PREPARATION AND CHARACTERIZATION OF ECO-FRIENDLY BIOPLASTICS FROM SAWDUST BIOMASS WASTE

Hnin Yu Wai[1], Yamin Ko[2]

Abstract

The present work is concerned with the preparation and characterization of sawdust-derived cellulose based bioplastics. Sawdust was collected from Family Saw Mill, North Okkalapa Township, Yangon Region, Myanmar. The physicochemical properties (such as moisture content, ash content, bulk density, and pH) of sawdust was determined by conventional methods and also characterized by modern techniques such as FT IR, SEM and TG DTA analyses. The cellulose in sawdust powder was prepared by using alkali treatment, bleaching process and hydrolysis method. The yield percent of the cellulose from sawdust powder was 36.6 %. Prepared cellulose was characterized by FT IR, SEM and XRD analyses. Bioplastics were prepared by mixing with various proportions (1, 2, 3, 4, 5, 6 g) of cellulose using 50 mL of water, 2 mL of acetic acid and 0.5 mL of sorbitol as plasticizer. The most favourable conditions for preparing bioplastic namely (SCB 3) was found to be 3 g of cellulose with 0.5 mL of sorbitol and 2 mL of acetic acid, was the most suitable for preparing bioplastic. It was found that the bioplastic (SCB 3) possesses tensile strength (9.40 MPa), elongation at break (45.00 %) and tear strength (56.70 kNm^{-1}). The selected bioplastic SCB 3 was characterized by FT IR, SEM and TG DTA analyses. All prepared bioplastics showed a plain, clear, smooth surface, were flexible, and pale yellow in colour. The prepared bioplastics can be used in packaging.

Keywords: cellulose, bioplastic, physicochemical properties, sorbitol

Introduction

Cellulose - based plastic has much potential in packaging applications, in transparent plastics or coatings, gel formulations, and as reinforcement in foams and composites (Henriksson *et al*., 2008). Widespread applications are currently restricted by the high cost due mainly to the difficulties in extraction of cellulose without chain cleavage during enzymic or chemical treatment. When dried, difficult to disperse lumps due to strong inter-molecular forces and entanglement of cellulose can give rise to problems in its dispersion into bioplastics (Chiellini *et al.,* 2002).Cellulose would be extracted from wood using some of the chemical and mechanical methods and they could be extracted in nano and micro forms by alkalization, bleaching and acid hydrolysis process (Piyaporn, 2015).One of such potential waste materials is sawdust which is relatively abundant and inexpensive. Sawdust is an industrial waste obtained as by-products from cutting, sawing or grinding of timber in the form of fine particle. Although sawdust consists largely of cellulose, it also contains soluble sugar, acids, resins, oils and waxes and other organic substances (Abdul Awal *et al.,* 2016). Sawdust is basically a waste of small particles available in saw-milling industries, pulp plant and paper industries as well as wood processing industries particularly, in the most of the country in a quite large volume in forms of heaps and mostly burnt off resulting in the environmental pollution (Rominiyi *et al.,* 2017). Bioplastic is form of plastic made from renewable biomass, instead of the conventional plastic derived from petroleum. It includes low accumulation of bulky plastic materials in the environment, increased soil fertility and reduced the cost of waste management. Bioplastic packaging options include bags for compost, agricultural foils, horticultural products, consumer goods, household appliances, stationery, cosmetic packaging, toys and textiles (Pramanik, *et al.,* 2015).

[1] Dr, Associate Professor, Department of Chemistry, Maubin University
[2] MSc Student, Department of Chemistry, University of Yangon

The aim of this research is to prepare and characterize the cellulose from sawdust and to prepare the bioplastic from cellulose.

Materials and Methods

The chemicals used in the experimental work were from British Drug House Chemical Ltd., England. In all the investigations, the recommended and standard procedures of both conventional and modern techniques were employed. The experiments were conducted at Physical Chemistry Research Laboratory, Department of Chemistry, University of Yangon.

Collection of Samples

In the experiment, sawdust was collected from Family Saw Mill, North Okkalapa Township, Yangon Region, Myanmar.

Preparation of Sawdust Powder

Sawdust was washed with distilled water 3 times, and cleaned samples were dried in the solar for 4 days. Then dried again at 80 °C for 2 h. They were ground by an electric grinder and sieved with 80 mesh sieve to get the fine sawdust powder.

