Preparation and Characterization of Cellulose Film from Sawdust of *Pterocarpus macrocarpus* Kurz

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Abstract

The sawdust of Padauk (*Pterocarpus macrocarpus* Kurz.) collected from Mogaung Township was selected for phytochemical screening, elemental analysis and the preparation of cellulosic pulps by using sodium hydroxide. In order to know the effect of time on the yield of cellulosic pulp, various refluxing periods were used. The yield percentages of the cellulosic pulps from Padauk were determined. The purity of Padauk cellulosic pulps was confirmed by Fourier Transform- Infrared (FT-IR) spectroscopic method. The cellulosic films were prepared by mixing with different ratios of plasticizer (Polyvinyl acetate, PVA). The mechanical properties of the prepared films were determined.

Keywords: Cellulose Film, FT-IR, mechanical properties, *Pterocarpus macrocarpus* Kurz., PVA

Introduction

Sawdust is the very fine particles of wood and produced mainly from sawmill industries. It is also a by-product from agricultural, forestry, furniture factories or architectural activities. (Adekunle, 2010 and Adekunle, 2007) It can be used as mulch, fuel, sorbent material and wood briquettes. But it is one of the major wastes from wood exploitation and processing under uncontrolled conditions. It may become one of the important factors of environmental pollution (water pollution and air pollution). (Deaca, 2016)

In Myanmar, the enormous quantities of sawdust generated from the different industries are commonly combusted as fuel for domestic uses, abandoned or thrown into rivers, streams or drainages. The resulting pollutants from the use of sawdust as a bioresource/ feedstock are ash and air pollutants emanated through carbon monoxide, volatile organic compounds, nitrogen oxides (NO_x), sulphur oxides (SO_x) and particular emissions. (Deaca, 2016)

Sawdust thrown in water contributes to increased organic matter particulates in the aquatic ecosystem, reflected by high biochemical oxygen demand (BOD) and reduced dissolved oxygen (DO). The disposal of sawdust into the drainage leads to blockages, transforming the resulting stagnant water to breeding ground for pathogenic organisms with associated water borne diseases and reduction of environmental aesthetics. (Adekunle, 2010 and Adekunle, 2007)

Sawdust is composed of basic chemical compositions such as cellulose, hemicellulose and lignin. Instead of using the raw sawdust particles as feedstock, one of its components cellulose may be used to make biodegradable film to reduce the environmental pollution. Thus, the sawdust of Padauk (widely distributed in Myanmar) was selected to do this research. The cellulose films were prepared from the extracted cellulose and the purity of the prepared film and its mechanical properties were determined.

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Materials and Methods

Materials

All the chemicals and reagents used were purchased from Able chemical shop, Mandalay. The sawdust of Padauk was collected from Mogaung Township, Kachin State. EDXRF Spectrometer (EDXRF-700) and FT-IR spectrometer (Shimadsu, Japan) were used for the elemental analysis and the determination of the purity of extracted cellulose.

Preliminary Phytochemical Tests

The phytochemical tests were carried out at Department of Chemistry, University of Mandalay to detect the different kinds of chemical constituents in the saw dust of Padauk. (Harbone, 1973, Thamaralselvi, 2012, Geetha, 2014 and Tiwari, 2011)

Elemental Analysis

The elemental compositions of the sawdust of Padauk were determined by EDXRF spectral data whether the heavy toxic metals were present or absent in the sawdut of Padauk.

Extraction of Cellulosic Pulps from Sawdust for Five Hours Refluxing Time

The extraction procedure of Cross and Bevan, described by David and Nobuo (Norashikin, 2010) which was based on chlorination and extraction with hot aqueous sodium sulphite was employed. In this research work, about 25 g of sawdust and 100 ml of 1% sodium sulphite were placed in flat bottom flask and refluxed for one hour. The flask was cooled under normal condition and filtered. 100 ml of 15% sodium hydroxide was added to the residue and refluxed for 5 hours (condition 1). It was cooled again and filtered. The residue was mixed with 100 ml of 5% potassium hydroxide and the mixture was shaken for one hour at 250 rpm and filtered .The residue was mixed with 100 ml of 5% sodium hypochloride. It was bleached for 24 hours and filtered again. The residue was washed with acetic acid and then distilled water until neutral. After that, the neutral cellulosic pulps were obtained.

Extraction of Cellulosic Pulps from Sawdust for Ten Hours Refluxing Time

The same procedure was repeated for ten hours refluxing time (condition 2).

