CHAPTER IV

RESULTS AND DISCUSSIONS

In this study, the microcrystalline waxes were prepared from bright stock using sweating process and oxidation procedure. The goal of this research was to obtain the appropriate condition for the production of the microcrystalline waxes by varying the temperature and time for sweating process. The oxidation procedure was studied for the effect of catalyst amount and reaction time. Finally, the physical properties of the wax were investigated.

4.1 Sweating Process

4.1.1 Study on the optimum condition for the sweating of wax

Sweating process was carried out at the temperature of 65, 70, 73, and 75 °C. Each temperature for sweating wax was varied for 12, 24, 36, and 48 hr. The final products of this step were called "sweated wax". The results from sweating process performed at various temperatures and time are shown in Table 4.1.

Table 4.1 The effect of temperature and time for sweating process on drop melting point, oil content, and %yield

Raw materials		Average of Drop Melting Point (°C)	Average of Oil Content (% by wt)	Average of % Yield (by wt)
Temperature (°C)	Time (hr.)			
65	12	78	4.57	100.00
	24	78	4.51	99.91
	36	78	4.48	99.87
	48	78	4.41	99.63
70	12	78.5	4.18	97.61
	24	79.3	4.22	94.05
	36	79.7	3.63	92.28
	48	80.2	3.34	89.06
73	12	79.3	3.78	91.85
	24	81.3	3.52	89.24
	36	82.2	3.07	82.50
	48	82.8	2.91	78.50
75	12	81.3	3.41	86.51
	24	82.0	3.01	80.63
	36	83.2	2.82	73.34
	48	83.8	2.6	58.83

The effect of temperature and time variation on drop melting point, oil content which was calculated from equation 3.1, and percentage yield of sweated wax are illustrated in Fig. 4.1, 4.2, and 4.3, respectively.

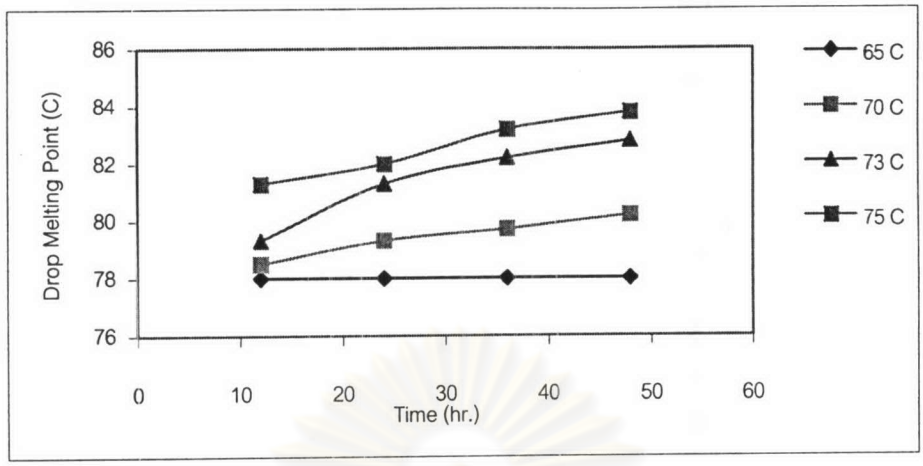

Figure 4.1 The effect of reaction time on drop melting point of each temperature for sweating wax

