	400.00	400.00	400.00	400.00	400.00	400.00	400.00
1st increment	200.00	200.00	200.00	-	200.00	200.00	200.00	200.00	200.00
2nd increment	200.00	200.00	200.00	200.00	200.00	200.00	200.00	200.00	200.00
washing	300.00	300.00	300.00	300.00	300.00	300.00	300.00	300.00	300.00
Initial Temperature, °C									
slack wax	72.00	80.00	70.00	72.00	80.00	69.00	72.00	71.00	74.00
MIBK	46.00	45.00	46.00	45.50	45.00	45.00	45.50	45.00	45.00
slack wax-MIBK solution	55.00	55.00	53.50	51.00	56.00	51.50	54.50	55.00	56.00
Final Decilling Temperature, °C	19.00	14.50	15.50	15.00	10.00	10.00	5.00	5.00	19.00
Filtration/washing Temperature, °C	20.00	15.00	15.50	16.00	10.50	10.00	6.00	6.00	19.00
Average Chilling Rate, °C/min									
from initial to 45°C	1.00	0.67	0.85	0.35	0.91	0.81	0.95	1.10	1.10
from 45°C to 30°C	0.75	0.38	0.83	0.83	0.83	1.25	1.00	1.00	1.08
from 30°C to 20°C	0.14	0.16	0.20	0.67	0.29	0.33	0.33	0.33	0.33
from 20°C to decilling temp.	-	0.13	0.17	0.13	0.29	0.29	0.30	0.30	-
Vacuum attained after 90 sec. mmHg									
filtration	360.00	360.00	330.00	370.0	360.00	380.00	390.00	380.00	320.00
washing	360.00	370.00	360.00	380.0	380.00	380.00	400.00	385.00	385.00
Filtrate collected, g									
filtration	584.50	746.50	565.50	408.0	563.00	454.00	497.00	-	440.00
washing	256.00	286.00	271.50	214.0	248.00	265.00	265.00	806.00	300.00
Cake obtained, g	532.00	602.00	528.00	560.0	594.00	568.00	599.00	501.00	536.00
Cake for Recovery, g	128.60	150.50	132.00	140.0	148.50	142.00	150.00	130.00	134.00
Product wax, g	74.50	76.50	78.20	74.5	75.50	76.50	79.50	77.00	74.50
Foots oil, g	89.50	79.00	82.80	80.0	71.00	71.50	65.00	54.00	74.00
MIBK from filtrate, g	704.00	912.00	701.50	540.0	688.00	624.00	669.00	722.00	640.00
MIBK from wax-cake, g	38.00	55.50	37.00	43.0	49.00	55.00	55.00	44.00	50.50

Table continued

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TABLE I-1.2 EXPERIMENTAL DATA FOR SECOND STAGE DEOILING, SOLVENT -TO-SLACK WAX RATIO 2:1

DATA	5.22	6.22	7.22	8.22	9.22	10.22	11.22	12.22	13.22
SECOND STAGE DEOILING									
Weight of wax cake, g	403.40	451.50	396.00	420.00	445.50	426.00	449.00	371.00	402.00
Weight of MIBK, g									
repulping	150.00	300.00	300.00	300.00	300.00	300.00	300.00	300.00	300.00
washing	225.00	225.00	225.00	225.00	225.00	225.00	225.00	225.00	225.00
Initial Slurry Temperature, °C	32.00	32.50	29.00	31.50	26.00	25.00	24.00	19.00	30.00
Final Deoiling Temperature, °C	19.00	15.50	15.00	15.00	10.00	10.00	5.00	5.00	19.00
Filtration/Washing Temperature, °C	20.00	16.00	15.50	15.50	10.50	10.50	6.00	8.00	19.00
Average Chilling Rate, °C/min.	0.24	0.35	0.28	0.32	0.31	0.41	0.38	0.40	0.32
Vacuum attained after 90 sec., mmHg									
filtration	-	360.00	190.00	320.00	-	360.00	375.00	310.00	350.00
washing	360.00	380.00	350.00	340.00	370.00	380.00	385.00	385.00	350.00
Filtrate collected, g									
filtration	-	356.00	172.00	342.00	-	301.00	337.00	147.00	307.50
washing	343.50	178.00	278.00	176.50	485.00	175.00	182.00	330.00	181.50
cake obtained, g	425.00	391.00	473.00	338.00	365.00	403.00	370.00	353.00	358.00
Product wax, g	215.00	228.00	223.00	207.00	217.50	236.00	239.00	220.00	218.00
Foots Oil, g	10.50	17.00	13.00	17.00	12.40	11.50	13.00	17.50	17.50
MIBK from filtrate, g	312.00	502.50	421.00	458.50	416.00	434.00	495.00	418.00	430.00
MIBK from wax-cake, g	184.00	116.00	111.00	111.00	99.00	110.00	98.00	130.50	118.0

TABLE I.2.1 EXPERIMENTAL DATA FOR FIRST-STAGE DEOILING, SOLVENT-TO-SLACK WAX RATIO 3:1

DATA	14.32	15.32	16.32	17.32	18.32	19.32	20.32	21.32
FIRST STAGE DEOILING								
Weight of slack wax, g	400.0	401.0	455.5	395.0	389.0	402.0	396.0	399.0
Weight of MIBK, g								
initially	600.0	600.0	674.0	600.0	600.0	600.0	600.0	600.0
1st increment	300.0	300.0	342.0	300.0	292.0	300.0	300.0	300.0
2nd increment	300.0	300.0	342.0	300.0	292.0	300.0	300.0	300.0
washing	300.0	300.0	342.0	300.0	292.0	300.0	300.0	300.0
Initial Temperature, °C								
slack wax	70.0	76.0	75.0	71.0	79.0	73.0	72.0	74.0
MIBK	45.0	45.5	45.0	45.0	45.0	45.0	45.5	45.0
slack wax-MIBK solution	53.0	54.0	55.0	51.0	54.0	54.0	56.0	54.5
Final Deoiling Temperature, °C	5.0	5.0	10.0	10.0	14.5	15.5	19.5	19.0
Filtration/washing Temperature, °C	5.5	6.0	11.0	10.0	16.0	16.5	20.0	20.5
Average Chilling Rate, °C/min								
from initial to 45°C	0.80	1.00	0.83	0.75	0.75	0.75	1.10	0.95
from 45°C to 30°C	1.50	1.36	1.00	1.25	1.15	0.83	0.50	0.75
from 30°C to 20°C	0.40	0.36	0.25	0.37	0.29	0.31	0.33	0.29
from 20°C to deoiling temp.	0.25	0.25	0.18	0.19	0.20	0.17	--	--
Vacuum attained after 90 sec.mmHg								
filtration	390.00	385.00	390.00	350.00	380.0	380.00	380.00	360.00
washing	390.00	390.00	390.00	380.00	385.0	390.00	380.00	380.00
Filtrate collected, g								
filtration	772.00	710.00	797.00	766.00	767.0	827.00	849.00	840.00
washing	290.00	294.50	360.00	294.00	288.5	288.00	330.00	275.00
Cake obtained, g	651.00	720.00	790.00	674.00	640.0	658.00	610.00	590.00
Cake for Recovery, g	163.00	180.00	197.50	168.50	160.0	165.00	152.00	148.00
Product wax, g	80.00	83.00	97.50	78.50	79.0	82.00	76.00	77.00
Foots Oil, g	70.00	61.50	78.00	65.00	73.0	81.00	85.50	92.00
MIBK from Filtrate, g	951.00	926.00	1059.0	968.50	964.0	1000.0	1066.00	1001.0
MIBK from wax-cake, g	77.00	92.50	94.0	84.00	72.5	79.00	69.00	60.00

TABLE I.2.2 EXPERIMENTAL DATA FOR SECOND-STAGE DEOILING, SOLVENT-TO-SLACK WAX RATIO 3:1

DATA	14.32	15.32	16.32	17.32	18.32	19.32	20.32	21.32
RUN NO.								
SECOND STAGE DEOILING								
Weight of wax cake, g	488.00	540.00	592.50	505.50	480.00	493.00	458.00	442.00
Weight of MIBK, g	450.00	450.00	513.00	450.00	450.00	450.00	450.00	450.00
repulping	337.50	225.00	256.50	225.00	225.00	225.00	225.00	225.00
washing								
Initial slurry Temperature, °C	27.50	29.00	25.50	22.00	27.00	29.0	30.00	30.50
Final Deoiling Temperature, °C	5.00	5.00	10.00	10.50	15.00	15.00	19.50	19.00
Filtration/Washing Temperature, °C	6.00	5.50	10.50	10.50	15.00	15.50	20.50	21.00
Average Chilling Rate, °C/min	0.45	0.48	0.28	0.29	0.29	0.31	0.40	0.29
Vacuum Attained after 90 sec; mm Hg								
filtration	380.00	380.00	370.00	340.00	380.00	390.00	370.00	340.00
washing	390.00	390.00	375.00	330.00	380.00	390.00	380.00	370.00
Filtrate collected, g								
filtration	509.00	444.00	565.00	510.00	468.00	460.00	474.00	476.00
washing	283.00	174.50	180.00	215.00	173.00	185.00	188.00	187.00
Cake obtained, g	419.00	463.00	515.00	393.00	416.00	392.00	386.00	361.00
Product wax, g	230.00	235.00	262.50	218.50	216.50	217.00	207.50	206.50
Foots Oil, g	17.00	12.50	17.00	15.00	15.50	14.00	16.00	17.00
MIBK from filtrate	757.00	575.00	693.00	688.00	601.00	611.00	612.00	610.00
MIBK from wax-cake, g	159.00	187.50	147.00	147.00	163.00	145.00	131.00	122.00

APPENDIX II. SLACK WAX AND PARAFFIN WAX SPECIFICATIONS

Table II.1 Specifications of Barauni Slack Wax

Characteristics	Specifications
Melting point, °C	62-64
Density at 15°C with respect to water at 4°C	0.8930
Kinetic viscosity at 98.9°C, cst.	4.8400
Colour (ASTM)	4.5-5.5
Oil Content	9-13%

Table II. I.S. Requirements for Paraffin Wax :

Characteristics	Requirement for			
	Type I	Type 2	Type 3	Type 4
Melting point(cooling curve)	← 45° to 70°C →			
As percent by weight, Max	0.03	0.03	0.03	0.03
Acidity:				
(a) Organic (mg of KOH per g), Max.	0.10	0.10	0.10	0.10
(b) Inorganic (mg of KOH per g), Max	0.02	0.02	0.02	0.02
Saponification value, Max.	1.00	1.00	1.00	1.00
Oil content, percent by weight, Max.	0.25	0.25	0.50	3.50
Colour, Lovibond, in 18-in cell, Max	0.5γ	0.5γ	1.5γ	5.0γ
Colour, Lovibond, after acid treatment, in 1/4 in cell, Max	2.0γ+ 1.0R	-	-	-
Odour, Max.	1	-	-	-

APPENDIX III: WAX DEOILING MODELS AND SAMPLE CALCULATIONS

III.1. SIMPLIFIED MODEL FOR WAX DEOILING AND COMPUTED RESULTS

A simple model for wax deoiling is obtained when simplifying assumptions regarding crystallization and mixing characteristics of wax-oil-solvent system, product wax yield and cake porosity are made. The assumptions used in the model are as follows:

1. Slack wax consists of hard wax and oil alone. There are no soft waxes which may be removed during deoiling.
2. Hard wax has zero solubility in the solvent.
3. Wax agglomerates entrapping the oil are not formed during crystallization or filtration.
4. Deoiling solvent mixes completely and rapidly with oil in all proportions.
5. Oil-solvent solution in wax cake mixes completely and rapidly with repulp-and wash-solvent during repulping and washing operations respectively.
6. Wax cake porosity remains constant.

With above assumptions and the knowledge of slack wax oil content and the quantities of solvent used, the oil content in product wax of different stages of operation can easily be calculated. General procedure for a

batchwise deciling operation is given below:

Basis: Weight of Slack Wax Feed 100

weight of oil in the slack wax x

weight of hard wax in the slack wax 100-x

$$\text{weight of liquid retained by cake} = \frac{\rho_{s-o}}{\rho_w} \cdot \frac{\theta}{1-\theta} (100-x)$$

where θ is the wax cake porosity,

ρ_w is the specific density of the oil free wax and

ρ_{s-o} is the density of solvent-oil solution.

Since the quantity of oil in solvent-oil solution is small, ρ_{s-o} can be taken to be equal to the solvent density ρ_s , weight of liquid retained by cake is constant and can be designated α , such that

$$\alpha = \frac{\rho_s}{\rho_w} \frac{\theta}{1-\theta} (100-x)$$

Weight fraction of oil in the liquid retained after first-stage crystallization and filtration, for a solvent-to-slack wax ratio of n is $\frac{x}{100n+x}$

Oil content after first stage crystallization and filtration, as weight percent of oil free wax, is given by

$$\alpha \cdot \frac{x}{100n+x} \frac{100}{100-x} = \left[\frac{100x}{100n+x} \cdot \frac{\theta}{1-\theta} \cdot \frac{\rho_s}{\rho_w} \right]$$

On washing, for a wash solvent-to-slack wax ratio of n_w , percent oil content reduces to

$$\left[\frac{100x}{100n+x} \cdot \frac{\theta}{1-\theta} \cdot \frac{s}{w} \right] \cdot \frac{\alpha}{100n_w + \alpha}$$

Repulping and filtration, for a repulp solvent-to-slack wax ratio of n_R , reduce the oil percent to

$$\left[\frac{100x}{100n+x} \cdot \frac{\theta}{1-\theta} \cdot \frac{\rho_s}{\rho_w} \right] \left[\frac{\alpha}{100n_w + \alpha} \right] \left[\frac{\alpha}{100n_R + \alpha} \right]$$

Second-stage washing after repulping and filtration reduce the oil percent to,

$$\left[\frac{100x}{100n+x} \cdot \frac{\theta}{1-\theta} \cdot \frac{\rho_s}{\rho_w} \right] \left[\frac{\alpha}{100n_w + \alpha} \right]^2 \left[\frac{\alpha}{100n_R + \alpha} \right]$$

In general, for a multi-stage deoiling operations involving a total number of $(1+p+q)$ filtration operations corresponding to p repulp and q solvent wash operations, the product wax oil content Y , as weight percent of oil free wax is given by

$$Y = \left[\frac{100x}{100n+x} \cdot \frac{\theta}{1-\theta} \cdot \frac{\rho_s}{\rho_w} \right] \left[\frac{\alpha}{100n_R + \alpha} \right]^p \left[\frac{\alpha}{100n_w + \alpha} \right]^q$$

From the value Y the oil content y , as weight percent of product wax, can be readily calculated using the relationship

$$y = \frac{100 Y}{100+Y}$$

Table III.1 shows the estimated percent oil content values based on this model, for solvent-to-slack wax ratios of 2:1, 3:1 and 4:1 for different number of filtration and wash stages and for wax-cake porosity values of 0.4 and 0.5.

