PREPARATION, CHARACTERIZATION AND APPLICATION OF MICRO-FIBRILLATED CELLULOSE FROM SAWDUST

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Abstract

In this research, micro-fibrillated cellulose (MFC) was prepared from sawdust of geland wood by three steps such as alkali treatment, bleaching and acid hydrolysis. The acid hydrolysis has been performed by two different H_2SO_4 concentrations (3N and 5N) whereas other conditions are remained unchanged. The physicochemical properties such as moisture content, ash content, particle density, porosity and water absorption capacity of sawdust and prepared MFC were determined by conventional methods. The bleached pulp and prepared MFC were characterized by EDXRF, FT-IR, TG-DTA, XRD and SEM spectroscopic methods. The relative abundance of elements present in MFC was obtained from the results of EDXRF. The oxidation reaction takes place during the preparation of MFC by using high concentrations of H_2SO_4 which has been detected by FTIR spectroscopy. The thermal behavior of bleached pulp and MFC were observed by TG-DTA. From the XRD results, the crystallinity of bleached pulp and prepared MFC was found to be 63.63% and 66.67% respectively. The films of polyvinyl alcohol (PVA) and PVA/MFC composites at different mass ratios of PVA:MFC (1:0.1, 1:0.2, 1:0.3, 1:0.4 and 1:0.5) were made by using the prepared MFC as reinforcement. The surface morphology of prepared films was revealed by SEM. The physical and mechanical properties such as thickness, tensile strength, elongation at break, tear strength and water vapour transmission rate of prepared films have been evaluated.

Keywords: Micro-fibrillated cellulose, sawdust, composite films, reinforcement

Introduction

The use of renewable materials for industrial applications is becoming impellent due to the increasing demand of alternatives to scarce and unrenewable petroleum supplies. Plant based fiber is sustainable in its supply and it is biodegradable and renewable. In particular, cellulose is the most abundant renewable polymer resource available today, and it is considered an almost inexhaustible source of

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raw material for the increasing demand for environmentally friendly and biocompatible products.

Substances used as raw materials should come from plants with a low economic and have no adverse effect on the environment. Among the natural plant wastes, sawdust is used as a raw material for the production of micro-fibrillated cellulose.

Sawdust refers to the tiny-sized and powdery wood waste produced by the sawing of wood. Sawdust is composed of three important constituents such as cellulose, lignin and extractives. The benefits of using sawdust as a woody biomass in compost manufacture are its favorable physical properties such as low apparent specific gravity (density), high porosity, high water retention, high bacteria tolerance and biodegradability at an acceptable rate (Nanci, et.al, 2016).

In this regard, micro-fibrillated cellulose (MFC), the most abundant biopolymer, is one of the most promising materials. Micro-fibrillated cellulose is a man-made substance which is obtained from the naturally occurring compound i.e., cellulose. On account of its small size, very high surface area and high aspect ratio, MFC is a potential reinforcing material with the advantages of being derived from renewable resources (Priya, et.al, 2015). MFC is really potential especially when it comes to replacing synthetic raw materials. Thus it is one of the most sustainable alternatives on the market (Ghani, et.al, 2014). Products which are produced from natural raw materials may be safe, hygiene and economic for customers. These are very important facts for the production of micro-fibrillated cellulose from sawdust.

Polyvinyl alcohol (PVA) has been widely used for the preparation of many blends and composites with several natural, renewable polymers and applied them in the development of green composite technology to achieve ecological sustainability. Composite films which consist of PVA with natural fibers are improved the biodegradability and physical properties of PVA. The interaction between fibers and PVA results a good composite property and satisfactory performance (Abas, 2016).

The aim of this research is to prepare and characterize the micro-fibrillated cellulose (MFC) from sawdust and to apply the prepared MFC as reinforcement for the preparation of composite films by using various mass ratios of PVA to MFC. The prepared films have been assessed according to their physicomechanical properties.

Materials and Methods

All chemicals used in this research were obtained from British Drug House (BDH), England. The experimental works were conducted at the Department of Chemistry, Myitkyina University. Sawdust sample from the geland wood used as a raw material for the preparation of MFC was collected from Hpokalar Sawmill, Tutgone Quarter, Myitkyina Township in Kachin State.