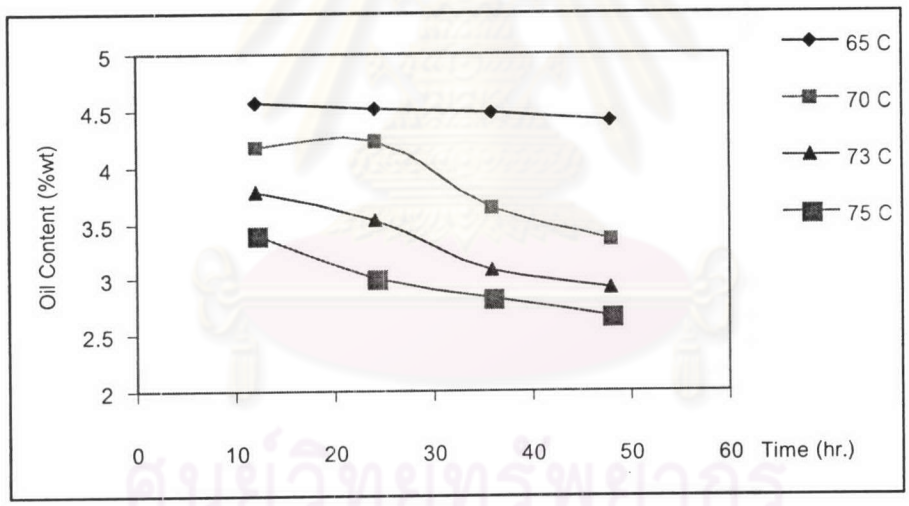

Figure 4.2 The effect of reaction time on oil content of each temperature for sweating wax

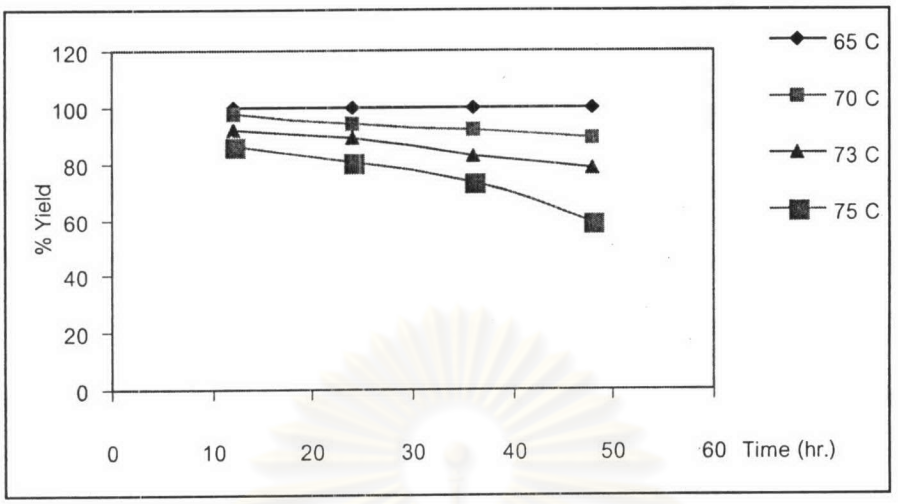

Figure 4.3 The effect of reaction time on % yield of each temperature for sweating wax

Table 4.1 and Figures 4.1-4.3 show that when the temperature was increased, the drop melting point increased, oil content decreased, and %yield decreased. Because at high temperature, the oils and low melting point waxes were melted. The remained wax had drop melting point higher than the bright stock and the oil content in wax was less than that of the bright stock.

For the specification of microcrystalline which was commercially available was more than 80 °C. In this research, the microcrystalline wax which drop melting point at 82 °C was used, thus, the optimum condition for sweating wax in this experimental was 73 °C for 36 hr which gave drop melting point of 82.2 °C which was comparable to that of std.microwax, while the oil content was decreased, and the yield was acceptable.

4.2 Oxidation Procedure

Even though, the drop melting point of sweated wax was excepted but the hardness and tackiness were not as good as std.micro.wax. So, the sweated wax was improved for hardness and toughness by oxidation procedure.

4.2.1 Study on the optimum condition for the oxidation of wax

The sweated wax obtained from the optimum condition in sweating step was oxidized at 110 °C. This procedure was carried out at the catalyst concentration of 1%, 2%, and 5% (by %wt of wax) and reaction time at 12, 15, 20, 24, 36, and 48 hr. The final products of this step were called "oxidized wax". The results from oxidation performed at various % catalyst concentration and reaction time are shown in Table 4.2.

Table 4.2 The effect of %catalyst and time for oxidation on drop melting point, acid Number, penetration, and kinematic viscosity

Sweated wax		Average of Drop melting Point (°C)	Average of Acid Number	Average of Penetration @25°C (mm/5s)	Average of ν @100°C (cSt)
%Catalyst (By wt of SW wax)	Time (hr.)				
1%	12	82.0	23.3	64.8	20.56
	15	81.5	30.1	63.3	24.76
	20	81.0	38.3	51.8	32.49
	24	80.5	46.3	47.0	39.85
	36	80.0	51.2	36.3	47.42
	48	79.0	53.6	28.8	49.63
2%	12	81.5	29.6	63.3	21.69
	15	81.0	35.5	59.3	31.20
	20	80.5	44.2	49.8	38.26
	24	80.0	56.7	43.5	46.28
	36	78.8	73.55	34.8	49.39
	48	78.0	80.9	27.8	51.99
5%	12	78.8	44.0	64.3	23.82
	15	78.3	52.7	58.8	36.89
	20	77.3	66.8	42.3	44.64
	24	76.3	74.6	34.3	49.02
	36	75.0	82.1	30.5	52.26
	48	74.0	85.7	25.3	53.41