Table III.1 Estimated Percent Oil Content Values Based on the Simplified Model.

Porosity θ	Solvent -to- slack wax ratio n	Estimated values of oil content in Product wax, wt. %.					
		Crystall- ization and filtration	After washing	Repulping and Filtration	After wash- ing	2nd Repulp- ing and Filtration	After wash- ing
0.5	2:1	4.97	2.60	1.16	0.598	0.263	0.135
	3:1	3.43	1.79	0.62	0.318	0.110	0.096
	4:1	2.62	1.36	0.39	0.197	0.056	0.029
0.4	2:1	3.37	1.41	0.49	0.202	0.070	0.029
	3:1	2.32	0.96	0.25	0.104	0.027	0.011
	4:1	1.76	0.73	0.15	0.063	0.013	0.005

Repulp solvent-to-slack wax ratio $n_R = 0.5 n$

Wash solvent-to-slack wax ratio $n_w = 0.75$

III.2 MODIFIED MODEL FOR WAX-DEOILING BASED ON ACTUAL WAX CAKE AND YIELD VALUES AND COMPUTED RESULTS

More realistic values of product wax oil content are obtained than the values calculated from the generalised simplified model for deoiling of slack wax when the actual values of wax cake porosity and product wax yield are incorporated in the generalized model. General procedure for a batchwise two-stage deoiling operation is as given below:

Basis: Weight of slack wax feed 100

Weight of oil in the slack wax x

After first stage crystallization and filtration, the quantity of oil retained is given by,

$$\frac{x}{100n} (K_1 - Y_1) \text{ where } K_1 \text{ and } Y_1 \text{ are weights of cake obtained and product wax.}$$

After washing, weight of oil retained is given by

$$\frac{x}{100n} \cdot (K_1 - Y_1) \cdot \frac{K_1 - Y_1}{K_1 - Y_1 + 100 n_w}$$

After washing, weight of oil retained is given by

$$\frac{x}{100n} \cdot (K_1 - Y_1) \cdot \frac{K_1 - Y_1}{K_1 - Y_1 + 100 n_w}$$

The percent oil P_1 and cake porosity θ_1 from first-stage deoiling operation involving crystallization, filtration and a single wash is given by

$$P_1 = \left[\frac{100x}{Y_1(100n)} \right] \left[\frac{(K_1 - Y_1)^2}{K_1 - Y_1 + 100n_w} \right]$$

$$\theta_1 = \frac{(K_1 - Y_1) + .01 P_1 Y_1}{(K_1 - Y_1) + 0.01 P_1 Y_1 + \frac{\rho_s}{\rho_w} (Y_1 - 0.01 P_1 Y_1)}$$

ρ_w and ρ_s are specific weight of wax and the solvent respectively.

Oil remaining with cake after second-stage filtration is given by

$$\left(\frac{P_1 Y_1}{100} \right) \frac{(K_2 - Y_2)}{(K_1 - Y_1) + 100 n_R}$$

where, K_2 and Y_2 are weights of cake obtained and product wax from two-stage deoiling.

or

$$\frac{x}{100n} \cdot \frac{(K_1 - Y_1)}{(K_1 - Y_1) + 100n_w} \cdot \frac{K_2 - Y_2}{(K_1 - Y_1) + 100n_w}$$

Oil remaining after second-stage wash is given by

$$\frac{x}{100n} \cdot \frac{(K_1 - Y_1)^2}{(K_1 - Y_1) + 100 n_w} \cdot \frac{(K_2 - Y_2)}{(K_2 - Y_2) + 100n_w} \cdot \frac{(K_2 - Y_2)}{(K_1 - Y_1) + 100n_R}$$

The oil percent P_2 and cake porosity θ_2 of the product wax and wax cake obtained from two-stage deoiling operation is given by

$$P_2 = \frac{100X}{Y_2(100n)} \cdot \frac{(K_1 - Y_1)^2}{(K_1 - Y_1) + 100n_w} \cdot \frac{K_2 - Y_2}{K_2 - Y_2 + 100n_w} \cdot \frac{K_2 - Y_2}{K_1 - Y_1 + 100n_R}$$

$$= \frac{100X}{Y_2 [100n]} \cdot \frac{K_2 - Y_2}{\left[1 + \frac{100n_w}{K_1 - Y_1} \right] \left[1 + \frac{100n_w}{K_2 - Y_2} \right] \left[1 + \frac{100n_R}{K_1 - Y_1} \right]}$$

$$\theta_2 = \frac{(K_2 - Y_2) + .01 P_2 Y_2}{(K_2 - Y_2) + .01 P_2 Y_2 + \frac{\rho_s}{\rho_w} (Y_2 - .01 P_2 Y_2)}$$

Table 5.2 shows the estimated percent oil content values for one-stage and two-stage deoiling operations for solvent to slack wax ratios of 2:1 and 3:1.

III.3: SAMPLE CALCULATIONS

III.3.1 Calculation of Oil Content of the Product Wax from the Simplified Model

Oil content for a two-stage deoiling and solvent-to-slack wax ratio of 3:1 is calculated. for slack wax containing 12.4 percent oil and cake porosity 0.5.

$$X = 12.4$$

$$\theta = 0.5$$

$$n = 3.0$$

$$\rho_w = 0.893$$

$$\rho_s = 0.80$$

$$n_R = 0.5n = 1.5$$

$$n_w = 0.75$$

and for 2-stage deoiling operation,

$$p = 1, q = 2$$

$$\text{Therefore, } \alpha = \frac{\rho_s}{\rho_w} \cdot \frac{\theta}{1-\theta} (100-x)$$

$$= \frac{0.8}{0.893} \cdot \frac{0.5}{1-0.5} (100-12.4)$$

$$= 78.495$$

$$Y = \left[\frac{100X}{100n+Y} \cdot \frac{\theta}{1-\theta} \cdot \frac{\rho_s}{\rho_w} \right] \left[\frac{\alpha}{100n_R+\alpha} \right]^p \left[\frac{\alpha}{100n_w+\alpha} \right]^q$$

$$= \left[\frac{12.4(100)}{100(3)+12.4} \cdot \frac{0.8}{0.893} \right] \left[\frac{78.495}{100(1.5)+78.495} \right] \left[\frac{78.495}{75+78.495} \right]^2$$

$$= 0.319$$

Oil content as weight percent of product wax, y, is

$$y = \frac{100Y}{100+Y} = \frac{31.9}{100.319} = 0.318$$

III.3.2 Calculation of Oil Content of the Product Wax from the Modified Model

Run No.11.22 is selected for this calculation.

Weight percent of oil in slack wax $X = 12.4$

Wax feed = 402.5 g

Weight of cake obtained in first stage = 5.99 g

Weight Percent Yield $y_1 = 83.0$

Weight of cake used for repulp = 449

Weight of cake obtained in second stage = 370

Weight percent yield of wax product $Y_2 = 79.47$

Solvent-to-slack wax ratio $n = 2.0$

Wash solvent-to-slack wax ratio $n_w = .75$

$$K_1 = \frac{599}{402.5} (100) = 148.5$$

Oil content in product wax from first stage is

$$\begin{aligned} P_1 &= \left[\frac{100 X}{Y_1 (X + 100 n)} \right] \left[\frac{(K_1 - Y_1)^2}{K_1 - Y_1 + 100 n_w} \right] \\ &= \left[\frac{12.4 (100.0)}{83.0 (12.4 + 200)} \right] \left[\frac{(148.5 - 83.0)^2}{(148.5 - 83.0 + 75)} \right] \\ &= 2.30 \end{aligned}$$

Repulp solvent to slack wax ratio $n_R = 0.5 n = 1.0$

$$K_2 = \frac{370}{402.5} \cdot \frac{599}{549} - (100) = 122.2$$

Oil content in product wax from second stage is

$$\begin{aligned} P_2 &= \frac{100 X}{Y_2 (X + 100 n)} \cdot \frac{(K_1 - Y_1)^2}{(K_1 - Y_1) + 100 n_w} \cdot \frac{K_2 - Y_2}{K_2 - Y_2 + 100 n_w} \cdot \frac{K_2 - Y_2}{K_1 - Y_1 + 100 n_R} \\ &= P_1 \cdot \frac{Y_2}{Y_1} \cdot \frac{(K_2 - Y_2)^2}{K_2 - Y_2 + 100 n_w} \cdot \frac{1}{K_1 - Y_1 + 100 n_R} \end{aligned}$$

-XV-

$$= 2.30 \frac{79.47}{83.0} \cdot \frac{(122.2-79.47)^2}{122.2-79.47+75} \cdot \frac{1}{148.5-83.0+100}$$
$$= 0.229$$

First-stage wax cake porosity is:

$$\theta_1 = \frac{(K_1 - Y_1) + 0.01 P_1 Y_1}{K_1 - Y_1 + 0.01 P_1 Y_1 + \frac{\rho_s}{\rho_w} (Y_1 - 0.01 P_1 Y_1)}$$
$$= \frac{148.5 - 83.0 + 2.3(.830)}{148.5 - 83.0 + 2.3(.830) + \frac{.8}{.893} (83.0)(1 - .023)}$$
$$= .483$$

Second-stage wax cake porosity is:

$$\theta_2 = \frac{K_2 - Y_2 + 0.01 P_2 Y_2}{K_2 - Y_2 \left(1 - \frac{\rho_s}{\rho_w}\right) + 0.01 P_1 Y_1 \left(1 - \frac{\rho_s}{\rho_w}\right)}$$
$$= \frac{162.8 - 79.47 + 2.29(.7947)}{162.8 - 83.0 \left(1 - \frac{.8}{.893}\right) + 2.3(.83) \left(1 - \frac{.8}{.893}\right)}$$
$$= .421$$

C O N T E N T

CERTIFICATE

ACKNOWLEDGEMENT

CONTENTS

1. INTRODUCTION	... 1
2. LITERATURE REVIEW	... 4
3. EXPERIMENTAL SET-UP	... 13
3.1 Deoiling System	... 13
3.2 Solvent Recovery System	... 20
4. EXPERIMENTAL PROCEDURE	... 21
4.1 First-stage Deoiling	... 21
4.2 Second-stage Deoiling	... 23
4.3 Recovery of Solvent	... 24
4.4 Oil Content Analysis	... 25
5. RESULTS AND DISCUSSION	... 28
5.1 Product Wax Yield	... 30
5.2 Oil Content In Product Wax	... 33
5.3 Relationship between Oil Content and Product Wax Yield	... 38
5.4 Simplified Model For Wax Deoiling	... 39
5.5 Refractive Index and Oil Content	... 44
6. CONCLUSIONS AND RECOMMENDATIONS	... 46
6.1 Conclusions	... 46
6.2 Recommendations	... 48
REFERENCES	... 50
APPENDIX I. EXPERIMENTAL DATA	... -i-
APPENDIX II. SLACK WAX AND PARAFFIN WAX SPECIFICATIONS	... -vi-
APPENDIX III. WAX DEOILING MODELS	... -vii-

CHAPTER 1

I N T R O D U C T I O N

Presently there is a world-wide shortage of petroleum waxes. Due to their many and diverse applications, there is considerable interest in the production of these waxes from traditional sources as well as to find new sources for their manufacture. Petroleum refiners are concentrating more and more on improving the existing dewaxing and deoiling processes to increase the production of these petroleum products with suitable functional properties for varied end uses.

Processes employed for the production of refined wax are aimed at deoiling the petroleum wax concentrate known as slack wax, a co-product from dewaxing unit. These processes⁽¹⁾ are physical in nature and are similar to those used in dewaxing of lubricating oil fractions, often varying only in the choice of treating conditions. Solvent deoiling, like solvent dewaxing, is by far the most widely used process for the production of refined wax of low oil content.

