Investigation on the Characterization of Thitsi for Lacquerware Processing

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Abstract

The oleo-resin of Thitsi tree (*Melanorrhoea usitata*) is obtained by tapping the trees in many of the forests of Myanmar. Thitsi samples were collected from Kaingtaung, Mabain and Yaksawk Toawnships, Shan State during October and November. The physico-chemical characteristics of raw and purified Thitsi such as colour, odour, ash, viscosity, boiling range, pH and specific gravity were determined. Moreover, chemical constituents such as moisture and volatile matter, thitsiol, nitrogenous matter, gummy matter and fatty or oily matter were also investigated. In order to get kurome lacquer (purified lacquer), Thitsi were purified, firstly prepolymerized with make-shift homogenizer and followed by filtration and their physico-chemical characteristics and chemical constituents were studied again. Various functional groups of thitsiol were characterized by Fourier Transform Infrared Spectroscopy and elemental compositions of Thitsi were analyzed by Energy Dispersive X-ray Fluorescence Spectrometer. Bamboo lacquerwares were prepared by applying raw and purified Thitsi severally and hardened in the underground cellar by polymerization process. Effect of number of coating on the hardening time of Thitsi-coat on bamboo substrates were investigated at the relative humidity (70–87)% and the temperature (27.1–31.8)ºC of underground cellar. Thisi, the very natural eco-friendly sap, could be significantly enhanced its characteristics by purifying it.

Keywords : Thitsi, kurome lacquer, underground cellar, polymerization, hardening

Introduction

Lacquer used in Myanmar is called “Thitsi”, literally meaning wood varnish. It is the sap of *Melanhorrea usitata*, a tree native to Southeast Asia. Thitsi is a natural polymer collected from Thitsi trees; and it has been used in Asia for a thousand years because of its beauty and durability. Collecting Thitsi sap is like collecting gum from a rubber tree. The bark of the Thitsi tree is tapped and the exuded milky-white saps can be collected. The sap obtained is an emulsion of water in oil, which is called raw Thitsi. After being stirred to homogenize it and heated to evaporate the water, fine (purified) Thitsi is produced (website 1).

Thitsi can be classified into different classes depending upon the growing region and the tapping season. Thitsi collected during the hot season is considered to be the best quality with deep black colour. Thitsi with brown colour collected during the winter is inferior while the one collected during the rainy season is regarded as Thitsi of lowest quality with reddish brown colour (Aung Myint, et al., 1967).

Thitsi sap is prepared by prepolymerization under oxygen atmosphere. The polymerization of thitsiol monomer was accelerated by the prepolymerization process. The content of monomer in the Thitsi sap swiftly decreased; the polymer increased and the oligomer increased; in 0.5 – 2 hr. In addition, the prepolymerization was also improved significantly the hardening property of lacquer film, especially at low relative humidity. At the same time, the sizes of the water drops in the Thitsi saps significantly decreased by increasing the prepolymerization time and the lacquer film surface became smoother, which resulted in its

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excellent gloss. Due to the prepolymerization, the pencil hardness and thermal stability of lacquer film were improved as well. Thus, the fast drying lacquer should also be able to be prepared under atmosphere (Jianhong, et al., 2005).

Thitsi is an excellent natural paint. It can be stored in wooden barrels for several weeks. It is highly resilient and durable. It becomes very resistant, water-proof, insect-proof, and mold-proof, and the product is prevented from cracking, bending, or warping under fluctuations of temperature and humidity. It provides a rich array of esthetic effects of textures, layers, colours, light, and shine (Fraser-Lu, 1996).

Lacquerwares are the most distinction of all Myanmar handicrafts which are extensively used for producing several items. It is considered a minor art in most countries, but it has been one of the show industries of Myanmar. Moreover, it used to be a favorite container for royalty to store precious jewellery and documents. Myanmar Kings often gave silk, precious stones, or lacquerwares to foreign envoys as gifts. Lacquered boxes were used to store royal jewels, and meals were served in lacquered containers. It was also used in important Buddhist religious ceremonies and used as a food-carrier to the monasteries on Sabbath days (Fraser-Lu, 1996).

The overall objective of this study was to investigate the characteristics of raw and purified Thitsi for lacquerware processing.