The effect of reaction time variation of each catalyst concentration on drop melting point, acid number, which was calculated from equation 3.2, penetration @ 25 °C, and kinematic viscosity @ 100 °C of sweated wax are illustrated in Fig. 4.4, 4.5, 4.6, and 4.7, respectively.

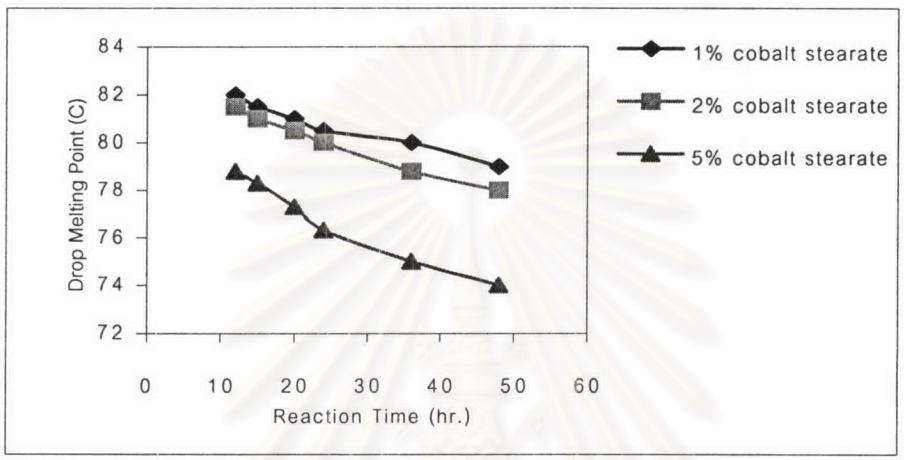

Figure 4.4 The effect of reaction time on drop melting point of each catalyst concentration for wax oxidation

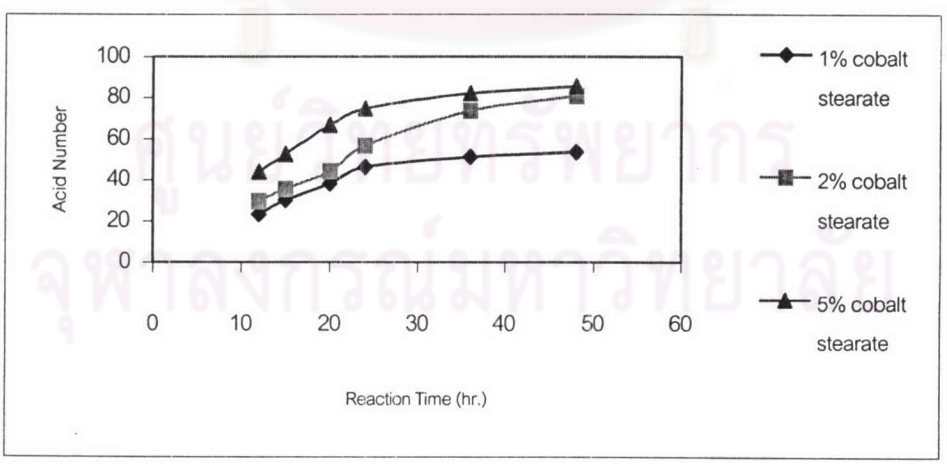

Figure 4.5 The effect of reaction time on acid number of each catalyst concentration for wax oxidation