Ketones have since long been described as good dewaxing and deoiling solvents by virtue of their low solubility for n-paraffin type waxes and relatively higher filtration rates⁽²⁾. Choice of a particular deoiling solvent depends upon its miscibility with oil, solubility of wax in it, rate of filtration, latent heat of evaporation, boiling

range stability, non-corrosiveness, water solubility, availability and its cost. (3)

The choice of operating variables, such as, deoiling temperature and solvent-to-feed ratio depends on the solvent, the nature of the feed-stock and the degree of refinement desired. Higher deoiling temperature results in a product of lower oil content but the wax yield is adversely affected. In opting for a higher solvent-to-feed ratio, possible gains in the degree of refinement achieved will have to be weighed against increased solvent losses. With methyl ethyl ketone, the range of deoiling temperatures commonly employed is -4° to $+21^{\circ}\text{C}$ and the solvent-to-feed ratio (volume/volume) varies from 1 to 8^(4,5). By proper utilization of techniques of incremental dilution and filtrate recirculation, fresh solvent-to-feed ratios as low as 1 to 2 can be used in a two stage process.⁽⁶⁾ Cooling is carried out very slowly since too rapid chilling results in poorly crystallized, bulky wax cake which crack badly during filtering and wash unevenly.⁽⁵⁾

The aim of the present investigation was to study the feasibility of using methyl-isobutyl-ketone which offers many performance advantages over methyl ethyl ketone (MEK) and MEK-aromatic mixtures, to produce wax of low oil content from the Barauni slack wax. Barauni refinery, which processes Nahorkatiya crude of paraffinic type, is expected to produce 75,000 tons per year of

CHAPTER 2

LITERATURE REVIEW

Paraffin wax is a purified mixture of solid hydrocarbons obtained from petroleum. It is more or less a translucent mass, either colourless or white, showing a crystalline structure without odour or taste with slightly greasy touch. Paraffin wax is an important petroleum product with widely different commercial application. It has been observed these waxes usually consist of upto 90 percent n-paraffin hydrocarbons with some iso-paraffins (10 to 20 percent) and less than 5 percent cycloparaffins in the range of C_{18} to C_{32} and solidifying between $27.0^{\circ}C$ and $68.9^{\circ}C$. (7,8)

Paraffin wax from petroleum was originally an unavoidable by-product obtained during dewaxing of lubricating oil fractions. Currently, because of its varied end uses it is also obtained as a primary product by dewaxing certain petroleum fractions and cracking the dewaxed portion to produce gasoline. However, most of the wax is still produced as a co-product of lubricating oil.

(9)

Paraffin wax is mainly used in the manufacture of waxed papers, waxed cartons and candles. Other consumers of paraffin wax include matches, fruit and vegetable coatings, cosmetics, carbon paper, preventives, electrical and communication equipment, and pharmaceutical, textile and leather industries. It is also used to manufacture

slack wax. This slack wax can be utilized to produce high melting point hard paraffin wax. Since the composition of different stocks varies with respect to type and distribution of various kinds of hydrocarbons and relative proportion of n-paraffins, iso-paraffins and the carbon distribution of non-paraffinic portion, it becomes essential that the possible application of a new solvent should be carefully investigated for each type of feed-stock.

In the present investigation, a total of twenty one runs were carried out at four different deoiling temperatures, viz. 20°, 15°, 10° and 5°C and for two solvent-to-feed ratios, volume/volume, of 2 and 3. From the experimental data, attempt has been made to determine the optimum conditions for the production of refined wax meeting Indian Standard specifications.

chlorinated paraffin wax and as a chemical raw material. Chlorinated paraffin wax is mainly used as plasticizer or extender, flame proofing agents and extreme pressure lubricants.

Crude petroleum on distillation at atmosphere pressure gives various fractions differing in boiling point and viscosity. Gaseous high ends, gasoline, naphtha, kerosene and gas oil fractions are removed in this order. The bottoms from the atmospheric distillation unit is further distilled under vacuum to obtain wax distillate and cylinder stock. The wax distillate obtained from paraffinic crudes consists of a mixture of light bodied oils and various wax fractions with nearly the same boiling range. Consequently it is not possible to separate them by distillation.

Wax distillate, the raw stock for the manufacture of light lubricating oils may contain upto 30% of paraffin wax and must be freed from it in order to fulfill pour point requirements for lubricating oils. Most oils are dewaxed to a pour point of 25°C or lower for use as lubricants. The wax is removed by chilling the distillate, generally mixed with a solvent, and filtering the wax. The petroleum wax concentrate obtained from dewaxing is known as slack wax and contains 10 to 50 percent oil⁽¹⁾. Slack wax is of little value as a speciality product because of its high oil content and presence of low melting point soft waxes and colour bodies. In general the

oil content of slack wax increases with the viscosity of feed-stock even though higher solvent dilution ratios are used with heavier stocks during dewaxing.

Commercial processes ^(11,14) in use for deoiling the slack wax are sweating, emulsion deoiling, spray deoiling and solvent deoiling. Sweating process is less efficient fractional melting method with poor yields and the product wax, in general, fails to meet the specifications of refined wax for most uses. However, the investment is low and more economical for small capacity units than the solvent deoiling process. Emulsion deoiling is also a low cost process from which semi-refined waxes can be obtained. In spray deoiling process, slack wax is first melted and atomized to form a dry powder with oil adhering to the surface. The adhering oil is dissolved in a solvent using a system of mixer-settlers and the wax from the last settler is freed from the solvent by passing it through a centrifuge and an evaporator. The process becomes uneconomical for feedstock capacities larger than 400 barrels per day. ⁽¹²⁾

Modern deoiling units employ selective solvents for better separation. It is now well established that the use of an appropriate solvent under proper conditions results in the improved recovery of valuable high melting point wax components on crystallization in an oil free condition. In this operation, molten slack wax is mixed with solvent, chilled indirectly in scraped surface

exchangers and chillers, wax crystals separated on a rotary filter and the solvent recovered from products by distillation: It is desirable to have large average crystal size, a few or no fines and dense crystals which occlude no oil. For good crystal formation, the wax is crystallized from its solution in the oil-solvent mixture, slowly enough, partly in scraped surface exchangers and partly in scraped surface chillers. In order to prevent the formation of fine crystals due to shock chilling, the solvent injected as incremental dilution at several points between the elements of the scraped surface exchangers and chillers is always close to the temperature of the slurry at the point of injection. The amount of solvent used should be only as much as is necessary to reduce the viscosity of the resulting oil-solvent solution to the desired value and additional solvent used for washing or redispersing. The deoiling filter operates at a higher temperature than dewaxing filters to dissolve out the low-melting-point soft waxes which are not desired in refined wax.

The suitability of a solvent for deoiling depends on following desirable characteristics: complete miscibility with the oil present in the crude wax, very low solubility for the wax, absence of swelling or solvation of the wax crystals, low boiling point and stability with respect to decomposition on distillation.

Solvents suggested for fractional crystallization of wax are similar to those recommended for dewaxing oils which may consist of a single solvent or a mixture of solvents.⁽¹³⁾ The solubility characteristics of single solvents are inflexible and can be controlled only by varying the solvent-to-feed ratio and the temperature. Propane, trichloroethylene and methyl-butyl-ketone are examples of single dewaxing solvents used commercially. The use of solvent mixtures permits altering their solvent characteristics by varying their composition. Many combinations of aromatic solvents acting as anti-solvents, and miscible solvents have been suggested such as benzol in combination with acetone, furfural, nitrobenzene, picoline, pyridine or sulfurdioxide.

Ketones have since long been described as good dewaxing and deoiling solvents⁽²⁾. Methyl ethyl ketone and its mixtures with upto 70 percent aromatics such as benzene and toluene have been widely in use. Lower ketones have the advantage of possessing higher filtration rates and low solubility for wax but their solubility in water and vapour pressures are relatively higher. Their oil solubility characteristics are also poor, thus necessitating the use of another solvent to hold the oil in solution at low dewaxing temperatures. In MEK-aromatic process, benzene and toluene act as solvent for oil and the ketone as the wax precipitant. But use of aromatics with lower ketones implies loss of the advantage of high

filtration rates. On the other hand, with increase in molecular weight of the ketone, the solubility of oil in the ketone increases but at the same time ketone loses the advantage of low wax solubility. Higher ketones being viscous, rate of filtration decreases with the increase in molecular weight.

Recent studies on the use of larger molecule ketones have shown that the methyl n-propyl ketone, diethyl ketone and methyl isobutyl ketone are the three most attractive higher ketone solvents. They show better performance as dewaxing solvents than MEK-aromatic mixtures. The actual magnitude and value of this improved performance depends on a comparison of the two types of solvents in a specific refining situation⁽²⁾. Butler and Maeleod⁽¹⁵⁾ observed that an anhydrous mixture containing 70 percent methyl isobutyl ketone and 30 percent methyl-ethyl-ketone gives a performance essentially equivalent to that obtained with the much more expensive, yet, up until now technically preferred methyl n-propyl ketone.

The use of MEK-aromatic or MEK-MIBK mixtures require a complex solvent recovery system. The potential advantage of methyl iso-butyl ketone as a deoiling solvent is the reduced complexity of the solvent recovery system because water saturated methyl isobutyl ketone can be used effectively in place of anhydrous methyl ethyl ketone. Increased solvent miscibility with higher

molecular weight foots oil, reduction in solvent consumption due to reduced volatility of MIBK, lower solvent content of rejection water, reduced steam consumption due to low values of latent heat of vaporization and specific heat, elimination of salt and drying steps and indigenous availability make water saturated MIBK very close to an ideal deoiling solvent. (16)

Work has been carried out at the Indian Institute of Petroleum, Dehradun to investigate the feasibility of using methyl-isobutyl ketone for the dewaxing and deoiling operations. Kumar, Ghosh and Gulati⁽¹⁷⁾ carried out a comparative study of MIBK and MEK-Benzene-Toulene mixture (40:30:30 by volume) for the dewaxing of Nahorkatiya low and medium viscosity index type lube raffinates produced by the Barauni Refinery. The important conclusions reached by these workers are as under:

— The substitution of MEK-aromatics by MIBK as dewaxing solvent can reduce refrigeration cost due to much lower value of temperature differential (dewaxing temperature-pour point):

— The MEK-aromatic mixtures require higher dilution ratio as compared to MIBK particularly when heavy stocks are dewaxed.

— Even though MIBK has higher viscosity compared to MEK-aromatic mixtures, it offers comparable filtration rates because of the higher dewaxing temperatures.

— The yield and quality of the products obtained with MIBK and MEK-aromatic mixture are comparable.

Kumar and coworkers⁽¹⁸⁾ studied the production of microcrystalline waxes from heavy residual slack waxes produced by Haldia Refinery during the processing of residual lube stock and from the wax-like solids from the tank bottoms at Ankleshwar, Kalol and Navagam oil fields. Good quality microcrystalline waxes of different grades were obtained by these workers using MIBK as a deoiling solvent.

Gudelis et.al. have reported a new development⁽¹⁹⁾ in the field of wax refining. They have developed the Dilchill Dewaxing Process for ketone dewaxing based on direct heat exchange with cold solvent in a highly sheared environment. They observed that highly discrete and stable wax crystal agglomerates are formed by this process which facilitated the separation of wax from oil at high yields and high throughput. Relatively large compact crystals with very little occluded oil were obtained and the soft wax was found to be deposited conceptually in the form of layers on the nucleus of hard wax in the centre. They further reported that the outer layers of the crystals consisting of soft waxes can be removed during warm-up deoiling operation without an intermediate deoiling operation. They claim lower investment, higher dewaxed oil

yields, potential ability to make refined wax directly without recrystallization and reduced maintenance and steadier plant operation as the key advantages with the Dilchill Solvent Dewaxing-Deoiling Process.

CHAPTER 3 EXPERIMENTAL SET UP

Solvent deoiling studies are carried out batch-wise in a bench scale unit consisting of the following:

1. Deoiling System,
2. Solvent Recovery System.

3.1 DEOILING SYSTEM

Solvent deoiling experiments are carried out in batch unit capable of processing nearly one kilogram of slack wax. The unit is similar to that described by Kumar et.al. (17,18). It consists of an externally heated wax melting vessel, a jacketed solvent tank, a scraped surface chiller, a filtration vessel and a filtrate receiver as shown schematically in Figure 3.1.

The following is the description of the important component units.

3.1.1 WAX MELTING VESSEL: A stainless steel vessel of approximately 1.8 litre capacity, about 15 cm. inside diameter and about 10 cm deep is used as the vessel for wax melting. It is fitted with a 400 watt heating mantle. A variac (voltage regulator) is used for controlling the heating rate. The vessel is provided with a cover which is kept in position with the help of six machine screws. A half inch outside diameter stainless steel pipe