Physicochemical Properties of Sawdust Powder

The physicochemical properties (moisture content, ash content, bulk density, and pH) of prepared sawdust powder were determined by conventional methods.

Characterization of Sawdust Powder

The sawdust powder was characterized by FT IR, SEM and TG-DTA analyses.

FT IR analysis was performed in order to characterize the functional groups of samples. A Perkin-Elmer Spectrum GX, USA was used for FT IR analysis.

The scanning electron micrograph of sawdust powder was performed by a Scanning Electron Microscope (JSM-5160, JEOL Ltd., Japan).

Thermal analysis of sawdust powder was determined by a DTA-60H (Hi-TGA 2950) thermal analyzer.

Preparation of Cellulose

Sawdust powder was washed with distilled water 3 times, and cleaned samples were dried in the solar for 4 days. Then dried again at 80 °C for 2 h. They were ground by an electric grinder and sieved with 80 mesh sieve to get the fine sawdust powder. And then cellulose was prepared by the chemical process: alkali treatment, bleaching process followed by acid hydrolysis.

Alkali treatment

Sodium hydroxide 16 g were dissolved in distilled water and the volume was made up to 100 mL with distilled water. The alkali treatment purified the cellulose by removing hemicellulose from sawdust. The sawdust powder was put in a round-bottomed flask with 16 % (w/v) sodium hydroxide solution and refluxed at 70 °C for 6 h, and then washed several times with distilled water until pH 7. Then they were filtered and dried at room temperature.

Bleaching process

The bleaching process was performed to purify the cellulose by removing lignin from sawdust. The alkali treated sawdust powder was put in a beaker with 30 % (v/v) sodium hypochlorite solution and bleached at 70 °C for 6 h, and then washed several times with distilled water until pH 7. Then they were filtered and dried at room temperature.

Acid hydrolysis

The acid hydrolysis treatment was performed on treated sample with 10 % (v/v) sulphuric acid solution at 40 °C for 30 min. The treated pulp was centrifuged at 6500 rpm for 30 min to remove acidic solution. The colloidal suspension was washed several times with distilled water until pH 7 and sonicated for 15 min. To obtain cellulose fiber, they were filtered and dried at room temperature for 3 days. The yield percent of the cellulose from sawdust powder was 36.6 %.

Characterization of the Prepared Cellulose

The prepared cellulose was also characterized by FT IR, SEM and XRD analyses.

Preparation of Bioplastics

In this research, all of bioplastics were prepared by blending casting method.

Each of 1, 2, 3, 4, 5 and 6 g of cellulose was mixed with 50 mL of water, 2 mL of acetic acid and 0.5 mL of sorbitol as plasticizer. The solution was stirred on the magnetic stirrer at 70 °C for 30 min. The solution was casted onto cleaned and dried melamine plate at room temperature. And then, allowed to air dry for 3 days.

Determination of the Physicomechanical Properties of the Prepared Bioplastics (SCB)

The physicomechanical properties (thickness, tensile strength, elongation at break, and tear strength) of the prepared bioplastics were determined by the conventional method and modern techniques.

Water uptake and degree of swelling of the prepared bioplastics were also determined.

Characterization of the Selected Bioplastic (SCB 3)

The selected bioplastic was characterized by FT IR, SEM and TG-DTA analyses.

Determination of Biodegradation by Soil Burial Test

Biodegradation of the prepared bioplastic was studied by soil burial test to examine the morphology changes.

Results and Discussion

Cellulose is a common natural polymer, so that it occupies an important position in the advancement of human civilization today. Cellulose can be used as the main material in the modern pharmaceutical industries, cosmetics, material industries, and other sectors. Table 1 shows that the physicochemical properties of sawdust determined by the conventional method.

Table 1 Physicochemical Properties of Sawdust

No.	Physicochemical Properties	Quantity
1	Moisture content (%)	8.25
2	Ash content (%)	0.96
3	Solid content (%)	91.75
4	Bulk density (g mL^{-1})	0.42
5	pH	6.32

Characteristics of Sawdust Powder

FT IR spectrum of raw sawdust powder shows that it contains cellulose which was proved by the presence of typical cellulose groups, -OH with absorption band at 3329 cm^{-1} (Figure 1 and Table 2).