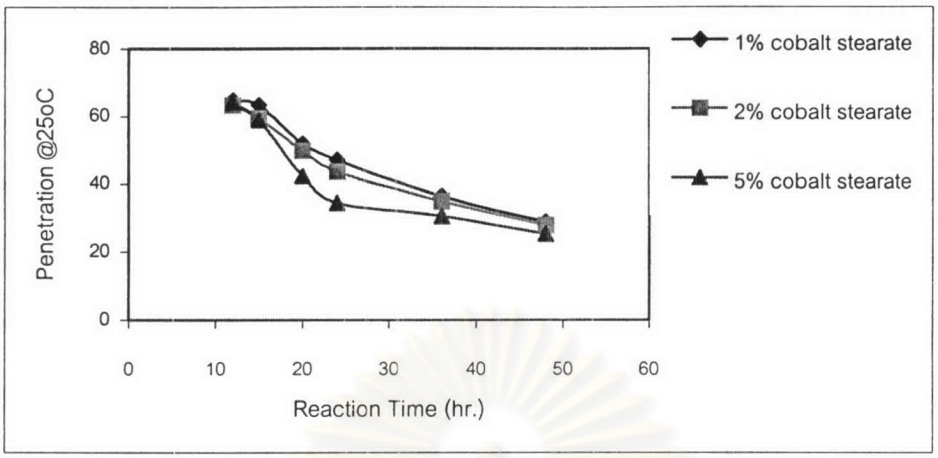

Figure 4.6 The effect of reaction time on penetration @ 25°C of each catalyst concentration for wax oxidation

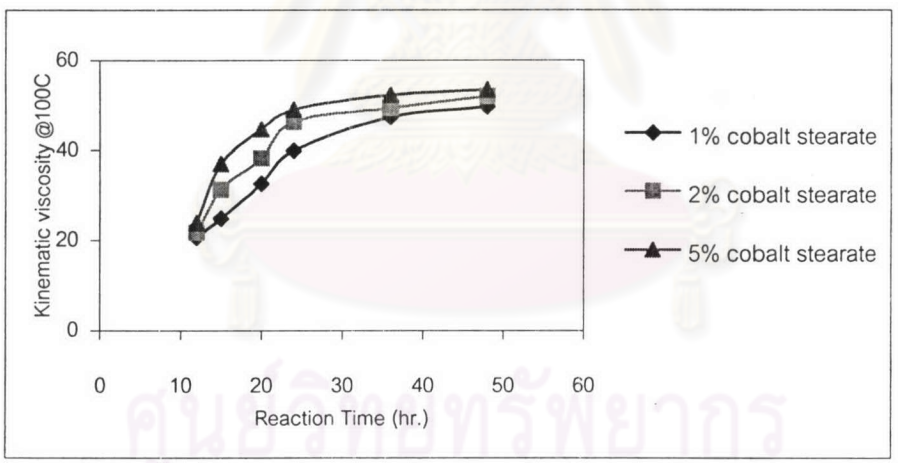

Figure 4.7 The effect of reaction time on kinematic viscosity @ 100°C of each catalyst concentration for wax oxidation

Table 4.2 and Figure 4.4-4.7 show that the acid number increased when the reaction time rose up from 10-48 hr. This may be because part of the wax was changed to fatty acids.

At higher catalyst concentration, the acid number increased faster than at the low catalyst concentration but the drop melting point decreased. It was found that at 5% of catalyst the acid number higher was and reaction time was faster than those at 1% and 2% of catalyst. However, the drop melting point was less than 80 °C. Therefore, the oxidized wax from the reaction using 5% catalyst was not appropriate to be applicable.

The acid number, drop melting point and kinematic viscosity of 1% and 2% of catalyst were compared in order to choose the best condition. It was found that, the 2% of catalyst had reaction time faster than that of 1% of catalyst. The hardness of this oxidized wax was similar to that of std.micro.wax thus, the oxidized wax in this reaction could be used as in commercial microcrystalline wax.

The optimum condition for oxidation of wax was chosen at 2% of catalyst (cobalt stearate) for 24 hr., drop melting point at 80 °C, acid number of 56.7, penetration @ 25 °C of 43.5 mm/5s, and kinematic viscosity @ 100 °C of 46.28 cSt.

4.3 Investigation of the waxes properties

In this study, the other properties of wax were measured by following the ASTM method. The properties of wax are shown in Table 4.3.

Table 4.3 The testing of waxes follow ASTM method

Testing Method	Standard
Drop melting point	ASTM D 127
Congealing point	ASTM D938
Specific gravity & Density @ 25°C	ASTM D 70
Penetration @ 25°C	ASTM D1321
Kinematic viscosity @ 100°C	ASTM D445
Flash point	ASTM D92
Estimate molecular weight	ASTM D2502
Characteristic & microscopy	-

4.3.1 Drop melting point (ASTM D 127)

The drop melting point of a wax is the temperature at which it liquefied when heated and dropped from the bottom of thermometer. For candle making, microcrystalline wax should have the drop melting point more than 80 °C.

