

University of Khartoum Faculty of Engineering & Architecture Chemical Engineering Department

Production of Petroleum Wax from Nile Blend Oil

A Thesis Submitted for fulfillment of the requirements for the degree of Ph.D in Chemical Engineering

By:

Amel Abdelatti Ahmed Nimir M.Sc, B.Sc, Chemical Engineering, Faculty of Engineering University of Khartoum

Supervisor: Dr.Abdelshakour Awadalkarim Mohamed Co-supervisor: Dr. Ali Abdelrahman Rabah

August 2008

Dedication

To the soul of my mother To my father To my beloved husband, daughters and son To my brothers and sisters To my teachers To my friends

Acknowledgments

I would like to express my profound gratitude to my supervisor, Dr. Abdelshakour Awadalkarim Mohammed, for his guidance, patience and understanding.

Grateful acknowledgment is made to **Dr. Ali Abdelrahman Rabah** for his continuous help and support during this study.

Sincere thanks to Khartoum Refinery Company, Ministry of Energy and Mining, Greater Nile Petroleum Operation Company (GNPOC), Unit Operation Lab Staff-University of Khartoum and special thanks to Central Petroleum Laboratories CPL staff for their help. Also my deep gratitude extends to the academic and technical staff of the Department of Chemical Engineering, University of Khartoum.

Abstract

Petroleum wax is a substance, which has many applications and economical value today. The production of petroleum wax in the world about 3.6×10^9 ton per year, valued at nearly \$2 billion.

The basic raw material for paraffin wax is petroleum crude oil and lubricating oil.

Although the availability of Sudan petroleum crude oil which contain large amount of paraffin wax and availability of refineries for crude oil, there are no units in these refineries for production of wax.

Sudan now imports paraffin wax to cover its needs (145.3058 ton).

In this research, paraffin wax is extracted from Nile Blend oil under the best and most economical operating conditions (the used sample contains 26% paraffin wax). The laboratorial experiments of extraction are done in three steps:

In the first step the asphaltene is removed, in the second step the solvents in which the paraffin wax dissolves is added and in the last step the solution is chilled to precipitate the paraffin wax.

For dissolution of paraffin wax in Nile Blend oil, the following solvents are used:

-Methyl ethyl ketone, -Methyl ethyl ketone(75%) + Toluene(25%) -Methyl ethyl ketone(50%) + Toluene(50%) -Methyl ethyl ketone(70%) + Toluene (20%)and Naphtha(10%)

The laboratorial experiments of extraction wax is done for a wide range of temperature $(28 - 70^{\circ}C)$, mixing time of solvent and sample (5 - 30 minutes) and solvent to sample ratio (16:1 - 32:1).

The results revealed that the best conditions to extract 100 % of paraffin wax from Nile Blend crude oil are:

Temperature 60°C Mixing time of solvent and sample is 25 minutes Solvent to sample ratio is 20:1. And the best solvent is methyl ethyl ketone.

ملخص البحث

Tables of Contents

1	Introduction1
	1. Waxes1
	1.1 Type of Waxes1
	1.1.2 Insect and Animal Waxes1
	1.1.3 Vegetable Waxes2
	1.1.3.1 Carnauba Wax2
	1.1.3.2 Candelilla Wax
	1.1.3.3 Japan Wax3
	1.1.3.4 Ouricury Wax
	1.1.3.5 Rice-Bran Wax4
	1.1.3.6 Jojoba Wax4
	1.1.3.7 Caster Wax4
	1.1.3.8 Bayberry Wax4
	1.1.4 Minerals Waxes
	1.1.5 Petroleum Wax
	1.1.5.1 Macrocrystalline Wax (Paraffin Wax)6
	1.1.5.2 Microcrystalline Wax (Micro Wax)
	1.1.6 Synthetic Waxes
	1.1.7 Polymerized a-Ollefins10
	1.1.8 Chemically Modified Waxes11
	1.1.9 Characteristic Properties of Waxes11
	1.1.10 Function and Effects of Waxes12
	1.2 Refinery of Petroleum12

1.3 Refinery Process	13
1.3.1 Separation Process	13
1.3.2 Petroleum Convertion Process	14
1.3.2.1 Catalytic Craking	14
1.3.2.2 Thermal Cracking (Visbreaking)	14
1.3.2.3 Coking	14
1.3.2.4 Alkylation and Polymerization	15
1.3.2.5 Isomerization and reforming	15
1.3.2 6 Petroleum Treating Process	15
1.3.2.6.1 Hydro treating	15
1.3.2.6.2 Chemical Sweating	16
1.3.2.6.3 Acid Gas Removal	16
1.3.2.6.4 Deasphalting	16
1.3.2.6.5 Asphalt Blowing	16
1.3.2.6.6 ewaxing Process	17
1.4 Solvent Dewaxing	17
1.5 Catalytic Dewaxing	18
1.6 Urea Dewaxing	18
1.7 Feedstock and Product Handling	19
1.8 Auxiliary Facilities	19
1.8.1 Steam Boilers	19
1.8.2 Waste Water and Solid Waste Treatment	20
1.8.3 Hydrogen Production	20
1.8.4 Sulfur Recovery Plants	20
1.9 Petroleum in The Sudan	21
1.10 Objective of the Study	24

2	Literature Review	v(Previous Works)	
3	Materials and M	ethods	29
	3.1 Introduction	l	29
	3.2 Materials		
	3.2.1 NILE	BLEND	29
	3.2.2 Solve	nts	
	3.3 Method		
	3.3.1 Deas	halting	
	3.3.2 Dewa	xing	
		C	
4	Results and Disc	cussion	
	4.1 Results		
	4.2 Discussion.		115
	4.3 Carbon Ato	m Analysis	117
5	Economic Evalu	ation	
	5.1 Material Ba	lance	
	5.1.5 Overa	Il Material Balance	121
	5.2 Energy Bala	nce	122
	5.2.1 Energy	y Balance around Heater	
	5.2.2 Energy	Balance around Mixer 1	
	5.2.3 Energ	v Balance around Mixer 2	124
	5.3 Cost Estima	tion (MEK at 60°C. 25 min and 20):1 solvent to
	sample ratio)	
	5.3.1 Purch	ase Cost of Major Equipment (PC	COME)125

	5.3.2 Total Ivestment Cost (TIC)	126
	5.3.3 Annual Operating Cost (AOC)	128
	5.4 Payback Period.	129
	5.5 Conclusions	130
	5.6 Recommendations	131
Re	ferences	132
Ap	ppendices	136
A.		136
A	Nile Blend Fields in the Sudan	136
B.,		137
В	Apparatus	137
	B.1 Balance	137
	B.2 Oven	138
	B.3 Filter	138
	B.4 Circulator Water Bath	139
	B.5 Rotary Evaporator	
	B.6 Chiller	140
	B.7 Water Suction Pump	140
	B.8 Flasks	141
	B.9 Magnetic Stirrer	141
	B.10 Desicator	142

С	The Method for Calculation of Extracted Wax	143
C.1	Solvent of Methyl Ethyl Ketone	143
C.2	Solvent of 75% MEK + 25% Toluene	150
C.3	Solvent of 50% MEK + 50% Toluene	157

C.4 Solvent of 70% MEK + 20% Toluene + 10% Naphtha.....165 List of Tables

3.1	Properties of Nile Blend Petroleum
3.2	Contribution of each field in the Nile Blend
3.3	Physical Properties of Solvents
4.1	Effect of Temperature on the Amount of Wax Extracted
	(% age) using 16:1 Solvent to Sample for 5 minutes
4.2	Effect of Temperature on the Amount of Wax Extracted
	(% age) using 20:1 Solvent to Sample for 5 minutes
4.3	Effect of Temperature on the Amount of Wax Extracted
	(% age) using 24:1 Solvent to Sample for 5 minutes40
4.4	Effect of Temperature on the Amount of Wax Extracted
	(% age) using 32:1 Solvent to Sample for 5 minutes41
4.5	Effect of Temperature on the Amount of Wax Extracted
	(% age) using 16:1 Solvent to Sample for 10 minutes42
4.6	Effect of Temperature on the amount of wax extracted
	(% age) using 20:1 Solvent to Sample for 10 minutes43
4.7	Effect of Temperature on the Amount of Wax Extracted
	(% age) using 24:1 Solvent to Sample for 10 minutes44
4.8	Effect of Temperature on the Amount of Wax Extracted
	(% age) using 32:1 Solvent to Sample for 10 minutes45
4.9	Effect of Temperature on the Amount of Wax Extracted
	(% age) using 16:1 Solvent to Sample for 15 minutes46
4.10	Effect of Temperature on the Amount of Wax Extracted
	(% age) using 20:1 Solvent to Sample for 15 minutes47

Х

4.11	Effect of Temperature on the Amount of Wax Extracted
	(% age) using 24:1 Solvent to Sample for 15 minutes 48
4.12	Effect of Temperature on the Amount of Wax Extracted
	(% age) using 32:1 Solvent to Sample for 15 minutes49
4.13	Effect of Temperature on the Amount of Wax Extracted
	(% age) using 16:1 Solvent to Sample for 20 minutes50
4.14	Effect of Temperature on the Amount of Wax Extracted
	(% age) using 20:1 Solvent to Sample for 20 minutes51
4.15	Effect of Temperature on the Amount of Wax Extracted
	(% age) using 24:1 Solvent to Sample for 20 minutes52
4.16	Effect of Temperature on the Amount of Wax Extracted
	(% age) using 32:1 Solvent to Sample for 20 minutes53
4.17	Effect of Temperature on the Amount of Wax Extracted
	(% age) using 16:1 Solvent to Sample for 25 minutes54
4.18	Effect of Temperature on the Amount of Wax Extracted
	(% age) using 20:1 Solvent to Sample for 25 minutes55
4.19	Effect of Temperature on the Amount of Wax Extracted
	(% age) using 24:1 Solvent to Sample for 25 minutes56
4.20	Effect of Temperature on the Amount of Wax Extracted
	(% age) using 32:1 Solvent to Sample for 25 minutes57
4.21	Effect of Temperature on the Amount of Wax Extracted
	(% age) using 16:1 Solvent to Sample for 30 minutes58
4.22	Effect of Temperature on the Amount of Wax Extracted
	(% age) using 20:1 Solvent to Sample for 30 minutes59
4.23	Effect of Temperature on the Amount of Wax Extracted
	(% age) using 24:1 Solvent to Sample for 30 minutes60

xi

4.24	Effect of Temperature on the Amount of Wax Extracted
	(% age) using 32:1 Solvent to Sample for 30 minutes61
4.25	Effect of Time on the Amount of Wax Extracted
	(% age) using 16:1 Solvent to Sample at 28°C62
4.26	Effect of Time on the Amount of Wax Extracted
	(% age) using 20:1 Solvent to Sample at 28°C63
4.27	Effect of Time on the Amount of Wax Extracted
	(% age) using 24:1 Solvent to Sample at 28°C64
4.28	Effect of Time on the Amount of Wax Extracted
	(% age) using 32:1 Solvent to Sample at 28°C65
4.29	Effect of Time on the Amount of Wax Extracted
	(% age) using 16:1 Solvent to Sample at 40°C66
4.30	Effect of Time on the Amount of Wax Extracted
	(% age) using 20:1 Solvent to Sample at 40°C67
4.31	Effect of Time on the Amount of Wax Extracted
	(% age) using 24:1 Solvent to Sample at 40°C68
4.32	Effect of Time on the Amount of Wax Extracted
	(% age) using 32:1 Solvent to Sample at 40°C69
4.33	Effect of Time on the Amount of Wax Extracted
	(% age) using 16:1 Solvent to Sample at 50°C70
4.34	Effect of Time on the Amount of Wax Extracted
	(% age) using 20:1 Solvent to Sample at 50°C71
4.35	Effect of Time on the Amount of Wax Extracted
	(% age) using 24:1 Solvent to Sample at 50°C72
4.36	Effect of Time on the Amount of Wax Extracted
	(% age) using 32:1 Solvent to Sample at 50°C73

4.37	Effect of Time on the Amount of Wax Extracted
	(% age) using 16:1 Solvent to Sample at 60°C74
4.38	Effect of Time on the Amount of Wax Extracted
	(% age) using 20:1 Solvent to Sample at 60°C75
4.39	Effect of Time on the Amount of Wax Extracted
	(% age) using 24:1 Solvent to Sample at 60°C76
4.40	Effect of Time on the Amount of Wax Extracted
	(% age) using 32:1 Solvent to Sample at 60°C77
4.41	Effect of Time on the Amount of Wax Extracted
	(% age) using 16:1 Solvent to Sample at 70°C78
4.42	Effect of Time on the Amount of Wax extracted
	(% age) using 20:1 Solvent to Sample at 70°C79
4.43	Effect of Time on the Amount of Wax Extracted
	(% age) using 24:1 Solvent to Sample at 70°C80
4.44	Effect of Time on the Amount of Wax Extracted
	(% age) using 32:1 Solvent to Sample at 70°C81
4.45	Effect of Solvent to Sample Ratio on the Amount
	of Wax Extracted (% age) at 28°C and 5 minutes82
4.46	Effect of Solvent to Sample Ratio on the Amount
	of Wax Extracted (% age) at 40°C and 5 minutes83
4.47	Effect of Solvent to Sample Ratio on the Amount
	of Wax Extracted (% age) at 50°C and 5 minutes84
4.48	Effect of Solvent to Sample Ratio on the Amount
	of Wax Extracted (% age) at 60°C and 5 minutes85
4.49	Effect of Solvent to Sample Ratio on the Amount
	of Wax Extracted (% age) at 70°C and 5 minutes86

4.50	Effect of Solvent to Sample Ratio on the Amount
	of Wax Extracted (% age) at 28°C and 10 minutes87
4.51	Effect of Solvent to Sample Ratio on the Amount
	of Wax Extracted (% age) at 40°C and 10 minutes88
4.52	Effect of Solvent to Sample Ratio on the Amount
	of Wax Extracted (% age) at 50°C and 10 minutes
4.53	Effect of Solvent to Sample Ratio on the Amount
	of Wax Extracted (% age) at 60°C and 10 minutes90
4.54	Effect of Solvent to Sample Ratio on the Amount
	of Wax Extracted (% age) at 70°C and 10 minutes91
4.55	Effect of Solvent to Sample Ratio on the Amount
	of Wax Extracted (% age) at 28°C and 15 minutes92
4.56	Effect of Solvent to Sample Ratio on the Amount
	of Wax Extracted (% age) at 40°C and 15 minutes93
4.57	Effect of Solvent to Sample Ratio on the Amount
	of Wax Extracted (% age) at 50°C and 15 minutes94
4.58	Effect of Solvent to Sample Ratio on the Amount
	of Wax Extracted (% age) at 60°C and 15 minutes95
4.59	Effect of Solvent to Sample Ratio on the Amount
	of Wax Extracted (% age) at 70°C and 15 minutes96
4.60	Effect of Solvent to Sample Ratio on the Amount
	of Wax Extracted (% age) at 28°C and 20 minutes97
4.61	Effect of Solvent to Sample Ratio on the Amount
	of Wax Extracted (% age) at 40°C and 20 minutes98
4.62	Effect of Solvent to Sample Ratio on the Amount
	of Wax Extracted (% age) at 50°C and 20 minutes99

4.63	Effect of Solvent to Sample Ratio on the Amount
	of Wax Extracted (% age) at 60°C and 20 minutes100
4.64	Effect of Solvent to Sample Ratio on the Amount
	of Wax Extracted (% age) at 70°C and 20 minutes101
4.65	Effect of Solvent to Sample Ratio on the Amount
	of Wax Extracted (% age) at 28°C and 25 minutes102
4.66	Effect of Solvent to Sample Ratio on the Amount
	of Wax Extracted (% age) at 40°C and 25 minutes103
4.67	Effect of Solvent to Sample Ratio on the Amount
	of Wax Extracted (% age) at 50°C and 25 minutes104
4.68	Effect of Solvent to Sample Ratio on the Amount
	of Wax Extracted (% age) at 60°C and 25 minutes105
4.69	Effect of Solvent to Sample Ratio on the Amount
	of Wax Extracted (% age) at 70°C and 25 minutes106
4.70	Effect of Solvent to Sample Ratio on the amount
	of Wax Extracted (% age) at 28°C and 30 minutes107
4.71	Effect of Solvent to sample Ratio on the amount
	of wax extracted (% age) at 40°C and 30 minutes108
4.72	Effect of Solvent to Sample Ratio on the Amount
	of Wax Extracted (% age) at 50°C and 30 minutes109
4.73	Effect of Solvent to Sample Ratio on the amount
	of wax extracted (% age) at 60°C and 30 minutes110
4.74	Effect of Solvent to Sample Ratio on the Amount
	of Wax Extracted (% age) at 70°C and 30 minutes111
4.75	Percentage of Wax Extracted at Different
	Temperatures, Times and Ratio of Solvent to Sample112

4.76	Percentage of Wax Extracted at Different
	Temperatures, Times and Ratio of Solvent to Sample113
4.77	The Maximum Extraction Conditions for
	Different Solvents114
5.1	Material Balance of Deaspalting and Dewaxing
	processes
5.2	Purchase Cost of Major Equipment126
5.3	Total Investment Cost (TIC)127
5.4	Annual Operating Cost (AOC)128
5.5	Best Conditions of Wax Extraction for Nile Blend
	Petroleum130
C.1	Pure MEK at 28°C, 16:1 Ratio of Solvent to Sample and
	Different Times143
C.2	Pure MEK at 40°C, 16:1 Ratio of Solvent to Sample and
	Different Times144
C.3	Pure MEK at 50°C, 16:1 Ratio of Solvent to Sample and
	Different Times144
C.4	Pure MEK at 60°C, 16:1 Ratio of Solvent to Sample and
	Different Times144
C.5	Pure MEK at 70°C, 16:1 Ratio of Solvent to Sample and

C.6	Different Times Pure MEK at 28°C, 20:1 Ratio of Solvent to Sample and	.145
	Different Times145	
C.7	Pure MEK at 40°C, 20:1 Ratio of Solvent to Sample and	
	Different Times145	
C.8	Pure MEK at 50°C, 20:1 Ratio of Solvent to Sample and	
	Different Times146	
C.9	Pure MEK at 60°C, 20:1 Ratio of Solvent to Sample and	
	Different Times146	
C.10	Pure MEK at 70°C, 20:1 Ratio of Solvent to Sample and	
	Different Times146	
C.11	Pure MEK at 28°C, 24:1 Ratio of Solvent to Sample and	
	Different Times147	
C.12	Pure MEK at 40°C, 24:1 Ratio of Solvent to Sample and	
	Different Times147	
C.13	Pure MEK at 50°C, 24:1 Ratio of Solvent to Sample and	
	Different Times147	
C.14	Pure MEK at 60°C, 24:1 Ratio of Solvent to Sample and	
	Different Times148	
C.15	Pure MEK at 70°C, 24:1 Ratio of Solvent to Sample and	
	Different Times148	

C.16	Pure MEK at 28°C, 32:1 Ratio of Solvent to Sample and
	Different Times148
C.17	Pure MEK at 40°C, 32:1 Ratio of Solvent to Sample and
	Different Times149
C.18	Pure MEK at 50°C, 32:1 Ratio of Solvent to Sample and
	Different Times149
C.19	Pure MEK at 60°C, 32:1 Ratio of Solvent to Sample and
	Different Times149
C.20	Pure MEK at 70°C, 32:1 Ratio of Solvent to Sample and
	Different Times150
C.21	Mixture of 75% MEK and 25% Toluene at 28°C, 16:1 Ratio
	of Solvent to Sample and Different Times150
C.22	Mixture of 75% MEK and 25% Toluene at 40°C, 16:1 Ratio
	of Solvent to Sample and Different Times151
C.23	Mixture of 75% MEK and 25% Toluene at 50°C, 16:1 Ratio
	of Solvent to Sample and Different Times151
C.24	Mixture of 75% MEK and 25% Toluene at 60°C, 16:1 Ratio
	of Solvent to Sample and Different Times151
C.25	Mixture of 75% MEK and 25% Toluene at 70°C, 16:1 Ratio
	of Solvent to Sample and Different Times152

C.26	Mixture of 75% MEK and 25% Toluene at 28°C, 20:1 Ratio
	of Solvent to Sample and Different Times

- C.27 Mixture of 75% MEK and 25% Toluene at 40°C, 20:1 Ratio of Solvent to Sample and Different Times......152
- C.28 Mixture of 75% MEK and 25% Toluene at 50°C, 20:1 Ratio of Solvent to Sample and Different Times......153
- C.29 Mixture of 75% MEK and 25% Toluene at 60°C, 20:1 Ratio of Solvent to Sample and Different Times......153
- C.30 Mixture of 75% MEK and 25% Toluene at 70°C, 20:1 Ratio of Solvent to Sample and Different Times......153
- C.31 Mixture of 75% MEK and 25% Toluene at 28°C, 24:1 Ratio of Solvent to Sample and Different Times......154
- C.32 Mixture of 75% MEK and 25% Toluene at 40°C, 24:1 Ratio of Solvent to Sample and Different Times......154
- C.33 Mixture of 75% MEK and 25% Toluene at 50°C, 24:1 Ratio of Solvent to Sample and Different Times......154
- C.34 Mixture of 75% MEK and 25% Toluene at 60°C, 24:1 Ratio of Solvent to Sample and Different Times......155
- C.35 Mixture of 75% MEK and 25% Toluene at 70°C, 24:1 Ratio of Solvent to Sample and Different Times......155