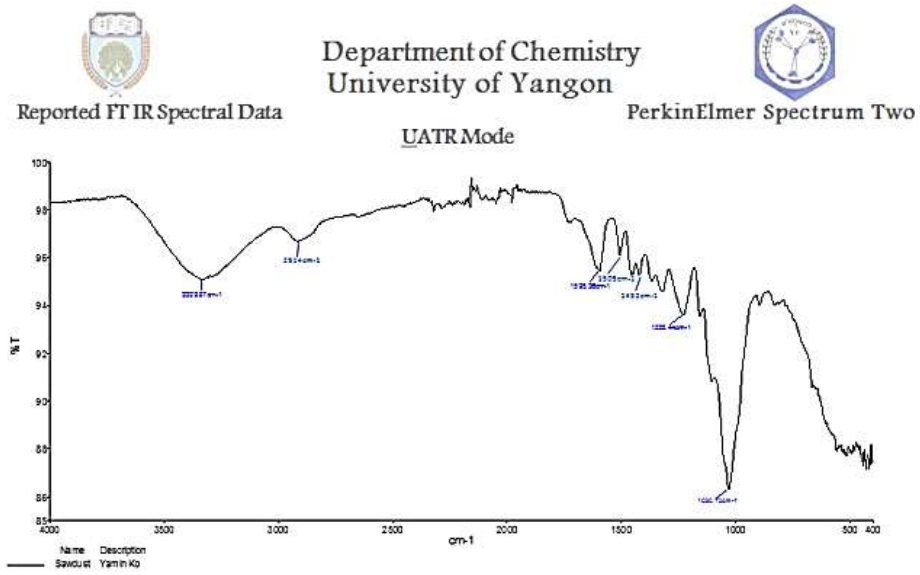

Figure 1 FT IR spectrum of sawdust powder

Table 2 FT IR Band Assignment of Sawdust Powder

Observed wavenumber (cm^{-1})	Literature wavenumber (cm^{-1}) *	Band assignment
3329	3100-3700	O-H stretching
2914	2850-2990	C-H stretching
1595, 1505, 1452	1450-1600	C=C stretching
1228, 1030	1020-1285	C-O stretching

* (Patcharaporn *et al.*, 2018)

The surface morphology of the sample was characterized by SEM analysis. From SEM image of sawdust powder, it is clearly observed that the sawdust material exhibits a dense fibrous structure (Figure 2).

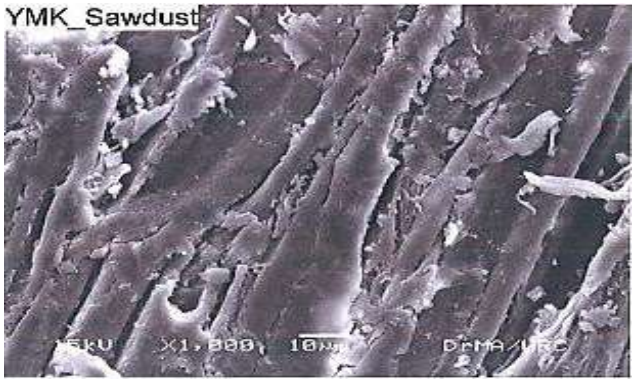

Figure 2 SEM photomicrograph of sawdust powder

Thermal stability of sawdust powder (Figure 3) and the TG-DTA Data (Table 3) are shown. The data show three distinct weight losses corresponded to the dehydration of water, the decomposition of organic residue and the carbonization of starting molecule.

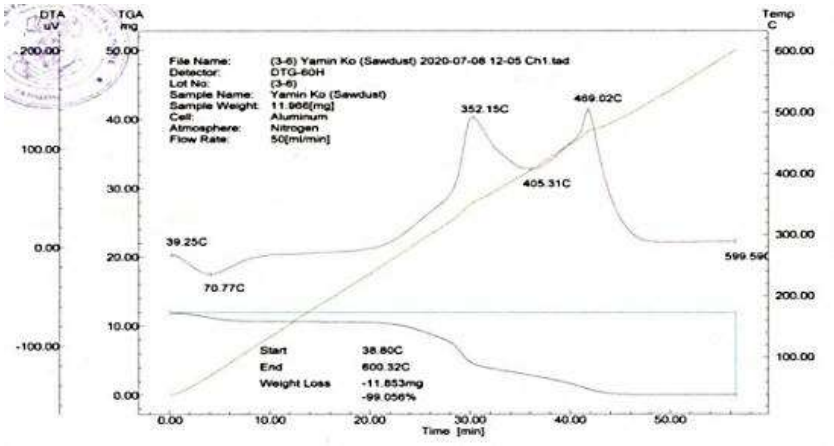

Figure 3 TG-DTA thermogram of sawdust powder

Table 3 TG-DTA Data of Sawdust Powder (SD)