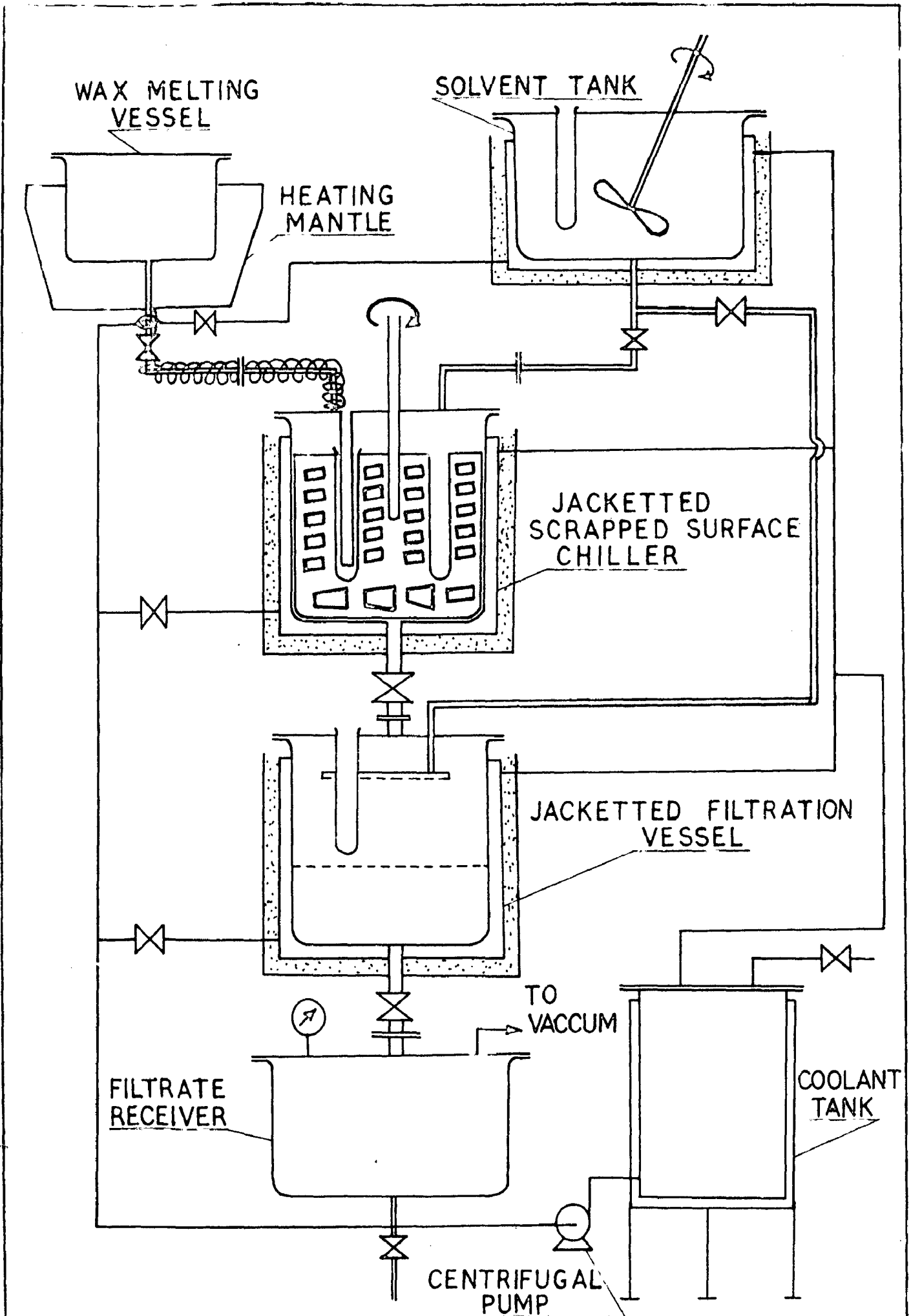


FIG.3.1 BENCH SCALE DEOILING UNIT

welded at the bottom is used to transfer molten wax by gravity to the scraped surface chiller. A brass glove valve is used to regulate the transfer of wax and the two vessels are connected with the help of a set of flanges. The transfer line has been provided with an external electric heating coil to prevent the clogging of the line due to the solidification of wax. A variac is used to control the rate of heat supply. Stirring during melting of the wax was not practised because it resulted in unwanted foaming. Wax temperature is measured with the help of a thermometer by inserting it through a central hole provided in the cover.

3.1.2 SOLVENT TANK: A five litre capacity and about 23 cm. inside diameter and 13 cm. deep stainless steel cylindrical vessel with mild steel jacket is used as a solvent tank. A half inch diameter stainless steel pipe welded at the bottom of the vessel is used to transfer solvent by gravity to the chiller. A brass glove valve is used to control the flow of solvent and the two vessels are connected with the help of a set of flanges. The transfer line is insulated with a half inch layer of glass wool.

The vessel is provided with a stainless steel cover and twelve machine screws are used for tightening. The cover is provided with two holes, one for stirrer and the other to transfer solvent into the vessel, and a 1/4 inch outside diameter stainless steel thermocouple

well. The solvent is cooled by circulating chilled water through the jacket. A variable speed stirrer is used to improve the heat transfer rate and to maintain chilled solvent at uniform temperature. A stirrer equipped with a fractional horse power universal motor is used and its speed is controlled with the help of a speed regulator. Temperature is measured with the help of a copper constantan thermocouple placed inside the thermocouple well containing small quantity of mercury for proper contact. The level of the solvent inside the vessel is read with the help of a glass tube level gauge.

3.1.3 SCRAPED SURFACE CHILLER: A five litre capacity and about 19 cm. inside diameter and 19 cm. deep stainless cylindrical vessel with mild steel jacket is used as a chiller. Wax-solvent mixture is cooled by circulating chilled water through the jacket. Inside the chiller, a stainless steel slotted scraper blade rotates so as to scrape the inner surface of the vessel. The clearance between the scraper blade and the chiller surface is kept to a minimum in order to prevent the build-up of wax layer during crystallization on the inner surface of the chiller. Build up of wax layer adversely affects the heat transfer rate. Rate of cooling during crystallization is controlled by varying the temperature and the rate of coolant recirculation through the jacket which is insulated on the outside with about one inch layer of

glass wool.

The vessel is provided with a stainless steel cover and the cover is kept in position using twelve 1/8 inch machine screws. A hole is provided in the centre of the cover for the rotating scraper blade shaft. Two half inch outside diameter stainless steel tubes are welded to the cover and are connected to the transfer lines coming from the wax melting vessel and the solvent tank. The stainless steel cover has two rectangular one and half inch by two inch slots and these are covered with perspex. These slots are used as peep-holes to watch the formation and growth of the crystals and the movement of the scraper blade during the run. One of the slots is also used to transfer the wax cake obtained from first stage crystallization and filtration for repulping with fresh solvent. In order to determine the temperature of the solvent-wax slurry at any time during the crystallization, a copper-constantan thermocouple has been provided. The thermocouple is placed in an open end 1/4 inch outside diameter stainless steel tube welded vertically to the cover. The scraper blade is provided with two vertical slots of suitable dimensions so that its rotation is not obstructed by the thermocouple well.

A 0.5 horse power motor fitted with a reduction gear and pulley system is used as a drive for the scraper blade. Three-jaw chuck is used to hold the

scraper blade shaft and its speed could be fixed at either 16 or 32 or 64 revolutions per minute.

A one inch inside diameter stainless steel pipe has been welded at the bottom of the chiller to connect, using a set of flanges, the chiller with the filtration vessel. A gate valve is provided to regulate the transfer of wax-solvent slurry. The wax-solvent slurry is transferred by gravity from the chiller to the filtration vessel.

3.1.4 FILTRATION VESSEL: This is also a five litre capacity and 19 cm. inside diameter and 19 cm. deep stainless steel cylindrical vessel with mild steel jacket. The vessel is provided with a stainless steel cover which is kept in position with the help of twelve 1/8 inch machine screws. The pipe connecting the vessel with the chiller is welded with the cover. A similar pipe is welded at the bottom of the filtration vessel to connect it to the filtrate receiver.

Inside the vessel, a perforated stainless steel plate with five millimeter holes at eight centimeter apart in a triangular pitch has been welded at a distance of six centimeters from the bottom of the vessel. Filter cloth, cut to proper size, is placed over the perforated plate and is kept in position with the help of a stainless o-ring, placed over the filter cloth, and six 1/8 inch brass machine screws.

A 1/4 inch outside diameter stainless steel tube with open ends is welded to the inside of the cover to keep a copper-constantan thermocouple for temperature measurement during filtration. The thermocouple bead is kept outside the tube at a distance less than half centimeter above the filter cloth so that the temperature could be measured during filtration accurately. Filtration temperature is maintained by controlling the temperature and the flow rate of coolant circulating through the jacket which is insulated on outer side with about one inch layer of glass wool.

A 15 cm. diameter ring made of 1/4 inch outside diameter copper tube with seventeen 1/16 inch holes and 8 cm. straight vertical end is fitted to the cover. The ring is positioned about 5 cm below the filtration vessel cover and is used to spray solvent above the wax cake for washing operation.

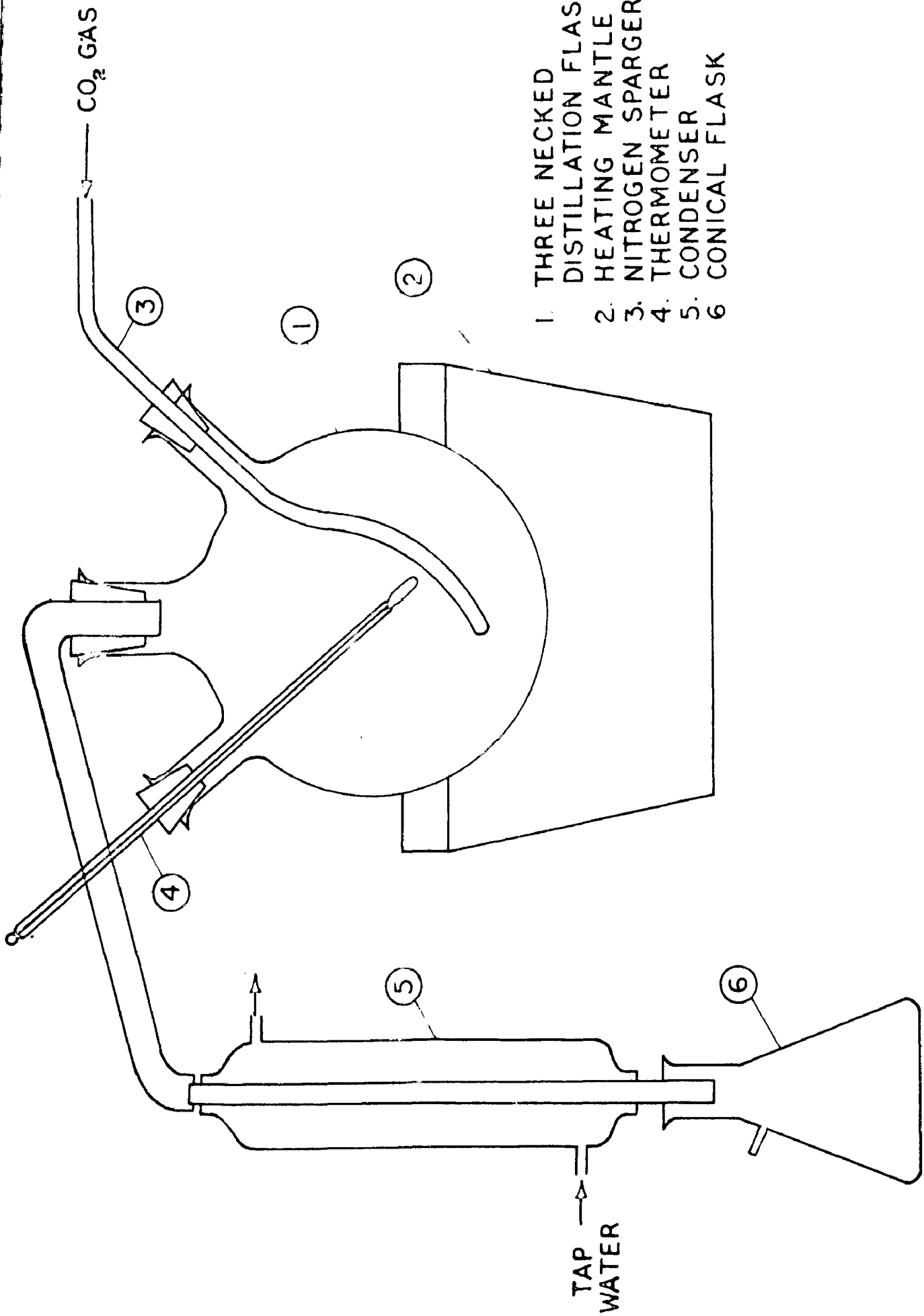
3.1.5 FILTERATE RECEIVER: A five litre capacity and about 23 cm. inside diameter and 13 cm deep cylindrical stainless steel vessel is used as filtrate receiver. It has been provided with a leak proof stainless steel cover secured tightly with the help of twelve machine screws and backing flanges using a rubber gasket in between. It has been placed just below the filtration vessel. A one inch diameter stainless steel pipe is welded with the cover to connect it to the bottom of the filtration

vessel, using a set of flanges. A brass gate valve is also provided in the transfer line to regulate the flow.

A stainless steel half inch outside diameter pipe is welded to the cover to connect the filtrate receiver to a portable vacuum pump. To facilitate the filtration, a vacuum pump of approximately 360 mm of Hg is maintained inside the vessel. A vacuum gauge is provided to read the actual value of the vacuum during each filtration run.

A half inch diameter stainless steel pipe with a brass gate valve in between is welded at the bottom of the vessel and is fitted with a brass gate valve. The filtrate is removed from the filtrate receiver by opening this valve at the end of the filtration after breaking the vacuum.

3.1.7 TEMPERATURE MEASUREMENT SYSTEM: The temperatures of methyl-isobutyl-ketone in the solvent tank, wax-solvent slurry in the chiller and the wax cake in the filtration vessel are measured by separate copper-constantan thermocouples provided for the purpose. The thermocouples are connected to a six point Toshniwal selector switch to facilitate easy measurement of the desired temperature at any time. The temperature of the molten slack wax and liquid temperatures during distillation for solvent recovery are directly measured by thermometers of suitable range.



1. THREE NECKED DISTILLATION FLASK
2. HEATING MANTLE
3. NITROGEN SPARGER
4. THERMOMETER
5. CONDENSER
6. CONICAL FLASK

FIG 3.2 SET UP FOR SOLVENT RECOVERY

3.2 SOLVENT RECOVERY SYSTEM:

The deoiled slack wax cake as well as the filtrate containing oil, low melting soft waxes and solvent are obtained during deoiling experiments and are stripped off the solvent by distillation using inert stripping gas. It is essential to strip the solvent from the wax and the filtrate to get the product wax of desired specifications and also to recover the valuable solvent.

Recovery of the solvent from the wax and the filtrate is done by separate distillation under nitrogen blanket to prevent oxidation. The use of nitrogen as stripping gas also permitted more or less complete removal of solvent at temperatures below 130°C . The solvent recovery system is shown schematically in Figure 3.2. It consists of a three necked distillation flask with a thermometer of $0-200^{\circ}\text{C}$ range, a heating mantle and a sparger for nitrogen, a glass condenser to condense the solvent vapours and a solvent receiving flask. Tap water is allowed to flow continuously through the condenser to condense essentially all of the MIBK vapours. Nitrogen flow rate is suitably controlled to limit the temperature during distillation below 130°C in order to prevent deterioration in wax properties. The heating rate is controlled by using a variac to prevent excessive boiling.