Preparation of Micro-fibrillated Cellulose from Sawdust

(a) Preparation of alkali treated fibers

The sawdust sample collected from sawmill was clarified by using several physical methods such as sieving and washing to remove large particles like as stone, concrete, bark etc.. The dry sawdust was immersed in 17% (w/v) sodium hydroxide solution at room temperature for 3 hours with occasional stirring by glass rod and the solid-liquor ratio was maintained at 1:50 (w/v). The alkali treated sawdust was washed several times thoroughly by distilled water and neutralized with very dilute acetic acid. The alkali treated sawdust fiber was dried in air and followed by an electric oven at 100 ± 5°C for 6 hours and stored in airtight plastic bag.

(b) Treatment of bleached pulp

The alkali treated fibers were bleached with 1% (w/v) sodium chlorite ($NaClO_2$) solution for 90 minutes at 85-95°C. The fiber-liquor ratio was maintained at 1:50 (w/v). In this process the pH was controlled at 4. A buffer mixture of pH 4 (acetic acid-sodium acetate) was added to the chlorite solution in the proportion of 1 mL of buffer solution for every 10mL of sodium chlorite solution. After the treatment, the resulting bleached pulp was filtered over a sintered funnel and washed thoroughly with distilled water. The pulp was then treated with 0.2% (w/v) sodium meta-bisulphite solution for 20

minutes with fiber-liquor ratio 1:20 (w/v). The pulps were filtered and washed thoroughly with distilled water and dried in an electric oven at 100 ± 5°C.

(c) Preparation of micro-fibrillated cellulose by acid hydrolysis

10g of each bleached pulps were added into the solutions of 3N and 5N sulphuric acid. The fiber-liquor ratio was maintained at 1:50 (w/v). The pulp suspensions were then placed on a magnetic stirrer and continued stirring up to 6 hours by a magnetic bar. After 6 hours acid hydrolysis, the white powder like MFC was formed and then it was filtered and washed thoroughly with distilled water. The prepared MFC's were kept in acetone and sonication was performed for 12 hours in an ultrasonic bath (Priya, et.al, 2015).

Preparation of Polyvinyl Alcohol (PVA) Film and PVA/MFC Composite Films

(a) Preparation of PVA film

Polyvinyl alcohol (PVA) was added to deionized water at a weight ratio of 1:9 to form a PVA solution. After that the solution was kept in a water bath at 90°C and stirred for 1.5 hours. The solution was cast on a glass plate and dried at 40°C to obtain a PVA film.

(b) Preparation of PVA/MFC composite films

PVA solution was added to the MFC dispersion at different mass ratios PVA : MFC (1:0.1, 1:0.2, 1:0.3, 1:0.4 and 1:0.5). These mixtures were stirred at 90°C for 1.5 hours and then cast on each of the glass plates. The corresponding PVA/MFC composite films were obtained by drying at 40°C.

Determination of the Physicochemical Properties of Sawdust and MFC
Determination of moisture content

Moisture present in sawdust and MFC were determined by oven-drying method at (100 ± 5ºC), according to AOAC., (2000). The results are shown in Table 1.

Determination of ash content

The ash contents of sawdust and MFC were determined by procedure according to AOAC., (2000). The results are shown in Table 1.

Determination of particle density

Each samples, sawdust and MFC, were placed into graduated volumetric cylinders to reach the marked 100 cm^3 volume (V_o), and its weight (gram) could then be known by subtracting the combined weight of sample and volumetric cylinder (W_b) with the weight of empty volumetric cylinder (W_a) alone. The particle density of sample could then be calculated using the formula presented in following formula. The results are shown in Table 1.

$$\text{Particle density (g cm}^{-3}) = (W_b - W_a) / V_o$$

Determination of porosity

Each of samples (sawdust and MFC) with apparent volume of 100 cm^3 and known weight (W_s in grams) was placed in a volumetric cylinder. Tap water was then poured gently into it until the surface of water reached a marked line at the 100 cm^3 level. Porosity of each sample was calculated as the formula represented in the following. The results are shown in Table 1.

$$\text{Porosity (\%)} = (V_a / V_o) \times 100\%$$

$$[V_a\ (cm^3) = W_{comb} - W_s - W_{vs}]$$

V_a = the volume of poured water (cm^3) together with the water in the sample

V_o = the volume of sample (100 cm^3)

W_{comb} = the combined weight of volumetric cylinder, sample particles and poured water (g)

W_s = the weight of sample particles (g) (oven-dry weight equivalent)

W_{vs} = the weight of volumetric cylinder (g)

Determination of water absorption test

The samples (sawdust and MFC) were first dried at 100 ± 5°C for 24 hours. 1 g of each sample was put in to a centrifuge tube and then tap water was poured into the each tube. The samples were centrifuged after soaking in water for a separated time period of 30 minutes and then decanted. After that the centrifuge tube was weighed.