- C.36 Mixture of 75% MEK and 25% Toluene at 28°C, 32:1 Ratio of Solvent to Sample and Different Times......155
- C.37 Mixture of 75% MEK and 25% Toluene at 40°C, 32:1 Ratio of Solvent to Sample and Different Times......156
- C.38 Mixture of 75% MEK and 25% Toluene at 50°C, 32:1 Ratio of Solvent to Sample and Different Times......156
- C.39 Mixture of 75% MEK and 25% Toluene at 60°C, 32:1 Ratio of Solvent to Sample and Different Time......156
- C.40 Mixture of 75% MEK and 25% Toluene at 70°C, 32:1 Ratio of Solvent to Sample and Different Times......157
- C42 Mixture of 50% MEK and 50% Toluene at 40°C, 16:1 Ratio of Solvent to Sample and Different Times......158
- C44 Mixture of 50% MEK and 50% Toluene at 60°C, 16:1 Ratio of Solvent to Sample and Different Times......158
- C45 Mixture of 50% MEK and 50% Toluene at 70°C, 16:1 Ratio of Solvent to Sample and Different Times......159

- C.46 Mixture of 50% MEK and 50% Toluene at 28°C, 20:1 Ratio of Solvent to Sample and Different Times......159
- C.47 Mixture of 50% MEK and 50% Toluene at 40°C, 20:1 Ratio of Solvent to Sample and Different Times......159
- C.48 Mixture of 50% MEK and 50% Toluene at 50°C, 20:1 Ratio of Solvent to Sample and Different Times......160
- C.49 Mixture of 50% MEK and 50% Toluene at 60°C, 20:1 Ratio of Solvent to Sample and Different Times......160
- C.50 Mixture of 50% MEK and 50% Toluene at 70°C, 20:1 Ratio of Solvent to Sample and Different Times......160
- C.51 Mixture of 50% MEK and 50% Toluene at 28°C, 24:1 Ratio of Solvent to Sample and Different Times......161
- C.52 Mixture of 50% MEK and 50% Toluene at 40°C, 24:1 Ratio of Solvent to Sample and Different Times......161
- C.53 Mixture of 50% MEK and 50% Toluene at 50°C, 24:1 Ratio of Solvent to Sample and Different Times......161
- C.54 Mixture of 50% MEK and 50% Toluene at 60°C, 24:1 Ratio of Solvent to Sample and Different Times......162
- C.55 Mixture of 50% MEK and 50% Toluene at 70°C, 24:1 Ratio of Solvent to Sample and Different Times......162

C.56	Mixture of 50% MEK and 50% Toluene at 28°C, 32:1 Ratio	
	of Solvent to Sample and Different Times162	
C.57	Mixture of 50% MEK and 50% Toluene at 40°C, 32:1 Ratio	
	of Solvent to Sample and Different Times163	
C.58	Mixture of 50% MEK and 50% Toluene at 50°C, 32:1 Ratio	
	of Solvent to Sample and Different Times163	
C.59	Mixture of 50% MEK and 50% Toluene at 60°C, 32:1 Ratio	
	of Solvent to Sample and Different Times163	
C.60	Mixture of 50% MEK and 50% Toluene at 70°C, 32:1 Ratio	
	of Solvent to Sample and Different Times164	
C.61	Mixture of 70% MEK and 20% Toluene and 10% Naphtha At	
	28°C, 16:1 Ratio of Solvent to Sample and Different Times164	
C.62	Mixture of 70% MEK and 20% Toluene and 10% Naphtha At	
	40°C, 16:1 Ratio of Solvent to Sample and Different Times165	
C.63	Mixture of 70% MEK and 20% Toluene and 10% Naphtha At	
	50°C, 16:1 Ratio of Solvent to Sample and Different Times165	
C.64:	Mixture of 70% MEK and 20% Toluene and 10% Naphtha At	
	60°C, 16:1 Ratio of Solvent to Sample and Different Times165	
C.65	Mixture of 70% MEK and 20% Toluene and 10% Naphtha At	
	70°C, 16:1 Ratio of Solvent to Sample and Different Times166	

- C.66 Mixture of 70% MEK and 20% Toluene and 10% Naphtha At 28°C, 20:1 Ratio of Solvent to Sample and Different Times......166
- C.67 Mixture of 70% MEK and 20% Toluene and 10% Naphtha At 40°C, 20:1 Ratio of Solvent to Sample and Different Times......166
- C.68 Mixture of 70% MEK and 20% Toluene and 10% Naphtha At
 50°C, 20:1 Ratio of Solvent to Sample and Different Times......167
- C.69 Mixture of 70% MEK and 20% Toluene and 10% Naphtha At60°C, 20:1 Ratio of Solvent to Sample and Different Times......167
- C.70 Mixture of 70% MEK and 20% Toluene and 10% Naphtha At 70°C, 20:1 Ratio of Solvent to Sample and Different Times......167
- C.71 Mixture of 70% MEK and 20% Toluene and 10% Naphtha At 28°C, 24:1 Ratio of Solvent to Sample and Different Times......168
- C.72 Mixture of 70% MEK and 20% Toluene and 10% Naphtha At 40°C, 24:1 Ratio of Solvent to Sample and Different Times.......168
- C.73 Mixture of 70% MEK and 20% Toluene and 10% Naphtha At50°C, 24:1 Ratio of Solvent to Sample and Different Times......168
- C.74 Mixture of 70% MEK and 20% Toluene and 10% Naphtha At60°C, 24:1 Ratio of Solvent to Sample and Different Times......169
- C.75 Mixture of 70% MEK and 20% Toluene and 10% Naphtha At 70°C, 24:1 Ratio of Solvent to Sample and Different Times......169

- C.76 Mixture of 70% MEK and 20% Toluene and 10% Naphtha At 28°C, 32:1 Ratio of Solvent to Sample and Different Times......169
- C.77 Mixture of 70% MEK and 20% Toluene and 10% Naphtha At
 40°C, 32:1 Ratio of Solvent to Sample and Different Times......170
- C.78 Mixture of 70% MEK and 20% Toluene and 10% Naphtha At
 50°C, 32:1 Ratio of Solvent to Sample and Different Times......170
- C.79 Mixture of 70% MEK and 20% Toluene and 10% Naphtha At 60°C, 32:1 Ratio of Solvent to Sample and Different Times......170
- C.80 Mixture of 70% MEK and 20% Toluene and 10% Naphtha At
 70°C, 32:1 Ratio of Solvent to Sample and Different Times......171

List of Figures

3.1	Experimental Steps
4.1	Effect of Temperature on the Amount of Wax Extracted
	(% age) using 16:1 Solvent to Sample for 5 minutes38
4.2	Effect of Temperature on the Amount of Wax Extracted
	(% age) using 20:1 Solvent to Sample for 5 minutes39
4.3	Effect of Temperature on the Amount of Wax Extracted
	(% age) using 24:1 Solvent to Sample for 5 minutes40
4.4	Effect of Temperature on the Amount of Wax Extracted
	(% age) using 32:1 Solvent to Sample for 5 minutes41
4.5	Effect of Temperature on the Amount of Wax Extracted
	(% age) using 16:1 Solvent to Sample for 10 minutes42
4.6	Effect of Temperature on the Amount of Wax Extracted
	(% age) using 20:1 Solvent to Sample for 10 minutes43
4.7	Effect of Temperature on the Amount of Wax Extracted
	(% age) using 24:1 Solvent to Sample for 10 minutes44
4.8	Effect of Temperature on the Amount of Wax Extracted
	(% age) using 32:1 Solvent to Sample for 10 minutes45
4.9	Effect of Temperature on the Amount of Wax Extracted
	(% age) using 16:1 Solvent to Sample for 15 minutes46
4.10	Effect of Temperature on the Amount of Wax Extracted
	(% age) using 20:1 Solvent to Sample for 15 minutes47
4.11	Effect of Temperature on the Amount of Wax Extracted
	(% age) using 24:1 Solvent to Sample for 15 minutes48
4.12	2Effect of Temperature on the Amount of Wax Extracted
	(% age) using 32:1 Solvent to Sample for 15 minutes49

4.13	Effect of Temperature on the Amount of Wax Extracted
	(% age) using 16:1 Solvent to Sample for 20 minutes50
4.14	Effect of Temperature on the Amount of Wax Extracted
	(% age) using 20:1 Solvent to Sample for 20 minutes51
4.15	Effect of Temperature on the Amount of Wax Extracted
	(% age) using 24:1 Solvent to Sample for 20 minutes52
4.16	Effect of Temperature on the Amount of Wax Extracted
	(% age) using 32:1 Solvent to Sample for 20 minutes53
4.17	Effect of Temperature on the Amount of Wax Extracted
	(% age) using 16:1 Solvent to Sample for 25 minutes54
4.18	Effect of Temperature on the Amount of Wax Extracted
	(% age) using 20:1 Solvent to Sample for 25 minutes55
4.19	Effect of Temperature on the Amount of Wax Extracted
	(% age) using 24:1 Solvent to Sample for 25 minutes56
4.20	Effect of Temperature on the Amount of Wax Extracted
	(% age) using 32:1 Solvent to Sample for 25 minutes57
4.21	Effect of Temperature on the Amount of Wax Extracted
	(% age) using 16:1 Solvent to Sample for 30 minutes58
4.22	Effect of Temperature on the amount of wax extracted
	(% age) using 20:1 Solvent to Sample for 30 minutes59
4.23	Effect of Temperature on the Amount of Wax Extracted
	(% age) using 24:1 Solvent to Sample for 30 minutes60
4.24	Effect of Temperature on the Amount of Wax Extracted
	(% age) using 32:1 Solvent to Sample for 30 minutes61
4.25	Effect of Time on the Amount of Wax Extracted
	(% age) using 16:1 Solvent to Sample at 28°C62

4.26	Effect of Time on the Amount of Wax Extracted
	(% age) using 20:1 Solvent to Sample at 28°C63
4.27	Effect of Time on the Amount of Wax Extracted
	(% age) using 24:1 Solvent to Sample at 28°C64
4.28	Effect of Time on the Amount of Wax Extracted
	(% age) using 32:1 Solvent to Sample at 28°C65
4.29	Effect of Time on the Amount of Wax Extracted
	(% age) using 16:1 Solvent to Sample at 40°C66
4.30	Effect of Time on the Amount of Wax Extracted
	(% age) using 20:1 Solvent to Sample at 40°C67
4.31	Effect of Time on the Amount of Wax Extracted
	(% age) using 24:1 Solvent to Sample at 40°C68
4.32	Effect of Time on the Amount of Wax Extracted
	(% age) using 32:1 Solvent to Sample at 40°C69
4.33	Effect of Time on the Amount of wax extracted
	(% age) using 16:1 Solvent to Sample at 50°C70
4.34	Effect of Time on the Amount of Wax Extracted
	(% age) using 20:1 Solvent to Sample at 50°C71
4.35	Effect of Time on the Amount of wax extracted
	(% age) using 24:1 Solvent to Sample at 50°C72
4.36	Effect of Time on the Amount of Wax Extracted
	(% age) using 32:1 Solvent to Sample at 50°C73
4.37	Effect of Time on the Amount of Wax Extracted
	(% age) using 16:1 Solvent to Sample at 60°C74
4.38	Effect of Time on the Amount of Wax Extracted
	(% age) using 20:1 Solvent to Sample at 60°C75

4.39	Effect of Time on the Amount of Wax Extracted
	(% age) using 24:1 Solvent to Sample at 60°C76
4.40	Effect of Time on the Amount of Wax Extracted
	(% age) using 32:1 Solvent to Sample at 60°C77
4.41	Effect of Time on the Amount of Wax Extracted
	(% age) using 16:1 Solvent to Sample at 70°C78
4.42	Effect of Time on the Amount of Wax Extracted
	(% age) using 20:1 Solvent to Sample at 70°C79
4.43	Effect of Time on the Amount of Wax Extracted
	(% age) using 24:1 Solvent to Sample at 70°C80
4.44	Effect of Time on the Amount of Wax Extracted
	(% age) using 32:1 Solvent to Sample at 70°C81
4.45	Effect of Solvent to Sample Ratio on the Amount
	of Wax Extracted (% age) at 28°C and 5 minutes82
4.46	Effect of Solvent to Sample Ratio on the Amount
	of Wax Extracted (% age) at 40°C and 5 minutes83
4.47	Effect of Solvent to Sample Ratio on the Amount
	of Wax Extracted (% age) at 50°C and 5 minutes84
4.48	Effect of Solvent to Sample Ratio on the Amount
	of Wax Extracted (% age) at 60°C and 5 minutes85
4.49	Effect of Solvent to Sample Ratio on the Amount
	of Wax Extracted (% age) at 70°C and 5 minutes86
4.50	Effect of Solvent to Sample Ratio on the Amount
	of Wax Extracted (% age) at 28°C and 10 minutes87
4.51	Effect of Solvent to Sample Ratio on the Amount
	of Wax Extracted (% age) at 40°C and 10 minutes88

4.52	Effect of Solvent to Sample Ratio on the Amount
	of Wax Extracted (% age) at 50°C and 10 minutes89
4.53	Effect of Solvent to Sample Ratio on the Amount
	of Wax Extracted (% age) at 60°C and 10 minutes90
4.54	Effect of Solvent to Sample Ratio on the Amount
	of Wax Extracted (% age) at 70°C and 10 minutes91
4.55	Effect of Solvent to Sample Ratio on the Amount
	of Wax Extracted (% age) at 28°C and 15 minutes92
4.56	Effect of Solvent to Sample Ratio on the Amount
	of Wax Extracted (% age) at 40°C and 15 minutes93
4.57	Effect of Solvent to Sample Ratio on the Amount
	of Wax Extracted (% age) at 50°C and 15 minutes94
4.58	Effect of Solvent to Sample Ratio on the Amount
	of Wax Extracted (% age) at 60°C and 15 minutes95
4.59	Effect of Solvent to Sample Ratio on the Amount
	of Wax Extracted (% age) at 70°C and 15 minutes96
4.60	Effect of Solvent to Sample Ratio on the Amount
	of Wax Extracted (% age) at 28°C and 20 minutes97
4.61	Effect of Solvent to Sample Ratio on the Amount
	of Wax Extracted (% age) at 40°C and 20 minutes98
4.62	Effect of Solvent to Sample Ratio on the Amount
	of Wax Extracted (% age) at 50°C and 20 minutes99
4.63	Effect of Solvent to sample Ratio on the Amount
	of Wax Extracted (% age) at 60°C and 20 minutes100
4.64	Effect of Solvent to Sample Ratio on the Amount
	of Wax Extracted (% age) at 70°C and 20 minutes101

4.65	Effect of Solvent to Sample Ratio on the Amount
	of Wax Extracted (% age) at 28°C and 25 minutes102
4.66	Effect of Solvent to Sample Ratio on the Amount
	of Wax Extracted (% age) at 40°C and 25 minutes103
4.67	Effect of Solvent to Sample Ratio on the Amount
	of Wax Extracted (% age) at 50°C and 25 minutes104
4.68	Effect of Solvent to Sample Ratio on the Amount
	of Wax Extracted (% age) at 60°C and 25 minutes105
4.69	Effect of Solvent to Sample Ratio on the Amount
	of Wax Extracted (% age) at 70°C and 25 minutes106
4.70	Effect of Solvent to Sample Ratio on the Amount
	of Wax Extracted (% age) at 28°C and 30 minutes107
4.71	Effect of Solvent to Sample Ratio on the Amount
	of Wax Extracted (% age) at 40°C and 30 minutes108
4.72	Effect of Solvent to Sample Ratio on the Amount
	of Wax Extracted (% age) at 50°C and 30 minutes109
4.73	Effect of Solvent to Sample Ratio on the Amount
	of Wax Extracted (% age) at 60°C and 30 minutes110
4.74	Effect of Solvent to Sample Ratio on the Amount
	of Wax Extracted (% age) at 70°C and 30 minutes111
5.1	Process Flow Sheet119
5.2	Overall Material Balance121
5.3	Diagram for Heater Energy Balance122
5.4	Diagram for Mixer (1) Energy Balance123
5.5	Diagram for Mixer (2) Energy Balance124

List of Maps

A.1 Nile Blend Fiel	ls136
---------------------	-------

List of Photographs

1.1 Paraffin Wax	7
B.1 Balance	
B.2 Filter	
B.3 Rotary Evaporator	139
B.4 Chiller	140
B.5 Magnetic Stirrer	142

CHAPTER ONE

INTRODUCTION

1. Waxes

Wax is one of the oldest and most versatile natural substances used by man. Egyptians used wax in the mummification process and for protective coatings 3000 years ago [1].

Wax is simply defined as substances which are solid at room temperature and get softer till it become liquid when raising their temperatures to about 40° C. It is a type of lipid that may contain a wide variety of long – chain alkanes, esters, polyesters and hydroxyl esters of long – chain primary alcohols and fatty acids [2].

1.1 Types of Wax [1][2][3][4]

Waxes are classified into natural and synthetic materials. Natural waxes are obtained from animals, insects and vegetable insects.

Waxes obtained from natural supplies, unlike synthetic waxes, are subject to weather conditions, which may severely affect the stability of supply and price and, to a lesser extent, the consistency of the products.

1.1.2 Insect and Animal waxes

White and yellow bee wax has been known for over 2000 years, especially through its use in the fine arts. Bees wax is secreted by bees and is used to

construct the combs in which bees store their honey. Removing the honey and melting the comb in boiling water harvest the wax; the melted product is then filtered and cast into cakes.

The yellow bee wax cakes can be bleached with oxidizing agents to white bee wax, a product much favored in the cosmetic industry. The major components of bee wax are esters of C_{31} to C_{32} alcohols with 16 acids, free C_{25} to C_{31} carboxylic acids, and C_{25} to C_{31} hydrocarbons. Bees wax has a melting point of 640°C, a penetration of 20 dmm at 25°C and 76

dmm at 43.3°C a viscosity of 1470 mm²/s at 98.90, an acid number of 20 and a specification number of 84.

Spermaceti is derived from the head oil of the perm whale. Owing to the present status of sperm whale as an endangered species.

1.1.3 Vegetable Waxes

Vegetable waxes include carnauba, candelilla, Japan wax, rice – bran wax, ouricury wax, joroba wax, caster wax and bayberry wax.

1.1.3.1Carnauba Wax

The source of carnauba wax is the palm trees, whose wax – producing stands grow almost exclusively. Carnuba wax forms on the fronds palm and is removed by cutting the fronds, drying and mechanically removing the impurities, which are removed from the wax by melting and filtering or centrifuging. The major components of carnauba wax are aliphatic and aromatic esters of long – chain alcohols and acids, with smaller amounts of free fatty acids and alcohols and resins.

1.1.3.2 Candelilla Wax

Candelilla wax is harvested from shrubs; the entire mature plant is uprooted and immersed in boiling water acidified with sulfuric acid. The wax floats to the surface and is filtered. The major components of candelilla wax are hydrocarbons, esters of long – chain alcohols and acids, long – chain alcohols, sterols, and neutral resins, and long – chain acids.

1.1.3.3 Japan Wax

Japan wax is a fat and is derived from the berries of a small tree native to Japan and China cultivated for its wax. Japan wax is composed of trigly cerides.

1.1.3.4 Ouricury Wax

Ouricury wax is a brown wax obtained from the fronds of a palm tree, which grows in Brazil. Ouricury is difficult to harvest, so sometimes used as a replacement for carnauba in applications that do not require a light – colored wax.

1.1.3.5 Rice – Bran Wax

Rice – bran wax is extracted from crude rice – bran oil, it can be degummed, the fatty acid content reduced by solvent extraction, and bleached

1.1.3.6 Jojoba Wax

Jojoba oil is obtained from the seeds of the jojoba plant, hydrogenated jojoba oil is a wax used in candles and other applications.

1.1.3.7 Castor Wax

Castor wax is catalytically hydrogenated castor bean oil. Castor wax is used primarily in the formulation of cosmetics. Derivative of castor wax are used as surfactants and plastics additives.

1.1.3.8 Bayberry Wax

Bayberry wax is removed from the surface of the berry of the bayberry shrub by boiling the berries in water and skimming the wax from the surface of the water. The wax is green and made up primarily of lauric, myristic, and palmitic acid esters.
1.1.4 Minerals Waxes

Mineral waxes include montan wax, peat waxes and ozokerite and ceresin waxes.

Montan wax is derived by solvent extraction. The composition of montan wax depends on the material from which it is extracted, but all contain varying amounts of wax, resin, and asphalt. Black montan wax may be further processed to remove resin and asphalt, is known as refined montan wax (White montan wax has been reacted with alcohols to form esters). The wax component of montan is a mixture of long – chain acids and long – chain alcohols, ketenes, and hydro – carbons.

Peat waxes contain three components: a wax fraction, a resin fraction, and an asphalt fraction. The wax fraction in peat wax is chemically similar to that of the wax fraction in montan wax.

True ozokerite no longer seems to be an article of commerce, and has been replaced with blends of petroleum derived paraffin and microcrystalline waxes. Ceresin wax originally was a refined and bleached ozokerite wax.

1.1.5 Petroleum Waxes [5][6][7][8][9][10]

Depending on their natural occurance and their crystalline nature, petroleum waxes are divided into:

- 1. Macrocrystalline waxes (paraffin waxes)
- 2. Microcrystalline waxes (micro waxes)

1.1.5.1 Macrocrystalline Waxes (Paraffin Waxes)

Paraffin waxes are obtained from light and middle lubricating oil cuts of vacuum distillation or from crude oil. Paraffin waxes also include waxes from heavy lubricating oil distillates, which are intermediates between macrocrystalline and microcrystalline waxes with regard to structure and composition (intermediate waxes).