Sample	TG		DTA		Remarks
	Temperature Range (°C)	Weight Loss (%)	Peak Temperature (°C)	Nature of Peak	
SD	39.25-100	13.67	70.77	endothermic	Dehydration of water
	100-360	43.55	352.15	exothermic	Decomposition of organic residue
	360-500	41.78	469.02	exothermic	Carbonization
		99.00			

Characteristics of the Prepared Cellulose

Cellulose is a very abundant polymer in nature. It can be extracted from various sources such as from plants, animals, algae, fungi, bacteria and minerals. Cellulose in nature is never found in pure form, but is always bound to other polysaccharides such as lignin, pectin, hemicellulose, wax, ash and xylan. In the present work, cellulose isolation from sawdust was conducted with chemical method using strong acid (H_2SO_4) for hydrolysis process. The yield percent of the cellulose from sawdust powder is 36.6 %.

FT IR analysis was performed in order to characterize the functional group of samples. The FT IR spectrum of cellulose (Figure 4) and the spectral data (Table 4) are given. The structural changes of each prepared (a) alkali treated sawdust powder, (b) bleached sawdust powder and (c) cellulose powder are shown in Figure 4 and band assignments are presented in Table 4. The broad peaks at 3300 cm^{-1} in all samples were due to the O-H stretching. The peak at 2894 cm^{-1} was due to stretching of aliphatic C-H on hemicelluloses and cellulose. Appearance of the band 1618-1650 cm^{-1} was owing to relative pure ring stretching mode similar to the aromatic ring C-O stretching in benzene as well as in pyrone ring. The bands at 1372 cm^{-1} and 1319 cm^{-1} were due to symmetric CH_2 bending and wagging. The weak band at 1031 cm^{-1} could be assigned to C-H bending vibration of polysaccharides. The band at 1024 cm^{-1} was due to the removal of lignin and hemicelluloses. The peak at 995 cm^{-1} also indicated stretching involving C-O-C and C-OH at C-5 and C-6 of cellulose.

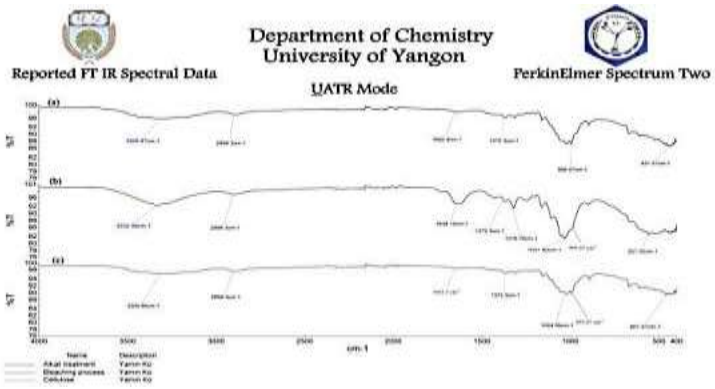

Figure 4 FT IR spectra of (a) alkali treated sawdust powder, (b) bleached sawdust powder and (c) cellulose powder

Table 4 FT IR Band Assignments of Alkali Treated Sample, Bleached Sample and Cellulose Powder

Observed wavenumber (cm^{-1})			Literature wavenumber (cm^{-1}) *	Band assignment
Alkali treated sample	Bleached sample	Cellulose powder		
3305	3332	3309	3300-3400	O-H stretching
2894	2894	2894	2820-2970	C-H stretching
1642	1638	1642	1618-1650	C-O stretching
1372	1372	1372, 1319	1300-1430	CH_2 bending
-	1031	1024	1000-1320	C-H bending
995	995	995	990-1050	C-O-C and C-OH ring stretching

* (Piyaporn, 2015)

The morphology of prepared cellulose studied by SEM analysis shows a microfibril structure (Figure 5).

Figure 5 SEM photomicrograph of cellulose

The XRD profile of the sawdust-derived cellulose shows that the characteristic peaks at 2θ values around 13.38°, 15.37° and 19.25° indicating typical semi-crystalline cellulose (Figure 6). This indicated that the sawdust powder completely transformed into cellulose through alkali treatment, bleaching and acid hydrolysis process.