The percentage of water absorption (%) for each sample was then calculated according to the following formula. The results are shown in Table 2 and Figure 4.

$$\text{Water absorption (\%)} = \frac{\text{weight of absorbed water}}{\text{weight of sample}} \text{ x } 100\%$$

Characterization of Prepared Pulp, MFC and Films

The prepared bleached pulp and MFC were characterized by modern techniques such as EDXRF, FTIR, TG-DTA, XRD and SEM.

Elemental analysis by EDXRF

The elements present in the sample, MFC, was measured by means of an EDX-8000 Shimadzu, Japan at Department of Chemistry, West Yangon University. The resultant data and EDXRF spectrum of MFC are present in Figure 5.

Fourier transform infrared spectroscopy (FTIR)

The procedure was in accordance with the catalogue instruction as reported in FTIR spectrophotometer, (IR Tracer-100, Shimadzu, Japan). The FTIR spectra of samples are shown in Figure 6 (a), (b) and (c), respectively.

Thermogravimetric and differential thermal analysis (TG-DTA)

The procedure was in accordance with the recommended standard procedure as reported in DTG-60H Thermal Analyzer (Shimadzu) instrument. The TG-DTA spectra of samples are shown in Figure 7 (a) and (b).

Degree of crystallinity determination by XRD

The procedure was in accordance with the recommended standard procedure as reported in XRD instrument. The XRD spectra of samples are shown in Figure 8 (a) and (b).

Scanning electron microscope analysis (SEM)

Prepared samples were examined by Scanning Electron Microscope (ZEISS, Germany), for a visual inspection of external

porosity and topological textures. The SEM micrographs of samples are shown in Figure 9 (a), (b), (c) and (d).

Determination of Physicomechanical Properties of PVA and PVA/MFC Composite Films

The evaluations of the physical and mechanical properties such as thickness, tensile strength, elongation at break and tear strength of PVA and PVA/MFC composite films were performed at the Department of Rubber Technology, Yangon.

(a) Determination of the tensile strength and elongation at break

The prepared films were cut off according to JISK 7127 and the dimensions of test pieces were described. Both ends of the test pieces were firmly clamped in the jaw of tensile strength machine. One jaw was fixed and other was movable. The movable jaw moved at the rate of 10 mm/min. The resultant data were shown in the recorder. This procedure was repeated three times for each test. The tensile strength and elongation at break were calculated as the following equations. The results are represented in Table 5 and Figure 10 (a) and (b).

$$T_s = \frac{F}{W \times t}$$

where, T_s = tensile strength in Mega Pascal (MPa)

F = the maximum force record in Newton (N), T = thickness of the test length (mm)

$$E_b = \frac{L - L_0}{L_0} \times 100$$

where, E_b = elongation at break in percentage

L_0 = initial length in mm, L = test length at break in mm

(b) Determination of the tear strength

The specimen to be tested was cut out by the die from the above films. Specimen was cut with a single nick (0.05 mm) at the entire of the inner concave edge by a special cutting device using a razor blade. The clamping of the specimen in the jaw of test machine

is aligned with travel direction of the grip at the rate of 100 mm/min. The recorder of the machine showed the highest force to tear from a specimen nicked. This procedure was repeated three times for each test. The tear strength was calculated as the following equations. The results are represented in Table 5 and Figure 10 (c).

$$T_s = L \times \frac{t_1}{t_2}$$

(c) Determination of water vapour transmission rate of films

The water vapour transmission rate (WVTR) was determined gravimetrically at room temperature. Beakers containing 3 g of anhydrous calcium chloride were prepared. The test films were placed inside the beakers and the assembly was weighed. The beakers were then placed in a desiccator containing saturated sodium chloride solution at room temperature. After that the assembly was weighed at a given time intervals and put in the desiccator again. Weight differences of the beakers were measured at 1 hour intervals for 1 day which were used to calculate the water transferred through the films, which were absorbed by the anhydrous calcium chloride. The results are represented in Table 6.

$$\text{Water vapour transmission rate} = \frac{\Delta W}{\Delta t \times A}$$

ΔW = weight difference at different interval time (g)

Δt = time interval (hour), A = area of film (m^2)

Figure 1: Photograph of the experiment for water vapour transmission of films

Results and Discussion

Preparation of Micro-fibrillated Cellulose (MFC)

Pure and quality product, micro-fibrillated cellulose (MFC), was prepared from sawdust of geland wood by performing three steps such as alkali treatment, followed by $NaClO_2$ bleaching and acid hydrolysis as represented in the experimental work. The following photographs are the results from each step of the preparation of MFC from sawdust of geland wood.