Paraffin waxes consist predominantly of mixtures of straight chain alkanes in a typical distribution of the homologous series whose masses depend on the boiling range of the lubricating oil distillate from which they are obtained long-chain, weakly branched isoalkanes are present in a much lower proportion, alone with a very small fraction of monocyclic alkanes[11].

Paraffin wax occurs as a colorless, or white, more or less translucent mass showing a crystalline structure. It is without odor or taste and is slightly greasy to the touch as shown in Photograph 1.1. The melting point is indicated as being above 40°C [3]. Paraffin waxes are insoluble in water and sparingly soluble in alcohols and ethers. They are more sloluble in ketones, chlorohydrocarbons, petroleum spirit, solvent naphtha, benzene, toluene, xylene, and higher aromatic, especially at elevated temperature. The solubility decreases markedly with increasing molar mass (higher melting point) of the waxes [3][12][13].



Photograph. 1.1: Paraffin Wax

Paraffin waxes are extremely unreactive under normal conditions. Oxidation reactions occur only at elevated temperatures (e.g., on storage and above 100°C), particularly in the presence of oxygen and catalytically active metals. These reactions can be recognized from the burnt odor produced and the yellow to brown coloration of the waxes. Nevertheless, under certain

thermally and catalytically control conditions, these waxes can undergo chemical reactions such as chlorination, oxidation, dehydrogenation, and cracking, which are important in industry [3][12][13].

1.1.5.2 Microcrystalline Waxes (Micro Waxes)

Microcrystalline waxes originate from vacuum residues and from the sediments of paraffinic crude oil (settling waxes). Waxes that are liquid at room temperature are mostly contained in deisel oil or gas oil fractions and can be isolated from them.

Like paraffin waxes, microcrystalline waxes consist of mixtures of saturated hydrocarbons that are predominantly solid at room temperature, such as isoalkanes, naphthenes, and alkyle- and naphthenes- substituted aromatics. Unliked paraffin waxes, isoparaffins and naphthenic compounds predominate here. The microcrystalline structure can be explained by the presence of strongly branched isoparaffins and naphthenes, which inhibit crystallization[11].

Microcrystalline waxes are insoluble in water. They are moderately sloluble in solvents such as chlorohydrocarbons, solvent naphtha, benzene, toluene, xylene, and turpentine, especially at elevated temperature. The solubility decreases markedly with increasing molar mass. Solvents and oils are retained very strongly by microcrystalline waxes, therefore evaporate very slowly. The quality of some consumer products, such as petroleum jellies or floor and shoe polish, is determined by these retention capacity microwaxes. Microcrystalline waxes are more reactive than paraffin waxes because their higher concentration of complex branched hydrocarbons with tertiary carbon atoms. These C-C bonds are not very thermally stable (i.e. the waxes darker resinify) on prolong heating. In addition, they form black tar like substances on contact with aggressive chemicals such as concentrated sulfuric acid or antimony pentachloride. The reaction of microwaxes with oxygen at elevated temperature and in the presence of catalytically acting heavy-metal soaps is used for the production of oxidized microwaxes.

Depending on the degree of refining, microwaxes are divided into the following classes:

- 1. Bright stock slack waxes (petrolatum)
- 2. Plastic microwaxes
- 3. Hard microwaxes [3][11]

1.1.6 Synthetic Waxes

Synthetic waxes include polyethylene waxes, fischer – tropsch waxes, substituted amide waxes and polymerized – olefins waxes.

Low molecular weight polyethylene having wax like properties are made either by high – pressure polymerization or low – pressure polymerization.

Polyethylene wax production is based on the Fischer – Tropsch synthesis, which is basically the polymerization of a carbon monoxide under high pressure and over special catalysts to produce hydrocarbons. Distillation is

then used to separate hydrocarbons into different products, including liquid fuels and waxes with melting points ranging from about 45 - 106 °C.

The product of fatty acid amidation has unique wax like properties. Probably the most widely produced material is N, N`distearylethy lendiamine, which has melting point of 140°C, an acid number of 7, and a low melt viscosity. Because of its unusually high melting point and unique functionality, it is used in additive quantities to raise the apparent melting point of thermo plastic resins and asphalts, as an internal, external lubricant in the compounding of a variety thermo plastic resins, and as a processing aid for elastomers.

1.1.7 Polymerized a – Olefins

Some polymers of higher a – olefins. e.g., C > 20, have wax like properties and are sold as synthetic waxes. The polymerization process yields highly branched materials, with broad molecular weight distributions. Properties of the individual products are highly dependent on a – olefin monomers and polymerization conditions. Melting points for the products range from 540°C to 740°C, with number average molecular weights 2600 – 2800. The unique structure makes these products very effective when use d in additive amounts to modify the properties of paraffin wax, primarily for use as candles. The products can increase the hardness and opacity of the paraffin, without increasing the cloud point or viscosity. Other uses include mold release polyurethane foams, additives for leather treating.

1.1.8 Chemically Modified Waxes[14][15][16]

of the Hydrocarbon microcrystalline polyethylene, waxes and polyethyethylene classes are chemically modified to meet specific market needs. In the vast majority of cases, the first step is air oxidation of wax with or without catalysts. The product has an acid number usually no higher than 30 and a saponification number usually no lower than 25. An alternative step is the reaction of the wax with a poly carboxylic acid e.g., maleic, at high temperature. Through its carboxyl groups, the oxidized wax can be further modified in such reactions as saponification or esterification. Oxidized wax is easily emulsified in water through the use of surfactants or simple soaps, and is widely used in many coating and polish applications.

1.1.9 Characteristic Properties of Waxes [1][15]

- Solid at 20°C varying in consistency from soft and plastic to brittle hard.

- As solid as coarse to finely crystalline, transparent to opaque. But not glass – like.

- Have relatively low viscosity slightly above the melting point.
- Melt above 40°C without decomposition.
- Consistency and solubility are highly temperature dependent.
- Buffable under slight pressure.
- Burn with yellow flame.
- Can form pastes or gels when dispersed in solvent.
- Exhibit low thermal and electrical conductivity.

1.1.10 Functions and Effects of Waxes [1][2][15]

Waxes have numerous functions and effects, some of them are listed below:-

- Flatting in paint systems.
- Fuel in candles and fire logs.
- Mold release.
- Lubrication in plastic procession.
- Protection against UV attack in rubber.
- Coating and lamination in flexible packaging.
- Moisture proofing in fibre and chipboard.
- Pigment carrier for colour concentrate and dispersions.
- Imparting gloss and protection in polishes.
- Viscosity regulation in hot melt adhesives.

1.2 Refining of Petroleum [17][18][19]

Petroleum is a complex mixture of organic liquids called crude oil and natural gas, which occurs naturally in the ground and was formed millions of years ago. Crude oil varies from oilfield to oilfield in color and composition, from a pale yellow viscosity liquid to heavy black 'treacle' consistencies.

Crude oil and natural gas are extracted from the ground, on land or under the oceans, by sinking an oil well and are then transported by pipeline and / or ship to refineries where their components are processed into refined products. Crude oil and natural gas of little use in their raw state; their Value lies in what is created from them: fuels, lubricating oils, waxes, asphalt, petrochemicals and pipeline quality natural gas.

1.3 Refinery process [20][21][22][23]

Every refinery begins with the separation of crude oil into different fractions by distillation.

There are five categories of general refinery processes and associated operations:

1.3.1 Separation Process

Crude oil is a complex mixture of hydrocarbon compounds, including paraffinic, naphthenic and aromatic hydrocarbons plus small amount of some impurities including salt, sulfur, nitrogen, oxygen and metals. Desalting separates the salt from the crude oil by water addition to dissolve the salt followed by physical separation of the crude oil and water phases prior to distillation of the crude oil.

Atmospheric distillation is repeated at atmospheric pressures in order to separate higher boiling point fractions of the crude oil at normal pressure.

Vacuum distillation is repeated the process at reduced pressures in order to separate higher boiling point fractions of the crude oil that are not accomplished by atmospheric distillation.

Light end recovery is the separation of non-condensable gases (refinery fuel gas) from condensable hydrocarbons that the fuel gas can be used as refinery energy source.

1.3.2 Petroleum Conversion Process

Petroleum conversion process include catalytic cracking, thermal cracking, coking, alkylation and polymerization, and isomerization and reforming.

1.3.2.1 Catalytic Cracking

Catalytic cracking is used to break down large petroleum molecules into smaller petroleum molecules by using a catalyst and high temperature in order to break part long chain hydrocarbons to yield lower boiling point hydrocarbons suitable for the production of gasoline or fuel oil.

1.3.2.2 Thermal Cracking (Visbreaking)

Visbreaking, which is less commonly used, accomplishes the same thing using heat and high pressure, but without a catalyst.

1.3.2.3 Coking

Coking produces volatile hydrocarbons that are valuable from low volatility petroleum fractions that are not valuable.

1.3.2.4 Alkylation and Polymerization

Alkylation and polymerization processes are used to combine small petroleum molecules into larger molecules more suitable for use in gasoline production. They need a catalyst (phosphoric, hydrofluoric, or sulfuric acid) with pressure and / or heat to accomplish this.

1.3.2.5 Isomerization and Reforming

Isomerization and reforming processing rearrange the structure of petroleum molecules in order to produce molecules of similar molecular size to the original molecule but of higher value in the final refined product. A catalyst and heat are required in both isomerization and reforming processes.

1.3.2.6 Petroleum Treating Process

Petroleum treating processes stabilize and upgrade the petroleum products by separating them from less desirable products and by removing objectionable elements. Treating processes that are used in oil refineries include:

1.3.2.6.1 Hydro treating

Hydro treating employs a catalyst to react hydrogen, at high pressure and temperature, with the sulfur, nitrogen or oxygen in the crude oil to form hydrogen sulfide, ammonia and water.

1.3.2.6.2 Chemical Sweating

Chemical sweating employs caustic and air to convert odorous mercaptans in the petroleum to less odours disulfides.

1.3.2.6.3 Acid Gas Removal

Acid gas removal absorbs sulfur compounds into solution in order to separate them from the refinery fuel gas.

1.3.2.6.4 Deasphalting

Deasphalting is primarily used for the separation of asphaltenes and resins from vacuum distillation residues by extraction with a solvent such as refinery propane.

1.3.2.6.5 Asphalt blowing

Asphalt blowing is used primarily for polymerization and stabilizing asphalt in order to improve its weathering characteristics. This is accomplished by heating the asphalt in the presence of a flowing air stream.

1.3.2.6.6 Dewaxing Process

Dewaxing process is used to remove hydrocarbons that have high freezing point, i.e., long chain n – paraffin and to decrease the pour point lubricating base stocks. The process starts with vacuum distillation residue (VDR) from the vacuum distillation unit, where, the asphalt is removed. The deasphalted oil (DAO) from the deasphalted unit is sent to the dewaxingt unit from where lube oil is obtained. There are three methods to remove waxes from the lube oil, these are: solvent dewaxing, catalytic dewaxing and urea dewaxing [24][25].

1.4 Solvent Dewaxing

Solvent dewaxing is a physical separation process, where in the separation takes place by selective solubility. The principal solvents used in this process are either propane or ketones, with the most popular being methyl isobutyl ketone, methyl ethyl ketone with toluene, naphtha, or propane.

The ketone solvent are the most widely used, where as the propane solvents also requires the additional use of a dewaxing acid. In this process, solvent is mixed with the oil feed and is cooled by a series of chillers and heat exchangers while the wax crystallizes. The slurry of solvent and wax then proceed to the rotary filter where the wax and the oil are separated. The solvent and wax mixture is then sent to a flash column where they are separated by steam stripping. The oil and solvent mixture are then sent to a distillation unit. The oil then goes to a finishing step to produce the lube oil base stock. The solvent and water mixture is then separated and the solvent is recycled to the feed stream.

1.5 Catalytic Dewaxing

Catalytic dewaxing is a chemical process; where the chemical composition of the feed is changed. This involves cracking the long chains and adding hydrogen to prevent coke formation. The DAO feed is combined with hydrogen and proceeds to a reactor at $280 - 400^{\circ}$ C and 20 - 102 atmosphere, the feed is mixed in the reactor with a zeolite catalyst where selective catalytic cracking of the n – alkanes occurs. The product is then sent to a distillation unit. The bottoms product is the lube oil and the over head stream from the distillation unit contains naphtha which is further converted into gasoline. Because only the n – paraffins are cracked, the ratio of i – paraffins to n – paraffins is increased. The oil base stock yield of catalytic dewaxing is lower than that of solvent dewaxing.

1.6 Urea Dewaxing

It is known to dewax hydrocarbon mixtures or mineral oil distillates by reacting them with urea to form solid adducts (mixture of urea and organic compounds) of the n-paraffins and separating these adducts from the dewaxed mineral oil [26]. This process is called urea dewaxing and has been used in different embodiments for the large-scale refining of mineral oil.

Usually in this process, the mineral oil distillate charge is diluted with urea dewaxing solvent, i.e., an organic solvent or mixture of solvents in order to reduce the viscosity of the reaction mixture, to improve its pumping and mixing properties, and to increase the degree of separation of the adduct and the dewaxing selectivity.

1.7 Feedstock and Product Handling

These processes consist of unloading storage, blending, and loading activities. Each of these is a critical step in the successful operation of a petroleum refinery.

1.8 Auxiliary Facilities

Crude oil is used in function vital to the operation of the refinery. Examples of these are:

1.8.1 Steam Boilers

Steam boilers burn natural gas, refinery fuel gas or oil to supply steam heating for many process operations in an oil refinery.

1.8.2 Waste Water and Solid Waste Treatment

Wastewater treatment separate oil from the refinery wastewater and further treats the water by physical/chemical and/or biological processes prior to discharge.

Solid waste treatment separates solid refining process residues from liquid hydrocarbon fractions or waste by physical means.

1.8.3 Hydrogen Production

Hydrogen plants produce hydrogen for use in the hydro treating of certain crude oil fractions. They use petroleum hydrocarbons as feed stock and multiple processing steps in order to transform the hydrocarbons into hydrogen and other useful gases.

1.8.4 Sulfur Recovery Plants

Sulfur recovery plants react hydrogen sulfide, extracted from the refinery fuel gas, with sulfur dioxide over a catalyst at high temperature in order to produce a solid sulfur byproduct.

Cooling towers remove excess heat from many of the oil refining processes by water evaporation.

Blow down systems convey the contents of any process operation, to a flare, condensing systems or storage vessel.

Compressor engines bring gaseous process streams up to their required processing pressure.

1.9 Petroleum in the Sudan

Oil exploration in Sudan started in 1959, when Italy's Agib Oil Company was granted concessions in the red sea area, carrying out seismic surveys and drilling six wells. All yielded nothing for the next fifteen years [27].

The actual exploration operations began after the signature of an agreement with the American Chevron company in 1974. According to the good results in the central Sudan another bilateral agreement with Chevron was concluded in 1979. After that other agreements were concluded with French total company and the American Sun oil company in 1980-1982.

After the conduction of geological and geophysical surveys in different parts of the country 95 experimental wells were digged, 46 of them were productive such as the fields of Sawakin, Abu jabra, Sharif, the unity, Talh, Hejelij, Adareel and Kaigang, 49 wells are dry. But these explorations were not followed by and productive activity.

During the years 1989-1999 the Sudanese government concluded a number of agreements with different petroleum companies including the two Canadian companies IPC and SPC in 1991-1993, the Gulf Company 1995, and the Chinese National Petroleum Company in Feb. 1997. The international company GNPOC was established in 1997. As a result a number of the exploration companies carried their work in different parts of the country.

The petroleum production in Sudan began in Abu jabra and Sharif fields, followed by Adareel and Hejilig fields. The total production of petroleum in Sudan up to 1998 was over three million barrels per year, that Abu jabra and Sharif produced 471629 barrels, Adareel 196347 barrels and Hejilij 2517705. By the end of June 1999 the real production reached 150 thousand barrels per day from Hejilij and Unity fields [27].

The qualities of the Sudanese petroleum differ according to the fields, but it's most important qualities can be summarized in the followings:

- The crude Sudanese petroleum has a medium thickness similar to the light materials.
- It is categorized as one of the materials that include paraffin wax, which is chemical component of oil, which is a good burning material, and of high productivity in complex refining circumstances.
- The Sudanese crude oil is characterized by including a little quantity of sulfur, there fore its one of the best oils in the Middle East because sulfur has harmful side effects on the environment and the engines.
- The Sudanese petroleum is also characterized by having the specification of diesel derivative as the sixty's number is high and that raises the burning competence.

Although Sudanese crude oils contain a large percent of wax and the import of paraffin wax increase yearly, beside that wax causes problems of transportation through the pipelines, there is no industry of wax in Sudan. So it is important to study the manufacture of petroleum wax in Sudan.

1.10 Objective of the Study

The main objective of the present study is: to determine the best and most economical operating conditions for extraction of paraffin wax from Nile Blend petroleum.

CHAPTER TWO

LITERATURE REVIEW (PREVIOUS WORKS)

Solvent dewaxing method is the first and most applicable method for removal of petroleum waxes from crude oil and lubricant oil until now[20] [28].

Most researchers use this method for production of petroleum wax from waxy oils. In the coming pages, the works of some authors are reviewed.

Shaw, etal [29], mentioned that Wax-containing oil is chilled in an elongated chilling zone by introducing cold dewaxing solvent along the length of the cooling zone. The mixing of the chilled dewaxing solvent and wax-containing oil in the cooling zone is accomplished by static mixing means under conditions of plug flow radial mixing, thereby avoiding shock chilling without the need for the intense agitation and/or dynamic agitators normally required for such processes. Specific examples of suitable solvents are mixtures of MEK and MIBK, MEK and toluene, dichloromethane and dichloroethane, propylene and acetone. Preferred solvents are ketones. A particularly preferred solvent is a mixture of MEK and MIBK.

Watts, etal [30] made a process for dewaxing waxy petroleum distillate oil stock wherein one volume waxy oil stock is mixed with about 0.5-2 volumes aromatic hydrocarbon dewaxing solvent, and the oil/aromatic solvent mixture heated to a temperature for dissolving all solid wax therein, after heating a gradual cooling is done. The solid wax and dewaxed oil are recovered as products of the process. Dewaxing solvents which may be used

in solvent dewaxing processes include known dewaxing solvents. Commonly used solvents include aliphatic ketones of 3-6 carbon atoms, C_2 - C_4 range hydrocarbons, C_6 - C_7 aromatic hydrocarbons, halogenated C_1 - C_4 hydrocarbons, and mixtures of such solvents.

Ackerson, etal [31], stated that there are disclosed processes for separating waxes of different melting points from a room temperature amorphous by selectively causing precipitation of crystallized waxes. The processes involve the use of a selected co-solvent totally miscible with light and intermediate hydrocarbons from a group consisting of acetone, ketene, propanone, 2-propanone, methanol, ethanol, isopropanol, N-propanol, acetic acid, formic acid, and propionic acid or combinations thereof as а precipitating agent. Hydrocarbon mixtures, especially those with elevated pour points are first diluted by solvents such as touene and/or methyl ethyl ketone. The diluted hydrocarbon mixture at above 50°F. is mixed with one or more of such selected co-solvents in a ratio preferably between 1:1 and 10:1 by weight to the heavy hydrocarbon content of the mixture; five minutes or more without artificial cooling is allowed to permit crystallization of waxes which are removed in solid form by a physical process such as filtering, settling, or the like.

Ackerson, etal [32], made a process for dewaxing including the steps of mixing a waxy feedstock near its pour point with an ambient or below ambient temperature solvent essentially free of a selected cosolvent, to form a solvent/feedstock mixture, and subsequently adding the cosolvent to the solvent/feedstock mixture to cause instantaneous precipitation of wax on addition of cosolvent with the amount of wax precipitation being controlled

by the quantity and temperature of cosolvent added. The cosolvent is essentially completely miscible with the solvent, but immiscible with the oil and wax. For example, alcohols (methanol, ethanol, propanol), ketones (ketene, acetone), amines, etc.

Ryan, Douglas G [33], stated that a continuous, combination ketoneautorefrigerant solvent dewaxing process is disclosed wherein a waxy oil is partially solvent dewaxed to in a first chilling zone, preferably comprising a plurality of agitated stages in the presence of a ketone dewaxing solvent to form a slurry containing solid wax particles, partially dewaxed oil and solvent.

This ketone-containing slurry is passed to a second chilling zone, which is an autorefrigerant chilling zone, preferably employing liquid propylene operates on a continuous basis, and comprises a vertical, multi-staged tower, operating at constant pressure, wherein additional wax is precipitated from the slurry. In the second chilling zone the slurry is chilled down to the wax filtration temperature by stagewise contact with liquid propylene which is injected into a plurality of said stages and evaporated therein so as to cool the waxy slurry at an average rate of between about 0.1° to 20° F. per minute with an average each stage of between about 2° temperature drop across and 20° F. Some of the propylene remains in the oil which serves to further dilute and reduce the viscosity of the slurry. The dewaxed oil-containing slurry may then be fed directly to wax filters without having to pass through scraped-surface chillers and filter feed drum.

27

Broadhurst, Thomas E [34], made an improved process for dewaxing waxy hydrocarbon oils, wherein waxy oil is cooled in an indirect chilling zone to a temperature greater than the wax separation temperature whereby wax is precipitated to form a wax-oil-solvent slurry, cooling the slurry to the wax separation temperature in an indirect chilling zone thereby precipitating a further portion of wax from waxy oil and separating precipitated wax from the wax-oil-solvent slurry in solid-liquid separation means, the improvement comprising using as the indirect chilling zone an indirect heat exchanger means operated at a high level of agitation. Expressed in terms of Impeller Reynolds Number the agitation is on the order of about 1,000 to 1,000,000. Alternatively, the direct chilling zone is totally replaced by the high agitation indirect heat exchanger means.