In this study, the result of drop melting point of sweated wax and oxidized wax were 82 and 80 °C, respectively. Both are in the range of commercial waxes. The drop melting point of oxidized wax was 80°C. The drop melting point of oxidized wax was lower than that of sweated wax. Because wax was cleaved into small fatty acid in the oxidation reaction.

4.3.2 Congealing Point (ASTM 938)

Congealing Point was the points, which liquid changes to solid. From Table 4.7, it was found that sweated wax which was deoiled had congealing point higher than that of the bright stock wax because oils and low melting point substances were removed from the wax. However, oxidized wax had congealing point lower than that of sweated wax, this may be because the wax was oxidized, so the structure of wax was changed to fatty acid and the molecules were shorter than bright stock wax and sweated wax. The congealing point is 73.9 °C; therefore the wax in this research is within the acceptable range of congealing point when it was compared with the std.micro.wax.

4.3.3 Specific Gravity and Density @25°C (ASTM D 70)

The specific gravity and density at 25°C were calculated according to the equation 3.4 and 3.5 and these values of each type of wax are shown in Table 4.4.

Table 4.4 The specific gravity and density of each wax at 25°C

Type of wax	Sp Gr @25°C	Density @25°C
Paraffin wax	0.9003	0.8977
Std.micro.wax	0.9100	0.9073
Bright Stock	0.9085	0.9032
Sweated wax	0.9104	0.9076
Oxidized wax	0.9608	0.9580

The specific gravity and the density of wax in this research were near the std.micro.wax and were within the acceptable range of specific gravity and density at 25°C of normal microcrystalline wax.

4.3.4 Penetration (ASTM D 1321)

In the case of wax, it is a measurement of the relative hardness or softness by the needle penetration after 100 g of load for 5 seconds. It is reported that the average value in mm per 5 seconds of penetration was 43.5.

In this study, the oxidized wax was harder than sweated wax and the value of penetration was as good as that of std.micro.wax. This may be because they contained fatty acid. However, for the oxidized wax, the worked penetration was similar to those of the other microcrystalline wax for candle making. Thus, the oxidized wax in this research can be used in a similar manner to other commercial available microcrystalline wax.

4.3.5 Kinematic Viscosity (ASTM D 445)

In this study, the kinematic viscosity was measured and calculated using the Automated viscometers. The oxidized wax, which was, improved for the hardness and toughness is reported to have kinematic viscosity at 46.28 centistock.

From Table 4.7, it indicated that the kinematic viscosity of oxidized wax was higher than those of sweated wax and std.micro.wax. Even though, sweated wax had the kinematic viscosity similar to that of std.micro.wax but sweated wax was brittle, while Std.micro.wax was sticky. Moreover, it was found that the oxidized wax had tackiness similar to that of the std.micro.wax.

4.3.6 Flash Point (ASTM D 92)

In this study, the flash point was measured by using an automated Cleveland open cup apparatus. The flash point of the oxidized wax was reported at 244 degree Celsius. The results of flash point analysis of oxidized wax, sweated wax and std.micro.wax are shown in Table 4.7.

It was found that the flash point of oxidized wax was less than that of the flash point of sweated wax and std.micro.wax. This may be because structure of oxidized wax was changed to fatty acid with shorter chain length.

4.3.7 Estimation of Molecular Weight (ASTM D 2502)

The molecular weight of wax was calculated according to the Table 3.2 and Figure 3.2 and this values are shown in Table 4.5.

Table 4.5 The estimate molecular weight of each wax

Type of wax	ν@100 °C	ν@ 150 °C	ν@37.80 °C	ν@98.90 °C	Estimate MW
Paraffin wax	3.75	1.53	37.92	3.85	237
Std.micro.wax	18.38	7.74	111.10	18.81	772
BS	18.60	7.41	132.60	19.12	742
Sw.wax	18.32	7.42	136.80	19.25	744
Ox.wax	46.28	18.89	268.30	47.39	1079

The estimated molecular weight of bright stock and sweated wax in this research was in the range of molecular weight of the microcrystalline wax (500-800). [5] However, the oxidized wax had high molecular weight because it was estimated from kinematic viscosity, which was higher than std.micro.wax, bright stock and sweating wax.