CHAPTER 4

EXPERIMENTAL PROCEDURE

4.1 FIRST STAGE DEOILING

A representative sample of approximately 400 grams of slack wax is taken for a bench scale deoiling experiment. In order to break the existing crystal structure, slack wax in a glass beaker is heated to a temperature 10 to 15°C above its melting point by placing it over a heating mantle and then the molten wax sample is transferred to the wax melting vessel. The exact weight of slack wax feed is determined by noting the weight difference of beaker, thermometer and wax before and after the wax sample is transferred. Measured quantity of solvent is heated separately to 45°C and is maintained at that temperature, after transferring it to the chiller, by circulating hot water through its jacket. The amount of solvent taken initially depends upon the solvent-to-feed ratio and the weight of the slack wax sample used for the run. Melted slack wax is transferred to the chiller while the scraper blade is rotating. Initial solvent temperature of 45°C is found to be sufficient to keep the wax-oil-solvent mixture in a completely homogeneous liquid phase before the solution is chilled under controlled rate of cooling for wax crystallization.

The additional quantities of dilution solvent are added at desired temperatures and stages of crystallization. It is usual to add the first dilution solvent increment at a stage where crystallization process has

just set in. A preliminary run indicated that it is desirable to add first dilution solvent increment when the temperature in the chiller falls to 30°C. Since the temperature of the solvent is close to 30°C, it is added without any precooling. The second dilution solvent increment is added at the final deoiling temperature after precooling the solvent in the solvent tank close to the final deoiling temperature.

On attaining the desired deoiling temperature and after adding the final dilution solvent increment, the coolant recirculation rate is reduced just sufficient to keep the temperature constant. Slurry is allowed to remain at this temperature in the chiller for about twenty minutes to crystallize the wax into well formed crystals. The slurry is then transferred to the filtration vessel which is kept cool at the desired temperature by circulating the coolant through its jacket. It is important that the temperature of the slurry does not change much during the transfer or during the filtration. Filtration is carried out under vacuum by connecting the filtrate receiver directly to a 0.25 horsepower portable vacuum pump. Filtration is carried out for 90 seconds and filtrate is removed from the filtrate receiver after breaking the vacuum. The quantity of the filtrate collected is measured.

For washing, measured quantity of wash solvent maintained at the final deoiling temperature is sprayed

over the wax cake in the filtration vessel. Filtration is again carried out for 90 seconds and the wash filtrate is similarly removed and measured.

Of the total solvent used for deoiling, two or three times the quantity of slack wax for solvent-to-wax ratios of 2:1 or 3:1 respectively, fifty percent is taken initially and twenty five percent each is added as the first and second dilution solvent increments. The ratio of wash solvent-to-slack wax is taken as 0.75:1.

The cake is scraped off from the filter cloth and a portion, normally twenty five percent of the wax cake obtained, is removed to find the oil content and other properties of the wax obtained from a single stage deoiling and washing.

4.2 SECOND STAGE DEOILING

Approximately seventy five percent of the wax cake obtained from first stage deoiling is transferred to the chiller through one of the rectangular slots provided for this purpose in the chiller cover. Measured quantity of solvent, one or one and half times the quantity of slack wax for solvent-to-wax ratios of 2:1 or 3:1 respectively, at the final deoiling temperature is added for second stage deoiling. The slurry is maintained at this temperature for nearly twenty minutes and then transferred to the filtration vessel for filtration and washing as described previously. The wax

cake from the second stage deoiling is scraped off from the filter cloth and is used to find oil content and other properties.

4.3 RECOVERY OF SOLVENT

The quantities of the deoiled wax and footsoil (or the reject stock) are obtained from first-stage or two-stage deoiling after stripping off the solvent from the wax cake and the filtrate respectively. Recovery of solvent as completely as possible is also important from the point of view of overall economics of the process. The wax cake and the filtrate obtained from different deoiling stages are separately distilled using nitrogen as the stripping gas. The rate of the heat input and the nitrogen flow rate are controlled to prevent excessive boiling as well as to maintain distillation temperature below 130°C . For complete recovery of the solvent, nitrogen flow rate is increased towards the end of the distillation and the distillation is continued till solvent drops cease to appear at the condenser exit. After the complete recovery of the solvent, exact weight of the deoiled wax and the footsoil is determined by subtracting the weight of the empty flask from the weight of the distillation flask containing wax or footsoil.

The samples of the deoiled wax obtained after distillation are kept in glass bottles for subsequent

analysis to determine the oil content and other physical properties of the product wax such as melting point, refractive index and viscosity.

4.4 OIL CONTENT ANALYSIS

Oil content analysis of the deoiled wax samples has been carried out at the Indian Institute of Petroleum, Dehradun. Test procedure described in Joint ASTM-IP Standard No. ASTM D721-IP 158/66T and adopted in IS:4654-1968 Specification For Paraffin Wax⁽²¹⁾, is followed for the determination of oil content. As outlined in the method, the sample is dissolved in methyl ethyl ketone, the solution cooled to -32°C to precipitate the wax and filtered. The oil content is determined by evaporating the methyl ethyl ketone and weighing the residue.

CHAPTER 5

RESULTS AND DISCUSSION

Barauni slack wax normally contains 10-13% oil and has a melting point in the range of 62-64°C. The general specifications are given in the Table II.1 in Appendix II. High melting point and predominantly paraffinic nature of the base stock indicates that the amount of soft waxes must not be large and a good quality hard paraffin wax could be obtained on removal of oil. Since the melting point of the slack wax already meets the specifications of all the four types of paraffin waxes given in IS:4654. Indian Specification for Paraffin Wax⁽²¹⁾, Table I.2 in Appendix I, the deoiling conditions are primarily chosen with the aim of achieving the product quality with respect to oil content.

The oil content of the Barauni slack wax sample used for experimental investigations is 12.4 percent⁽²²⁾. Details of the experimental data obtained from laboratory bench scale deoiling experiments are given in Table II.1 and II.2 in Appendix II. The data is collected for four different deoiling temperatures, 5°, 10°, 15° and 20°C and two different solvent-to-slack wax ratios, 2:1 and 3:1. Experimental data and calculated results for two-stage deoiling experiments, Run Nos. 5-22 to 21.32 are summarized in Table 5-1. The data obtained during the first four trial runs, Run Nos. 1.22 to 4.22, for single stage deoiling experiments are not included in Table 5.1.

TABLE 5.1 I- EXPERIMENTAL DATA AND RESULTS FOR SOLVENT-TO-SLACK WAX RATIO 2:1

RUN NO.	12.22	11.22	10.22	9.22	8.22	7.22	6.22	5.22	13.22
FIRST-STAGE DEOILING									
Final Deoiling Temperature, °C	5.00	5.00	10.00	10.50	15.00	15.50	14.50	19.00	19.00
Solvent/slack wax ratio (by wt.)	2.00	2.00	2.00	2.00	1.50	2.00	2.00	2.00	2.00
initially at 45°C	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00
1st increment at 30°C	0.50	0.50	0.50	0.50	nil	0.50	0.50	0.50	0.50
2nd increment at deoiling temp.	0.50	0.50	0.50	0.50	0.50	0.50	0.50	0.50	0.50
Wash solvent/slack wax ratio (by wt.)	0.75	0.75	0.75	0.75	0.75	0.75	0.75	0.75	0.75
Av. rate of chilling, °C/min									
initial to 300C	1.04	0.98	1.05	0.86	0.60	0.84	0.51	0.46	1.09
300C to deoiling temp.	0.31	0.31	0.31	0.29	0.27	0.19	0.15	0.15	0.33
Wax yield, wt.-% slack wax									
actual	73.82	78.87	77.40	75.31	75.25	78.40	76.50	76.19	74.59
corrected	84.42	83.00	81.09	80.96	78.83	79.05	79.48	77.48	80.10
Foots oil yield, wt.-% slack wax									
actual	13.43	16.15	18.06	17.71	20.20	20.75	76.00	22.13	18.52
corrected	15.58	17.00	18.91	19.04	21.17	20.95	76.00	22.52	19.90
Product wax oil content, wt.-%	N.A.	2.08	1.83	N.A.	3.30	N.A.	N.A.	N.A.	4.36
TWO-STAGE DEOILING									
Deoiling Temperature, °C	5.00	5.00	10.00	10.00	15.00	15.00	15.00	19.00	19.00
Repulp solvent/slack wax ratio (by wt.)	1.00	1.00	1.00	1.00	1.00	1.00	1.00	0.50	1.00
Wash solvent/slack wax ratio (by wt.)	1.125	0.75	0.75	0.75	0.75	0.75	0.75	0.75	0.75
Wax yield, wt.-% slack wax									
Actual	73.90	79.22	79.46	72.32	69.70	74.52	76.00	70.10	72.76
corrected	79.28	79.47	78.36	76.83	72.88	74.80	76.00	73.35	74.92
Foots oil yield, wt.-% slack wax									
actual,	19.31	20.46	21.93	21.83	25.93	25.10	24.00	25.55	24.36
corrected	20.72	20.53	21.64	23.13	27.12	26.65	24.00	25.08	25.08
Product wax oil content, wt.-%	1.50	1.38	1.10	1.64	0.79	0.51	N.A.	1.52	1.12
Total solvent loss, % solvent used.	80.90	81.70	75.30	77.00	80.90	78.20	97.60	83.90	76.20

N.A. = not analysed.

Table 5.1 II-EXPERIMENTAL DATA AND RESULTS FOR SOLVENT-TO-SLACK WAX RATIO 3:1

	14.32	15.32	16.32	17.32	18.32	19.32	20.32	21.32
RUN NUMBER								
FIRST-STAGE DEOILING								
Final deoiling temperature, °C	5.00	5.00	10.00	10.00	14.50	15.00	19.50	19.00
Solvent/slack wax ratio (by wt.)	3.00	3.00	3.00	3.00	3.00	3.00	3.00	3.00
initially at 45°C	1.50	1.50	1.50	1.50	1.50	1.50	1.50	1.50
1st increment at deoiling temp.	0.75	0.75	0.75	0.75	0.75	0.75	0.75	0.75
2nd increment at deoiling temp.	0.75	0.75	0.75	0.75	0.75	0.75	0.75	0.75
Wash solvent/slack wax ratio (by weight)	0.75	0.75	0.75	0.75	0.75	0.75	0.75	0.75
AV. rate of chilling, °C/min	1.15	1.20	0.92	1.05	0.96	0.80	0.65	0.82
Initial to 30°C	0.29	0.28	0.21	0.25	0.25	0.24	0.33	0.29
30°C to deoiling temp.	79.88	82.79	82.11	79.49	81.20	81.34	77.02	76.93
Wax yield, wt. %	82.09	84.37	82.74	82.86	81.24	80.11	78.12	76.94
actual	17.50	15.34	17.12	16.46	18.75	20.15	21.59	23.06
corrected	17.91	15.63	17.26	17.14	18.76	19.89	21.88	23.06
Product wax oil content, wt. %	N.A.	N.A.	N.A.	N.A.	N.A.	N.A.	N.A.	N.A.
TWC-STAGE DEOILING								
Decoiling Temperature, °C	9.00	5.00	10.00	10.50	15.00	15.00	19.50	19.00
Repulp solvent/slack wax ratio (by wt.)	1.50	1.50	1.50	1.50	1.50	1.50	1.50	1.50
Wash solvent/slack wax ratio (by wt.)	1.125	0.75	0.75	0.75	0.75	0.75	0.75	0.75
Wax yield, wt. %	76.71	78.14	76.84	73.76	74.21	72.05	69.79	69.08
actual	76.80	79.92	77.33	77.44	75.51	74.40	71.40	70.63
corrected	23.17	19.49	22.10	21.52	24.08	24.80	26.97	28.74
Product wax oil content, wt. %	23.20	20.08	22.67	22.56	24.49	25.60	28.60	29.37
Total solvent loss, %	N.A.	0.39	N.A.	0.46	N.A.	0.48	N.A.	0.37
actual	85.00	81.90	83.30	86.80	83.70	84.40	86.30	82.40
corrected								
Product wax oil content, wt. %								
Total solvent loss, %								

N.A. - Not Analyzed.

The actual total yield of product wax and oil varied from 95 to 101.5 percent weight of the slack wax used except for Run Nos. 9.22 and 12.22 which show yield values of less than 95 percent. The average yields for all the seventeen runs reported in Table 5.1 are 96.81 and 97.64% wt. slack wax for 1-stage and 2-stage respectively. The yield values are slightly lower for first-stage deoiling operations as compared to two-stage deoiling operation because only twenty five percent of the wax cake obtained after the first-stage filtration and washing is used for product wax recovery as against seventy five percent used for repulping and second stage filtration, washing and product wax recovery. Considering the number of operations involved in deoiling experiments and the problems associated with the handling of thick slurry, the above yield values are quite satisfactory.

The Table 5.1 also gives the corrected values of product wax and foots oil yield by neglecting the losses. These corrected values are more meaningful than the actual values because they neglect the error of measurements and the losses in material handling.

As indicated previously in Section 4.4, the oil content analysis of the deoiled wax sample is carried out at the Indian Institute of Petroleum, Dehradun. Oil content analysis of each sample takes nearly six hours and require large quantities of solid carbon dioxide (dry ice)

for chilling upto -32°C . Due to time consuming and expensive nature of the analysis and because of non-availability of the facilities in our laboratory, only sixteen deoiled wax samples out of a total of 38 samples could be analyzed for the oil content. Even for these samples duplicate analysis could not be carried out to check the reliability of the analytical results. It is also important to note that the accuracy of oil content analysis is quite low for wax of low oil content. The I.S. Specification for Paraffin Wax⁽²¹⁾ gives the following values for the repeatability and reproducibility of the analytical results for oil content:

Repeatability = $\pm(0.06 + 8 \text{ percent of mean value})$

Reproducibility = $\pm(0.20 + 11 \text{ percent of mean value})$

Accordingly, the analytical results on oil content of a wax sample with 0.5 percent oil can vary between 0.245 and 0.755 percent.

It is also observed, as discussed in Section 5.5, that the reliable estimate of the oil content values of the deoiled wax samples is not possible from refractive index for oil content values of less than two percent.