Materials and Methods

Materials

Thitsi (Myanmar Lac) samples were collected from Kaingtaung, Mabain and Yaksawk Townships, Shan State during October and November to investigate the characterization of raw and purified Thitsi and to study the hardening time of Thitsi coat on bamboo substrates.

Methods

Purification or Prepolymerization of Thitsi

About 500 g. of raw Thitsi was taken and homogenized by make-shift homogenizer for 0.5 hr., 1 hr., 1.5 hr. and 2 hr. respectively. During homogenizing the water content was slowly reduced. After that it was filtered by using a filter cloth. During filtering and homogenization process, heating was made by halogen lamp (1000W.) from 1 ft. above the homogenizer and filtering medium to facilitate the flow of Thitsi and evaporation of water. Finally, purified Thitsi (kurome lacquer) was obtained.

Determination of Physico-chemical Characteristics of Raw and Purified Thitsi

Physico-chemical characteristics of raw and purified Thitsi like ash content, viscosity, boiling range, pH and specific gravity were determined (Annual Book of ASTM Standards, 1986, *Paints, Related Coatings and Aromatics,* Volume 06.01 and Volume 06.02).

Determination of Constituents of Raw and Purified Thitsi

Constituents of raw Thitsi and purified samples such as moisture and volatile matter, thitsiol, nitrogenous matter, gummy matter and fatty or oily matter contents were determined (Pearson, R. S., 1908).

Characterization of Functional Groups of Thitsiol

The various functional groups of thitsiol of purified Thitsi were examined by Fourier Transform Infrared Spectroscopy (FT-IR, Perkin Elmer, 8400, Shimadzu).

Mineralogical Analysis of Raw and Purified Thitsi

The ED-XRF (Energy Dispersive X-ray Fluorescence Spectrometer) (Spectro Xepos, Benchtop XRF Spectrometer) was used for the determination of elemental composition of Thitsi.

Preparing the Bamboo Substrates

Air-dried matured stalks of bamboo (Wa-ya) (*Gigantochloa rostrata Wong*) substrates were cut into 2 in. length and 1 in. width and polished with abrasive paper (paper No. 100, 200, 400, 600) respectively to smooth the surface and cleaned with a piece of clean cotton cloth to free from dust and dirt.

Lacquering and Hardening in Local Underground Cellar

The prepared bamboo substrates were lacquered with raw and purified Thitsi from Shan State. Lacquering was made with a flat brush carefully in all coating to form a thin layer of uniform coating. The hardening of lacquer coating was taken place in the local underground cellar. After each coating, the freshly dried coat was rubbed with abrasive paper (paper No. 400 and 600) to get smooth surface. The specimen was said to be dried if no tackiness was felt by a finger press on the lacquered surface.

Results and Discussion

As shown in Table (1), it was observed that the colour of Thitsi changed from brown and deep brown to black and also from reddish brown to deep brown due to the purification process. The colour of Thitsi represented their quality. Thitsi samples possessed the peculiar sweetish odour. It was also found that ash content of purified Thitsi samples was slightly increased by the purification and highest ash content, 0.132 %w/w was occurred in purified Thitsi from Yaksawk Township. After purification, it was clearly observed that the viscosity of all purified Thitsi were greatly higher than that of raw Thitsi. Among Thitsi samples, the highest viscosity (12160 cP) was occupied by purified Thitsi from Kaingtaung Township. It was occurred that the viscosity of Thitsi affected on the dry film thickness of lacquerwares. But specific gravity was rather decreased. Thus, specific gravity of purified Thitsi became lighter than that of raw Thitsi and also that of water. pH of all Thitsi samples were between 5.6 and 5.9. By the purification process, their physico-chemical characteristics did not entirely deviate. These physico-chemical characteristics of Thitsi were within the range of literature's Thitsi values.