It is clear from the above review that the basic method for extraction of wax from crude petroleum, namely, solvent extraction followed by chilling remained unchanged. The contribution of the above authors concentrated on improvements either to increase the efficiency of the process or to reduce the cost of production.

CHAPTER 3

MATERIALS AND METHODS

3.1 Introduction

It was mentioned before (section 1.2.3) that the method of solvent extraction is widely used to separate wax from petroleum. Solvent extraction is a process which depends on the nature of the extractive solvent, the temperature of the solvent, the time of contact between the solvent and the extracted material and the ratio of the quantity of the solvent to the extracted material.

In this experimental part, the effect of these variables on the quantity of wax extracted from Nile Blend oil will be studied.

3.2 Materials

3.2.1 NILE BLEND

Two litres of Nile Blend mixture (in a plastic container) have been obtained from The Central Petroleum Laboratories (CPL), Ministry of Energy and Mining on 6/2/2007. The properties of the Nile Blend are shown in Table 3.1.

Property	Test Method	Unit	Result
API Gravity	IP Table51	Degree	34.81
Salt Content	ASTM D 3230	mgNaCl/L	9.12
Density at 15°C	ASTM D 5002	Kg/m ³	850.4
Wax Content	UOP 46	%m	25.87
Flash Point	IP 170	°C	21.5
Pour Point	ASTM D97	°C	+33.0
Kinematic Viscosity at50°C	ASTM D 445	mm²⁄s	22.64
Kinematic Viscosity at 80°C	ASTM D 445	mm²⁄s	8.884
Water Content by Distillation	ASTM D 4006	%V	0.09
Ash content	ASTM D 482	%m∕ m	0.043
Asphaltene Content	IP 143	wt%	0.11
Sulfur Content	ASTM D4294	wt%	0.051

Tab. 3.1: Properties of Nile Blend Petroleum 2005 [27].

Nile Blend is a mixture of oil from various fields: Hegilig, Unity, Elnar, Eltoor, bamboo, Manga, Diffra and Toma South. These fields are located in the Muglad Basin in the Unity State (Fig (A.1)). The quality of Nile Blend is similar to Indonesia's Minas crude. The producers of this field make up a consortium known as the Greater Nile Petroleum Operating Company (GNPOC). GNPOC owns the 930000 km pipe line which transports oil from oil field to Bashair sea port, Red Sea State. Table 3.2 shows the different contribution of the each field in the Nile Blend.

No	Field	Ratio (vol %)
1	Toma South	24.48
2	Eltoor	4.31
3	Elnar	19.05
4	Bambo	4.79
5	Manga	7.37
6	Diffra	7.12
7	Unity	20.03
8	Hejlieg	12.85

Tab.3.2: Contribution of each field in the Nile Blend

Main marketing characteristics of Nile Blend:

- Sweet (5% wt sulfur).
- Light (34.81 API Gravity). API (27 38) for light petroleum crude oil.
- Waxy (25.87%).

3.2.2 Solvents

Methyl ethyl ketone (MEK), Toluene and Petroleum Naphtha are used as solvents for wax separation. These three solvents have been chosen as extractive liquids for petroleum wax by many other researchers [35][36] due to their high ability for dewaxing crude oils.

MEK is used separately, mixed with Toluene, or with Toluene and Naphtha. MEK and Toluene is used due to the ability of MEK to minimize the wax solubility, toluene is a good solvent for hydrocarbons and has a quite high solubility for wax so the two solvents were mixed with a high percentage of MEK [37][38].

A mixture of three solvents is used because Naphtha-Toluene is necessary to avoid the separation of two liquid-phases at lower temperatures, which could occur in heavy distillates.

The physical properties of these solvents are given in Table 3.3

No	Physical Properties	Solvent				
		MEK	Toluene	Naphtha		
1	Molecular Formula	C ₄ H ₈ O	C ₇ H ₈	C ₅ H ₁₂		
2	Molecular Weight	72	92.1	72		
3	Boiling Point °C	79.6	111	155-217		
4	Melting Point °C	-86	95	0		
5	Flash Point °C	-6	4	40-60		
6	Vapor Pressure at 20 °C (mmHg)	77.5	22	0.1-0.3		
7	Vapor Density (air=1 at boiling point of MEK and Toluene kg/m ³ at 15 °C	2.42	3.14	718.9		
8	Lower Explosive Limit %	2	1.2	0.7		
9	Upper Explosive Limit %	10	77	6.0		
10	Solubility in Water at 20 °C	27.5%	Nearly Insoluble	Insoluble		
11	Manufacture	England	England	England		
12	Purity %	98	98	97		

Tab. 3.3: Physical Properties of Solvents [27].

3.3 Method

Production of wax takes place in two steps: Deasphalting and Dewaxing of crude oil as shown in Fig 3.1. The details of each step are described below.

3.3.1 Deasphalting

The removal of asphaltene prior to dewaxing is necessary, due to the fact that asphaltene will contaminate the extracted wax and cause solvent and heat losses during the extraction step [39].

For each sample under test, two grams of crude oil was weighed and placed in 500 ml conical flask, heated to 60°C in an electrical oven for 30 min. after then, the sample was shaken till homogeneous solution is obtained.

Three hundred mills of Naphtha and 15 grams of dry Fuller's Earth was then added to the sample. The mixture was, then, thoroughly agitated using magnetic stirrer for 20 minutes, allowed to stand for 20 minutes before it was filtered using filter paper(50 mm) and glass wool in a Gooch crucible (see Appendix B, section B.10) to remove Fuller's Earth and asphaltene as residue. The asphaltene free filtrate was dried from Naphtha using evaporator under vacuum suction.

3.3.2 Dewaxing

Dewaxing step was made in two steps; in the first step the deasphalted oil was mixed with the certain amount of test solvent, manually shaked,

wrapped with aluminum foil to prevent evaporation, put on a magnetic stirrer with heater and allowed to be heated at certain temperature and time.

The experiment was repeated with other deasphalted oil at different temperatures $(28 - 70^{\circ}C)$, different time (5 - 30 min.) and different volumes of solvent to samples (16:1 - 32:1). In the second step, the solution of oil in solvent was then chilled to $- 17^{\circ}C$ in a chiller (Appendix 2, section B.10) for 30 minutes. The separated wax from the mother solution was filtered under vacuum suction through glass wool in a Gooch crucible, The flask containing the mother solution was rinsed with 20 ml of fresh solvent at $- 17^{\circ}C$ to ensure that all components have been removed.

The wax collected on the glass wool was dissolved by washing with 100 ml of warm Naphtha. Naphtha was then removed from the wax by evaporation using vacuum evaporator. The wax was then dried from any traces of Naphtha by leaving it for 24 hours in a dessicator containing P_2O_5 before being weighed.

The method described above was used for the following solvents

1- Pure MEK.

- 2- Mixer of 75% MEK and 25% Toluene.
- 3- Mixer of 50% MEK and 50% Toluene.
- 4- Mixer of 70% MEK and 20% Toluene and 10% Naphtha.

The amount of wax extracted by each test, was calculated as a percentage from the test sample (2 grams) using the following equation:

Wax, wt % =
$$[(W_2 - W_1)/S] \times 100 \%$$
 (3.1)

Where W_1 = weight of empty flask, gram. W_2 = weight of flask + wax, grams. S = Sample weight, 2 grams.



Fig. 3.1: Experimental Steps

CHAPTER FOUR

4. RESULTS AND DISCUSSIONS

4.1 Results

The results of the experiments are shown in page (37) to page (97). The calculations are present on the Appendix C.

The sample of Nile Blend crude oil which was used for the experiments contains 25.87% by mass paraffin wax (Table 3.1) which was determined by Central Petroleum Laboratories.

The amount of wax extracted at different conditions of temperatures, time and ratio of solvent to oil sample are shown on Tables 4.1 - 4.74, and Figures 4.1 - 4.74.

Tables 4.1 to 4.74 are summarized in Tables 4.75 and 4.76 respectively.

Temperature C	28	40	50	60	70
Solvent					
MEK	19	20	21	21	21
75% MEK, 25%Toluene	18	19	20	21	22
50%MEK, 50% Toluene	18	19	20	20	20
70%MEK, 20%Toluene	18	20	20	20	20
and 10% Naphtha					

Tab. 4.1: Effect of Temperature on the amount of wax extracted (% age) using 16:1 Solvent to Sample for 5 minutes.



Fig. 4.1: Effect of Temperature on the amount of wax extracted (% age) using 16:1 Solvent to Sample for 5 minutes.

Temperature [•] C	28	40	50	60	70
Solvent					
MEK	20	21	23	23	24
75% MEK, 25%Toluene	19	20	20	21	22
50%MEK, 50% Toluene	19	20	20	21	21
70%MEK, 20%Toluene	19	20	20	20	21
and 10% Naphtha					

Tab. 4.2: Effect of Temperature on the amount of wax extracted (% age) using 20:1 Solvent to Sample for 5 minutes.



Fig. 4.2: Effect of Temperature on the amount of wax extracted (% age) using 20:1 Solvent to Sample for 5 minutes.

Temperature [•] C	28	40	50	60	70
Solvent					
MEK	20	21	23	23	23
75% MEK, 25%Toluene	19	20	21	21	23
50%MEK, 50% Toluene	19	20	21	23	23
70%MEK, 20%Toluene	19	20	21	21	22
and 10% Naphtha					

Tab. 4.3: Effect of Temperature on the amount of wax extracted (% age) using 24:1 Solvent to Sample for 5 minutes.



Fig. 4.3: Effect of Temperature on the amount of wax extracted (% age) using 24:1 Solvent to Sample for 5 minutes.
Temperature C	28	40	50	60	70
Solvent					
MEK	20	21	23	23	24
75% MEK, 25%Toluene	19	20	21	23	23
50%MEK, 50% Toluene	19	20	21	23	23
70%MEK, 20%Toluene	19	20	21	22	22
and 10% Naphtha					

Tab. 4.4: Effect of Temperature on the amount of wax extracted (% age) using 32:1 Solvent to Sample for 5 minutes.



Fig. 4.4: Effect of Temperature on the amount of wax extracted (% age) using 32:1 Solvent to Sample for 5 minutes.

Temperature C	28	40	50	60	70
Solvent					
MEK	20	21	22	22	23
75% MEK, 25%Toluene	19	21	21	22	22
50%MEK, 50% Toluene	19	21	21	21	22
70%MEK, 20%Toluene	19	21	21	21	22
and 10% Naphtha					

Tab. 4.5: Effect of Temperature on the amount of wax extracted (% age) using 16:1 Solvent to Sample for 10 minutes.



Fig. 4.5: Effect of Temperature on the amount of wax extracted (% age) using 16:1 Solvent to Sample for 10 minutes.

Temperature C	28	40	50	60	70
Solvent					
MEK	22	22	23	23	24
75% MEK, 25%Toluene	20	22	22	23	23
50%MEK, 50% Toluene	20	21	22	22	23
70%MEK, 20%Toluene	19	21	21	22	22
and 10% Naphtha					

Tab. 4.6: Effect of Temperature on the amount of wax extracted (% age)using 20:1 Solvent to Sample for 10 minutes.



Fig. 4.6: Effect of Temperature on the amount of wax extracted (% age) using 20:1 Solvent to Sample for 10 minutes.

Temperature [•] C	28	40	50	60	70
Solvent					
MEK	22	23	23	23	24
75% MEK, 25%Toluene	20	22	22	23	24
50%MEK, 50% Toluene	20	21	22	23	23
70%MEK, 20%Toluene	21	21	22	22	22
and 10% Naphtha					

Tab. 4.7: Effect of Temperature on the amount of wax extracted (% age)using 24:1 Solvent to Sample for 10 minutes.



Fig. 4.7: Effect of Temperature on the amount of wax extracted (% age) using 24:1 Solvent to Sample for 10 minutes.

Temperature C	28	40	50	60	70
Solvent					
MEK	22	23	23	24	24
75% MEK, 25%Toluene	21	22	22	23	24
50%MEK, 50% Toluene	21	22	23	23	23
70%MEK, 20%Toluene	21	21	22	22	22
and 10% Naphtha					

Tab. 4.8: Effect of Temperature on the amount of wax extracted (% age) using 32:1 Solvent to Sample for 10 minutes.



Fig. 4.8: Effect of Temperature on the amount of wax extracted (% age) using 32:1 Solvent to Sample for 10 minutes.

Temperature [•] C	28	40	50	60	70
Solvent					
MEK	21	22	22	23	23
75% MEK, 25%Toluene	20	22	22	22	23
50%MEK, 50% Toluene	21	21	21	21	22
70%MEK, 20%Toluene	20	21	21	21	22
and 10% Naphtha					

Tab. 4.9: Effect of Temperature on the amount of wax extracted (% age) using 16:1 Solvent to Sample for 15 minutes.



Fig. 4.9: Effect of Temperature on the amount of wax extracted (% age) using 16:1 Solvent to Sample for 15 minutes.

Temperature [•] C	28	40	50	60	70
Solvent					
MEK	23	23	23	24	24
75% MEK, 25%Toluene	21	22	22	23	23
50%MEK, 50% Toluene	21	22	22	22	23
70%MEK, 20%Toluene	20	21	21	22	22
and 10% Naphtha					

Tab. 4.10: Effect of Temperature on the amount of wax extracted (% age) using 20:1 Solvent to Sample for 15 minutes.



Fig. 4.10: Effect of Temperature on the amount of wax extracted (% age) using 20:1 Solvent to Sample for 15 minutes.

Temperature [•] C	28	40	50	60	70
Solvent					
MEK	23	23	23	24	24
75% MEK, 25%Toluene	21	22	22	23	24
50%MEK, 50% Toluene	21	22	22	23	23
70%MEK, 20%Toluene	21	22	22	22	22
and 10% Naphtha					

Tab. 4.11: Effect of Temperature on the amount of wax extracted (% age)using 24:1 Solvent to Sample for 15 minutes.



Fig. 4.11: Effect of Temperature on the amount of wax extracted (% age) using 24:1 Solvent to Sample for 15 minutes.

Temperature [•] C	28	40	50	60	70
Solvent					
MEK	23	23	23	24	24
75% MEK, 25%Toluene	21	22	23	23	24
50%MEK, 50% Toluene	21	22	23	23	23
70%MEK, 20%Toluene	22	22	22	23	23
and 10% Naphtha					

Tab. 4.12: Effect of Temperature on the amount of wax extracted (% age) using 32:1 Solvent to Sample for 15 minutes.



Tab. 4.12: Effect of Temperature on the amount of wax extracted (% age) using 32:1 Solvent to Sample for 15 minutes.

Temperature C	28	40	50	60	70
Solvent					
MEK	21	23	24	24	25
75% MEK, 25%Toluene	21	22	23	23	24
50%MEK, 50% Toluene	21	21	22	22	23
70%MEK, 20%Toluene	20	22	22	22	23
and 10% Naphtha					

Tab. 4.13: Effect of Temperature on the amount of wax extracted (% age)using 16:1 Solvent to Sample for 20 minutes.



Fig. 4.13: Effect of Temperature on the amount of wax extracted (% age) using 16:1 Solvent to Sample for 20 minutes.

Temperature [•] C	28	40	50	60	70
Solvent					
MEK	23	24	25	25	26
75% MEK, 25%Toluene	21	23	24	24	25
50%MEK, 50% Toluene	21	22	22	23	24
70%MEK, 20%Toluene	20	22	22	23	24
and 10% Naphtha					

Tab. 4.14: Effect of Temperature on the amount of wax extracted (% age)using 20:1 Solvent to Sample for 20 minutes.



Fig. 4.14: Effect of Temperature on the amount of wax extracted (% age) using 20:1 Solvent to Sample for 20 minutes.

Temperature [•] C	28	40	50	60	70
Solvent					
MEK	23	24	25	25	26
75% MEK, 25%Toluene	22	23	24	25	25
50%MEK, 50% Toluene	22	23	23	24	25
70%MEK, 20%Toluene	21	23	23	23	24
and 10% Naphtha					

Tab. 4.15: Effect of Temperature on the amount of wax extracted (% age)using 24:1 Solvent to Sample for 20 minutes.



Fig. 4.15: Effect of Temperature on the amount of wax extracted (% age) using 24:1 Solvent to Sample for 20 minutes.

Temperature [•] C	28	40	50	60	70
Solvent					
MEK	24	24	25	25	26
75% MEK, 25%Toluene	22	24	24	25	25
50%MEK, 50% Toluene	22	23	23	25	25
70%MEK, 20%Toluene	22	23	23	23	24
and 10% Naphtha					

Tab. 4.16: Effect of Temperature on the amount of wax extracted (% age)using 32:1 Solvent to Sample for 20 minutes.



Fig. 4.16: Effect of Temperature on the amount of wax extracted (% age) using 32:1 Solvent to Sample for 20 minutes.

Temperature [•] C	28	40	50	60	70
Solvent					
MEK	21	23	24	25	25
75% MEK, 25%Toluene	21	22	23	23	24
50%MEK, 50% Toluene	22	22	22	22	23
70%MEK, 20%Toluene	21	22	22	23	24
and 10% Naphtha					

Tab. 4.17: Effect of Temperature on the amount of wax extracted (% age)using 16:1 Solvent to Sample for 25 minutes.



Fig. 4.17: Effect of Temperature on the amount of wax extracted (% age) using 16:1 Solvent to Sample for 25 minutes.

Temperature C	28	40	50	60	70
Solvent					
MEK	24	24	25	26	26
75% MEK, 25%Toluene	22	23	24	24	25
50%MEK, 50% Toluene	22	23	23	23	24
70%MEK, 20%Toluene	21	23	23	24	24
and 10% Naphtha					

Tab. 4.18: Effect of Temperature on the amount of wax extracted (% age)using 20:1 Solvent to Sample for 25 minutes.



Fig. 4.18: Effect of Temperature on the amount of wax extracted (% age) using 20:1 Solvent to Sample for 25 minutes.

Temperature [•] C	28	40	50	60	70
Solvent					
MEK	24	25	25	26	26
75% MEK, 25%Toluene	22	24	24	25	25
50%MEK, 50% Toluene	22	23	23	24	25
70%MEK, 20%Toluene	22	23	23	24	24
and 10% Naphtha					

Tab. 4.19: Effect of Temperature on the amount of wax extracted (% age)using 24:1 Solvent to Sample for 25 minutes.



Fig. 4.19: Effect of Temperature on the amount of wax extracted (% age) using 24:1 Solvent to Sample for 25 minutes.

Temperature [•] C	28	40	50	60	70
Solvent					
MEK	24	25	25	26	26
75% MEK, 25%Toluene	23	24	24	25	25
50%MEK, 50% Toluene	23	23	24	25	25
70%MEK, 20%Toluene	22	23	24	24	24
and 10% Naphtha					

Tab. 4.20: Effect of Temperature on the amount of wax extracted (% age)using 32:1 Solvent to Sample for 25 minutes.



Fig. 4.20: Effect of Temperature on the amount of wax extracted (% age) using 32:1 Solvent to Sample for 25 minutes.

Temperature [•] C	28	40	50	60	70
Solvent					
MEK	22	23	24	25	26
75% MEK, 25%Toluene	22	22	23	23	24
50%MEK, 50% Toluene	22	22	22	22	23
70%MEK, 20%Toluene	21	22	22	23	23
and 10% Naphtha					

Tab. 4.21: Effect of Temperature on the amount of wax extracted (% age)using 16:1 Solvent to Sample for 30 minutes.



Fig. 4.21: Effect of Temperature on the amount of wax extracted (% age) using 16:1 Solvent to Sample for 30 minutes.

Temperature [•] C	28	40	50	60	70
Solvent					
MEK	24	24	25	25	26
75% MEK, 25%Toluene	22	23	24	24	25
50%MEK, 50% Toluene	22	23	23	23	25
70%MEK, 20%Toluene	21	23	23	24	24
and 10% Naphtha					

Tab. 4.22: Effect of Temperature on the amount of wax extracted (% age)using 20:1 Solvent to Sample for 30 minutes.



Fig. 4.22: Effect of Temperature on the amount of wax extracted (% age) using 20:1 Solvent to Sample for 30 minutes.

Temperature [•] C	28	40	50	60	70
Solvent					
MEK	24	25	25	26	26
75% MEK, 25%Toluene	23	24	24	25	26
50%MEK, 50% Toluene	23	23	23	24	25
70%MEK, 20%Toluene	22	23	23	24	25
and 10% Naphtha					

Tab. 4.23: Effect of Temperature on the amount of wax extracted (% age)using 24:1 Solvent to Sample for 30 minutes.



Fig. 4.23: Effect of Temperature on the amount of wax extracted (% age) using 24:1 Solvent to Sample for 30 minutes.

Temperature [•] C	28	40	50	60	70
Solvent					
MEK	24	25	26	26	26
75% MEK, 25%Toluene	23	25	25	26	26
50%MEK, 50% Toluene	23	23	24	25	25
70%MEK, 20%Toluene	22	23	24	24	25
and 10% Naphtha					

Tab. 4.24: Effect of Temperature on the amount of wax extracted (% age) using 32:1 Solvent to Sample for 30 minutes.



Fig. 4.24: Effect of Temperature on the amount of wax extracted (% age) using 32:1 Solvent to Sample for 30 minutes.

Time (min)	5	10	15	20	25	30
Solvent						
MEK	19	20	21	21	21	22
75%MEK,25%Toluene	18	19	20	21	21	22
50%MEK, 50%Toluene	18	19	21	21	22	22
70% MEK, 20%Toluene	18	19	20	20	21	21
and 10% Naphtha						

Tab. 4.25: Effect of Time on the amount of wax extracted (% age) using 16:1 Solvent to Sample at 28°C.