5.1 PRODUCT WAX YIELD

Fig. 5.1 shows the variation in product wax yield with deoiling temperature for first-stage and two-stage deoiling operations for solvent-to-slack wax ratios of

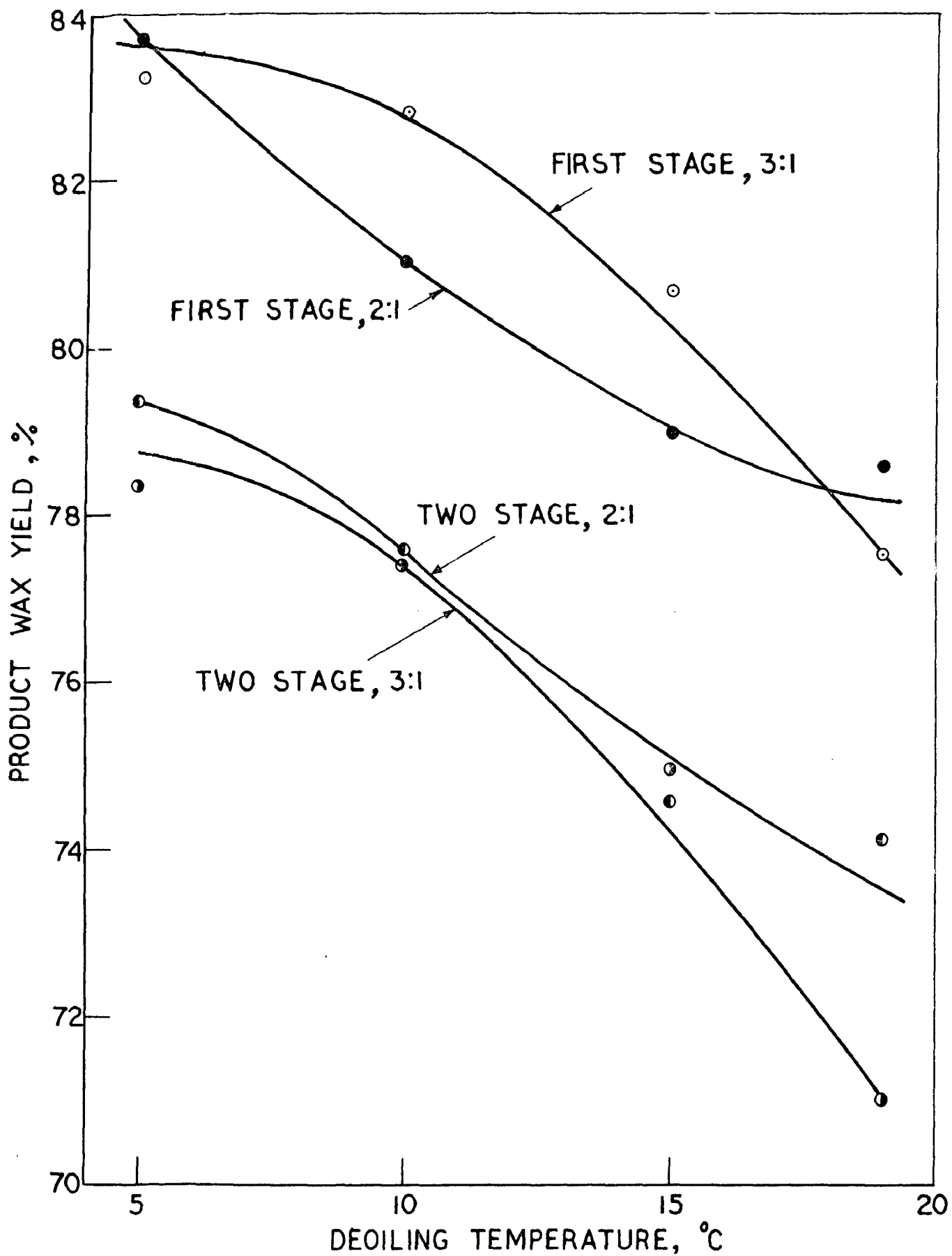


FIG. 5.1 PRODUCT WAX YIELD AS A FUNCTION OF DEOILING TEMPERATURE

2:1 and 3:1. For more meaningful interpretation of results corrected values of yield are used in place of the actual yield values.

5.1.1 EFFECT OF TEMPERATURE: It is observed that the increase in deoiling temperature results in a decrease in product wax yield both for first-and two-stage deoiling operations at solvent-to-slack wax ratios of 3:1 and 2:1. In nearly all cases the decrease in product wax yield is relatively small when the deoiling temperature is increased from 5°C to 10°C. The decrease in yield becomes more significant as the temperature is increased to 15° and 20°C. Increase in wax solubility and removal of soft waxes are primarily responsible for lower product yields at higher deoiling temperatures. However, the product wax yields higher than 71 percent at deoiling temperature as high as 19°C suggest that the amount of soft waxes in Barauni slack-wax is not large and its melting point, 60-64°C, is high enough to meet the specifications of all the four types of paraffin waxes⁽²⁾.

5.1.2 EFFECT OF SOLVENT-TO-SLACK WAX RATION: The effect of increasing the solvent-to-slack wax ratio from 2:1 to 3:1 is to decrease the product wax yield but the decrease is not very appreciable for two-stage deoiling operation for deoiling temperatures of 15°C or less. At 20°C, the product wax yields are nearly three percent lower for solvent-to slack wax ratio of 3:1 as compared to 2:1

probably due to more efficient removal of soft waxes in presence of larger quantities of the solvent.

For first-stage deoiling operation at 5°C and 20°C the product wax yields are slightly lower at the higher solvent-to-slack wax ratio, but at 10°C and 15°C the product wax yields are found to be nearly 1.7 percent higher for solvent-to-slack wax ratio of 3:1 as compared to 2:1. No explanation can be suggested for this unusual behaviour at 10°C and 15°C because larger amount of wax is expected to go into solution as the quantity of solvent and the deoiling temperature are increased.

5.1.3 EFFECT OF NUMBER OF DEOILING STAGES: For solvent-to-slack wax ratio of 3:1 the nature of the product wax yield versus temperature curve in Fig.5-1 for first- and two-stage deoiling operation is similar with two-stage yields remaining lower by about 5 to 6.5 percent. The difference in yields increases from 4.87 percent to 6.52 percent with increase in deoiling temperature from 5° to 20°C.

For solvent-to-slack wax ratio of 2:1, the product wax yields are 3.4 to 4.6 percent lower for two-stage deoiling as compared to first-stage deoiling. The difference in the two yields again shows an increase with increase in deoiling temperatures except at a deoiling temperature of 5°C.

The loss in product wax yields of $5 \pm 1.5\%$ in the two-stage deoiling can not be attributed, in general, to the decrease in oil content and the solubility of high melting wax in the solvent alone. It appears that additional quantities of soft waxes are removed during the second-stage of deoiling.

5.2 OIL CONTENT IN PRODUCT WAX:

Figure 5.2 shows the variation in oil content, as weight percent, of product wax with deoiling temperature for two stage deoiling operation at a solvent-to-slack wax ratio of 2:1 only. First- and two-stage deoiling oil content values of run No.8.22 and two-stage deoiling oil content value of run No.5.22 are not shown in the Figure 5.2 because, solvent quantities less than the normal are used in the first-stage and the second-stage deoiling operations respectively. In view of the limited number of samples analysed for oil content and the absence of duplicate oil content analysis for the samples analysed, it is difficult to draw very definite conclusions. However, the following general observations can be made.

5.2.1 EFFECT OF TEMPERATURE: For a solvent-to-slack wax ratio of 3:1, oil content of the product wax obtained in a two-stage deoiling operation does not exhibit any appreciable variation with temperature.

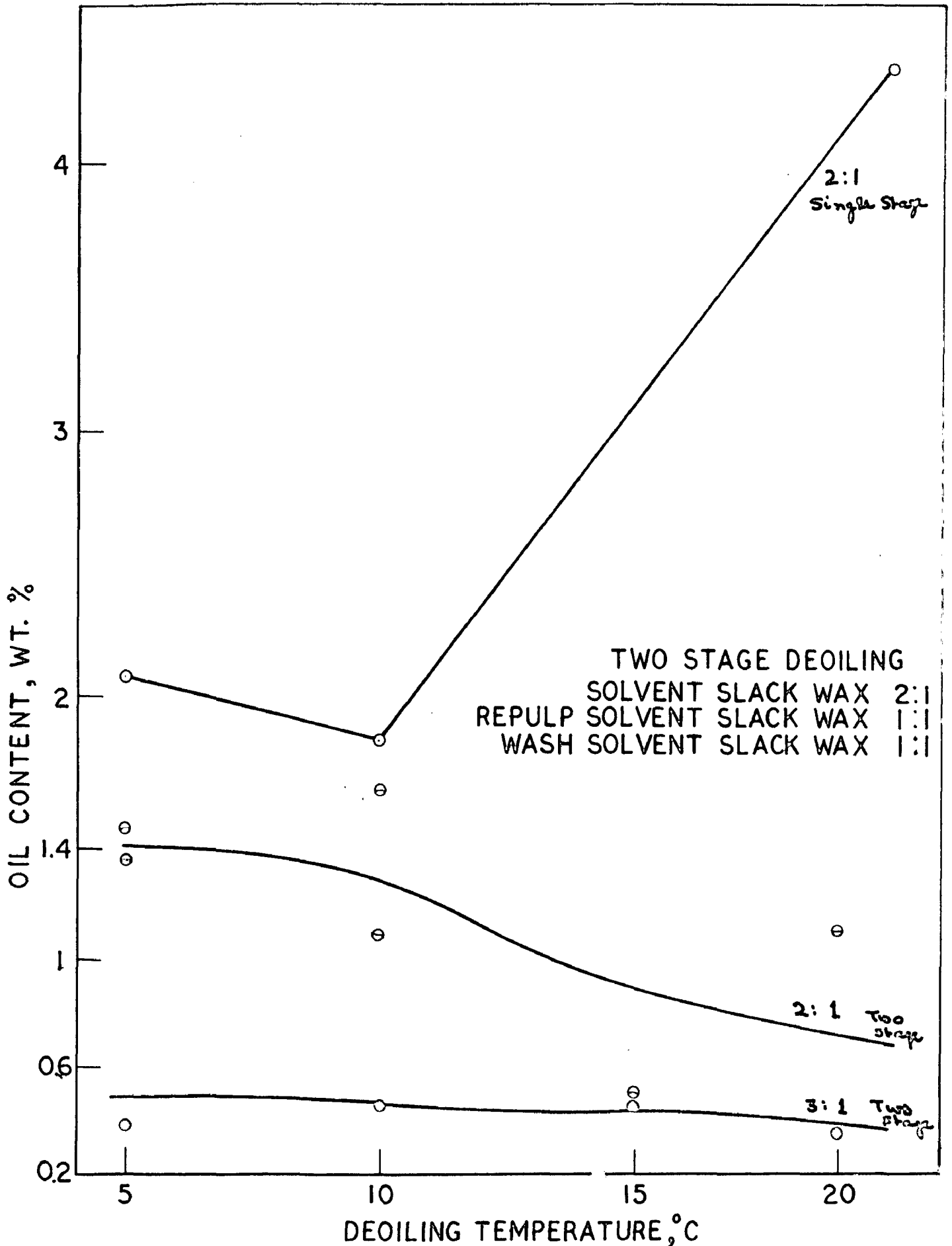


FIG.5.2 OIL CONTENT OF THE PRODUCT WAX AS A FUNCTION OF DEOILING TEMPERATURE

variation in oil content from 0.37 to 0.48 percent in deoiled wax samples suggest that any deoiling temperature between 5° to 20°C can be chosen at a solvent-to-slack wax ratio of 3:1. It appears that the use of higher temperature is only marginally advantageous. For a solvent-to-slack wax ratio of 2:1, the oil content of the product wax obtained in a two-stage operation is observed to decrease slightly as the deoiling temperature is increased from 5°C to 15°C. However, the higher oil content of product wax sample deoiled at 19°C as compared to that deoiled at 15°C makes it difficult to draw any firm conclusion.

For solvent-to-slack wax ratio of 2:1, oil content values for some samples obtained after the first-stage deoiling are also shown in the Figure 5.2. The oil content value of product wax deoiled at 10°C is slightly lower as compared to that obtained at a deoiling temperature of 5°C. But the product wax from first-stage deoiling at 19°C shows an unusually high oil content for which no logical explanation is available. It appears that the poor first-stage deoiling operation, for some unknown reasons, is primarily responsible for the unusually high oil content in the product wax obtained from first-stage and two-stage deoiling operation for solvent to slack wax ratio of 2:1.

Tiedje and Macleod⁽²⁾ reported that the miscibility temperature of 2:1 mixtures of methyl isobutyl ketone and oil is less than -20°C even for the most viscous oil

(viscosity of 111.9 S.S.U. at 99°C). Deoiling temperatures used in the present investigation are high enough to have complete miscibility of oil in the solvent. Therefore, the temperature can affect the oil content of product wax obtained from deoiling operation only through its effect on slurry viscosity. At a higher solvent-to-slack wax ratio of 38:1, the slurry is dilute enough and the use of lower deoiling temperature does not affect the crystallization, filtration and washing characteristics appreciably. Accordingly, oil content values are essentially unaffected by the deoiling temperatures in the range of 5° to 20°C. But at a lower solvent-to-slack wax ratio of 2:1, the slurry is much more viscous and the use of lower deoiling temperature adversely affects the wax crystallization and enhances the tendency to form agglomerates with poor filtration and washing characteristics. Therefore, the oil content in the deoiled wax product is expected to increase with decreasing deoiling temperature at low solvent-to-slack wax ratio.