Figure (1). Extraction of cellulose from sawdust by refluxing with sodium hydroxide

Identification of Cellulosic Pulps by FT-IR Spectral Data

The extracted cellulosic pulps in both conditions (1) and (2) were identified by FT IR spectral data measured at Department of Chemistry, Monywa University.

Preparation of Cellulose Film by Using Different Amounts of PVA Plasticizer

The preparation procedure described by Norashikin and Ibrahim, 2010 which was based on chemical treatment with concentrated NaOH solution followed by hydrolysis with concentrated HCl solution at 80ºC for 2 hrs. The alkaline treated pulp was washed several times with distilled water until the pH became neutral before being dried at room temperature. 2 gram starch and 100 milliliters distilled water was gelatinized by heating at 90°C. 2 gram chitosan powder was dissolved with 100 milliliters acetic acid and both solution were mixed and stirred until become homogeneous. Then, the solution was added with 2 gram sawdust fiber and 3 milliliters additive. After that the solution was degassed for 24 hours. The solution was poured onto a glass plate and dried at room temperature. The film was carefully removed by peeling from the glass plate. (Norashikin, 2010)

In this research, 1 g of cellulose pulp was treated with 15 mL of 3 M HNO_3 acid and a few drops of acetic acid was added to dissolve it. Then it was filtered and the filtrate was mixed with 10 mL of 3 M NaOH solution and 1 g of PVA powder. The mixture was stirred for about 2 hours and was poured onto a glass Petridis and dried at room temperature. The film was carefully removed from the Petridis.

The same procedure was used to prepare different films by using different amounts of PVA powder (0.5 g, 0.25 g, 0.125 g and 0.0625 g) and other different films by using different amounts of 3 M NaOH solution (10 mL, 5 mL and 2.5 mL) respectively.

Determination of Mechanical Properties of Prepared Cellulose Films

The mechanical properties of the prepared cellulose films such as thickness, tensile strength, elongation at break and tear strength were determined at Yangon University Research Centre, University of Yangon.

Results and Discussion

Preliminary Phytochemical Screening of Padauk Sawdust

The results obtained for the phytochemical screening from Padauk sawdust was tabulated in Table (1) as follows.

Table (1). Preliminary Phytochemical Tests of Sawdust of Padauk

No	Test	Reagents	Result
1.	Alkaloids	(a) Dragendorff's reagent	+
		(b) Wagner's reagent	+
2.	Flavonoids	Conc: HCl, Mg tunnings, conc: H_2SO_4	-
3.	Glycosides	10% Lead acetate	+
4.	Reducing Sugars	Benedict's solution	+
5.	Steroids	Acetic anhydride, $CHCl_3$, conc: H_2SO_4	-
6.	Saponins	Shake vigorously	+
7.	Phenolic compounds	10 % Fe Cl_3	+
8.	Terpenes	Pet ether, Acetic anhydride, conc: H_2SO_4, $CHCl_3$	+
9.	Lipophenols	0.5M KOH	-
10.	Polyphenols	10% Fe Cl_3, 1% $K_3[Fe(CN)_6]$	+
11.	Tannins	3 drops 5% $FeCl_3$	+

(+) = presence of constituents, (-) = absence of constituent

According to this table, the sawdust contains alkaloids, flavonoids, glycosides, phenols, polyphenols, sugars, reducing sugars, saponins and terpenes respectively.

Determination of Elemental Compositions of Padauk Sawdust

The relative abundance of the mineral elements present in Padauk sawdust was shown in table (2).

According to the EDXRF report, there are ten mineral elements in the sawdust of Padauk. Among them, silicon is the most abundant element followed by calcium, sulphur and potassium. The least elements present in the selected sawdust are copper, zinc and manganese.

Table (2). Relative Abundance of the Mineral Elements Present in Padauk Sawdust

No	Element	Symbol	Relative Abundance (%)
1	Silicon	Si	0.332
2	Calcium	Ca	0.244
3	Sulphur	S	0.072
4	Potassium	K	0.047
5	Iron	Fe	0.024
6	Phosphorus	P	0.023
7	Titanium	Ti	0.003
8	Copper	Cu	0.001
9	Zinc	Zn	0.001
10	Manganese	Mn	0.001

Extraction of Cellulose Pulps from Padauk Sawdust

The different weights of the cellulosic pulps extracted from the sawdust sample for 5 and 10 refluxing hours were weighed by using electrical balance. Their yield percents were calculated and described in the tables (3) and (4).