Fig. 4.25 Effect of Time on the amount of wax extracted (% age) using 16:1 Solvent to Sample at 28°C.

Time (min)	5	10	15	20	25	30
Solvent						
MEK	20	22	23	23	24	24
75%MEK,25%Toluene	19	20	21	21	22	22
50%MEK, 50%Toluene	19	20	21	21	22	22
70% MEK, 20%Toluene	19	19	20	20	21	21
and 10% Naphtha						

Tab. 4.26: Effect of Time on the amount of wax extracted (% age) using 20:1 Solvent to Sample at 28°C.



Fig. 4.26: Effect of Time on the amount of wax extracted (% age) using 20:1 Solvent to Sample at 28°C.

Time (min)	5	10	15	20	25	30
Solvent						
MEK	20	22	23	23	24	24
75%MEK,25%Toluene	19	20	21	22	22	23
50%MEK, 50%Toluene	19	20	21	22	22	23
70% MEK, 20%Toluene	19	21	21	21	22	22
and 10% Naphtha						

Tab. 4.27: Effect of Time on the amount of wax extracted (% age) using 24:1 Solvent to Sample at 28°C.



Fig. 4.27: Effect of Time on the amount of wax extracted (% age) using 24:1 Solvent to Sample at 28°C.

Time (min)	5	10	15	20	25	30
Solvent						
MEK	20	22	23	24	24	24
75%MEK,25%Toluene	19	21	21	22	23	23
50%MEK, 50%Toluene	19	21	21	22	23	23
70% MEK, 20%Toluene	19	21	22	22	22	22
and 10% Naphtha						

Tab. 4.28: Effect of Time on the amount of wax extracted (% age) using 32:1 Solvent to Sample at 28°C.



Tab. 4.28: Effect of Time on the amount of wax extracted (% age) using 32:1 Solvent to Sample at 28°C.

Time (min)	5	10	15	20	25	30
Solvent						
MEK	20	21	22	23	23	23
75%MEK,25%Toluene	19	21	22	22	22	22
50%MEK, 50%Toluene	19	21	21	21	22	22
70% MEK, 20%Toluene	20	21	21	22	22	22
and 10% Naphtha						

Tab. 4.29: Effect of Time on the amount of wax extracted (% age) using 16:1 Solvent to Sample at 40°C.



Fig. 4.29: Effect of Time on the amount of wax extracted (% age) using 16:1 Solvent to Sample at 40°C.

Time (min)	5	10	15	20	25	30
Solvent						
MEK	21	22	23	24	24	24
75%MEK,25%Toluene	20	22	22	23	23	23
50%MEK, 50%Toluene	20	21	22	22	23	23
70% MEK, 20%Toluene	20	21	21	22	23	23
and 10% Naphtha						

Tab. 4.30: Effect of Time on the amount of wax extracted (% age) using 20:1 Solvent to Sample at 40°C.



Fig. 4.30: Effect of Time on the amount of wax extracted (% age) using 20:1 Solvent to Sample at 40°C.

Time (min)	5	10	15	20	25	30
Solvent						
MEK	21	23	23	24	25	25
75%MEK,25%Toluene	20	22	22	23	24	24
50%MEK, 50%Toluene	20	21	22	23	23	24
70% MEK, 20%Toluene	20	21	22	23	23	24
and 10% Naphtha						

Tab. 4.31: Effect of Time on the amount of wax extracted (% age) using 24:1 Solvent to Sample at 40°C.



Fig. 4.31: Effect of Time on the amount of wax extracted (% age) using 24:1 Solvent to Sample at 40°C.

Time (min)	5	10	15	20	25	30
Solvent						
MEK	21	23	23	24	25	25
75%MEK,25%Toluene	20	22	22	24	24	25
50%MEK, 50%Toluene	20	21	22	23	23	23
70% MEK, 20%Toluene	20	21	22	23	23	23
and 10% Naphtha						

Tab. 4.32: Effect of Time on the amount of wax extracted (% age) using 32:1 Solvent to Sample at 40°C.



Fig. 4.32: Effect of Time on the amount of wax extracted (% age) using 32:1 Solvent to Sample at 40°C.

Time (min)	5	10	15	20	25	30
Solvent						
MEK	21	22	22	24	24	24
75%MEK,25%Toluene	20	21	22	23	23	23
50%MEK, 50%Toluene	20	21	21	22	22	22
70% MEK, 20%Toluene	20	21	21	22	22	22
and 10% Naphtha						

Tab. 4.33: Effect of Time on the amount of wax extracted (% age) using 16:1 Solvent to Sample at 50°C.



Fig. 4.33: Effect of Time on the amount of wax extracted (% age) using 16:1 Solvent to Sample at 50°C.

Time (min)	5	10	15	20	25	30
Solvent						
MEK	23	23	23	25	25	25
75%MEK,25%Toluene	20	22	22	24	24	24
50%MEK, 50%Toluene	20	22	22	22	23	23
70% MEK, 20%Toluene	20	21	21	22	23	23
and 10% Naphtha						

Tab. 4.34: Effect of Time on the amount of wax extracted (% age) using 20:1 Solvent to Sample at 50°C.



Fig. 4.34: Effect of Time on the amount of wax extracted (% age) using 20:1 Solvent to Sample at 50°C.

Time (min)	5	10	15	20	25	30
Solvent						
MEK	23	23	23	25	25	25
75%MEK,25%Toluene	21	22	22	24	24	24
50%MEK, 50%Toluene	21	22	22	23	23	23
70% MEK, 20%Toluene	21	22	22	23	23	23
and 10% Naphtha						

Tab. 4.35: Effect of Time on the amount of wax extracted (% age) using 24:1 Solvent to Sample at 50°C.



Fig. 4.35: Effect of Time on the amount of wax extracted (% age) using 24:1 Solvent to Sample at 50°C.

Time (min)	5	10	15	20	25	30
Solvent						
MEK	23	23	23	25	25	26
75%MEK,25%Toluene	21	22	23	24	24	25
50%MEK, 50%Toluene	21	23	23	23	24	24
70% MEK, 20%Toluene	21	22	22	23	24	24
and 10% Naphtha						

Tab. 4.36: Effect of Time on the amount of wax extracted (% age) using 32:1 Solvent to Sample at 50°C.



Fig. 4.36: Effect of Time on the amount of wax extracted (% age) using 32:1 Solvent to Sample at 50°C.

Time (min)	5	10	15	20	25	30
Solvent						
MEK	21	22	23	24	25	25
75%MEK,25%Toluene	21	22	22	23	23	23
50%MEK, 50%Toluene	20	21	21	22	22	22
70% MEK, 20%Toluene	20	21	21	22	23	23
and 10% Naphtha						

Tab. 4.37: Effect of Time on the amount of wax extracted (% age) using 16:1 Solvent to Sample at 60°C.



Fig. 4.37: Effect of Time on the amount of wax extracted (% age) using 16:1 Solvent to Sample at 60°C.

Time (min)	5	10	15	20	25	30
Solvent						
MEK	23	23	24	25	26	26
75%MEK,25%Toluene	21	23	23	24	24	24
50%MEK, 50%Toluene	21	22	22	23	23	23
70% MEK, 20%Toluene	20	22	22	23	24	24
and 10% Naphtha						

Tab. 4.38: Effect of Time on the amount of wax extracted (% age) using 20:1 Solvent to Sample at 60°C.



Fig. 4.38: Effect of Time on the amount of wax extracted (% age) using 20:1 Solvent to Sample at 60°C.

Time (min)	5	10	15	20	25	30
Solvent						
MEK	23	23	24	25	26	26
75%MEK,25%Toluene	21	23	23	25	25	25
50%MEK, 50%Toluene	23	23	23	24	24	24
70% MEK, 20%Toluene	21	22	22	23	24	24
and 10% Naphtha						

Tab. 4.39: Effect of Time on the amount of wax extracted (% age) using 24:1 Solvent to Sample at 60°C.




Time (min)	5	10	15	20	25	30
Solvent						
MEK	23	24	24	25	26	26
75%MEK,25%Toluene	23	23	23	25	25	26
50%MEK, 50%Toluene	23	23	23	25	25	25
70% MEK, 20%Toluene	22	22	23	23	24	24
and 10% Naphtha						

Tab. 4.40: Effect of Time on the amount of wax extracted (% age) using 32:1 Solvent to Sample at 60°C.





Time (min)	5	10	15	20	25	30
Solvent						
MEK	21	23	23	25	25	26
75%MEK,25%Toluene	22	22	23	24	24	24
50%MEK, 50%Toluene	20	22	22	23	23	23
70% MEK, 20%Toluene	20	22	22	23	23	23
and 10% Naphtha						

Tab. 4.41: Effect of Time on the amount of wax extracted (% age) using 16:1 Solvent to Sample at 70°C.



Fig. 4.41: Effect of Time on the amount of wax extracted (% age) using 16:1 Solvent to Sample at 70°C.

Time (min)	5	10	15	20	25	30
Solvent						
MEK	24	24	24	26	26	26
75%MEK,25%Toluene	22	23	23	25	25	25
50%MEK, 50%Toluene	21	23	23	24	24	25
70% MEK, 20%Toluene	21	22	22	24	24	24
and 10% Naphtha						

Tab. 4.42: Effect of Time on the amount of wax extracted (% age) using 20:1 Solvent to Sample at 70°C.



Fig. 4.42: Effect of Time on the amount of wax extracted (% age) using 20:1 Solvent to Sample at 70°C.

Time (min)	5	10	15	20	25	30
Solvent						
MEK	24	24	24	26	26	26
75%MEK,25%Toluene	23	24	24	25	25	26
50%MEK, 50%Toluene	23	23	23	25	25	25
70% MEK, 20%Toluene	22	22	22	24	24	25
and 10% Naphtha						

Tab. 4.43: Effect of Time on the amount of wax extracted (% age) using 24:1 Solvent to Sample at 70°C.



Fig. 4.43: Effect of Time on the amount of wax extracted (% age) using 24:1 Solvent to Sample at 70°C.

Time (min)	5	10	15	20	25	30
Solvent						
MEK	24	24	24	26	26	26
75%MEK,25%Toluene	23	24	24	25	25	26
50%MEK, 50%Toluene	23	23	23	25	25	25
70% MEK, 20%Toluene	22	22	23	24	24	25
and 10% Naphtha						

Tab. 4.44: Effect of Time on the amount of wax extracted (% age) using 32:1 Solvent to Sample at 70°C.



Fig. 4.44: Effect of Time on the amount of wax extracted (% age) using 32:1 Solvent to Sample at 70°C.

Tab	. 4.45:	Effect	of sol	vent to	sample	Ratio	on t	he a	mount	of v	vax	extra	cted
(% a	ge) at	28°C a	nd 5 m	ninutes									

Ratio of Solvent to Sample	16:1	20:1	24:1	32:1
Solvent				
MEK	19	20	20	20
75%MEK,25%Toluene	18	19	19	19
50%MEK, 50%Toluene	18	19	19	19
70% MEK, 20% Toluene and 10%	18	19	19	19
Naphtha				



Fig. 4.45 Effect of Solvent to Sample Ratio on the Extraction of Wax at 28°C and 5 minutes.

Ratio of Solvent to Sample	16:1	20:1	24:1	32:1
Solvent				
MEK	20	21	21	21
75%MEK,25%Toluene	19	20	20	20
50%MEK, 50%Toluene	19	20	20	20
70% MEK, 20% Toluene and 10%	20	20	20	20
Naphtha				

Tab. 4.46: Effect of Solvent to Sample Ratio on the amount of wax extracted (% age) at 40°C and 5 minutes.



Fig. 4.46 Effect of Solvent to Sample Ratio on the Extraction of Wax at 40°C and 5 minutes.

Ratio of Solvent to Sample	16:1	20:1	24:1	32:1
Solvent				
MEK	21	23	23	23
75%MEK,25%Toluene	20	20	21	21
50%MEK, 50%Toluene	20	20	21	21
70% MEK, 20% Toluene and 10%	20	20	21	21
Naphtha				

Tab. 4.47: Effect of Solvent to Sample Ratio on the amount of wax extracted (% age) at 50°C and 5 minutes.



Fig. 4.47 Effect of Solvent to Sample Ratio on the Extraction of Wax at 50°C and 5 minutes.

Ratio of Solvent to Sample	16:1	20:1	24:1	32:1
Solvent				
MEK	21	23	23	23
75%MEK,25%Toluene	21	21	21	23
50%MEK, 50%Toluene	20	21	23	23
70% MEK, 20% Toluene and 10%	20	20	21	22
Naphtha				

Tab. 4.48: Effect of Solvent to Sample Ratio on the amount of wax extracted (% age) at 60°C and 5 minutes.



Fig. 4.48 Effect of Solvent to Sample Ratio on the Extraction of Wax at 60°C and 5 minutes.

Ratio of Solvent to Sample	16:1	20:1	24:1	32:1
Solvent				
MEK	21	24	24	24
75%MEK,25%Toluene	22	23	23	23
50%MEK, 50%Toluene	20	21	23	23
70% MEK, 20% Toluene and 10%	20	21	22	22
Naphtha				

Tab. 4.49: Effect of Solvent to Sample Ratio on the amount of wax extracted (% age) at 70°C and 5 minutes.



Fig. 4.49 Effect of Solvent to Sample Ratio on the Extraction of Wax at 70°C and 5 minutes.

Ratio of Solvent to Sample	16:1	20:1	24:1	32:1
Solvent				
MEK	20	22	22	22
75%MEK,25%Toluene	19	20	20	21
50%MEK, 50%Toluene	19	20	20	21
70% MEK, 20% Toluene and 10%	19	19	21	21
Naphtha				

Tab. 4.50: Effect of Solvent to Sample Ratio on the amount of wax extracted (% age) at 28°C and 10 minutes.



Fig. 4.50 Effect of Solvent to Sample Ratio on the Extraction of Wax at 28°C and 10 minutes.

Ratio of Solvent to Sample	16:1	20:1	24:1	32:1
Solvent				
MEK	21	22	23	23
75%MEK,25%Toluene	21	22	22	22
50%MEK, 50%Toluene	21	21	21	22
70% MEK, 20% Toluene and 10%	21	21	21	21
Naphtha				

Tab. 4.51: Effect of Solvent to Sample Ratio on the amount of wax extracted (% age) at 40°C and 10 minutes.



Fig. 4.51 Effect of Solvent to Sample Ratio on the Extraction of Wax at 40°C and 10 minutes.

Ratio of Solvent to Sample	16:1	20:1	24:1	32:1
Solvent				
MEK	22	23	23	23
75%MEK,25%Toluene	21	22	22	22
50%MEK, 50%Toluene	21	22	22	23
70% MEK, 20% Toluene and 10%	21	21	22	22
Naphtha				

Tab. 4.52: Effect of Solvent to Sample Ratio on the amount of wax extracted (% age) at 50°C and 10 minutes.



Fig. 4.52 Effect of Solvent to Sample Ratio on the Extraction of Wax at 50°C and 10 minutes.

Ratio of Solvent to Sample	16:1	20:1	24:1	32:1
Solvent				
MEK	22	23	23	24
75%MEK,25%Toluene	22	23	23	23
50%MEK, 50%Toluene	21	22	23	23
70% MEK, 20% Toluene and 10%	21	22	22	22
Naphtha				

Tab. 4.53: Effect of Solvent to Sample Ratio on the amount of wax extracted (% age) at 60°C and 10 minutes.





Ratio of Solvent to Sample	16:1	20:1	24:1	32:1
Solvent				
MEK	23	24	24	24
75%MEK,25%Toluene	22	23	24	24
50%MEK, 50%Toluene	22	23	23	23
70% MEK, 20% Toluene and 10%	22	22	22	22
Naphtha				

Tab. 4.54: Effect of Solvent to Sample Ratio on the amount of wax extracted (% age) at 70°C and 10 minutes.



Fig. 4.54: Effect of Solvent to Sample Ratio on the Extraction of Wax at 70°C and 10 minutes.

Ratio of Solvent to Sample	16:1	20:1	24:1	32:1
Solvent				
MEK	21	23	23	23
75%MEK,25%Toluene	20	21	21	21
50%MEK, 50%Toluene	21	21	21	21
70% MEK, 20% Toluene and 10%	20	20	21	22
Naphtha				

Tab. 4.55: Effect of Solvent to Sample Ratio on the amount of wax extracted (% age) at 28°C and 15 minutes.



Fig. 4.55: Effect of Solvent to Sample Ratio on the amount of wax extracted (% age) at 28°C and 15 minutes.

Ratio of Solvent to Sample	16:1	20:1	24:1	32:1
Solvent				
MEK	22	23	23	23
75%MEK,25%Toluene	22	22	22	22
50%MEK, 50%Toluene	21	22	22	22
70% MEK, 20% Toluene and 10%	21	21	22	22
Naphtha				

Tab. 4.56: Effect of Solvent to Sample Ratio on the amount of wax extracted (% age) at 40°C and 15 minutes.



Fig. 4.56: Effect of Solvent to Sample Ratio on the amount of wax extracted (% age) at 40°C and 15 minutes.

Ratio of Solvent to Sample	16:1	20:1	24:1	32:1
Solvent				
MEK	22	23	23	23
75%MEK,25%Toluene	22	22	22	23
50%MEK, 50%Toluene	21	22	22	23
70% MEK, 20% Toluene and 10%	21	21	22	22
Naphtha				

Tab. 4.57: Effect of Solvent to Sample Ratio on the amount of wax extracted (% age) at 50°C and 15 minutes.



Fig. 4.57: Effect of Solvent to Sample Ratio on the amount of wax extracted (% age) at 50°C and 15 minutes.

Ratio of Solvent to Sample	16:1	20:1	24:1	32:1
Solvent				
MEK	23	24	24	24
75%MEK,25%Toluene	22	23	23	23
50%MEK, 50%Toluene	21	22	23	23
70% MEK, 20% Toluene and 10%	21	22	22	23
Naphtha				

Tab. 4.58: Effect of Solvent to Sample Ratio on the amount of wax extracted (% age) at 60°C and 15 minutes.



Fig. 4.58: Effect of Solvent to Sample Ratio on the amount of wax extracted (% age) at 60°C and 15 minutes.

Ratio of Solvent to Sample	16:1	20:1	24:1	32:1
Solvent				
MEK	23	24	24	24
75%MEK,25%Toluene	23	23	24	24
50%MEK, 50%Toluene	22	22	23	23
70% MEK, 20% Toluene and 10%	22	22	22	23
Naphtha				

Tab. 4.59: Effect of Solvent to Sample Ratio on the amount of wax extracted (% age) at 70°C and 15 minutes.



Fig. 4.59: Effect of Solvent to Sample Ratio on the amount of wax extracted (% age) at 70°C and 15 minutes.

Ratio of Solvent to Sample	16:1	20:1	24:1	32:1
Solvent				
MEK	21	23	23	24
75%MEK,25%Toluene	21	21	22	22
50%MEK, 50%Toluene	21	21	22	22
70% MEK, 20% Toluene and 10%	20	20	21	22
Naphtha				

Tab. 4.60: Effect of Solvent to Sample Ratio on the amount of wax extracted (% age) at 28°C and 20 minutes.



Fig. 4.60: Effect of Solvent to Sample Ratio on the amount of wax extracted (% age) at 28°C and 20 minutes.

Ratio of Solvent to Sample	16:1	20:1	24:1	32:1
Solvent				
MEK	23	24	24	24
75%MEK,25%Toluene	22	23	23	24
50%MEK, 50%Toluene	21	22	23	23
70% MEK, 20% Toluene and 10%	22	22	23	23
Naphtha				

Tab. 4.61: Effect of Solvent to Sample Ratio on the amount of wax extracted (% age) at 40°C and 20 minutes.



Fig. 4.61: Effect of Solvent to Sample Ratio on the amount of wax extracted (% age) at 40°C and 20 minutes.

Ratio of Solvent to Sample	16:1	20:1	24:1	32:1
Solvent				
MEK	24	25	25	25
75%MEK,25%Toluene	23	24	24	24
50%MEK, 50%Toluene	22	22	23	23
70% MEK, 20% Toluene and 10%	22	22	23	23
Naphtha				

Tab. 4.62: Effect of Solvent to Sample Ratio on the amount of wax extracted (% age) at 50°C and 20 minutes.





Ratio of Solvent to Sample	16:1	20:1	24:1	32:1
Solvent				
MEK	24	25	25	25
75%MEK,25%Toluene	23	24	25	25
50%MEK, 50%Toluene	22	23	24	25
70% MEK, 20% Toluene and 10%	22	22	23	23
Naphtha				

Tab. 4.63: Effect of Solvent to Sample Ratio on the amount of wax extracted (% age) at 60°C and 20 minutes.



Fig. 4.63: Effect of Solvent to Sample Ratio on the amount of wax extracted (% age) at 60°C and 20 minutes.

Ratio of Solvent to Sample	16:1	20:1	24:1	32:1
Solvent				
MEK	25	26	26	26
75%MEK,25%Toluene	24	25	25	25
50%MEK, 50%Toluene	23	24	25	25
70% MEK, 20% Toluene and 10%	23	24	24	24
Naphtha				

Tab. 4.64: Effect of Solvent to Sample Ratio on the amount of wax extracted (% age) at 70°C and 20 minutes.



Fig. 4.64: Effect of Solvent to Sample Ratio on the amount of wax extracted (% age) at 70°C and 20 minutes.