5.2.2 EFFECT OF SOLVENT-TO-SLACK WAX RATIO:

Figure 5.2 shows the effect of solvent-to-slack wax ratio on the oil content in the product wax obtained from two-stage deoiling operation. Higher solvent-to-slack wax ratio of 3:1 results in product wax with oil content about one percent lower as compared to that obtained at a solvent-to-slack wax ratio of 2:1. The difference in oil

content is more significant at lower deoiling temperatures because of poor crystallization and increasing tendency to form agglomerates with entrapped oil due to higher slurry viscosity at low solvent-to-slack wax ratio. Nothing can be said regarding the effect of solvent ratio on product wax from first-stage because oil content analysis is not available for first-stage and solvent-to-slack wax ratio 3:1.

If it is assumed that the entire quantity of wax present in the slack wax is recovered as the product wax, no wax crystal agglomerates entrapping oil are formed, oil mixes completely in the solvent, cake porosity is constant and the oil-solvent solution in cake mixes completely with repulp solvent and wash solvent during repulping and washing respectively, then the oil content in the product wax can be calculated theoretically. Details of these calculations and the validity of various assumptions used in the model are given in Section 5.4. The calculated values of the oil content in product wax obtained from first-stage and two-stage deoiling operations at solvent-to-slack wax ratio of 2:1 are 2.63 and 0.60 percent and corresponding values at solvent-to-slack wax ratio of 3:1 are 1.79 and 0.32 percent, if slack wax contains 12.4 percent oil and the wax cake porosity is 0.5. According to these calculations a solvent-to-slack wax ratio larger than 3.50:1 is required to obtain a product wax containing less than 0.25 percent oil from two-stage deoiling operation.

5.2.3 EFFECT OF NUMBER OF STAGES:

For a solvent-to-slack wax ratio of 2:1, oil content analysis is carried out for the product wax samples from first-stage as well as two-stage deoiling operations. From Figure 5.2 it is clear that the two-stage deoiling gives product wax of much lower oil content as compared to one-stage deoiling product wax, irrespective of the deoiling temperature. The second-stage deoiling operation reduces the oil content by 0.7, 0.8 and 3.2 percent at 5°, 10° and 19° C deoiling temperatures respectively. Due to an unusually high value of oil content, 4.36 percent, in product wax from first stage deoiling at 19°C, a larger quantity of oil is removed in the second stage showing a reduction in oil content value by 3.2 percent.

From theoretical calculations; based on the model developed in Section 5.4, it is observed that for a slack wax with 12.4 percent oil and cake porosity of 0.5, the oil content after crystallization and first filtration is 4.97 percent and 4.43 percent for solvent-to-slack wax ratios of 2:1 and 3:1 respectively. Each subsequent washing, for a wash solvent-to-slack wax ratio of 0.75, reduces the oil content to 51.1 percent of its initial value and each subsequent repulping and filtration reduces the oil content to 44.0 or 34.4 percent of the oil content value in wax cake feed to repulping stage depending upon whether the repulp solvent-to-slack wax ratio of

1:1 or 1.5:1 is used. These calculations indicate that in order to reduce the oil content to 3.5 percent, as specified for type 4 wax⁽²¹⁾, first stage deoiling at a solvent-to-slack wax ratio of 2:1 along with a single washing will be sufficient; to reduce oil content to 0.5 percent, as specified for type 3 wax⁽²¹⁾, two-stage deoiling at a solvent-to-slack wax ratio of 3:1 with single washing at each stage will be sufficient and to reduce the oil content to below 0.25 percent, as specified for type 1 and type 2 waxes⁽²¹⁾, a three-stage deoiling with solvent-to-slack wax ratio less than 2:1 or else a two-stage deoiling with solvent-to-slack wax ratio of 3.5:1 is required.

5.3 RELATIONSHIP BETWEEN OIL CONTENT AND PRODUCT WAX YIELD:

Figure 5.3 represents the variation in product wax yield as a function of its oil content for two-stage deoiling operation and solvent-to-slack wax ratio of 2:1 and 3:1. For a solvent-to-slack wax ratio of 2:1, the data show a considerable slope, but product wax yield, in general, decreases with decrease in oil content value. For a solvent-to-slack wax ratio of 3:1, the effect of product wax yield on oil content is not significant and can be considered to decrease only marginally with decrease in the yield. The product wax yield may decrease due to decrease in oil content, due to increase in wax solubility in the deoiling solvent

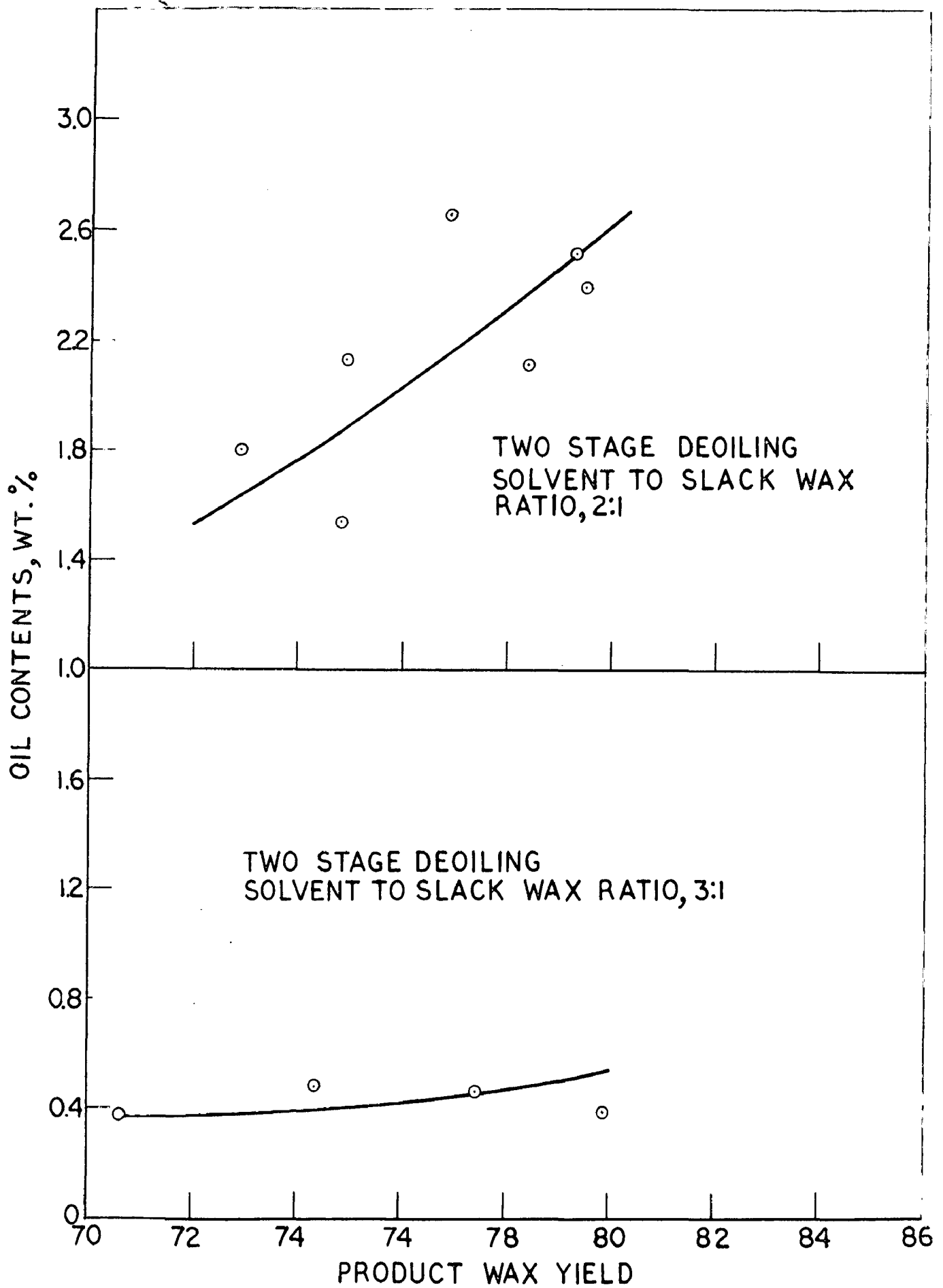


FIG.5.3 VARIATION OF OIL CONTENT AS A FUNCTION OF PRODUCT WAX YIELD

at increasing deoiling temperature and solvent quantity or due to the removal of additional quantities of soft waxes at higher temperatures. It appears that at solvent-to-slack wax ratio of 3:1 or higher, increase in deoiling temperature from 5° to 20°C does not change the oil content appreciably but reduces the yield significantly because of the removal of additional quantities of soft waxes.

5.4 SIMPLIFIED MODEL FOR WAX DEOILING:

Theoretical calculations can be made to estimate oil content of the product wax using a model based on crystallization and mixing characteristics of wax-oil-solvent system, the product wax yield, cake porosity, slack wax oil content and the quantities of solvent used at various stages. Details of the simplified wax deoiling model and computed results are given in Appendix-III.

Figure 5.4 shows the estimated values of percent oil content in product wax as a function of solvent-to-slack wax ratio for first-stage, two-stage and three-stage deoiling operations and for the assumed cake porosity values of 0.4 and 0.5. In order to obtain product wax of 0.25 percent oil content, for wax-cake porosity of 0.5, Figure 5.4 shows the solvent-to-slack wax ratio values of 3.5: and 1.35:1 will be required for two-and three-stage deoiling operation respectively. A two-stage deoiling operation at solvent-to-slack wax ratio of 1.75:1

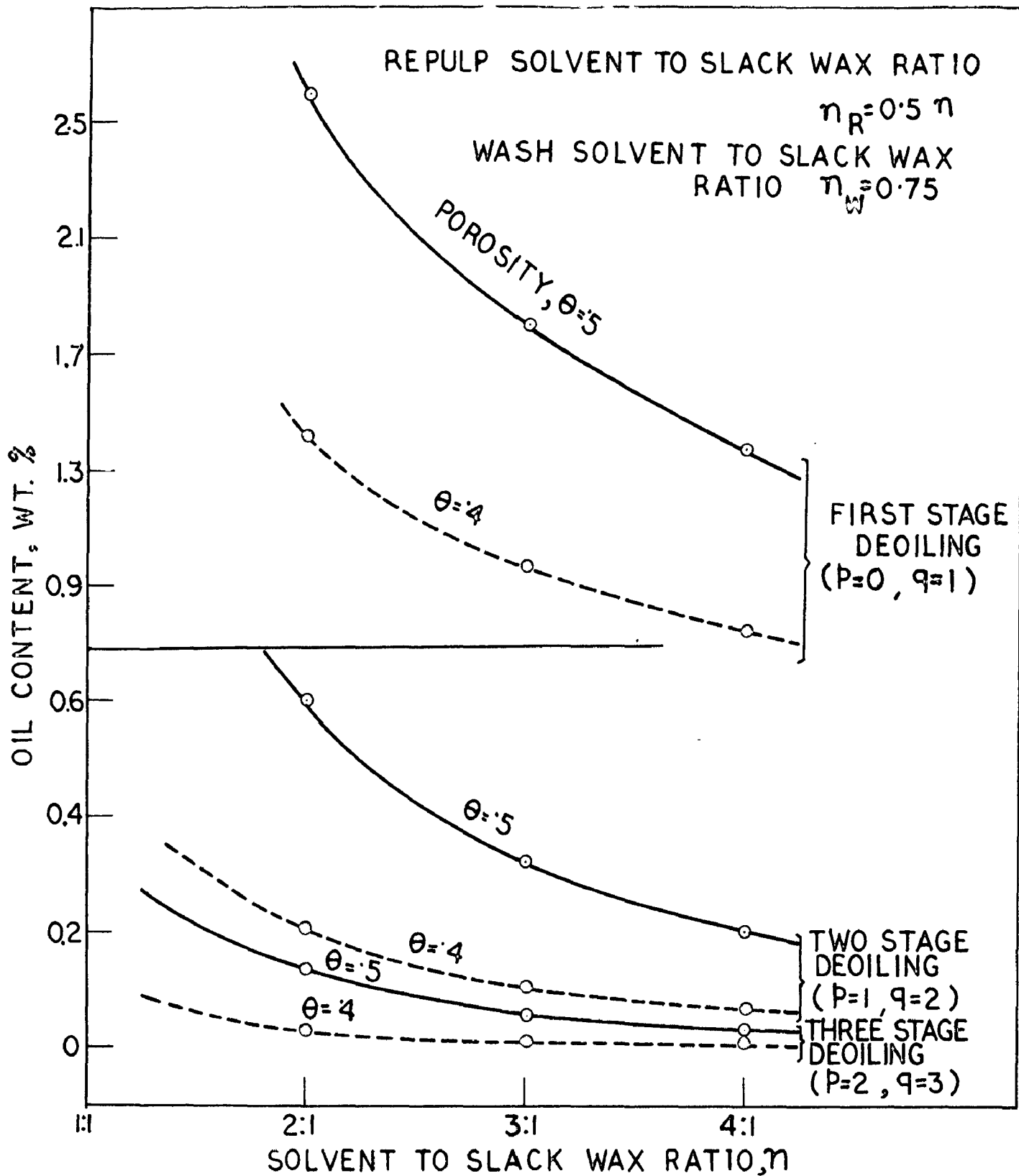


FIG. 5.4 OIL CONTENT OF PRODUCT WAX BASED ON SIMPLIFIED MODEL

will be sufficient to produce product wax of 0.25 percent oil content if the wax-cake porosity is only 0.4. However, the oil content of the product wax will be adversely affected by the use of solvent-to-slack wax ratio values of less than 2:1 due to poor wax crystallization and repulping and also for wax of low porosity due to poor washing and repulping.

5.4.1 IMPROVEMENTS IN WAX-DEOILING MODEL:

The simplified model is unrealistic because of the following reasons:

1. Slack wax always contains some soft waxes which are removed during deoiling. The extent of the removal of soft waxes depends on the deoiling temperature.

2. Hard wax is soluble to some extent in the solvent. For any given solvent, hard wax yield as product wax will decrease with increase in deoiling temperature as well as with increase in solvent-to-slack wax ratio.