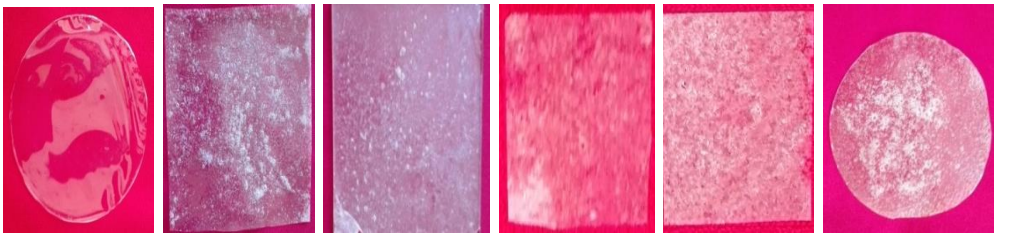
(a) (b) (c)

(d) (e) (f)

Figure 2: Photographs of (a) sawdust (b) alkali treated fiber (c) bleached pulp (d) dried pulp (e) acid hydrolysis of bleached pulp and (f) dried MFC

MFC serves as a prom ising material to various applications, such as bio-composites and packaging materials, due to its abundance, high strength, low weight and biodegradability. Polyvinyl alcohol (PVA) is an environmentally-friendly, biodegradable and renewable polymer (Siró and Plackett, 2010).

This research is focused on the use of MFC as reinforcement of coating for packaging. So, the films of polymer based composites with various mass ratios of (PVA/MFC) were made by using prepared MFC. A finding of relatively small improvement in barrier properties would make MFC suitable for various industrial applications. Figure 3(a) is represented for PVA film and Figure 3 (b), (c), (d), (e) and (f) are represented for the prepared PVA/MFC films.



(a) (b) (c) (d) (e) (f)

Figure 3: Photographs of prepared films (a) PVA (b) Composite-1 (c) Composite-2 (d) Composite-3 (e) Composite-4 and (f) Composite-5

Plastic materials are indispensable in our lives but they pose of environmental pollution. In order to reduce the environmental load generated from the disposal of used plastic materials, a growing interest has been focused on biodegradable polymers based materials. The composites materials should be able to recycle, reuse, reprocesses or biodegradable, to minimize its impact to ecosystem.

Determination of the Physicochemical Properties of Sawdust and MFC

Regarding the utilization of woody biomass into compost, it is important to pay attention to its physicochemical properties. This study was carried out to determine such appropriate physicochemical properties including moisture content, ash content, particle density, porosity and water absorption capacity for sawdust particle and MFC.The resultant data are presented in Table 1 and 2.The contents of moisture, ash and particle density decrease in MFC than that in sawdust. Porosity of samplecan be defined as a measure of voidsvolume of sample grains, composed principally of inter-spaces among and intra-spaces within the particles or the percentage of samplevolume occupied by air and water that filled voids. The

porosity (%) of MFC is more present than that of sawdust due to the consisting of large voids that occupied by water. From the experimental results, water absorption capacity of MFC is also greater than that of sawdust for each of separated times. That is why the quality material MFC produced from sawdust is preferable use for a reinforcement of polymer composite films in the manufacture of packaging materials.