Ratio of Solvent to Sample	16:1	20:1	24:1	32:1
Solvent				
MEK	21	24	24	24
75%MEK,25%Toluene	21	22	22	23
50%MEK, 50%Toluene	22	22	22	23
70% MEK, 20% Toluene and 10%	21	21	22	22
Naphtha				

Tab. 4.65: Effect of Solvent to Sample Ratio on the amount of wax extracted (% age) at 28°C and 25 minutes.



Fig. 4.65: Effect of Solvent to Sample Ratio on the amount of wax extracted (% age) at 28°C and 25 minutes.

Ratio of Solvent to Sample	16:1	20:1	24:1	32:1
Solvent				
MEK	23	24	25	24
75%MEK,25%Toluene	22	23	24	24
50%MEK, 50%Toluene	22	23	23	23
70% MEK, 20% Toluene and 10%	22	23	23	23
Naphtha				

Tab. 4.66: Effect of Solvent to Sample Ratio on the amount of wax extracted (% age) at 40°C and 25 minutes.



Fig. 4.66: Effect of Solvent to Sample Ratio on the amount of wax extracted (% age) at 40°C and 25 minutes.

Ratio of Solvent to Sample	16:1	20:1	24:1	32:1
Solvent				
MEK	24	25	25	25
75%MEK,25%Toluene	23	24	24	24
50%MEK, 50%Toluene	22	23	23	24
70% MEK, 20% Toluene and 10%	22	22	23	24
Naphtha				

Tab. 4.67: Effect of Solvent to Sample Ratio on the amount of wax extracted (% age) at 50°C and 25 minutes.



Fig. 4.67: Effect of Solvent to Sample Ratio on the amount of wax extracted (% age) at 50°C and 25 minutes.

Ratio of Solvent to Sample	16:1	20:1	24:1	32:1
Solvent				
MEK	25	26	26	26
75%MEK,25%Toluene	23	24	25	25
50%MEK, 50%Toluene	22	23	24	25
70% MEK, 20% Toluene and 10%	23	24	24	24
Naphtha				

Tab. 4.68: Effect of Solvent to Sample Ratio on the amount of wax extracted (% age) at 60°C and 25 minutes.



Fig. 4.68: Effect of Solvent to Sample Ratio on the amount of wax extracted (% age) at 60°C and 25 minutes.

Ratio of Solvent to Sample	16:1	20:1	24:1	32:1
Solvent				
MEK	25	26	26	26
75%MEK,25%Toluene	24	25	25	25
50%MEK, 50%Toluene	23	24	25	25
70% MEK, 20% Toluene and 10%	23	24	24	24
Naphtha				

Tab. 4.69: Effect of Solvent to Sample Ratio on the amount of wax extracted (% age) at 70°C and 25 minutes.



Fig. 4.69: Effect of Solvent to Sample Ratio on the amount of wax extracted (% age) at 70°C and 25 minutes.

Ratio of Solvent to Sample	16:1	20:1	24:1	32:1
Solvent				
MEK	22	24	24	24
75%MEK,25%Toluene	22	22	23	23
50%MEK, 50%Toluene	22	22	23	23
70% MEK, 20% Toluene and 10%	21	21	22	22
Naphtha				

Tab. 4.70: Effect of Solvent to Sample Ratio on the amount of wax extracted (% age) at 28°C and 30 minutes.



Fig. 4.70: Effect of Solvent to Sample Ratio on the amount of wax extracted (% age) at 28°C and 30 minutes.

Ratio of Solvent to Sample	16:1	20:1	24:1	32:1
Solvent				
MEK	23	24	25	23
75%MEK,25%Toluene	22	23	24	25
50%MEK, 50%Toluene	22	23	23	23
70% MEK, 20% Toluene and 10%	22	23	23	23
Naphtha				

Tab. 4.71: Effect of Solvent to Sample Ratio on the amount of wax extracted (% age) at 40°C and 30 minutes.



Fig. 4.71: Effect of Solvent to Sample Ratio on the amount of wax extracted (% age) at 40°C and 30 minutes.

Ratio of Solvent to Sample	16:1	20:1	24:1	32:1
Solvent				
MEK	24	25	25	26
75%MEK,25%Toluene	23	24	24	25
50%MEK, 50%Toluene	22	23	23	24
70% MEK, 20% Toluene and 10%	23	23	23	24
Naphtha				

Tab. 4.72: Effect of Solvent to Sample Ratio on the amount of wax extracted (% age) at 50°C and 30 minutes.



Fig. 4.72: Effect of Solvent to Sample Ratio on the amount of wax extracted (% age) at 50°C and 30 minutes.

Ratio of Solvent to Sample	16:1	20:1	24:1	32:1
Solvent				
MEK	25	26	26	26
75%MEK,25%Toluene	23	24	25	26
50%MEK, 50%Toluene	22	23	24	25
70% MEK, 20% Toluene and 10%	23	24	24	24
Naphtha				

Tab. 4.73: Effect of Solvent to Sample Ratio on the amount of wax extracted (% age) at 60°C and 30 minutes.



Fig. 4.73: Effect of Solvent to Sample Ratio on the amount of wax extracted (% age) at 60°C and 30 minutes.

Ratio of Solvent to Sample	16:1	20:1	24:1	32:1
Solvent				
MEK	26	26	26	26
75%MEK,25%Toluene	24	25	26	26
50%MEK, 50%Toluene	23	25	25	25
70% MEK, 20% Toluene and 10%	23	24	25	25
Naphtha				

Tab. 4.74: Effect of Solvent to Sample Ratio on the amount of wax extracted (% age) at 70°C and 30 minutes.



Fig. 4.74: Effect of Solvent to Sample Ratio on the amount of wax extracted (% age) at 70°C and 30 minutes.

S	R	16:1					20:1					24:1					32:1				
	Te	28	40	50	60	70	28	40	50	60	70	28	40	50	60	70	28	40	50	60	70
	Ťi																				
А	5	19	20	21	21	21	20	21	23	23	24	20	21	23	23	24	20	21	23	23	24
В		18	19	20	21	22	19	20	20	21	23	19	20	21	21	23	19	20	21	23	23
С		18	19	20	20	20	19	20	20	21	21	19	20	21	23	23	19	20	21	23	23
D		18	20	20	20	20	19	20	20	20	21	19	20	21	21	22	19	20	21	22	22
A	10	20	21	22	22	23	22	22	23	23	24	22	23	23	23	24	22	23	23	24	24
В		19	21	21	22	22	20	22	22	23	23	20	22	22	23	24	21	22	22	23	24
С		19	21	21	21	22	20	21	22	22	23	20	21	22	23	23	21	22	23	23	23
D		19	21	21	21	22	19	21	21	22	22	21	21	22	22	22	21	21	22	22	22
А	15	21	22	22	23	23	23	23	23	24	24	23	23	23	24	24	23	23	23	24	24
В		20	22	22	22	23	21	22	22	23	23	21	22	22	23	24	21	22	23	23	24
С		21	21	21	21	22	21	22	22	22	23	21	22	22	23	23	21	22	23	23	23
D		20	21	21	21	22	20	21	21	22	22	21	22	22	22	22	22	22	22	23	23
А	20	21	23	24	24	25	23	24	25	25	26	23	24	25	25	26	24	24	25	25	26
В		21	22	23	23	24	21	23	24	24	25	22	23	24	25	25	22	24	24	25	25
С		21	21	22	22	23	21	22	22	23	24	22	23	23	24	25	22	23	23	25	25
D		20	22	22	22	23	20	22	22	23	24	21	23	23	23	24	22	23	23	23	24
А	25	21	23	24	25	25	24	24	25	26	26	24	25	25	26	26	24	25	25	26	26
В		21	22	23	23	24	22	23	24	24	25	22	24	24	25	25	23	24	24	25	25
С		22	22	22	22	23	22	23	23	23	24	22	23	23	24	25	23	23	24	25	25
D		21	22	22	23	23	21	23	23	24	24	22	23	23	24	24	22	23	24	24	24
А	30	22	23	24	25	26	24	24	25	26	26	24	25	25	26	26	24	25	26	26	26
В		22	22	23	23	24	22	23	24	24	25	23	24	24	25	26	23	25	25	26	26
С		22	22	22	22	24	22	23	23	23	25	23	23	23	24	25	23	23	24	25	25
D		21	22	23	23	23	21	23	23	24	24	22	23	23	24	25	22	23	24	24	25

Tab.4.75 : Percentage of Wax Extracted at Different Temperatures, Times and Ratio of Solvent to Sample

S: Solvent

R: Ratio

Te: Temperature°C

Ti: Time Minutes

A: MEK

- B: 75% MEK and 25% Toluene
- C: 50% MEK and 25% Toluene
- D: 70% MEK, 20% Toluene and 10% Naphtha
| | Ratio | | | 16:1 | | | | | 20:1 | | | | | 24:1 | | | | | 32:1 | | |
|---|----------------------------------|----|----|------|----|----|----|----|------|----|----|----|----|------|----|----|----|----|------|----|----|
| s | Te [•] C
Ti
(min) | 28 | 40 | 50 | 60 | 70 | 28 | 40 | 50 | 60 | 70 | 28 | 40 | 50 | 60 | 70 | 28 | 40 | 50 | 60 | 70 |
| | 5 | 19 | 20 | 21 | 21 | 21 | 20 | 21 | 23 | 23 | 24 | 20 | 21 | 23 | 23 | 24 | 20 | 21 | 23 | 23 | 24 |
| | 10 | 20 | 21 | 22 | 22 | 23 | 22 | 22 | 23 | 23 | 24 | 22 | 23 | 23 | 23 | 24 | 22 | 23 | 23 | 24 | 24 |
| | 15 | 21 | 22 | 22 | 23 | 23 | 23 | 23 | 23 | 24 | 24 | 23 | 23 | 23 | 24 | 24 | 23 | 23 | 23 | 24 | 24 |
| | 20 | 21 | 23 | 24 | 24 | 25 | 23 | 24 | 25 | 25 | 26 | 23 | 24 | 25 | 25 | 26 | 24 | 24 | 25 | 25 | 26 |
| A | 25 | 21 | 23 | 24 | 25 | 25 | 24 | 24 | 25 | 26 | 26 | 24 | 25 | 25 | 26 | 26 | 24 | 25 | 25 | 26 | 26 |
| | 30 | 22 | 23 | 24 | 25 | 26 | 24 | 24 | 25 | 26 | 26 | 24 | 25 | 25 | 26 | 26 | 24 | 25 | 26 | 26 | 26 |
| | 5 | 18 | 19 | 20 | 21 | 22 | 19 | 20 | 20 | 21 | 23 | 19 | 20 | 21 | 21 | 23 | 19 | 20 | 21 | 23 | 23 |
| | 10 | 19 | 21 | 21 | 22 | 22 | 20 | 22 | 22 | 23 | 23 | 20 | 22 | 22 | 23 | 24 | 21 | 22 | 22 | 23 | 24 |
| B | 15 | 20 | 22 | 22 | 22 | 23 | 21 | 22 | 22 | 23 | 23 | 21 | 22 | 22 | 23 | 24 | 21 | 22 | 23 | 23 | 24 |
| | 20 | 21 | 22 | 23 | 23 | 24 | 21 | 23 | 24 | 24 | 25 | 22 | 23 | 24 | 25 | 25 | 22 | 24 | 24 | 25 | 25 |
| | 25 | 21 | 22 | 23 | 23 | 24 | 22 | 23 | 24 | 24 | 25 | 22 | 24 | 24 | 25 | 25 | 23 | 24 | 24 | 25 | 25 |
| | 30 | 22 | 22 | 23 | 23 | 24 | 22 | 23 | 24 | 24 | 25 | 23 | 24 | 24 | 25 | 26 | 23 | 25 | 25 | 26 | 26 |
| | 5 | 18 | 19 | 20 | 20 | 20 | 19 | 20 | 20 | 21 | 21 | 19 | 20 | 21 | 23 | 23 | 19 | 20 | 21 | 23 | 23 |
| | 10 | 19 | 21 | 21 | 21 | 22 | 20 | 21 | 22 | 22 | 23 | 20 | 21 | 22 | 23 | 23 | 21 | 22 | 23 | 23 | 23 |
| С | 15 | 21 | 21 | 21 | 21 | 22 | 21 | 22 | 22 | 22 | 23 | 21 | 22 | 22 | 23 | 23 | 21 | 22 | 23 | 23 | 23 |
| | 20 | 21 | 21 | 22 | 22 | 23 | 21 | 22 | 22 | 23 | 24 | 22 | 23 | 23 | 24 | 25 | 22 | 23 | 23 | 25 | 25 |
| | 25 | 22 | 22 | 22 | 22 | 23 | 22 | 23 | 23 | 23 | 24 | 22 | 23 | 23 | 24 | 25 | 23 | 23 | 24 | 25 | 25 |
| | 30 | 22 | 22 | 22 | 22 | 24 | 22 | 23 | 23 | 23 | 25 | 23 | 23 | 23 | 24 | 25 | 23 | 23 | 24 | 25 | 25 |
| | 5 | 18 | 20 | 20 | 20 | 20 | 19 | 20 | 20 | 20 | 21 | 19 | 20 | 21 | 21 | 22 | 19 | 20 | 21 | 22 | 22 |
| D | 10 | 19 | 21 | 21 | 21 | 22 | 19 | 21 | 21 | 22 | 22 | 21 | 21 | 22 | 22 | 22 | 21 | 21 | 22 | 22 | 22 |
| | 15 | 20 | 21 | 21 | 21 | 22 | 20 | 21 | 21 | 22 | 22 | 21 | 22 | 22 | 22 | 22 | 22 | 22 | 22 | 23 | 23 |
| | 20 | 20 | 22 | 22 | 22 | 23 | 20 | 22 | 22 | 23 | 24 | 21 | 23 | 23 | 23 | 24 | 22 | 23 | 23 | 23 | 24 |
| | 25 | 21 | 22 | 22 | 23 | 23 | 21 | 23 | 23 | 24 | 24 | 22 | 23 | 23 | 24 | 24 | 22 | 23 | 24 | 24 | 24 |
| | 30 | 21 | 22 | 23 | 23 | 23 | 21 | 23 | 23 | 24 | 24 | 22 | 23 | 23 | 24 | 25 | 22 | 23 | 24 | 24 | 25 |

Tab: 4. 76: Percentage of Wax Extracted at Different Temperatures, Times and Ratio of Solvent to Sample

S: Solvent

R: Ratio

Te: Temperature°C

Ti: Time Minutes

A: MEK

C: 50% MEK and 25% Toluene

D: 70% MEK, 20% Toluene and 10% Naphtha

B: 75% MEK and 25% Toluene

Ratio				
	16:1	20:1	24:1	32:1
Solvent				
	26% at 70°Cand	26% at 60°C and	26% at 60°C and	26% at 60°C and
MEK	30 minutes	25 minutes	25 minutes	25 minutes.
75% MEK, 25%	24% at 70°C and	25% at 70°C and	26% at 70°C and	26% at 60°C and
Toluene	20 minutes	20 minutes	30 minutes	30 minutes
50% MEK, 50%	24% at 70°C and	25% at 70°C and	25% at 70°C and	25% at 60°C and
Toluene	30 minutes	30 minutes	20 minutes	20 minutes
70%MEK, 20%	23% at 60°C and	24% at 60°C and	25% at 70°C and	25% at 70°C and
Toluene and 10%	25 minutes	25 minutes	30 minutes	30 minutes
Naphtha				

Tab. 4.77: The Maximum Extraction Conditions for Different Solvents.

4.2 Discussion

On examining the results shown on Table 4.75 and Table 4.76, many observations can be drawn:

- (i) The amount of wax extracted from each sample increases as the temperature, time and solvent to sample ratio increase.
- (ii) For each solvent, the amount of wax extracted reaches its maximum value at or above 60°C, 20 minutes and ratio of solvent to sample 20:1.
- (iii) Generally the amount of wax extracted decreases as the amount of MEK in the solvent decreases.
- (iv) For each solvent, the increase in wax extracted from each sample increases by 3.5 to 4.3 % by changing for any set of temperature, time and solvent to sample ratio to another set of these variables.
- (v) From Table 4.77 the maximum amount of wax extracted (26 % of the sample: 100 % the wax in the sample if we ignore the escaped atoms of fuller's earth and asphaltene during filtration.) is obtained by :
 - MEK : 70°C, 30 minutes, ratio of 16:1 60°C, 25 minutes, ratio of 20:1 70°C, 20 minutes, ratio of 20:1 60°C, 25 minutes, ratio of 24:1 70°C, 20 minutes, ratio of 24:1 60°C, 25 minutes, ratio of 32:1 50°C, 30 minutes, ratio of 32:1

MEK (75%) + Toluene (25%): 70°C, 30 minutes, ratio of 24:1 60°C, 30 minutes, ratio of 32:1 70°C, 30 minutes, ratio of 32:1

From the cost estimation (Chapter 5) page (), it is found that using the solvent of MEK(75%) + Toluene(25%) at 70°C is more expensive than MEK at 60°C and 25 minutes and 20:1 ratio of solvent to sample for extraction of wax.

4.3 Carbon Atom Analysis

The wax produced from Nile Blend at the conditions arrived to (section 4.2) was analyzed using (ASTM D 5442) Gas Chromatography method. The wax is generally characterized by number of carbon atoms. The presence of carbon atom was measured by its retention time (the time that every carbon atom takes to appear in Gas Chromatography Analysis). The wax was compared with reference wax obtained in The Laboratory Petroleum Centre. Figure 4.75 shows the number of carbon atoms in the reference wax and Nile blend wax. It was found that the carbon atoms for both the reference wax and Nile blend wax are ranging from C10 to C44, which means that both samples have the same composition of hydrocarbons.



Fig. 4.75: Carbon Atom Gas Chromatography for Reference and Extracted Wax.

CHAPTER FIVE

ECONOMIC EVALUATION

The economic evaluation is based on the following assumption:

- 1. The plant capacity is 1200 ton/day crude Nile Blend. This figure is arrived to by taking 75% of the pumped crude to Khartoum Refinery which is 67 ton /hr.
- 2. The operation time is 300 day/year;
- 3. The plant location is in Khartoum Bahri near Khartoum Refinery Company.

The economic evaluation was done using Factorial method [40].

5.1 Material Balance

Basis: 1hr.

The low of the conversion of Mass states that mass is neither created nor destroyed.

```
Mass input = Mass output + Mass accumulated (5.1)
```

$$M_{input} = M_{ouput} + M_{accumulated}$$
(5.2)

Where M = the mass

Figure 5.1 represents the process flow diagram and Tables 5.1 show material balance of deasphalting and dewaxing processes . The extraction of wax is done in a number of process units. The main units include:

- 1. Mixers
- 2. Vacuum filters
- 3. Evaporators



Fig. 5.1: Process Flow Sheet

Stream		1,2		3	E	VPA		4	4	5,6,7		8		9		10		11		12
Mass in	%	Kg/hr	%	Kg/hr	%	Kg/hr	%	Kg/hr	%	Kg/hr	%	Kg/hr	%	Kg/hr	%	Kg/hr	%	Kg/hr	%	Kg/hr
Oil	59	26947.5	0.49	29647.5	0.49	29647.5	72	29647.5	2.8	26947.5	0.00	0.00	0.96	14244.02	0.00	0.00	0.00	0.00	0.00	0.00
Wax	41	20602.5	0.32	20602.5	0.34	20602.5	28	11256	1.1	11256	37	26659.5	0.00	0.00	0.00	0.00	94	26659.5	0.00	0.00
Solvent + Fullar's Earth	0.00	0.00	99.22	6386775	99.17	6000553.5	0.00	0.00	96.1	1005000	63	45225	99.04	1462275	100	35942.24	6.0	1789.5	100	79377
Total	100	50250	100	6437025	100	6050803.5	100	40903.5	100	1045903.5	100	71884.5	100	1476519	100	35942.24	100	28449	100	79377

Tab:5.1. Material Balance of Deasphalting and Dewaxing Processes

5.1.2 Overall Material Balance





Fig. 5.2: Overall Material Balance

5.2 Energy Balance

5.2.1 Energy Balance around Heater

Basis for calculation:

- 1. Enthalphy of crude oil is 123 kJ/kg.
- 2. Enthalphy of heated crude oil is 194.75 kJ/kg.
- 3. Crude feed temperature (ambient temperature) 25°C.
- 4. Out let temperature is 60° C.
- 5. The heat consumption = $M_2(H_2 H_1) = 50250(194.75 123) = 1001.5$ kw.

Where H_1 and H_2 the enthalphy of heated crude oil and crude oil.



Fig. 5.3: Diagram for Heater Energy Balance

5.2.2 Energy Balance around Mixer (1)

Basis for calculation:

- 6. Specific heat of mixture $cp = \sum x_i \cdot cp_i = 2.05 \text{kJ/kgC}$.
- 7. Crude feed temperature is 60°C.
- 8. Out let temperature (ambient temperature) 25°C.
- 9. The heat consumption = m cp ΔT = 6437025 x 2.05 x (60 25) = 461856543 kJ/hr = 461856543/3600 = 128293.5 kw



Fig .5.4: Diagram for Mixer (1) Energy Balance

5.2.3 Energy Balance around Mixer (2)

Basis for calculation:

- 10. Specific heat of mixture cp = 3.63 kJ/kgK.
- 11.Crude feed temperature is 60°C.
- 12.Out let temperature (ambient temperature) 25°C.
- 13. The heat consumption = m cp ΔT = 1045903.5 x 3.63 x (60 25) = 132882040 kJ/hr = 132882040/3600 = 36911.68 kw.
- 14. Assume the solvent losses during process is 5%.