The simplest and most widely used method for estimating the average crystalline size is from the full width at half maximum (FWHM) of a diffraction peak using the Scherrer equation,

$$d = K\lambda / \beta \cos\theta$$

where, d is the crystallite size, λ is the diffraction wavelength, β is the corrected FWHM,

θ is the diffraction angle and K is a constant which is close to unity.

The crystallite size of prepared cellulose calculated by this method is shown in Table 5.

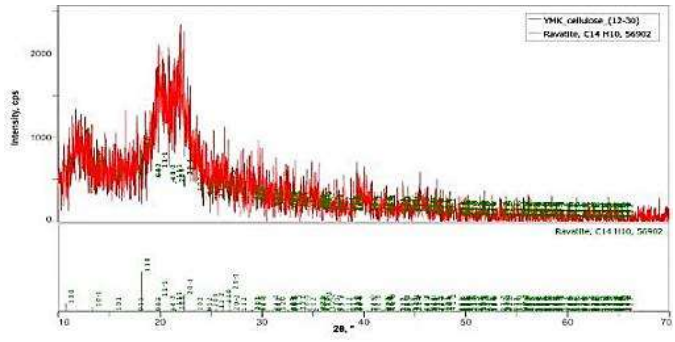

Figure 6 X-ray diffractogram of cellulose powder

Table 5 XRD Data of Cellulose Powder

No.	2θ (°)	Miller indices (h k l)	β(FWHM)	Crystallite size (nm)
1.	13.38	(1 0 $\bar{1}$)	0.4074	20.51
2.	15.37	(1 0 1)	0.4074	20.56
3.	19.25	(0 0 2)	0.4074	20.67
Average Crystallite Size (nm)				20.58

Preparation and Physicomechanical Properties of SCB Bioplastics

For all of the prepared bioplastics, physicomechanical parameters were determined. Among these parameters, tensile strength is more specific than other for determining bioplastics quality. The six types of bioplastics were prepared. The results of the physicochemical properties of prepared bioplastics are presented in Table 6 and Figures 7, 8 and 9. The effect of cellulose content on tensile strength and elongation at break of the cellulose blended bioplastics were studied. The tensile strength and elongation at break (% of cellulose-based bioplastic increased along with an increase in cellulose content. The highest tensile strength was found in bioplastic SCB 3 which contains 3 g of cellulose having tensile strength of 9.4 MPa. When the cellulose content exceeded 3 g, the tensile strength of the blend bioplastic decreased along with an increase in cellulose content, but it was still much higher than that of the unreinforced bioplastic.

Table 6 Physicomechanical Properties of SCB Bioplastics

Properties	Bioplastics					
	SCB-1	**SCB-2**	**SCB-3**	**SCB-4**	**SCB-5**	**SCB-6**
Thickness (mm)	0.15	0.16	0.18	0.19	0.10	0.10
Tensile Strength (MPa)	3.90	6.30	9.40	9.23	8.97	7.80
Elongation at Break (%)	27.00	36.00	45.00	43.20	40.70	39.60
Tear Strength (kN / m)	29.50	44.40	56.70	53.86	50.95	49.42

SCB 1 = (1 g) Cellulose + (50 mL) H_2O + (0.5 mL) sorbitol + (2 mL) acetic acid
SCB 2 = (2 g) Cellulose + (50 mL) H_2O + (0.5 mL) sorbitol + (2 mL) acetic acid
SCB 3 = (3 g) Cellulose + (50 mL) H_2O + (0.5 mL) sorbitol + (2 mL) acetic acid
SCB 4 = (4 g) Cellulose + (50 mL) H_2O + (0.5 mL) sorbitol + (2 mL) acetic acid
SCB 5 = (5 g) Cellulose + (50 mL) H_2O + (0.5 mL) sorbitol + (2 mL) acetic acid
SCB 6 = (6 g) Cellulose + (50 mL) H_2O + (0.5 mL) sorbitol + (2 mL) acetic acid