Table 1: Physicochemical Properties of Sawdust and Micro-fibrillated Cellulose (MFC)

Sr. No	Test Parameter	Result	
		Sawdust	MFC
1	Moisture (%)	11.10	8.96
2	Ash (%)	15.27	5.31
3	Particle density (gmL^{-1})	0.46	0.23
4	Porosity (%)	71.24	86.52

Table 2:Water Absorption Capacities of Sawdust and Micro-fibrillated Cellulose (MFC)

Time (min)	Water absorption capacity (%)	
	Sawdust	MFC
30	12.35	16.71
60	29.57	37.43
90	40.63	58.01
120	71.86	84.12
150	72.05	84.25
180	72.42	84.58

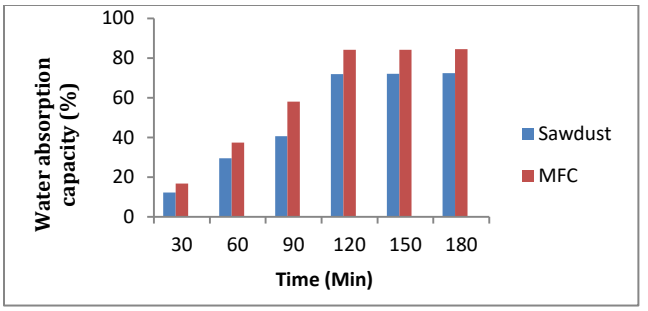


Figure 4: Water absorption capacities (%) of sawdust and micro-fibrillated cellulose

Characterization of Sawdust, Bleached Pulp and Micro-fibrillated Cellulose (MFC)

The elemental analysis, thermal property, degree of crystallinity and morphology of MFC and reference bleached pulp prepared from sawdust were characterized by energy dispersive X-ray fluorescence (EDXRF), fourier transform infrared spectroscopy (FTIR), thermogravimeteric and differential thermal analysis (TG-DTA), X-ray diffractogram (XRD) and scanning electron microscopy (SEM).

Elemental analysis by EDXRF

Figure 5 shows the EDXRF spectrum of MFC. The relative abundance of elements present in MFC is observed from the resultant data. According to the results, there is no element that causes harm for the people. Thus, MFC is suitable for a coating material in polymer composite films used for food packaging.

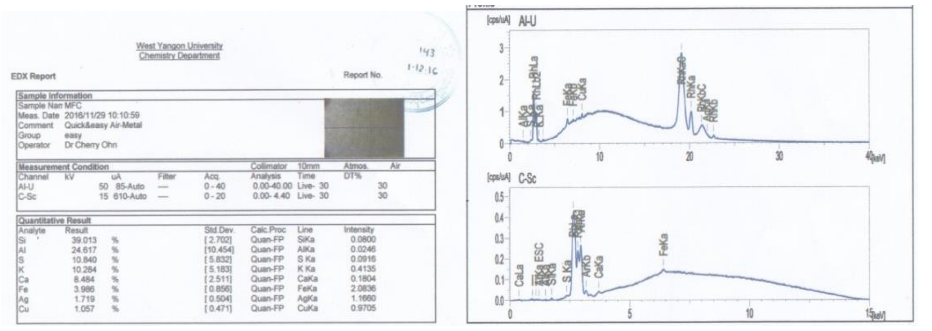


Figure 5: EDXRF spectrum of MFC

Functional Groups Determination by FT IR Spectra

T The FTIR spectra of bleached pulp and MFC's are shown in the Figure 6 (a), (b) and (c). The hydrophilic tendency of the cellulose and micro-cellulose samples is reflected in the broad absorption band in the 3700-3100 cm^{-1} region, which is related to the -OH groups present in their main components. In the 1600- 900 cm^{-1} region, it is possible to appreciate in fibers vibrations of chemical components of the lignin, at frequencies of between 1597 cm^{-1} and 1508 cm^{-1} for O-H bending of guaiacyl and 1427 cm^{-1}, 1369 cm^{-1} and 1317 cm^{-1} associated with syringyl. These absorptions are consistent with those of the typical cellulose backbone (Silverstein, 1963). Furthermore, almost the same absorption peaks as shown in the cellulose fibers are observed in the spectrum of the MFC. This indicates that the structure of cellulose has not been damaged after the treatments.

On the other hand, the peak centered at 1643 cm^{-1} in the FTIR spectrum of cellulose and MFC may be due to the C=O bond of hemicellulose. The intensity of the peak decreases from cellulose to MFC as the hemicellulose is removed gradually by acid hydrolysis.