3. Wax crystals will have a tendency to form loose agglomerates during crystallization. The oil-solvent solution entrapped in the wax crystals can not be easily removed during washing.

4. Some cracks invariably develop during filtration which reduce the filtration and washing efficiency.

5. Washing operation is usually much less efficient than repulping operation.

6. Porosity of wax cake will depend on the filtration and washing conditions.

7. Formation of wax-cake of very low porosity is not desirable due to poor washing and repulping characteristics.

8. Deoiling is adversely affected at low temperatures, especially at low solvent-to-slack wax ratio due to increase in slurry viscosity.

Some of the improvements can be easily incorporated to develop a more realistic wax deoiling model if reliable relevant data is available.

5.4.2 MODIFIED MODEL FOR WAX-DEOILING BASED ON ACTUAL WAX-CAKE, YIELD VALUES:

Due to paucity of data it is not possible to interpret all the modifications necessary in the model. However, an attempt is made to calculate the oil content values in product wax using the experimental values of wax cake and product wax yields for each run. Details of the modified wax deoiling model based on the experimental yield values are given in Appendix III. Table 5.2 gives experimental values of oil content and wax cake porosity along with the values of oil content using modified wax deoiling model.

For solvent-to-slack wax ratio of 3:1, estimated values of oil content for a two-stage deoiling operation varies in the range of 0.14 to 0.26 percent as compared

to the experimental values of 0.37 to 0.48 percent. Considering the fact that the reproducibility of oil content analysis for low oil content values itself is quite poor as indicated earlier, the applicability of the model can be considered as reasonable. However, since the estimated values of oil content are always lower than the experimental values, considerable refinement in deoiling model is possible by using realistic values for repulping and washing efficiencies.

For solvent-to-slack wax ratio of 2:1, estimated values for oil content for a two-stage deoiling operation varies in the range of 0.12 to 0.50 percent as compared to experimental values of 0.51 to 1.64 percent. The applicability of the modified model for deoiling is distinctly poor in this case. Probably, low wax-cake porosity is responsible for poor repulping and washing efficiencies and, therefore, actual(experimental) oil content values are, in general, 1.0 ± 0.4 percent higher than those predicted by the model.

For first-stage deoiling operation only limited analytical results are available for oil content in product wax. However, it is rather surprising that the actual (experimental) values of oil content are lower, except for Run No.13.22, than those predicted by the model. More experimental verification is necessary before offering any explanation for this unusual behaviour.

Table 5.2 Estimated Oil Content Values Based on the Modified Wax Deoiling Model

Solvent -to- slack wax Ratio n	Run No.	Experimental Values				Estimated Values of oil content, percent	
		Porosity θ		Oil content, percent		First- stage deoiling	Two- stage deoiling
		First- stage deoiling	Two- stage deoiling	First- stage deoiling	Two- stage deoiling		
3:1	15.32	.565	.511	N.A.	0.39	1.61	0.26
	17.32	.551	.429	N.A.	0.46	1.91	0.18
	19.32	.545	.460	N.A.	0.48	1.83	0.21
	21.32	.514	.441	N.A.	0.37	1.47	0.14
	21.32	.514	.441	N.A.	0.37	1.47	0.14
2:1	11.22	.483	.421	2.08	1.38	2.30	0.23
	10.22	.469	.450	1.83	1.10	2.09	0.33
	8.22	.488	.411	3.30	0.79	4.09	0.39
	13.22	.440	.400	4.36	1.12	1.75	0.20
	12.22	.355	.349	N.A.	1.50	1.04	0.12
	9.22	.493	.397	N.A.	1.64	2.44	0.26
	7.22	.445	.552	N.A.	0.51	1.77	0.50
	5.22	.454	.512	N.A.	1.52	1.89	0.42

N.A. - not analysed.

5.5 REFRACTIVE INDEX AND OIL CONTENTS:

The standard method used for the determination of oil content is discussed in Section 4.4. The procedure is not only expensive but also takes nearly six to eight hours for each analysis. Possible use of the refractive index for analysing oil content⁽²⁴⁾ in product wax sample is very desirable and, therefore, refractive index of all wax samples is determined at 75°C using Carl Zeiss Jena Refractometer and Veb Praegerate Werk Medingen Thermostat. Table 5.3 gives the refractive index values at 75°C for all the samples. In Figure 5.5 the experimental values of oil content, as determined by the standard method, are plotted against the refractive index values. For oil content values higher than 3.5 percent, a linear relationship between oil content and refractive index is observed upto 12.4 percent oil content in slack wax. However, for oil content in the range 0 to 2 percent not only the deviation from straight line is large but also scatter in the data is so much that the refractive index is not useful even for estimating the approximate value of oil content. The refractive index can, therefore, be used only for routine checking to reject product wax samples of high oil content. For the production of paraffin wax of Types 1,2 and 3 it is necessary to use the standard method for oil content determination.

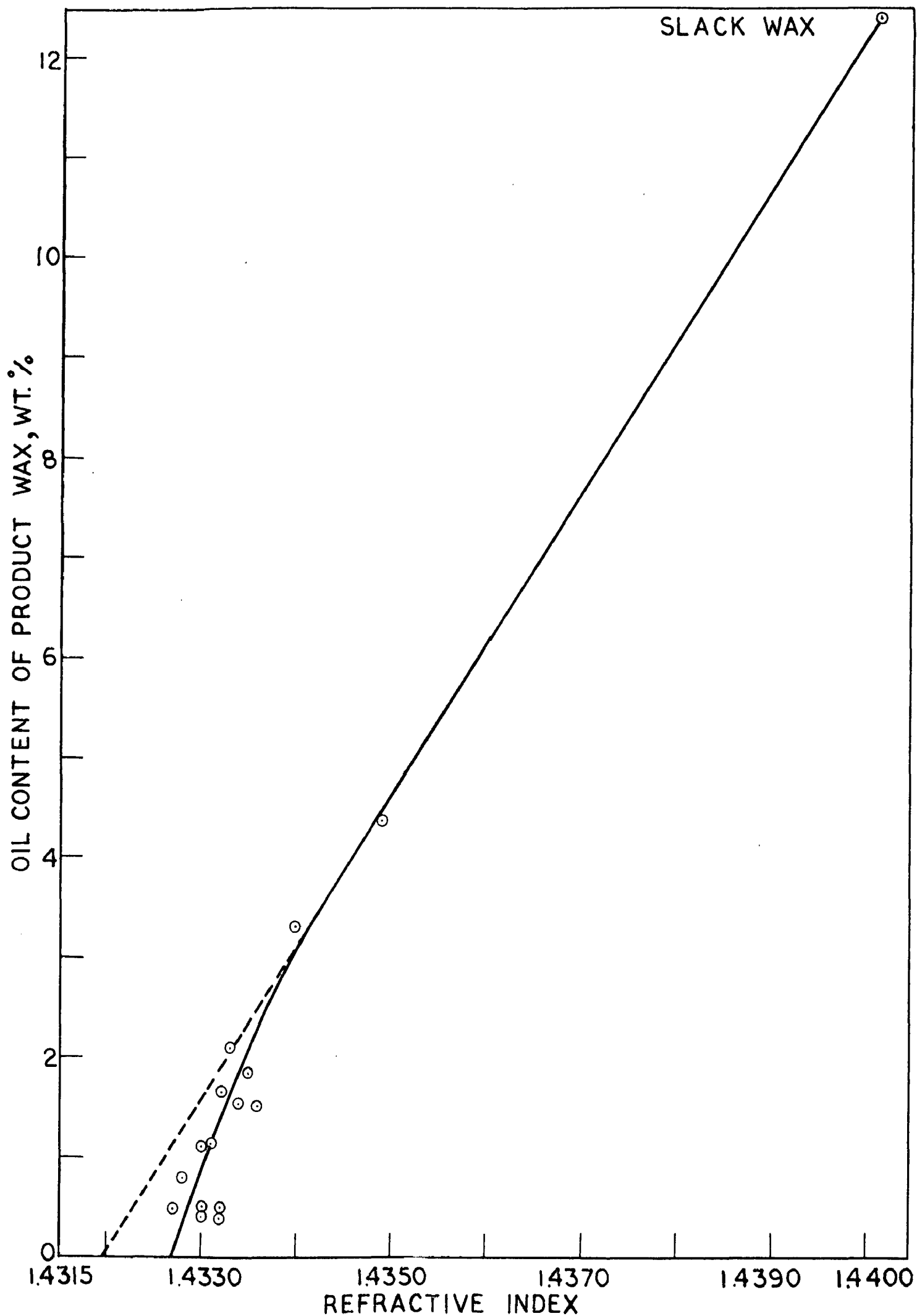


FIG.55 RELATIONSHIP BETWEEN OIL CONTENT AND REFRACTIVE

Table 5.3 Oil Content Values on the basis of R.I. and as determined by Standard ASTM method.

Run No.	First-stage Deoiling			Two-stage Deoiling		
	Refractive Index at 75°C	Oil percent based on R.I.	Oil percent by Standard ASTM method	Refractive Index at 75°C	Oil percent based on R.I.	Oil percent by Standard ASTM method
1.21	1.4335	2.00	N.A.	N.A.	N.A.	N.A.
2.21	1.4345	3.85	N.A.	N.A.	N.A.	N.A.
3.21	1.4341	3.15	N.A.	N.A.	N.A.	N.A.
4.21	1.4341	3.15	N.A.	N.A.	N.A.	N.A.
5.22	1.4340	3.00	N.A.	1.4334	1.80	1.52
6.22	N.A.	N.A.	N.A.	1.4331	1.10	N.A.
7.22	1.4335	2.00	N.A.	1.4330	0.90	0.51
8.22	1.4340	3.00	3.30	1.4328	0.35	0.79
9.22	1.4340	3.00	N.A.	1.4332	1.35	1.64
10.22	1.4335	2.00	1.83	1.4330	0.90	1.10
11.22	1.4333	1.60	2.08	1.4332	1.35	1.38
12.22	1.4350	4.65	N.A.	1.4336	2.20	1.50
13.22	1.4349	4.50	4.36	1.4331	1.10	1.12
14.32	1.4350	4.65	N.A.	1.4335	2.00	N.A.
15.32	1.4347	4.20	N.A.	1.4332	1.35	0.39
16.32	1.4346	4.05	N.A.	1.4333	1.60	N.A.
17.32	1.4336	2.20	N.A.	1.4327	0.05	0.46
18.32	1.4340	3.00	N.A.	1.4333	1.60	N.A.
19.32	1.4345	3.90	N.A.	1.4332	1.35	0.48
20.32	1.4344	3.75	N.A.	1.4334	1.80	N.A.
21.32	1.4338	2.70	N.A.	1.4330	0.90	0.37

CHAPTER 6

CONCLUSIONS AND RECOMMENDATIONS

6.1 CONCLUSIONS:

The present investigation leads to the following important conclusions:

1. Methy-isobutyl ketone is an effective deoiling solvent and can be used with advantage for the deoiling of Barauni slack wax.
2. In deoiling temperature range 5° to 20°C ; the product wax yield is adversely affected by the increase in deoiling temperature. Decrease in yield is much more pronounced at deoiling temperature 15°C or higher.
3. For solvent-to-slack wax ratios used in present investigation, namely, 2:1 and 3:1, lower product wax yields are obtained at higher solvent-to-slack wax ratio. Difference in the yield values depend on deoiling temperature and becomes appreciable at 20°C .
4. Deoiling temperature in the range 5° to 20°C has no direct effect on the product wax oil content. However, at lower solvent-to-slack wax ratio of 2:1 higher oil content is expected at low deoiling temperature due to increase in slurry viscosity.

5. For two-stage deoiling operation and at solvent-to-slack wax ratio of 3:1, product wax oil content can be predicted with reasonable accuracy using a simple model based on actual wax-cake porosity. However, accuracy of the model at solvent-to-slack wax ratio of 2:1 was appreciably lower due to poor repulping and washing characteristics of wax cake of low porosity.
6. Proposed deoiling model indicates that type 1 and type 2 paraffin waxes with oil content less than 0.25 percent can be produced in a two-stage deoiling operation with MIBK-to-slack wax ratio of 3.5:1. Type 3 paraffin wax with oil content less than 0.5 percent can be produced in a two-stage deoiling operation with MIBK-to-slack wax ratio 2.25:1. Type 4 paraffin wax with oil content less than 3.5 percent can be produced in a single-stage deoiling operation with MIBK-to-slack wax ratio of 2:1 or lower.
7. The use of solvent-to-slack wax ratio less than 2:1 is not recommended because of poor flow characteristics of slurry.
8. The choice of deoiling conditions essentially depend on profitability analysis based on yields and oil

content and refrigeration cost, product wax and solvent loss. Two-stage deoiling operation at 10°C and solvent-to-slack wax ratio of 3.5:1 is suggested for producing type 1 and type 2 paraffin waxes.

6.2 RECOMMENDATIONS:

1. More intensive investigations using solvent-to-slack wax ratios of 2:1 to 4:1 and deoiling temperatures of 5° to 20°C are necessary to develop correlations for predicting the effect of these variables on the product wax yield and oil content.
2. It is necessary to study the filtration and solvent washing operations under carefully controlled conditions to obtain a reliable correlation between wax cake properties and filtration and washing variables.
3. The effect of temperature, solvent-to-slack wax ratio and chilling rate on the property of wax-oil-solvent slurry needs to be investigated.
4. Optimization of incremental dilutions may be studied by varying the number of increments for the same solvent-to-slack wax ratio.
5. Studies can be carried out with MEK, MEK-Aromatic Mixtures and MEK-MIBK Mixtures

for a comparative study of the effectiveness of these as deoiling solvents.

6. Predominance of n-alkanes in the slack wax generally give rise to lateral growth and fairly large crystals are expected even with shock chilling using cold solvent. Experimental investigation using Dilchill Deoiling Process is therefore recommended.

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