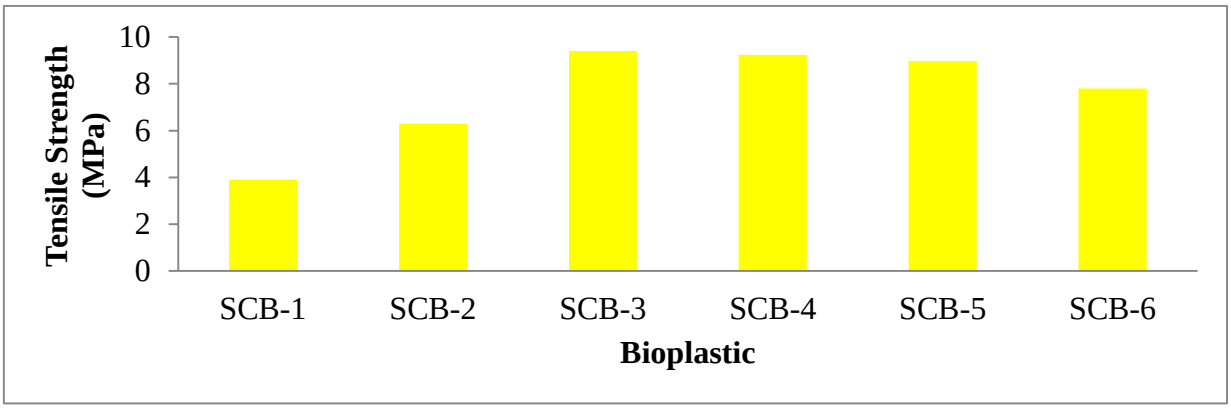

Figure 7 Tensile strength of different types of bioplastic

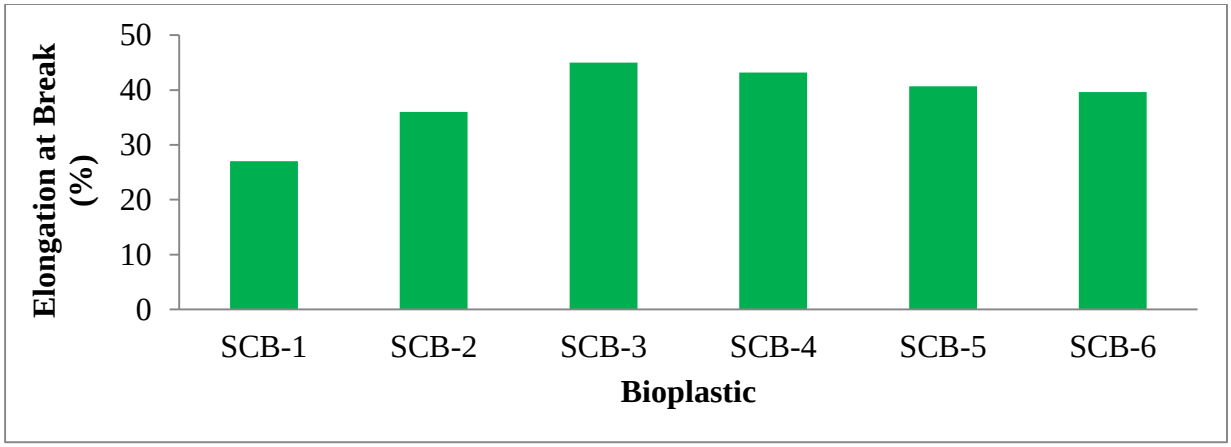

Figure 8 Elongation at break of different types of bioplastic

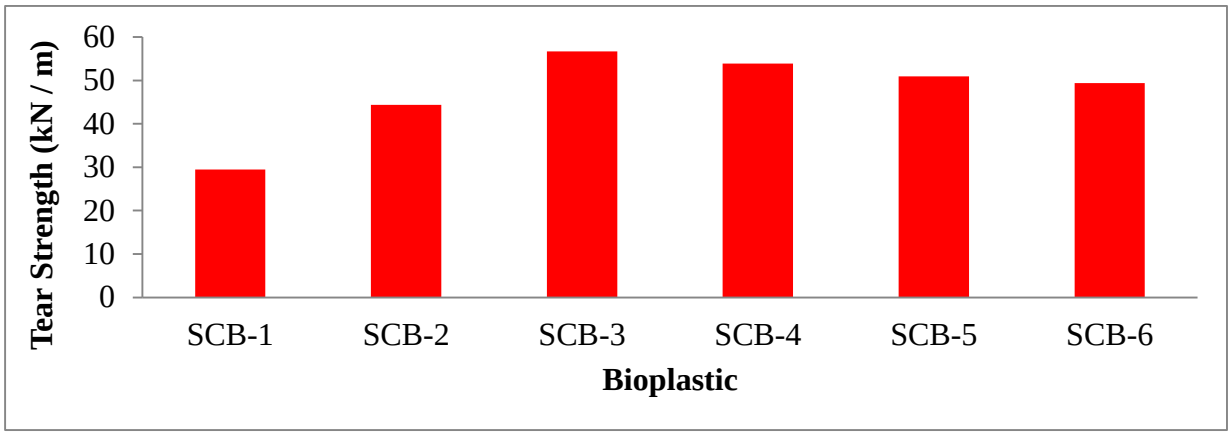

Figure 9 Tear strength of different types of bioplastic

Water Uptake Properties of SCB Bioplastics

The water uptake was investigated with increasing immersion time. The water uptake is one of the most significant parameters when a bioplastic is intended to be used as making utensils. The water uptake was the amount of water entrapped in the matrix including bound water. The water absorption properties of SCB bioplastics were studied for varying time intervals such as 5 min, 10 min, 15 min, 20 min, 25 min and 30 min. The water uptakes as a function of time for SCB bioplastics are shown in Table 7 and Figure 10. The bioplastic SCB 3 has the equilibrium water uptake percentage among them. Therefore, bioplastic SCB 3 was chosen to make the most suitable bioplastic.