Constituents of raw and purified Thitsi are displayed in Table (2). The main constituent of Thitsi, the highest thitsiol content, 85.0% w/w was found in purified Thitsi from Mabain Township and the lowest thitsiol content, 62.5% w/w was occurred in raw Thitsi from Yaksawk Township. Due to the purification process, the contents of thitsiol and nitrogenous matter were increased whereas remaining constituents, moisture and volatile matter, gummy matter and fatty or oily matter were decreased. The lowest moisture and volatile matter content, 2.5%w/w and the highest thitsiol content, 85%w/w were occurred in purified Thitsi from Mabain Township. Therefore, they could possess the best quality. In purified Thitsi, decreasing the moisture and volatile matter content and fatty or oily matter and increasing the nitrogenous matter facilitated the hardening, meanwhile increasing the thitsiol content impaired the hardening, but it could provide the best gloss and high resistance to physical and chemical attack. The quality of Thitsi depends on its content of Thitsic acid, now termed thitsiol. The quality of Thitsi depends on its content of Thitsic acid, now termed thitsiol.

To improve the quality of lacquerware products and to speed up the polymerization time, raw Thitsi were prepolymerized or purified. Table (3) shows the effect of prepolymerization time on the moisture and volatile matter content and thitsiol content of Thitsi. It was found that moisture and volatile matter content decreased meanwhile thitsiol content increased with increased prepolymerization time. It was also found that the most suitable prepolymerization time observed in 1 hr. because of the needs for the reliable Thitsi to be minimum moisture and volatile matter content, 3–5% w/w. At this time, the highest thitsiol content was 85%w/w. For some Thitsi samples, it was observed that better constituents were still provided beyond 1 hr. prepolymerization time. But there would be great expense on production time between 1 hr. and 1.5 hr.

Various functional groups of thitsiol from Thitsi were examined by FTIR and it was proved by these spectra that the samples were Thitsi. As shown in Table (4) and (Fig. 1), the frequencies at 3470 cm^{-1}, 3441 cm^{-1} and 3427 cm^{-1} were represented respectively the presence of phenolic groups which has O – H stretching vibration assignment. The frequencies at 3009 cm^{-1} of all thitsiol samples were corresponded to the presence of = CH – group of the aromatic system. The bands at 2924 cm^{-1} and 2926 cm^{-1} frequency were indicated the saturated alkyl group which has – CH_2 – asymmetric stretching vibration and the frequencies at 2852 cm^{-1} of all samples showed the saturated alkyl group which had – CH_2 – symmetric stretching vibration. The bands at 1278 cm^{-1} and 1280 cm^{-1} frequency were represented the characteristics of O – H in-plane bending vibration of phenolic group whereas at the frequencies of 731 cm^{-1} and 732 cm^{-1} were corresponded to the O – H out-of-plane bending vibration of phenolic group. The infrared spectrum of thitsiol obtained was found to be same of urushiol from Japanese urushi virtually.

The elemental compositions of Thitsi samples are shown in Tables (5), (6) and (7). It was observed that the most plentiful element in all Thitsi samples was calcium, 0.04325 % - 0.09867% in raw Thitsi and 0.20320% - 0.39000% in purified Thitsi. Other obvious elements were potassium and iron. The concentration of these elements in raw Thitsi increased because of the purification process. Some harmful elements such as arsenic, cadmium, mercury and lead, all of which appear in the World Health Organization's list of 10 chemicals of major public concern, were also observed. It can be clearly seen from these Tables that the concentration of these elements mostly decreased due to the purification process. Some concentrations greatly decreased to nearly 0%. Other elements contained only in trace amount. Therefore, Thitsi is a kind of natural organic polymer.

Preliminary determination of standard deviation for hardening time of raw Thitsi-coat on bamboo substrates is presented in Table (8). It was found that standard deviation for hardening time of raw Thitsi-coat on bamboo substrates hold between 0.58 hr. and 1 hr. For further studies, this deviation time covered on the hardening time of raw and purified Thitsi-coat on bamboo lacquerwares. As described in Table (9), for both raw and purified Thitsi, high moisture and volatile matter content and high nitrogenous matter content facilitate the hardening whereas high thitsiol content curtails the hardening. The presence of nitrogenous constituent was essential for the drying of Thitsi. It was found that the hardening time of all Thitsi samples were slightly decreased in consecutive increased number of coating. Moreover, high relative humidity of underground cellar diminished the hardening time. Since, purification or prepolymerization accelerated the polymerization of thitsiol monomer to the lacquer dimer, trimer, oligomer and polymer, it can be clearly observed that the hardening time of coated lacquerwares with purified Thitsi in all coating was greatly shorter than the wares coated with raw Thitsi. It was also found that the increased numbers of coating decreased the hardening time.