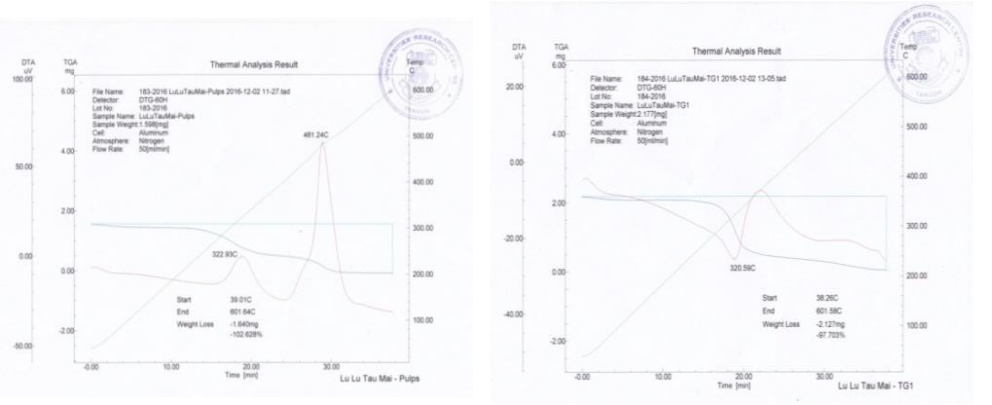
(a) (b) (c)

Figure 6: FTIR spectra of (a) bleached pulp (b) MFC (hydrolysis by 3 N H_2SO_4) and(c) MFC (hydrolysis by 5 N H_2SO_4)

Thermogravimetric and differential thermal analysis (TG-DTA)

The thermal behavior of bleached pulp and MFC are presented in Figure 7 (a) and 7 (b). The summary of weight loss in different temperature ranges is also depicted by Table 3. For convincing

explanation, three different temperature ranges (39-280°C, 281-440°C and 441-601°C) were considered on the basis of the degradation of the constituent of fibers. The first weight loss at the range 39-280°C is found due to removal of moisture and hemicellulose. The further degradation occurs at the range of 281-440°C for removal of cellulose and then final weight loss happens for degradation of residual cellulose and lignin together at the range of 441-601°C. According to the results, it is observed that the amount of moisture content in bleached pulp is more present than that in MFC.



(a) (b)

Figure 7: TG-DTA thermograms of (a) bleached pulp and (b) MFC

Table 3: Weight Loss of Bleached Pulp and Micro-fibrillated Cellulose (MFC) at Various Temperature Ranges

Samples	Weight loss (%) at 39-280 ºC	Weight loss (%) at 281-440 ºC	Weight loss (%) at 441-601 ºC
Bleached pulp	24.90	74.97	87.48
MFC	8.13	67.03	76.22

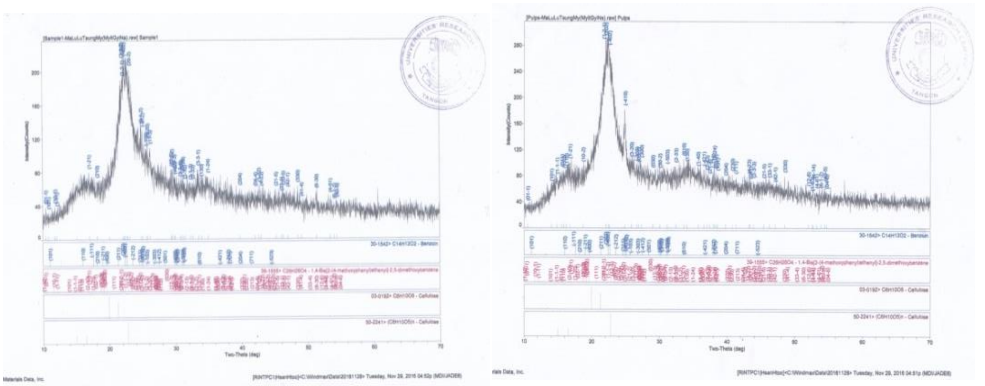
Degree of crystallinity determination by XRD

The degree of crystallinity in the cellulosic materials may be measured in several ways by an X-ray diffractogram. This review compiles peak height method to evaluate the degree of crystallinity reported in the literature (Priya, et al., 2015). In this approach, the X-ray apparent crystallinity (%) of cellulose is calculated from the height ratio between the intensity of the crystalline peak and the total intensity after the subtraction of the background signal (non-crystalline) measured without cellulose according to the following equation:

$$C\ (\%) = \frac{I_{crystalline} - I_{noncrystalline}}{I_{crystalline}} \times 100$$

where C expresses the apparent crystallinity (%). $I_{crystalline}$ and $I_{noncrystalline}$ represent the intensities of diffraction of the crystalline and non-crystalline materials.