Table (3). Weight and Yield Percent of the Extracted Cellulosic Pulps from Padauk Sawdust (Condition-1)

No.	Weight of Sawdust (g)	Time (hr)	% NaOH	Weight of Pulp(g)	Yield Percent of Pulp (%)
1.	25.00	5	15%	10.75	43.0
2.	25.00	5	15%	11.00	44.0
3.	25.00	5	15%	10.60	42.4
					43.0 ± 1.3

Table 4. Weight and Yield Percent of the Extracted Cellulosic Pulps from Padauk Sawdust (Condition-2)

No.	Weight of Sawdust (g)	Time (hr)	% NaOH	Weight of Pulp(g)	Yield Percent of Pulp (%)
1.	25.00	10	15%	5.3	21.2
2.	25.00	10	15%	5.4	21.6
3.	25.00	10	15%	5.2	20.8
					21.6 ± 0.63

According to these tables, the yield percent of cellulose pulp from experiment using 15% sodium hydroxide for 5 hours was high.

FT-IR Assignments of Cellulosic Pulps for Different Refluxing Hours

In the FT- IR spectrum of the cellulosic pulp extracted for **five** hours refluxing time (Fig. 2), the band at 3333.14 cm^{-1} indicates the O-H stretching vibration of hydroxyl group.

The peak at 2900.4 cm^{-1} represents the unsymmetrical C-H stretching vibrations of sp^3 hydrocarbons. The C=C stretching vibration of sp^2 hydrocarbons was found at 1595 cm^{-1}. The C-O-C stretching vibration of ether group is found at 1159, 1107.2 and 1031.37 cm^{-1}. The C-H out of plane bending vibration of sp^3 hydrocarbons is found at 894.63 cm^{-1}.

Therefore, the IR spectral data confirm the formation of cellulosic pulps extracted from the sawdust of Padauk. But a little amount of lignin is still present in the extracted cellulosic pulp. (Silverstein, 1981)

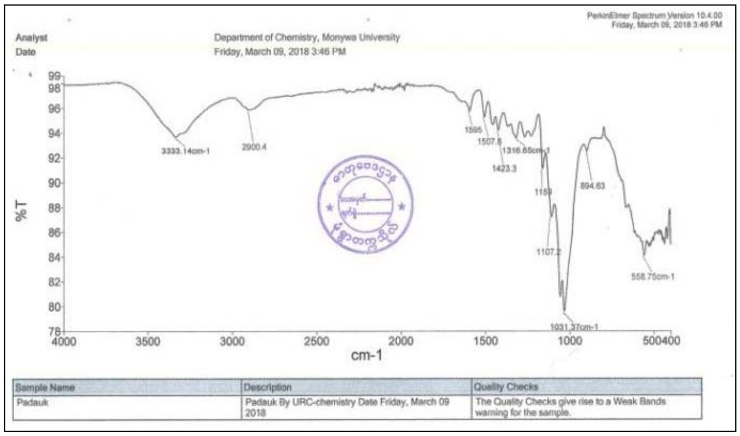


Figure (2). FT-IR spectrum of extracted cellulosic pulp for five hours refluxing time.

In the FT- IR spectrum of the cellulosic pulp extracted for **ten** hours refluxing time (Fig. 3), the band at 3338.53 cm^{-1} indicates the O-H stretching vibration of OH group. The peak at 2903.1 cm^{-1} represents the unsymmetrical C-H stretching vibrations of sp^3 hydrocarbons. The peaks at 1592.3 cm^{-1} reveals the C=C stretching vibrations of aromatic ring skeleton. The C-O-C stretching vibration of ether group is found at 1159, 1107.2, 1055.41 and 1031.90 cm^{-1}. The C-H out of plane bending vibration of cis or Z alkenic group is found at 894.63 cm^{-1}.

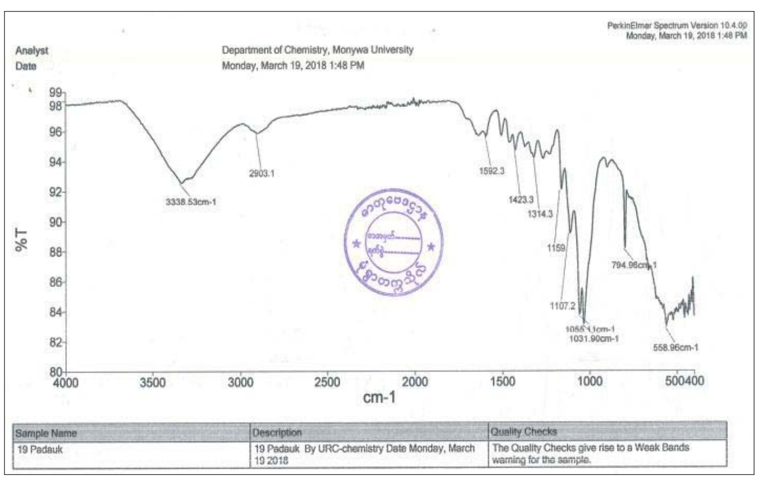


Figure (3). FT-IR spectrum of extracted cellulosic pulp for ten hours refluxing time