Total hardening time of coat of raw and purified Thitsi are shown in Table (10). For the coated lacquerwares with purified Thitsi, the total hardening time required were more saved than for the wares coated with raw Thitsi. For 8th coated wares, if raw Thitsi from Mabain Township was used to paint the bamboo substrates, 344.84 hr. would be required to harden. But purified Thitsi from Mabain Township required 260.75 hr. to harden that 8th coated bamboo substrates, and the same situation was encountered in all the remaining Thitsi.

Table (1) Physico-chemical Characteristics of Raw and Purified Thitsi

Sr. No.	Characteristic	Source of Thitsi						Literature Value
		KT Raw	KT Purified	MB Raw	MB Purified	YS Raw	YS Purified	
1	Colour	brown	black	deep brown	black	reddish brown	deep brown	grayish – black
2	Odour	peculiar sweetish	peculiar sweetish	peculiar sweetish	peculiar sweetish	peculiar sweetish	peculiar sweetish	peculiar sweetish
3	Ash (%w/w)	0.092	0.103	0.082	0.094	0.126	0.132	0.041–0.130
4	Viscosity (cP.)	3862	12160	2501	5700	3494	8960	200 – 14100
5	Boiling Range (ºC)	40 – 90	43 – 84	36 – 88	41 – 85	30 – 92	54 – 87	18.8 – 387.7
6	pH	5.7	5.7	5.9	5.9	5.6	5.6	4 – 6
7	Specific Gravity	1.009	0.936	1.010	0.948	1.018	0.938	0.985–1.013

KT = Kaingtaung, MB = Mabain, YS = Yaksawk

Table (2) Constituents of Raw and Purified Thitsi

Sr. No.	Constituent	Source of Thitsi						Literature Value (Japanese Urushi)
		KT (R)	KT (P)	MB (R)	MB (P)	YS (R)	YS (P)	
1	Moisture and Volatile Matter (%w/w)	15.0	10.0	5.0	2.5	23.5	15.0	20 – 30
2	Thitsiol (%w/w)	72.5	80	80.0	85.0	62.5	71.5	60 – 70
3	Nitrogenous Matter (%w/w)	0.5	1.5	0.45	1.5	1.0	2.0	1.5 – 5
4	Gummy Matter (%w/w)	2.5	2.0	1.5	1.0	3.0	2.5	4 – 10
5	Fatty or Oily Matter (% w/w)	0.3	0.2	0.2	0.1	0.6	0.4	0 – 1

KT = Kaingtaung, MB = Mabain, YS = Yaksawk, R = raw, P = purified

Table (3) Effect of Prepolymerization Time on the Moisture, Volatile Matter and Thitsiol Contents of Raw Thitsi

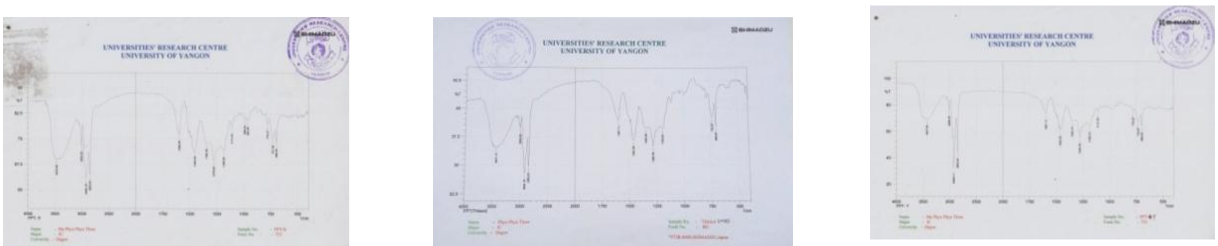
Sr. No.	Prepolymerization Time (hr.)	Constituent of Different Sources of Thitsi						Remarks
		Moisture and Volatile Matter (% w/w)			Thitsiol (%w/w)			
		KT	MB	YS	KT	MB	YS	
1	0.0	17.0	8.0	25.5	72.5	80.0	62.5	1. Thitsi should have minimum moisture and volatile matter content (3 – 5)%w/w. 2. All constituents are essential for hardening of Thitsi.
2	0.5	14.0	5.5	20.0	76.0	82.5	67.5	
3	1.0*	10.0	2.5	15.0	80.0	85.0	71.5	
4	1.5	4.5	0.0	6.5	85.0	88.0	80.0	
5	2	0.0	0.0	1.0	88.0	88.0	86.5	