According to Figure 8 (a) and 8 (b), it is observed that all the cellulose peaks consist near 2θ =22.4 degree. Comparison between the crystallinity of bleached pulp and MFC are found to be 63.63% and 66.67% respectively. The increase in diffraction intensity indicates that the acid hydrolysis induces the crystallinity (%) due to the removal of amorphous materials like hemicellulose, lignin, and some other non-cellulosic materials, which is revealed by the resultant data in Table 4.



(a) (b)

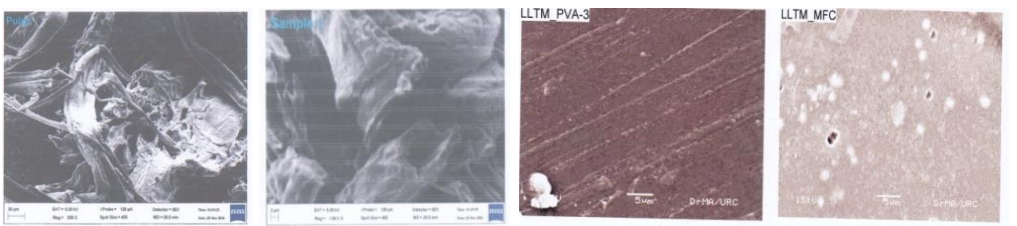
Figure 8:X- ray diffractograms of (a) bleached pulp and (b) MFC

Table 4: Crystallinity (%) of Bleached Pulp and Micro-fibrillated Cellulose (MFC)

Samples	Intensity of crystalline peak (2□=22.4°)	Intensity of non-crystalline peak (2□ =16.2°)	Crystallinity (%)
Bleached pulp	220	80	63.63
MFC	180	60	66.67

Scanning electron microscope analysis (SEM)

Figure 9 (a), (b), (c) and (d) show the images of SEM for the cross-sectional surface of bleached pulp, MFC, PVA film and PVA/MFC composite-4 film. From the Figure 9 (a) and 9 (b), it is found that microporous materials consist of a regular organic or inorganic framework supporting a porous structure. The SEM image of Figure 9 (c) shows the homogeneous smooth surface texture of PVA. The SEM image of Figure 9 (d) shows that MFC is uniformly distributed in PVA and the rough surface in PVA/MFC film which is composed of aggregate particles.



(a) (b) (c) (d)

Figure 9: SEM images of (a) bleached pulp (b) MFC (c) PVA film and (d) PVA/MFC composite film

Physicomechanical Properties of PVA and Composite Films

The physical and mechanical properties in terms of thickness, tensile strength, elongation at break and tear strength are important parameters for the evaluation of the prepared composite films. The results of the physicomechanical properties of PVA and PVA/MFC composite films are represented in Table 5 and Figure 10 (a), (b) and (c).

The tensile properties of natural fiber reinforce polymers are mainly influenced by the interfacial adhesion between the matrix and the fibers. Tensile properties are frequently included in material specifications to ensure quality. Tensile properties are often measured during development of new materials and processes, so that different materials and processes can be compared...

Table 5: Physical and Mechanical Properties of PVA and Composite Films

No	Test	PVA	Composite-1	Composite-2	Composite-3	Composite-4	Composite-5
1	Thickness (mm)	0.15	0.16	0.18	0.20	0.30	0.35
2	Tensile Strength (MPa)	6.7	9.9	10.8	11.2	11.4	11.3
3	Elongation at break (%)	98	68	59	41	39	36
4	Tear Strength (kN/m)	66.5	78.3	80.5	87.2	87.7	86.4