4.3.8 Characteristics and Microscopy of waxes

The paraffin wax, std.micro.wax, bright stock, sweated wax, and oxidized wax were visually inspected using optical microscopy. The characteristics of wax and microscopy of wax are summarized in Table 4.6.

Table 4.6 Summary of visual characteristics and microscopy of waxes

Type of wax	Characteristics		
	Color	Observation	Microscopy
Paraffin wax	White	Hard & Brittle	Plate-type crystal
Std.micro.wax	Yellow	Buttery, Hard & Sticky	Fine crystal
Bright stock	Light brown	Buttery & Brittle	Need-type crystal
Sweated wax	Light brown	Buttery & Brittle	Need-type crystal
Oxidized wax	Brown	Buttery, Hard & Sticky	Need-type & Fine crystal

The crystal behaviors of waxes were studied microscopically (250x) after the waxes were cooled down from high temperature at a set rate (Figures 4.8– 4.12).

Figure 4.8 Crystal of Paraffin wax

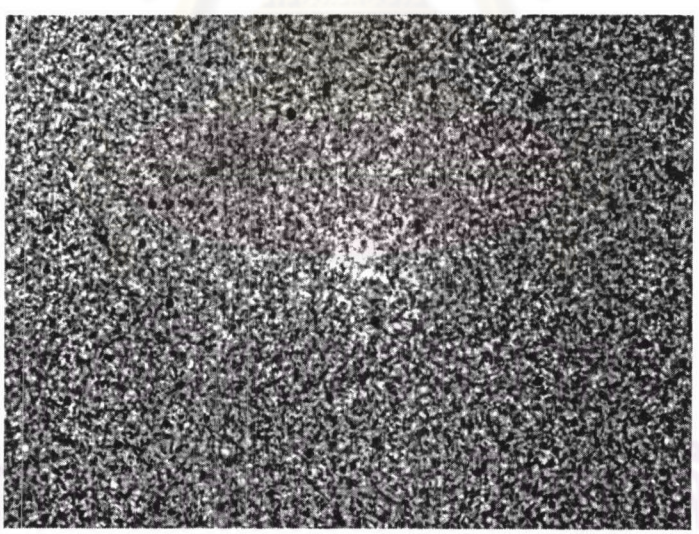

Figure 4.9 Crystal of std.micro.wax

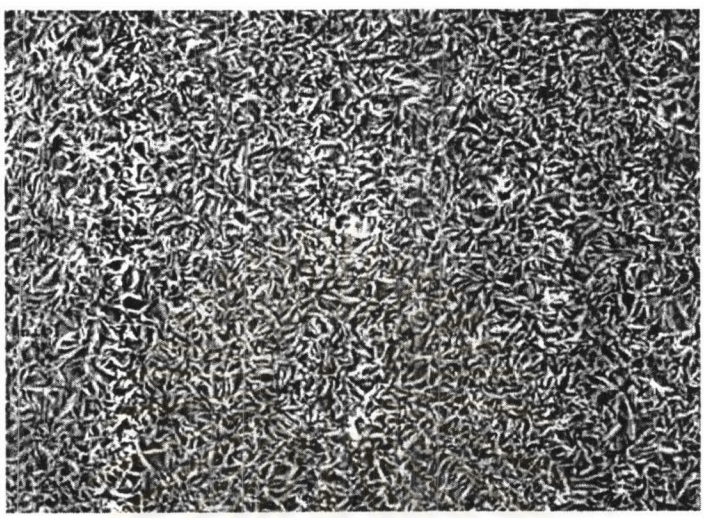

Figure 4.10 Crystal of Bright Stock Wax

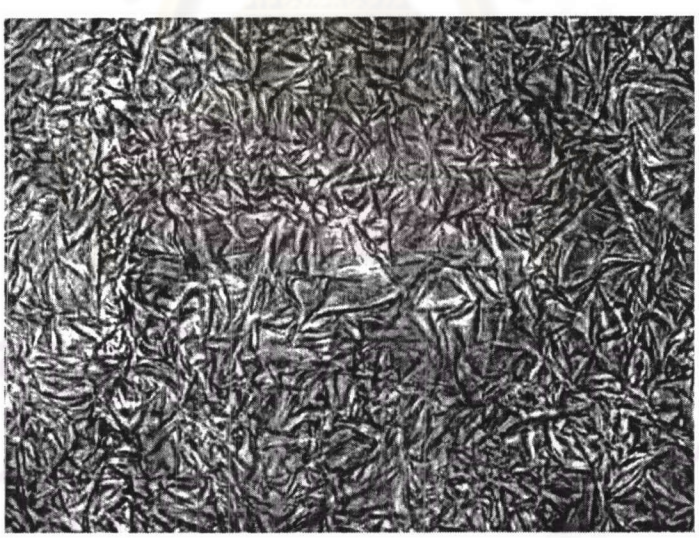

Figure 4.11 Crystal of Sweated wax

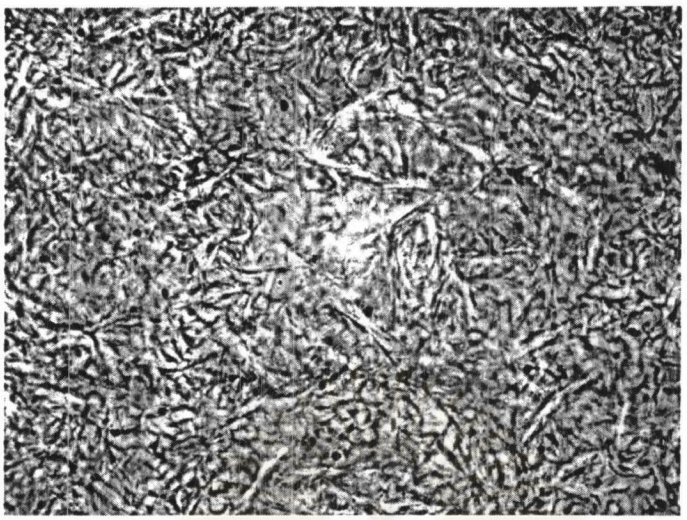

Figure 4.12 Crystal of Oxidized Wax (2%catalyst, 24 hr.)

The wax crystal structures are used in the characterization of the waxes in that plate-type crystals represent straight-chain hydrocarbons, needle-type crystals represent branched-chain hydrocarbons, and fine crystals represent microcrystalline waxes (see in Chapter 2.3).