*Most suitable condition

Table (4) FTIR Absorption Frequencies of Functional Groups of Thitsiol from Purified Thitsi

Sr. No.	Observed Frequency (cm^{-1})			Literature Frequency* (cm^{-1}) (Urushiol)	Band Assignment	Remarks
	KT	MB	YS			
1	3470	3441	3427	3431	O – H stretching vibration	Phenol
2	3009	3009	3009	3012	= CH – stretching vibration	Aromatic ring
3	2924	2924	2926	2926	– CH_2 – asymmetric stretching vibration	Saturated alkyl group
4	2852	2852	2852	2853	– CH_2 – symmetric stretching vibration	Saturated alkyl group
5	1278	1280	1280	1310-1200	O – H in-plane bending vibration	Phenol
6	731	732	731	750-650	O – H out-of-plane bending vibration	Phenol

KT = Kaingtaung, MB = Mabain, YS = Yaksawk



(a) (b) (c)

Fig (1) FTIR Spectrum of Thitsiol in Thitsi from (a) Kaingtaung (b) Mabain and (c) Yaksawk Townships

Table (5) Elemental Composition of Thitsi from Kaingtaung Township

Sr. No.	Element	Concentration (% w/w)		Sr. No.	Element	Concentration (% w/w)	
		Raw	Purified			Raw	Purified
1	Phosphorus	0.00506	0.00825	11	Nickel	0.00006	0.00010
2	Sulfur	0.00706	0.00962	12	Copper	0.00202	0.00295
3	Chlorine	0.02706	0.13610	13	Zinc	0.00078	0.00793
4	Potassium	0.01296	0.04947	14	Arsenic	0.00008	0.00010
5	Calcium	0.05987	0.21640	15	Silver	<0.00020	-
6	Titanium	0.00825	0.00972	16	Cadmium	0.00007	<0.00020
7	Chromium	0.00107	0.00365	17	Iodine	<0.00030	0.00007
8	Manganese	0.00710	0.01943	18	Mercury	0.00049	0.00034
9	Iron	0.05169	0.15100	19	Lead	0.00011	0.00022
10	Cobalt	<0.00030	<0.00030	20	Uranium	<0.00002	-

Table (6) Elemental Composition of Thitsi from Mabain Township

Sr. No.	Element	Concentration (% w/w)		Sr. No.	Element	Concentration (% w/w)	
		Raw	Purified			Raw	Purified
1	Phosphorus	0.00635	0.01269	11	Nickel	0.00007	0.00015
2	Sulfur	0.00533	0.01297	12	Copper	0.00253	0.00263
3	Chlorine	0.03260	0.12240	13	Zinc	0.00313	0.00204
4	Potassium	0.02304	0.07376	14	Arsenic	0.00008	0.00010
5	Calcium	0.09867	0.39000	15	Silver	<0.00020	-
6	Titanium	0.04712	0.01534	16	Cadmium	0.00009	0.00001
7	Chromium	0.00136	0.00421	17	Iodine	<0.00030	<0.00030
8	Manganese	0.01160	0.00699	18	Mercury	0.00027	0.00026
9	Iron	0.52930	0.14130	19	Lead	0.00069	0.00032
10	Cobalt	<0.00030	<0.00030	20	Uranium	0.00003	-

Table (7) Elemental Composition of Thitsi from Yaksawk Township

Sr. No.	Element	Concentration (% w/w)		Sr. No.	Element	Concentration (% w/w)	
		Raw	Purified			Raw	Purified
1	Phosphorus	0.00359	0.00809	11	Nickel	0.00007	0.00010
2	Sulfur	0.00460	0.00986	12	Copper	0.00181	0.00271
3	Chlorine	0.01735	0.09323	13	Zinc	0.00039	0.00147
4	Potassium	0.01146	0.04354	14	Arsenic	0.00006	0.00006
5	Calcium	0.04325	0.20320	15	Silver	<0.00020	-
6	Titanium	0.00615	0.00950	16	Cadmium	0.00009	0.00001
7	Chromium	0.00114	0.00264	17	Iodine	0.00016	<0.00030
8	Manganese	0.00921	0.00751	18	Mercury	0.00028	0.00023
9	Iron	0.00211	0.07450	19	Lead	0.00016	0.00020
10	Cobalt	<0.00030	<0.00030	20	Uranium	0.00005	-