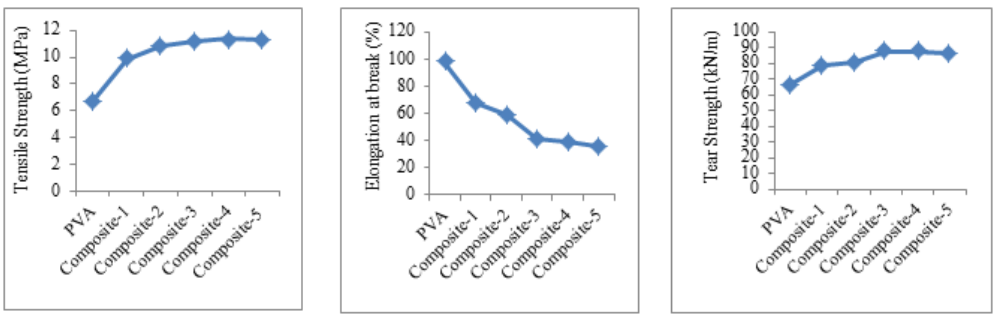


Figure 10: Physical and mechanical properties of PVA and composite films

The addition of MFC increases the tensile strength and tear strength of the composite films. This indicates a reinforcement of the relatively compliant PVA matrix by the stiffer and stronger cellulose microfibrils. For the present samples, when the addition of MFC is greater than mass ratio (1: 0.4) PVA/MFC, no further increases in the tensile strength and tear strength of the composites are observed. This may be due to the formation of fibril aggregates, which reduces the effective aspect ratio.

According to the resultant data, it is observed that the composite-3 mass ratio (1: 0.3) of PVA/MFC (thickness = 0.2 mm, tensile strength = 11.2 MPa, elongation at break = 41% and tear strength = 87.2 kN/m) and composite-4, mass ratio (1: 0.4) of PVA/MFC (thickness = 0.3 mm, tensile strength = 11.4 MPa, elongation at break = 39% and tear strength = 87.7 kN/m) are more suitable films for packaging compare to those of composite-1, composite-2 and composite-5.

Determination of the water vapour transmission rate of films

The barrier performance of a film is measured by the water vapor transmission rate (WVTR). WVTR measures the rate at which water vapor permeates through the film at a specified temperature and relative humidity. The barrier properties of MFC films are determined largely by the crystallinity and the network structure formed by fibers in a dry film. It is difficult for other molecules to penetrate the crystalline parts or the very dense network (Kumar et al., 2014). The results from determination of the water vapour transmission rate of films are represented in Table 6. According to the experimental results, it is found that the composite films can prevent the transmission of water vapour. The greater the mass content of MFC present in the film, the less transmission rate of water vapour. So, the PVA/MFC composite films are more suitable for packaging materials than the film of neat PVA.

Table 6:Determination of the Water Vapour Transmission Rate of Films

No	Type of film	WVTR(g/h m^2)
1	PVA	3.26
2	Composite-1	2.45
3	Composite-2	2.31
4	Composite-3	1.97
5	Composite-4	1.78
6	Composite-5	1.71

Conclusion

In this research, highly purified MFC was prepared from clarified sawdust by hydrolysis with different concentrations of H_2SO_4 solutions. The composite films with various mass ratios of PVA/MFC were made by using the MFC as reinforcement.

The prepared MFC were confirmed by modern techniques such as XRD, SEM, EDXRF, FT-IR and TG-DTA. The relative abundant of elements present in MFC were obtained from the EDXRF spectrum. According to the resultant data, there is no element that causes harm for people in an unacceptable limit. According to the FTIR analysis, it was observed that fibers contain the typical vibration bands of the component mainly corresponding to cellulose, hemicellulose and lignin. From the results of XRD, the crystallinity of prepared bleached pulp and MFC can be estimated to be 63.63% and 66.67% respectively. According to SEM spectrum, it is found that micro-porous materials consist of a regular organic or inorganic framework supporting with porous texture. As the TGDTA spectra, the weight losses of the bleached pulp and MFC were determined for three different temperature ranges. The first weight loss at the range 39-280 ºC is found due to removal of moisture and hemi-cellulose. The weight loss at the range of 281-440 ºC happens for degradation of cellulose and that of above 440 ºC is due to the degradation of residual cellulose and lignin together.

The evaluation of the composite films were made by the determination of their physical and mechanical properties such as thickness, tensile strength, elongation at break, tear strength and water vapour transmission rate.

According to the physical and mechanical properties of prepared films, the composite-3 (1:0.3) and composite-4 (1:0.4) of PVA/MFC films have the suitable results for coating of packaging materials in tensile strength (11.2 MPa and 11.4 MPa) and tear strength (87.5 kN/m and 87.7 kN/m) respectively.

Water vapour transmission rate of prepared films were determined to evaluate their barrier properties. As the experimental results, it is observed that the PVA/MFC composite films are more suitable for packaging materials than neat PVA film.

From the assessments of the prepared films, it is observed that the composite-3 and composite-4 PVA/MFC films may be substituted in packaging of plastic polymers. The enhancement in mechanical properties of composites by addition of MFC represents a strong opportunity for industrial sector.

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