Fig .5.5: Diagram for Mixer (2) Energy Balance

5.3 Cost Estimation (MEK at 60°C, 25 min and 20:1 solvent to sample ratio)

The total cost consists of

- 1- Total Investment Cost (TIC)
 - (a) Purchace Cost of Major Equipment (PCOME)
 - (b) Physical Plant Cost (PPC)
 - (c) Fixed Capital (FC)
 - (d) Working Capital Cost (WCC)
- 2- Annual Operating Cost (AOC)
 - (a) Total Variable Cost (TVC)
 - (b) Total Fixed Cost (TFC)

5.3.1 Purchase Cost of Major Equipments (PCOME)

The purchase cost for each equipment is calculated using the following equation

$$C = C S^n \tag{5.1}$$

Where C is a constant and S is size or capacity of the equipment.

Table 5.2 list the cost of each item.

Item	Unit	Size (S)	Constant (C)	Index n	Cost (\$)			
fired heater	kw	1001.50	560.00	0.77	114469			
Heater 2	Kg/hr	1045903.50	100.00	0.80	6540234			
Mixer 1	Kw	128293.50	1900.00	0.50	680543			
Mixer 2	kw	36912	1900.00	0.50	365036			
Vacuum filter 1	m^2	50.00	4400,00	0.60	46008			
Filter 2	m^2	38.00	17000.00	0.60	150771			
Evaporator 1	m^2	55.00	11500.00	0.53	96181			
Evaporator 2	m^2	37.00	11500.00	0.53	77955			
chiller	Kg/hr	1045903.50	60.00	0.80	3924140			
Purchase Cost of I	Purchase Cost of Major Equipment (PCOME) 11995339							

Tab.5.2: Purchase cost of major equipments

5.3.2 Total Investment Cost (TIC)

TIC is the sum of fixed capital and working capital. Table 5.3 lists the component of TIC. Table 5.4 lists the annual operating cost AOC.

Item	(PCOME)%	(\$)
Purchase Cost of MajorEquipment(PCOME))		11995339
Equipments erection	0.40	4798136
Piping	0.30	3598601
Instrumentation	0.10	1199533
Electrical	0.10	1199533
Building process	0.10	1199533
Utilities	0.025	299883
Storages	0.20	2399068
Site development	0.05	599767
Ancillary building	0.10	1199534
PPC		28488930
Design and Engineering	0.20	2399068
Contractor's fee	0.05	599767
Contingency	0.05	599767
Fixed Capital (IDC)		8546679
Total Fixed Capital (TFC) = PCC + IDC		37035610
Working Capital Cost (WCC) 5% of IDC	0.05	1851780
Total Investment $Cost = (TFC + WCC)$		38887390

Tab. 5.3: Total Investment Cost (TIC)

5.3.3 Annual Operating Cost (AOC)

Item	Description	Rate	QTY	\$
		(\$/ton)	(ton/day)	
1		Raw Materia	ls	
	Solvents	2500	1200	3000000
2	Miscellaneous 10% f	rom Maintena	ance	185178
3		Utility		
	Steam	0.25	371.68	93
	Water	0.01	3.3	0.033
	Electrical	0.10	200	20
4	Shipping and	l Packing		-
	Subtotal A: Variab	les Cost		3185291
5	Maintenance 5% of FC	0.05		1851780
6	Operating Labour	job		100000
7	Lab. Cost (20% of item 6)	0.20		20000
8	Supervision (20% of item 6)	0.20		20000
9	Overheads (50% of item 6)	0.50		50000
10	Capital charge (15% of FC)	0.15		5555341
11	Insurance (1% of FC)	0.01		370356
12	Local Taxes (20% of FC)	0.02		740712
13	Royalties (1% of FC)	0.01		370356
	Subtotal B: Fixed	d Cost		9078546
	Direct Production Co	st(A+B)		12263837
14	Sales Expenses	0.20		2452767.4
15	General Overheads	0.20		2452767.4
16	Research and Development	0.20		2452767.4
	Subtotal C		7358302	
	Annual Operating Cost		19622139	

Tab.5.4: Annual Operating Cost

5.4 Payback Period

a- Total product revenue = 1000x191948.256 = 191948256 \$

b- Total annual product cost = (Total cost + 10% depreciation = 19622139+1962213.9 = 21584353\$

c- Net Annual Profit (taxes are excluded) = 191948256 - 21584353= 170363903

d- Payback period = 170363903/38887390 = 4 years

5.5 Conclusions

Nile Blend is a waxy crude oil, this wax increases by the addition of new fields.

The results of this study showed that the extracted wax percentage increases with temperature. The maximum extraction which is 26% at 25 minutes occurs at a temperature 60°C and 20:1 solvent to sample ratio, so 60°C was selected as optimum temperature for extraction of wax.

The results show that the extraction of wax initially increases with time of mixing the solvent and sample till it reaches a point beyond which the time has no influence on the extraction.

The produced wax is light yellow. It is odorless and is slightly greasy to the touch. The melting point is about 40°C.

It is soluble in naphtha, toluene, xylene, kerosene, benzene, ethyl alcohol, hot acetone, and carbon tetra chloride.

From the results obtained, the best operating conditions for wax extraction are shown in Table 5.7 below:

Parameter	Best Condition
Solvent	MEK
Temperature	60°C
Operation Time	25 minutes
Ratio of Solvent to Sample	20:1

Tab.5.5: Best Conditions of Wax Extraction for Nile Blend Petroleum

5.6 Recommendations

• The extraction of wax performed using only four solvents. Other investigator can try many other solvents like methyl isobutyl ketone on the production of wax would of importance, at different conditions.

• Optimization procedure for production of wax depends on the removal of asphaltene from crude oil. Hence further investigation of deasphalting process of petroleum is recommended.

• Further investigation on extraction of wax from ElDar Blend and ElFula crude oil to make a comparison between them would of importance.

• Further investigation to study the effect of blending the crude Nile Blend with the required amount of dewaxing solvents in the Control Process Facility (CPF) and to delay the chilling step to be performed in Khartoum Refinery. By so doing the cost of transportation may be reduced.

References

- Baker, E. A. "Chemistry and Morphology of Plant Epicuticular Waxes". In The Plant Cuticle. Ed. DF Cutler, KL Alvin, CE Price. Academic Press. ISBN 0 12 199920 3. (1982).
- 2- Web site (Wikipedia, the free encyclopedia.htm. 3/6/2008).
- 3- Welcome To Wax India.htm 13/2/2006.
- 5- Himran Syukri, Suwono Aryadi; and Mansoori, G. A. "Characterization of Alkanes and Paraffin Waxes for Application as Phase Change Energy Storage Medium ".Energy sources, vol – 16. (1994).
- 6- Bennett, H. f. a. IC. "Industrial Waxes". Chemical Publishing Company, Inc. New York. (1963).
- 7- Agahi, R. P. Bershghi, M. C. Lin and. Mansoori, A. "Thermo dynamic Behaviour of Hydrogen / Natural Gas Mixtures", Proceeding of the 74th Annual Convention of the Gas processors associatom, GPA, Tulsa, ok. (1995).
- 8- Adolfo P Pires, Rahoma S. Mohamed and Mansoori, G. A. July . "An Analytic Theory for the Production of Thermo dynamic Properties of Hydrocarbon – Water Systems", Proceedings of the 5th World Congress of Chemical Engineering, San Diego. (1996).
- Mansoori, G. A. "Modeling of Asphaltene and Other Heavy Organic Depositions", Journal of Petroleum Science & Engineering. Vol. 17. (1997).
- 10- "Mansoori, G. A, Lindsey Barriers, H. and Webster, Glen M. "Petroleum Waxes", West Conshohocken. (2003)

- 11- Minchin, S. T. "An Account of Some Solid State Properties of Petroleum Waxes in Terms of Their Composition". J. Inst. Petrol. (1948).
- 12- Carpenter, J. W. "The Physical and Chemical Properties of Paraffin Wax, Particularly in the Solid State". J. Inst. Petrol. (1962).
- Mazee, W. M. "On Properties of Paraffin Wax in the Solid State". J. Inst. Petrol. (1949).
- 14- William, A. Cruse and Donaldr Steven. "Chemical Technology of Petroleum". Mc Graw – Hill Book Company, Inc. New York, Toranto, London. (1960).
- Verlagsgesellschaft, VCH. 1996. "Ullman's Encyclopedia", Vol.A 28.
 1996.
- 16- Bennet, H. "Commercial Waxes". New York, Chemical Publishing Co. (1956).
- 17- Kalichevsky, V.A. and Kobe, K. A. "Petroleum Refining with Chemicals". Elsevier, Amsterdam. (1956).
- 18- Gerding, Mildred, (ed). "Fundamentals of Petroleum", Petroleum Extension Service, University of Texas at Austin. (1979).
- 19- <u>http://www.hghouston.com/refining.html</u>. (2005).
- Nelson, W. L. "Petroleum Refinery Engineering". Mc Graw Hill Book Company. New York. (1958).
- 21- <u>http://www.msu.edu/course/cep/255/petroleum</u> Case Study.htm. (2005).
- 22- Lewis, Alfred. "The New World of Petroleum", NewYork, Dodd, Mead & Co. (1966).
- 23- Sequeria, A. Jr. "Lubric Base oil and Wax Processing". Marcel Dekker, Inc., Chemical Industries Series. August. (1994).

- 24- Marple. S. J. and Landy, L. J. "Modern Dewaxing Technology." Adv. Pet. Chem. Refin. (1965).
- 25- Wilson, M. W., Mueller, T. A., and Kraft, G. W. "Commercialization of Iso Dewaxing – a new Technology for Dewaxing to Manufacture High Quality Lube Base Stocks", NPRA. (1994).
- 26- File:/A:\shell Global Solutions-Base oil Group.htm. (2005).
- 27- Central Petroleum Laboratory, Ministry of Energy and Mining, 2006, 2007.
- 28- Ferris, S. W, Harvey W. T. and Ritter J. F. "Petroleum Waxes, Research and Development Division", Sun Oil Company. (1960).
- 29- Shaw, David H and Hall, Ralph R, US patent 4514260. 1978.
- 30- Watts, Leonard R and Brewer, Norman L, US patent 4088565. 1977.
- 31- Ackerson, Michael D, Arabshahi, Seyed-Hamid and Babcock, RobertE, US patent 5196116. 1991.
- 32- Ackerson, Michael D, Arabshahi, Seyed-Hamid and Babcock, Robert E, US patent 5196116. 1998.
- 33- Ryan, Douglas G, US patent 4216075. 1987.
- 34- Broadhurst, Thomas E, patent 4441987. 1981.
- 35- Evans, E. B. "Modern Petroleum Technology". E. B. Evans. Institute of Petroleum. London. (1962).
- 36- John J. Meketta. "Petroleum Processing hand book". New York. (1992).
- 37- Tiedje and Maclead. "Higher Ketones as Dewaxing Solvents". (1956).
- 38- Ackerson, Michael D. Arabshahi, Seyed-Hamid And Babcock, Robert
 E. "Process for Petroleum wax separation at or above room temperature". http://www.freepatentsonline.com/5196116.html. (1993).

- 39- Penn Well, J. R. "Crude Oil Waves, Emulsions, and Asphaltenes".Publishing Company. (1997).
- 40- Sinnot, R. K. "Chemical Engineering Volume 6". Delhi, India. (1999).

Appendix: A



Fig.A.1: Nile Blend Fields

(Source: Ministry of Energy and Mining, 2007)

Appendix B. Apparatus

B.1 Balance

AMETTLER TOLEDO (Switzerland) balance type was used for weighing the crude oil and wax. The balance weighs in grams to four decimals; it operates at 8 - 145 V and 50/60 Hz. A photograph of the apparatus is shown below.



Photograph. B.1: Balance

B.2 Oven

A heating oven of the L.O.V SOCIAD type 200 DIG was used for the heating of the crude oil. It is design in accordance with relevant English regulation. The temperature scale is in °C. It has temperature range to 200°C. It operates with 415 volts and 50/60 Hz.

B.3 Filter

A Germany Gooch crucible with a glass wall was used for filtering the solution of crude oil and Naphtha and Fuller's earth through a filter paper and another one was used for filtering the solution of wax and solvent. A photograph of the apparatus is shown below.



Photograph. B.2: Filter

B.4 Circulator Water Bath

An English RE 100 (BIBBY) circulator water bath type was used for heating the crude oil and was samples to facilitate the evaporation of solvent, its temperature until 95°C. A photograph of the apparatus is shown below.

B.5 Rotary Evaporator

An English RE 100 (BIBBY) rotary evaporator type was used for evaporation the solvent from crude oil and wax. A photograph of the apparatus is shown below.



Photograph. B.3: Rotary Evaporator, Water Bath and Water Suction Pump

B.6 Chiller

A German Julabo FP50 (HD) chiller type was used for chilling the crude oil and solvent. It operates at 230 volts and 50 Hz. It used glycol for cooling. A photograph of the apparatus is shown below.



Photograph. B.4: Chiller

B.7 Water Suction Pump

An English D - 79112 Freiburg, N 035 AN. 18 type was used for increasing the velocity of the evaporator and the velocity of the filtration. It operates at 230 volts and 50 Hz. A photograph of the apparatus is shown in photograph B.3.

B.8 Flasks

- A- An English Duran / Schott 24/29, 500 ml was used for weighing crude oil and 1000 ml was used for chilling.
- B- English filtring flasks, 3 units, 500 ml were used.
- C- A German round bottom flasks 100 and500 ml were used for weighing the wax and 1000 liter for evaporation the naphtha before adding the solvent.

B.9 Magnetic Stirrer

An English magnetic stirrer was used for shaking the sample with solvent. A photograph of the apparatus is shown below.



Photograph. B.5: Magnetic Stirrer

B.10 Desiccator

ACZECHOSLOVAKIKA, 249 KP 9 type was used for keeping the wax sample until weighing.

Appendix C. The Method for Calculation of Extracted Wax

The wax content was calculated by the equation

Wax, wt % =
$$(W_2 - W_1) \times 100$$
 (C1)
S
Where W_1 = weight of round bottom flask, g
 W_2 = weight of round bottom flask + wax, g

S = sample weight, 2 gm

The experimental results for W_1 , W_2 and the amount of wax extracted under each condition of temperature, time and ratio of solvent to sample, calculated by equation C1 are shown below.

C.1 Solvent of Methyl Ethyl Ketone

Tab. C.1: At 28°C, 16:1 Ratio of Solvent to Sample and Different

Time	W ₁	W_2	Wax %
(min)			
5	99.2611	99.6360	19
10	99.3676	99.7650	20
15	102.7131	103.1283	21
20	105.9453	106.3640	21
25	99.1676	99.5859	21
30	105.6736	106.1192	22

Tab. C.2: At 40°C, 16:1 Ratio of Solvent to Sample and Different

Times

Time	\mathbf{W}_1	\mathbf{W}_2	Wax %
(min)			
5	100.2565	100.6539	20
10	100.3897	100.8068	21
15	99.8958	100.3414	22
20	102.9166	103.3827	23
25	102.8045	103.2656	23
30	100.0267	100.4899	23

Tab. C.3: At 50°C, 16:1 Ratio of Solvent to Sample and Different

Times

Time	W ₁	\mathbf{W}_2	Wax %
(min)			
5	103.6108	104.0294	21
10	102.1181	102.5635	22
15	105.3402	105.7857	22
20	105.4425	105.9222	24
25	99.6527	100.1297	24
30	102.8759	103.3518	24

Tab. C.4: At 60°C, 16:1 Ratio of Solvent to Sample and Different

Time (min)	W ₁	W ₂	Wax %
5	106.8430	107.2615	21
10	100.3621	100.8073	22
15	100.1389	100.6049	23
20	105.0980	105.5757	24
25	99.2365	99.7345	25
30	100.4587	100.9569	25

Tab. C.5: At 70°C, 16:1 Ratio of Solvent to Sample and Different

Times

Time	W ₁	W ₂	Wax %
(min)			
5	103.1652	103.5842	21
10	99.3601	99.8270	23
15	99.5823	100.0473	23
20	102.6809	103.1798	25
25	106.9042	107.4010	25
30	102.5231	103.0449	26

Tab. C.6: At 28°C, 20:1 Ratio of Solvent to Sample and Different

Times

Time	W ₁	\mathbf{W}_2	Wax %
(min)			
5	102.1518	102.5424	20
10	108.5853	109.0293	22
15	99.2389	99.7065	23
20	102.4621	102.9283	23
25	104.3102	104.7886	24
30	106.5324	107.0105	24

Tab. C.7: At 40°C, 20:1 Ratio of Solvent to Sample and Different

Time (min)	W ₁	W ₂	Wax %
5	105.4809	105.9043	21
10	99.9075	100.3503	22
15	105.6853	106.1504	23
20	99.7536	100.2327	24
25	102.9758	103.4538	24
30	104.1980	104.6765	24

Tab. C.8: At 50°C, 20:1 Ratio of Solvent to Sample and Different

Times

Time	W ₁	W ₂	Wax %
(min)			
5	106.9075	107.3747	23
10	99.1297	99.5968	23
15	103.3519	103.8189	23
20	100.1264	100.6241	25
25	99.3487	99.8464	25
30	102.5709	103.0693	25

Tab. C.9: At 60°C, 20:1 Ratio of Solvent to Sample and Different

Times

Time	W ₁	\mathbf{W}_2	Wax %
(min)			
5	105.5741	106.0385	23
10	106.7963	107.2648	23
15	99.4202	99.8982	24
20	104.7921	105.2897	25
25	100.3109	100.8339	26
30	99.1887	99.7118	26

Tab. C.10: At 70°C, 20:1 Ratio of Solvent to Sample and Different

Time	W ₁	\mathbf{W}_2	Wax %
(min)			
5	102.6421	103.1194	24
10	105.8653	106.3437	24
15	106.0985	106.5759	24
20	102.9465	103.4674	26
25	103.7243	104.2485	26
30	105.5021	106.0256	26

Tab. C.11: At 28°C, 24:1 Ratio of Solvent to Sample and Different

Times

Time	W ₁	\mathbf{W}_2	Wax %
(min)			
5	104.7055	105.1006	20
10	108.1297	108.5726	22
15	102.2185	102.6787	23
20	102.6944	103.1525	23
25	103.3218	103.7982	24
30	105.5441	106.0203	24

Tab. C.12: At 40°C, 24:1 Ratio of Solvent to Sample and Different

Times

Time	W ₁	W_2	Wax %
(min)			
5	102.2697	102.6929	21
10	105.4712	105.9308	23
15	100.2490	100.7076	23
20	99.2203	99.7011	24
25	106.0145	106.5129	25
30	103.2367	103.7344	25

Tab.C.13: At 50°C, 24:1 Ratio of Solvent to Sample and Different

Time (min)	W ₁	W ₂	Wax %
5	107.0168	107.4749	23
10	104.7942	105.2517	23
15	106.5723	107.0294	23
20	100.4519	100.9498	25
25	104.6241	105.1228	25
30	105.8712	106.3686	25

Tab. C.14: At 60°C, 24:1 Ratio of Solvent to Sample and Different

Times

Time	W ₁	W ₂	Wax %
(min)			
5	99.3501	99.8082	23
10	107.1279	107.5854	23
15	100.4325	100.9134	24
20	99.0934	99.5898	25
25	99.3799	99.9032	26
30	102.1567	102.6778	26

Tab. C.15: At 70°C, 24:1 Ratio of Solvent to Sample and Different

Times

Time	W ₁	\mathbf{W}_2	Wax %
(min)			
5	102.0281	102.5099	24
10	104.8069	105.2885	24
15	102.6847	103.1667	24
20	106.9345	107.4548	26
25	105.7123	106.2348	26
30	103.7523	104.2739	26

Tab. C.16: At 28°C, 32:1 Ratio of Solvent to Sample and Different

Time (min)	W ₁	W ₂	Wax %
5	108.7072	109.1124	20
10	102.3516	102.7947	22
15	99.9057	100.3613	23
20	104.4625	104.9486	24
25	100.2434	100.7264	24
30	107.0245	107.5042	24
Tab. C.17: At 40°C, 32:1 Ratio of Solvent to Sample and Different

Times

Time	W ₁	\mathbf{W}_2	Wax %
(min)			
5	99.0464	99.4695	21
10	105.6835	106.1494	23
15	102.4613	102.9188	23
20	104.8023	105.2868	24
25	107.3416	107.8388	25
30	99.0143	99.5154	25

Tab. C.18: At 50°C, 32:1 Ratio of Solvent to Sample and Different

Times

Time (min)	W ₁	W ₂	Wax %
5	106.2391	106.6979	23
10	99.0169	99.4759	23
15	108.7947	109.2518	23
20	103.7981	104.3018	25
25	99.0203	99.5246	25
30	105.9675	106.4868	26

Tab. C.19: At 60°C, 32:1 Ratio of Solvent to Sample and Different

Time (min)	W ₁	W ₂	Wax %
5	100.4725	100.9298	23
10	106.6801	107.1656	24
15	107.3789	107.8634	24
20	104.7081	105.2125	25
25	99.1897	99.7079	26
30	102.4119	102.9299	26

Tab. C.20: At 40°C, 32:1 Ratio of Solvent to Sample and Different

Times

Time	W_1	\mathbf{W}_2	Wax %
(min)			
5	104.1567	104.6394	24
10	102.9345	103.4151	24
15	106.7176	107.2029	24
20	105.6341	106.1528	26
25	105.8563	106.3746	26
30	100.0785	100.5967	26

C.2 Solvent of 75% Methyl Ethyl Ketone + 25% Toluene

Tab. C.21: At 28°C, 16:1 Ratio of Solvent to Sample and Different

Time	W ₁	W_2	Wax %
(min)			
5	108.5671	108.9360	18
10	99.0399	99.4199	19
15	99.0336	99.4320	20
20	102.8243	103.2481	21
25	105.6022	106.0258	21
30	102.2402	102.6832	22