Table (8) Preliminary Determination of Standard Deviation for Hardening Time of Raw Thitsi-Coat on Bamboo Substrates

Sr. No.	Thitsi Sample	Hardening Time of Thitsi-Coat (hr.)			Average Deviation Time (hr.)	Standard Deviation Time (hr.)
		1st Time	2nd Time	3rd Time		
1	KT	67.00	69.00	68.00	67.00	1.00
2	MB	72.00	70.00	71.00	71.00	1.00
3	YS	39.00	40.00	39.00	39.33	0.58

Table (9) Hardening Time of Coat of Raw and Purified Thitsi

Sr. No.	Number of Coat	Hardening Time of Thitsi-Coat (hr.)					
		KT (R)	KT (P)	MB (R)	MB (P)	YS (R)	YS (P)
1	1st coat	67.00	34.67	71.50	42.83	38.83	30.33
2	2nd coat	45.83	33.33	49.00	39.75	36.67	28.00
3	3rd coat	39.00	32.17	45.67	37.00	34.17	26.58
4	4th coat	31.00	27.50	38.58	33.17	29.50	23.25
5	5th coat	28.75	23.75	36.42	29.00	26.92	20.00
6	6th coat	27.42	21.17	35.42	27.50	25.25	18.75
7	7th coat	26.25	20.33	34.50	26.17	24.33	18.00
8	8th coat	25.00	19.50	33.75	25.33	23.17	17.17

R = Raw, P = Purified, KT = Kaingtaung, MB = Mabain, YS = Yaksawk

Table (10) Total Hardening Time of Coat of Raw and Purified Thitsi

Sr. No	Number of Coat	Total Hardening Time of Thitsi-Coat (hr.)					
		KT (R)	KT (P)	MB (R)	MB (P)	YS (R)	YS (P)
1	1st coat	67.00	34.67	71.50	42.83	38.83	30.33
2	2nd coat	112.83	68.00	120.50	82.58	75.50	58.33
3	3rd coat	151.83	100.17	166.17	119.58	109.67	84.91
4	4th coat	182.83	127.67	204.75	152.75	139.17	108.16
5	5th coat	211.58	151.42	241.17	181.75	166.09	128.16
6	6th coat	239.00	172.59	276.59	209.25	191.34	146.91
7	7th coat	265.25	192.92	311.09	235.42	215.67	164.91
8	8th coat	290.25	212.42	344.84	260.75	238.84	182.08

Fig (2) Raw Thitsi Sample

Fig (3) Purified Thitsi Sample

Fig (4) Make-shift Homogenizer

Fig (5) Drying of Bamboo Substrates in Local Underground Cellar

Conclusion

In conclusion, the constituents and properties of Thitsi varied with the age of tree, grown region and collected season. Moreover, the quality of Thitsi mainly depended on its thisiol content. In both raw and purified Thitsi, Thitsi sample from Mabain Township was the best quality with the highest thitsiol content. Moreover, the best quality Thitsi could give the illustrious lacquerwares, possessing outstanding qualities with marvelous gloss, great resistance and high durability. It was found that purification of Thitsi could produce fine lacquer, since viscosity and thitsiol content markedly increased with decreased in moisture and volatile matter content. It was claimed that purified Thitsi could provide further appropriate seasoning, viscosity and glossiness when laid upon any surfaces. Myanma Thitsi has necessary compositions that required in creditable Thitsi.

Lacquer could not set by the aid of heat, sun light and dry air and hence, polymerization of Thitsi should be taken place in the dark underground cellar to dry the lacquerwares. The shortest hardening time occurred in both raw and purified Thitsi from Yaksawk Township. Purification process saved the time required for polymerization of raw Thitsi and also gave the high quality of lacquerwares. In addition, the prepolymerization was to hopefully be used to prepare a fast drying Thitsi on a large scale and promote the application of Thitsi as an industrial paint.

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