The data of visual characteristics and microscopy of waxes in Table 4.4 (Figures 4.6 to 4.10) indicated the crystal types of waxes. These data can be expected that the bright stock and sweated wax in this research contain branched-chain hydrocarbons. When wax was oxidized, it was found that the crystal of oxidized wax was needle-plate and fine crystal. Parts of crystals were changed to resemble the crystal of microcrystalline wax. Thus, this wax was optically similar to the other commercial microcrystalline wax.

4.4 A comparison of paraffin wax, std.micro.wax and wax in this work

The important properties of this wax were compared with Paraffin wax and std.micro.wax as shown in Table 4.7.

Table 4.7 Comparison of paraffin wax, std.micro.wax and wax in this work

Testing Method	Standard	Unit	Std.micro. wax	Paffin wax	wax in this work		
					BS wax	SW wax	Ox.wax
Drop Melting Point	ASTM D 127	°C	82	56	78	82	80
Oil Content	ASTM D 721	%wt	2.48	1.06	4.57	3.07	3.07
Congealing Point	ASTM D 938	°C	73.9	57.2	76.7	77.8	73.9
Specific gravity@25°C	ASTM D 70	-	0.9100	0.9003	0.9085	0.9104	0.9608
Density @25°C	ASTM D 70	-	0.9073	0.8977	0.9032	0.9076	0.9580
Penetration@25°C	ASTM D 1321	mm/5s	44	13.5	51	48.5	43.5
Kinematic Viscosity@100°C	ASTM D 445	cSt	18.38	3.75	18.60	18.32	46.28
Flash Point	ASTM D 92	°C	288	234	320	316	244
Estimate Molecular Weight	ASTM D 2502	-	772	237	742	744	1079

The drop melting point, oil content and penetration, which are important properties of these waxes were found to be within the specification of the general microcrystalline wax. Although kinematic viscosity of this wax was higher than the general microcrystalline wax but it had appropriate tackiness for candle making. Thus, the wax in this work can be used in a similar manner to the commercial microcrystalline wax.