Table 7 Water Uptake of SCB Bioplastics

Bioplastics / Time (min)	Water Uptake (%)					
	5	**10**	**15**	**20**	**25**	**30**
SCB-1	55.76	61.53	67.31	71.15	76.92	82.69
SCB-2	61.12	68.51	74.07	79.62	83.34	92.37
SCB-3	80.15	86.37	93.31	99.82	105.87	112.29
SCB-4	98.16	100.80	110.38	114.67	119.43	125.63
SCB-5	116.36	119.20	126.34	135.50	140.34	149.15
SCB-6	121.9	128.37	131.50	137.87	146.28	152.48

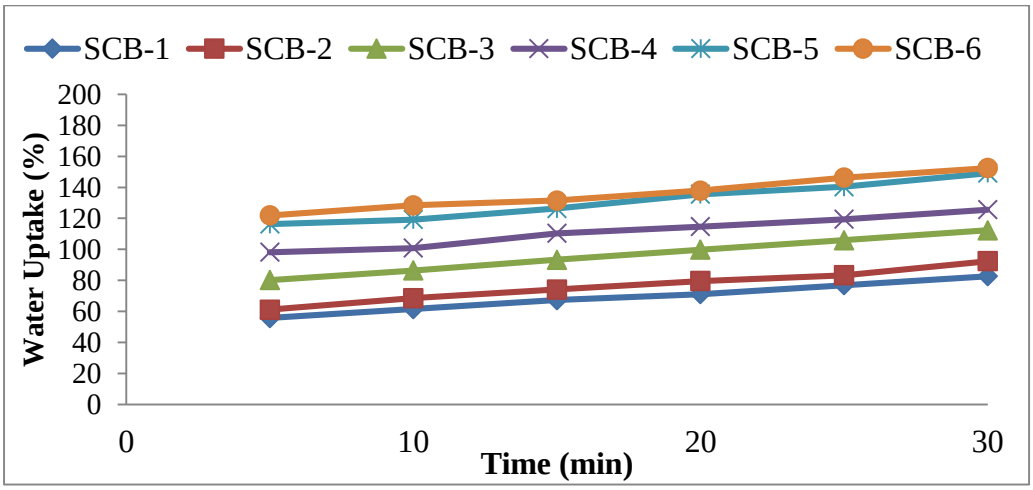

Figure 10 Water uptake of various types of bioplastic at different contact times

Degree of Swelling Properties of SCB Bioplastics

The degree of swelling of SCB bioplastic with different compositions as a function of immersion time in distilled water at room temperature is shown in Table 8 and Figure 11. For a given blend composition time, mostly the degree of swelling increased with increasing immersion time. The degree of swelling from 5 min to 30 min was slightly different for all prepared bioplastics.