Tab. C.22: At 40°C, 16:1 Ratio of Solvent to Sample and Different

Times

Time	W ₁	\mathbf{W}_2	Wax %
(min)			
5	108.8074	109.1856	19
10	99.7520	100.1764	21
15	100.4623	100.9054	22
20	105.6845	106.1275	22
25	103.4564	103.8975	22
30	105.2342	105.6752	22

Tab. C.23: At 50°C, 16:1 Ratio of Solvent to Sample and Different

Times

Time	W ₁	\mathbf{W}_2	Wax %
(min)			
5	105.9633	106.3678	20
10	102.9753	103.3988	21
15	99.0120	99.4532	22
20	106.2499	106.7160	23
25	102.5832	103.0485	23
30	104.9165	105.3786	23

Tab. C.24: At 60°C, 16:1 Ratio of Solvent to Sample and Different

Time (min)	W ₁	W ₂	Wax %
5	105.1976	105.6200	21
10	102.7898	103.2310	22
15	106.5678	107.0088	22
20	106.2498	106.7138	23
25	102.5831	103.0467	23
30	106.9174	107.3788	23

Tab. C.25: At 70°C, 16:1 Ratio of Solvent to Sample and Different

Times

Time (min)	W ₁	W ₂	Wax %
5	105.3456	105.7857	22
10	107.1234	107.5635	22
15	105.2507	105.7159	23
20	99.4924	99.9747	24
25	102.7758	103.2565	24
30	99.1091	99.5934	24

Tab. C.26: At 28°C, 20:1 Ratio of Solvent to Sample and Different

Times

Time	W ₁	\mathbf{W}_2	Wax %
(min)			
5	107.9630	108.3421	19
10	102.4302	102.8367	20
15	106.7344	107.1611	21
20	99.3629	99.7782	21
25	99.9001	100.3413	22
30	102.6779	103.1190	22

Tab. C.27: At 40°C, 20:1 Ratio of Solvent to Sample and Different

Time (min)	W ₁	W ₂	Wax %
5	100.0062	100.4124	20
10	106.4437	106.8798	22
15	108.2215	108.6585	22
20	105.5840	106.0493	23
25	100.9173	101.3823	23
30	105.2306	105.6948	23

Tab. C.28: At 50°C, 20:1 Ratio of Solvent to Sample and Different

Times

Time	W ₁	W ₂	Wax %
(min)			
5	109.5376	109.9431	20
10	105.8993	106.3384	22
15	99.6771	100.1161	22
20	100.4424	100.9234	24
25	104.7657	105.2492	24
30	103.1038	103.5845	24

Tab. C.29: At 60°C, 20:1 Ratio of Solvent to Sample and Different

Times

Time	W ₁	W_2	Wax %
(min)			
5	99.6715	100.0991	21
10	99.4889	99.9543	23
15	104.9222	105.3868	23
20	106.4224	106.9068	24
25	106.6657	107.1468	24
30	104.8990	105.3848	24

Tab. C.30: At 70°C, 20:1 Ratio of Solvent to Sample and Different

Time	W ₁	\mathbf{W}_2	Wax %
(min)			
5	103.2543	103.7194	23
10	107.4876	107.9537	23
15	104.8209	105.2879	23
20	106.9364	107.4419	25
25	99.1586	99.6638	25
30	106.3877	106.8929	25

Tab. C.31: At 28°C, 24:1 Ratio of Solvent to Sample and Different

Times

Time	W ₁	W_2	Wax %
(min)			
5	99.4040	99.7913	19
10	106.2254	106.6326	20
15	100.5946	101.0093	21
20	107.4567	107.8938	22
25	99.2345	99.6727	22
30	102.5833	103.0394	23

Tab. C.32: At 40°C, 24:1 Ratio of Solvent to Sample and Different

Times

Time	W ₁	\mathbf{W}_2	Wax %
(min)			
5	100.6554	100.2530	20
10	102.7069	103.1525	22
15	100.0402	100.4828	22
20	100.2503	100.7071	23
25	105.2323	105.7148	24
30	102.5655	103.0476	24

Tab.C.33: At 50°C, 24:1 Ratio of Solvent to Sample and Different

Time	\mathbf{W}_1	\mathbf{W}_2	Wax %
(min)			
5	107.6883	108.1003	21
10	99.3735	99.8158	22
15	105.7068	106.1491	22
20	104.8948	105.3799	24
25	99.2252	99.7082	24
30	99.6485	100.1315	24

Tab. C.34: At 60°C, 24:1 Ratio of Solvent to Sample and Different

Times

Time	W ₁	W ₂	Wax %
(min)			
5	108.1327	108.5447	21
10	99.9210	100.3786	23
15	105.5877	106.0453	23
20	105.6186	106.1257	25
25	103.8393	104.3465	25
30	100.0625	100.5695	25

Tab. C.35: At 70°C, 24:1 Ratio of Solvent to Sample and Different

Times

Time	W ₁	\mathbf{W}_2	Wax %
(min)			
5	102.3634	102.8190	23
10	105.9858	106.4715	24
15	100.3131	100.7978	24
20	99.3851	99.8925	25
25	102.5020	103.0096	25
30	104.3007	104.8187	26

Tab. C.36: At 28°C, 32:1 Ratio of Solvent to Sample and Different

Time (min)	W ₁	W ₂	Wax %
5	100.2120	100.5991	19
10	103.4720	103.8933	21
15	99.4798	99.8909	21
20	100.0401	100.4821	22
25	106.1301	106.5878	23
30	107.8068	108.2635	23

Tab.C.37: At 40°C, 32:1 Ratio of Solvent to Sample and Different

Times

Time	W ₁	\mathbf{W}_2	Wax %
(min)			
5	99.5894	99.9876	20
10	99.3734	99.8155	22
15	102.7067	103.1489	22
20	99.1092	99.5889	24
25	102.7758	103.2542	24
30	105.7342	106.2413	25

Tab. C.38: At 50°C, 32:1 Ratio of Solvent to Sample and Different

Times

Time (min)	W ₁	W ₂	Wax %
5	99.0141	99.4313	21
10	106.0400	106.4799	22
15	99.5745	100.0305	23
20	105.5425	106.0199	24
25	106.6758	107.1548	24
30	102.9574	103.4656	25

Tab. C.39: At 60°C, 32:1 Ratio of Solvent to Sample and Different

Time	W ₁	\mathbf{W}_2	Wax %
(min)			
5	105.3412	105.7976	23
10	99.0119	99.4686	23
15	102.6806	103.1390	23
20	100.2786	100.7859	25
25	99.5698	100.0678	25
30	103.5319	104.0499	26

Tab. C.40: At 70°C, 32:1 Ratio of Solvent to Sample and Different

Times

Time	W_1	\mathbf{W}_2	Wax %
(min)			
5	105.2473	105.7059	23
10	100.2192	100.6986	24
15	103.8859	104.3639	24
20	105.9021	106.3994	25
25	103.5688	104.0678	25
30	106.7451	107.2646	26

C.3 Solvent of 50% Methyl Ethyl Ketone + 50% Toluene

Tab. C41: At 28°C, 16:1 Ratio of Solvent to Sample and Different

Time	W ₁	\mathbf{W}_2	Wax %
(min)			
5	105.1599	105.5286	18
10	103.3377	103.7208	19
15	102.5962	103.0189	21
20	107.3635	107.7827	21
25	102.4733	102.9098	22
30	102.8076	103.2461	22

Tab. C.42: At 40°C, 16:1 Ratio of Solvent to Sample and Different

Times

Time	W ₁	W ₂	Wax %
(min)			
5	100.9758	101.3623	19
10	100.5653	100.9887	21
15	108.0365	108.4607	21
20	105.8143	106.2375	21
25	100.1419	100.5796	22
30	99.4752	99.9149	22

Tab. C.43: At 50°C, 16:1 Ratio of Solvent to Sample and Different

Times

Time	W ₁	W ₂	Wax %
(min)			
5	105.0969	105.5022	20
10	99.5921	100.0151	21
15	109.3699	109.7928	21
20	105.8185	106.2582	22
25	103.1518	103.5899	22
30	107.4851	107.9245	22

Tab. C.44: At 60°C, 16:1 Ratio of Solvent to Sample and Different

Time	W ₁	\mathbf{W}_2	Wax %
(min)			
5	100.3035	100.7060	20
10	100.3154	100.7410	21
15	105.3579	105.7748	21
20	99.1432	99.5873	22
25	105.8101	106.2543	22
30	99.5658	100.0060	22

Tab. C.45: At 70°C, 16:1 Ratio of Solvent to Sample and Different

Times

Time	W ₁	W_2	Wax %
(min)			
5	100.0343	100.4316	20
10	99.2325	99.6725	22
15	106.9092	107.3513	22
20	106.9140	107.3721	23
25	107.6207	108.0792	23
30	100.3974	100.8554	23

Tab. C.46: At 28°C, 20:1 Ratio of Solvent to Sample and Different

Times

Time	W ₁	W ₂	Wax %
(min)			
5	106.1900	106.5725	19
10	106.8032	107.2100	20
15	102.9135	103.3304	21
20	99.2900	99.7157	21
25	105.6759	106.1187	22
30	102.3426	102.7857	22

Tab. C.47: At 40°C, 20:1 Ratio of Solvent to Sample and Different

Time	W ₁	\mathbf{W}_2	Wax %
(min)			
5	103.3154	103.7201	20
10	108.8456	109.2667	21
15	100.0094	100.4534	22
20	106.6761	107.1191	22
25	99.3610	99.8271	23
30	102.8054	103.2727	23

Tab. C.48: At 50°C, 20:1 Ratio of Solvent to Sample and Different

Times

Time	\mathbf{W}_1	W_2	Wax %
(min)			
5	107.0343	107.4410	20
10	102.9011	103.3382	22
15	99.3455	99.7846	22
20	105.7889	106.2270	22
25	100.2498	100.7178	23
30	105.6938	106.1622	23

Tab. C.49: At 60°C, 20:1 Ratio of Solvent to Sample and Different

Times

Time	W ₁	\mathbf{W}_2	Wax %
(min)			
5	100.4012	100.8213	21
10	100.2332	100.6714	22
15	106.6676	107.1058	22
20	107.1362	107.5978	23
25	106.5802	107.0412	23
30	108.0246	108.4852	23

Tab. C.50: At 70°C, 20:1 Ratio of Solvent to Sample and Different

Time (min)	W ₁	W ₂	Wax %
5	105.9568	106.3767	21
10	105.4679	105.9293	23
15	107.9113	108.3736	23
20	103.4523	103.9296	24
25	102.1190	102.5978	24
30	99.2375	99.7359	25

Tab. C.51: At 28°C, 24:1 Ratio of Solvent to Sample and Different

Times

Time	W ₁	W ₂	Wax %
(min)			
5	108.4122	108.7947	19
10	100.4788	100.8844	20
15	102.5124	102.9312	21
20	105.1120	105.5513	22
25	102.7564	103.1955	22
30	102.3655	102.8280	23

Tab. C.52: At 40°C, 24:1 Ratio of Solvent to Sample and Different

Times

Time	W ₁	\mathbf{W}_2	Wax %
(min)			
5	99.4566	99.8591	20
10	99.1680	99.5856	21
15	106.2108	106.6498	22
20	103.2542	103.7176	23
25	104.8098	105.2729	23
30	104.7986	105.2606	23

Tab. C.53: At 50°C, 24:1 Ratio of Solvent to Sample and Different

Time	W ₁	\mathbf{W}_2	Wax %
(min)			
5	108.6114	109.0301	21
10	107.6452	108.0833	22
15	105.0896	105.5276	22
20	103.2420	103.7041	23
25	105.6864	106.1496	23
30	100.4822	100.9383	23

Tab. C.54: At 60°C, 24:1 Ratio of Solvent to Sample and Different

Times

Time	W ₁	W_2	Wax %
(min)			
5	99.0478	99.5042	23
10	102.6034	103.0593	23
15	105.2590	105.7161	23
20	105.7837	106.2617	24
25	99.4504	99.9279	24
30	105.1271	105.6056	24

Tab. C.55: At 70°C, 24:1 Ratio of Solvent to Sample and Different

Times

Time	W ₁	\mathbf{W}_2	Wax %
(min)			
5	106.8116	107.2689	23
10	105.3672	105.8240	23
15	99.9228	100.3808	23
20	104.9042	105.4017	25
25	107.5014	107.9987	25
30	102.2681	102.7658	25

Tab. C.56: At 28°C, 32:1 Ratio of Solvent to Sample and Different

Time (min)	W ₁	W ₂	Wax %
(11111)	00.0770	100 2 (02	10
5	99.9778	100.3602	19
10	102.8347	103.2534	21
15	105.6125	106.0323	21
20	99.4340	99.8712	22
25	106.4784	106.9367	23
30	104.0341	104.4912	23

Tab. C.57: At 40°C, 32:1 Ratio of Solvent to Sample and Different

Times

Time	W ₁	W_2	Wax %
(min)			
5	100.4900	100.8935	20
10	105.6784	106.1164	22
15	99.2292	99.6748	22
20	106.5907	107.0469	23
25	104.1563	104.6126	23
30	105.7119	106.1692	23

Tab. C.58: At 50°C, 32:1 Ratio of Solvent to Sample and Different

Times

Time	W ₁	\mathbf{W}_2	Wax %
(min)			
5	108.3903	108.8112	21
10	103.2685	103.7255	23
15	102.8231	103.2821	23
20	99.3897	99.8469	23
25	106.7938	107.2619	24
30	105.4605	105.9369	24

Tab. C.59: At 60°C, 32:1 Ratio of Solvent to Sample and Different

Time (min)	W ₁	W ₂	Wax %
5	106.8453	107.3038	23
10	100.4009	100.8593	23
15	106.9565	107.4138	23
20	102.6810	103.1822	25
25	100.0143	100.5163	25
30	106.3476	106.8479	25

Tab. C.60: At 70°C, 32:1 Ratio of Solvent to Sample and Different

Times

Time	W ₁	W ₂	Wax %
(min)			
5	99.5123	99.9693	23
10	102.7356	103.1915	23
15	100.2345	100.6898	23
20	102.5899	103.0942	25
25	104.9252	105.4308	25
30	103.2583	103.614	25

C.4 Solvent of 70% Methyl Ethyl Ketone, 20% Toluene and

10% Naphtha

Tab. C.61: At 28°C, 16:1 Ratio of Solvent to Sample and Different

Time	W ₁	W ₂	Wax %
(min)			
5	99.4758	99.8383	18
10	105.6445	106.0267	19
15	106.9175	107.3215	20
20	108.4560	108.8550	20
25	99.4809	99.9040	21
30	106.7135	107.1341	21

Tab. C.62: At 40°C, 16:1 Ratio of Solvent to Sample and Different

Times

Time	W ₁	\mathbf{W}_2	Wax %
(min)			
5	106.9123	107.3206	20
10	106.8520	107.2710	21
15	105.2401	105.6521	21
20	102.7958	103.2368	22
25	100.3513	100.7958	22
30	104.8169	105.2579	22

Tab. C.63: At 50°C, 16:1 Ratio of Solvent to Sample and Different

Times

Time	W ₁	\mathbf{W}_2	Wax %
(min)			
5	108.4265	108.8321	20
10	105.6233	106.0500	21
15	99.1407	99.5560	21
20	106.4925	106.9368	22
25	107.0514	107.4899	22
30	99.1447	99.5961	23

Tab. C.64: At 60°C, 16:1 Ratio of Solvent to Sample and Different

Time (min)	W ₁	W ₂	Wax %
5	102.0407	102.4313	20
10	109.7826	110.2002	21
15	99.1402	99.5649	21
20	106.6120	107.0494	22
25	103.6944	104.1595	23
30	107.9140	108.3709	23

Tab. C.65: At 70°C, 16:1 Ratio of Solvent to Sample and Different

Times

Time	W ₁	W ₂	Wax %
(min)			
5	107.9140	108.3210	20
10	99.2586	99.6969	22
15	105.7152	106.1548	22
20	99.3500	99.8125	23
25	102.0400	102.4971	23
30	107.2571	107.7224	23

Tab. C.66: At 28[•]C, 20:1 Ratio of Solvent to Sample and Different

Times

Time	W ₁	\mathbf{W}_2	Wax %
(min)			
5	103.1500	103.5340	19
10	99.9300	100.3110	19
15	106.8746	107.2842	20
20	105.6300	106.0345	20
25	105.1357	105.5526	21
30	106.2587	106.6829	21

Tab.C.67: At 40°C, 20:1 Ratio of Solvent to Sample and Different

Time	W ₁	W_2	Wax %
(min)			
5	106.8920	107.3012	20
10	103.6942	104.1155	21
15	107.9190	108.3382	21
20	105.3418	105.7789	22
25	107.3422	107.8114	23
30	99.1402	99.6031	23

Tab. C.68: At 50°C, 20:1 Ratio of Solvent to Sample and Different

Times

Time	W ₁	W_2	Wax %
(min)			
5	103.6944	104.0995	20
10	105.1252	105.5424	21
15	99.0103	99.4332	21
20	105.5736	106.0168	22
25	102.0400	102.4971	23
30	105.0377	105.4991	23

Tab. C.69: At 60°C, 20:1 Ratio of Solvent to Sample and Different

Times

Time	W ₁	\mathbf{W}_2	Wax %
(min)			
5	102.0401	102.4471	20
10	108.7958	109.2388	22
15	99.0180	99.4610	22
20	99.1402	99.5649	23
25	109.9185	110.4012	24
30	107.9140	108.3909	24

Tab. C.70: At 70°C, 20:1 Ratio of Solvent to Sample and Different

Time	W ₁	W_2	Wax %
(min)			
5	102.0407	102.4634	21
10	105.1456	105.5920	22
15	106.2309	106.6788	22
20	108.4425	108.9222	24
25	106.4531	106.9310	24
30	99.1450	99.6226	24

Tab. C.71: At 28°C, 24:1 Ratio of Solvent to Sample and Different

Times

Time	W ₁	\mathbf{W}_2	Wax %
(min)			
5	99.1500	99.4875	19
10	107.9105	108.3225	21
15	99.1465	99.5576	21
20	109.3154	109.7410	21
25	99.0087	99.4543	22
30	105.6402	106.0705	22

Tab. C.72: At 40°C, 20:1 Ratio of Solvent to Sample and Different

Times

Time (min)	W ₁	W ₂	Wax %
5	99.1450	99.5432	20
10	108.4265	108.8521	21
15	109.7125	110.1530	22
20	102.0400	102.4971	23
25	99.0678	99.5248	23
30	103.5120	103.9693	23

Tab. C.73: At 50°C, 24:1 Ratio of Solvent to Sample and Different

Time (min)	W ₁	W ₂	Wax %
5	105.7207	106.1397	21
10	106.2309	106.6788	22
15	99.7929	100.2399	22
20	105.7118	106.1681	23
25	100.1462	100.6024	23
30	104.3541	104.8161	23

Tab. C.74: At 60°C, 24:1 Ratio of Solvent to Sample and Different

Times

Time	\mathbf{W}_1	\mathbf{W}_2	Wax %
(min)			
5	100.0364	100.4598	21
10	106.2365	106.6831	22
15	105.4578	105.9023	22
20	103.1419	103.6051	23
25	104.1364	104.6136	24
30	105.8031	106.2797	24

Tab. C.75: At70[•]C, 24:1 Ratio of Solvent to Sample and Different

Times

Time	W ₁	\mathbf{W}_2	Wax %
(min)			
5	107.0133	107.4567	22
10	105.6714	106.0970	22
15	108.1476	108.5818	22
20	102.0078	102.4851	24
25	105.6647	106.1444	24
30	99.5911	100.0945	25

Tab. C.76: At 28°C, 32:1 Ratio of Solvent to Sample and Different

Time (min)	W ₁	W ₂	Wax %
5	102.2407	102.6133	19
10	102.9452	103.3623	21
15	102.5999	103.0410	22
20	106.7135	107.1551	22
25	105.0345	105.4819	22
30	109.8183	110.2535	22

Tab. C.77: At 40°C, 32:1 Ratio of Solvent to Sample and Different

Times

Time (min)	W ₁	W ₂	Wax %
5	102.0407	102.4313	20
10	104.1663	104.5849	21
15	99.1675	99.6049	22
20	99.5801	100.0407	23
25	108.0134	108.4748	23
30	99.4668	99.9291	23

Tab. C.78: At 50°C, 32:1 Ratio of Solvent to Sample and Different

Times

Time (min)	W ₁	W_2	Wax %
(11111)	105 5501	1061855	
5	105.7531	106.1755	21
10	107.9105	108.3445	22
15	106.4531	106.9010	22
20	102.9165	103.3826	23
25	100.3313	100.8097	24
30	105.4900	105.9705	24

Tab. C.79: At 60°C, 32:1 Ratio of Solvent to Sample and Different

Time	W ₁	\mathbf{W}_2	Wax %
(min)			
5	109.8236	110.2641	22
10	106.9031	107.3510	22
15	105.3609	105.8282	23
20	106.8053	107.2733	23
25	106.2731	106.7584	24
30	99.0479	99.5301	24

Time (min)	W ₁	W ₂	Wax %
(11111)	105.0525	106 4020	
5	105.9735	106.4038	22
10	106.9175	107.3581	22
15	104.9156	105.3825	23
20	107.9190	108.3382	24
25	99.1450	99.6226	24
30	99.1456	99.6478	25

Tab. C.80: At 70°C, 32:1 Ratio of Solvent to Sample and Different