Therefore, the IR spectral data confirm the formation of cellulosic pulps extracted from the sawdust of Padauk. But a little amount of lignin is still present in the extracted cellulosic pulp. The high percent transmittance of the IR spectrum for 10 hours refulxing time

R Spectrum of Isolated Organic Compound (KB-1)

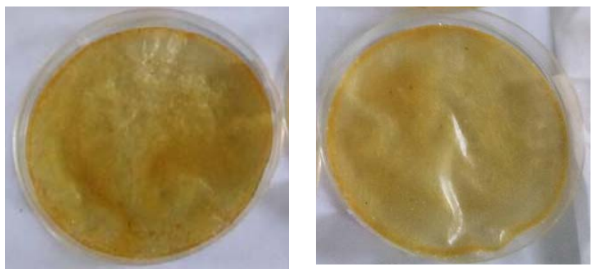
compared with that of the previous spectrum indicated that the extracted pulps for 10 hours refluxing time is more pure than the old one.

Thus the cellulose pulp extracted for 10 hours refluxing time is used to prepare the cellulosic films.

Preparation of Cellulose Film by Using Different Amounts of PVA Plasticizer

In the preparation of cellulose film, 1 g of cellulose pulp was treated with 15 mL of 3 M HNO_3 acid and a few drops of acetic acid was added to dissolve it. Then it was filtered and the filtrate was mixed with 10 mL of 3 M NaOH solution and 1 g of PVA powder. The mixture was stirred for about 2 hrs and was poured onto a glass Petridis and dried at room temperature. The film was carefully removed from the Petridis. (Condition A)

The same procedure was applied for the preparation of cellulose file with 0.5 g of PVA plasticizer for condition B. The cellulosic pulp prepared using ten hour refluxing time was used for both conditions as it was purer than that prepared using five hour refluxing hours.



Condition (A) Condition (B)

Figure (4). Cellulose films by using different amounts of PVA plasticizer

Determination of Mechanical Properties of Prepared Cellulose Films

The results obtained from the determination of the mechanical properties of the prepared cellulose films such as thickness, tensile strength, elongation at break and tear strength were described in Table (5).

Table (5) Results of Mechanical Properties of Cellulosic Film from PVA.

Conditions	**Thickness**	**Tensile strength (MPa)**	**Elongation at break (%)**	**Tear strength (kN/m)**
Condition (A)	0.20	0.8	24	0.6
Condition (B)	0.50	0.6	10	0.6

From the measurement of the mechanical properties, the prepared film in condition (B) is thicker than that in condition (A). The tensile strength and elongation at break of the film prepared in condition (A) are higher than that in condition (B). But the tear strengths of the films in both conditions are the same.

Conclusion

The sawdust Padauk collected from Mogaung Township, Kachin State was used for the determination of phytochemical constituents and elemental compositions by standard method and EDXRF spectral analysis. According to the phytochemical screening, this sawdust contains alkaloid, glycoside, reducing sugar, saponin, phenolic compound, polyphenol and tannin respectively.

EDXRF report showed that 10 mineral elements (Si, Ca, S, K, Fe, P, Ti, Cu, Zn, and Mn) were present in the Padauk sawdust. The amounts of silicon and calcium are higher than that of the other mineral elements. The cellulosic pulps were prepared by refluxing with sodium hydroxide in different refluxing periods to determine the effect of time on the yield of the cellulosic pulp. The yield percent of the cellulosic pulp from Padauk were 43.13 % for condition (1) and 21.1% for condition (2).

The purity of cellulosic pulps extracted from Padauk cellulosic pulps were by FT- IR spectroscopic method. According to IR spectral data (C=C stretching vibration of lignin at 1595 and 1592.3 cm^{-1}), both cellulosic pulps (conditions 1 and 2) still contain a little amount of lignin.

The cellulosic films were prepared from using different amounts of plasticizer (PVA) and sodium hydroxide. The mechanical properties of the prepared films were determined. The thickness, tensile strength, elongation at break and tear strength of the films prepared in conditions (A) and (B) are 0.20 mm, 0.8 MPa, 24 %, 6.0 kN/m, and 0.50 mm, 0.6 MPa, 10% and 6.0 kN/m respectively. Therefore, the film prepared in condition (A) shows better mechanical properties than the films prepared in conditions (B).

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