Table 8 Degree of Swelling of Different Types of Bioplastic

Bioplastics / Time (min)	Degree of Swelling (%)					
	5	**10**	**15**	**20**	**25**	**30**
SCB-1	35.80	38.09	40.23	41.57	43.47	45.26
SCB-2	37.93	40.65	42.36	44.32	45.98	48.85
SCB-3	43.48	47.56	51.85	56.84	62.86	68.23
SCB-4	48.50	53.85	57.59	63.73	68.73	73.98
SCB-5	54.96	59.48	65.94	69.25	74.58	78.42
SCB-6	59.72	64.73	72.35	75.93	80.79	84.74

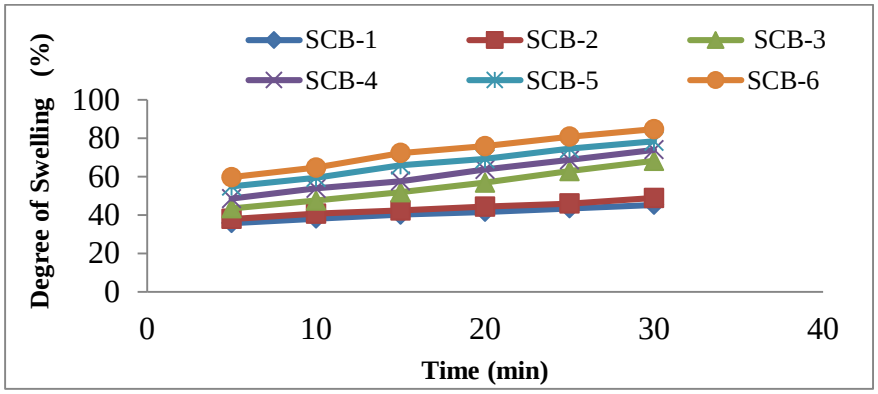

Figure 11 Degree of swelling of SCB bioplastics as a function of contact time

Characteristics of the Selected Bioplastic (SCB 3)

FT IR spectrum for SCB 3 bioplastic recorded in the range of 400-4000 cm^{-1} is shown in (Figure 12 and table 9). In the FT IR spectrum of SCB 3 bioplastic, the differences seem to be in the region 3200-3600 cm^{-1}, 1650 cm^{-1} and 1350 cm^{-1}. But there is an enhancement of OH group wavenumber from 3309 cm^{-1} at cellulose become 3275 cm^{-1} at SCB 3 bioplastic. The decrease in OH group value is due to cellulose their combined in bioplastic.

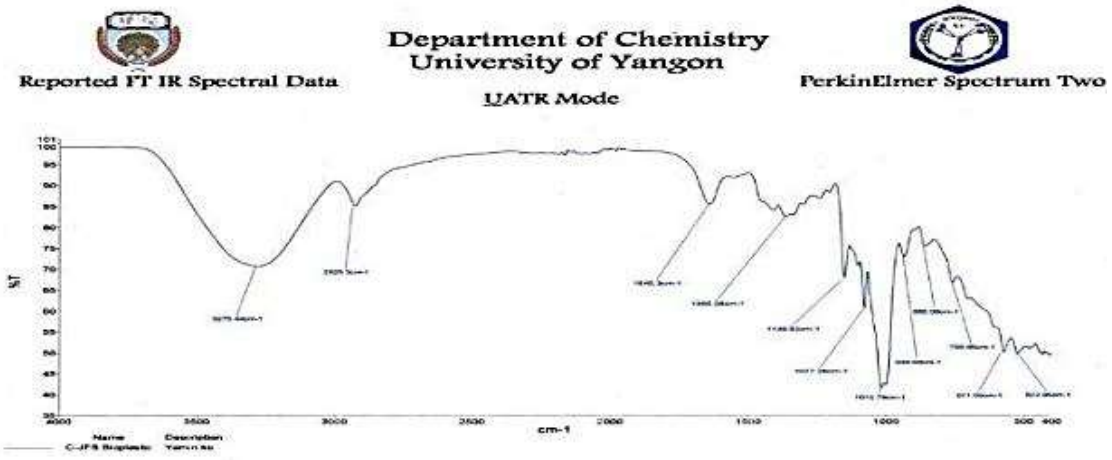

Figure 12 FT IR spectrum of SCB 3 bioplastic

Table 9 FT IR Band Assignments of SCB 3 bioplastic

Observed wavenumber (cm^{-1})	Literature wavenumber (cm^{-1}) *	Band assignment
3275	3200-3600	O-H stretching
2928	2850-2940	C-H stretching
1640	1600-1650	C-O bending
1365, 1149	1350-1380	C-H or C-O bending
1077, 1015	1000-1125	CH_2-O-CH_2 pyranose ring stretching
934, 860	800-995	C-O-C asymmetric stretching

* (Piyaporn, 2015)

Figure 13 shows the SEM microphotograph of SCB 3 bioplastic. It was observed rough and agglomerates were seen on the surface of the SCB 3 bioplastic.

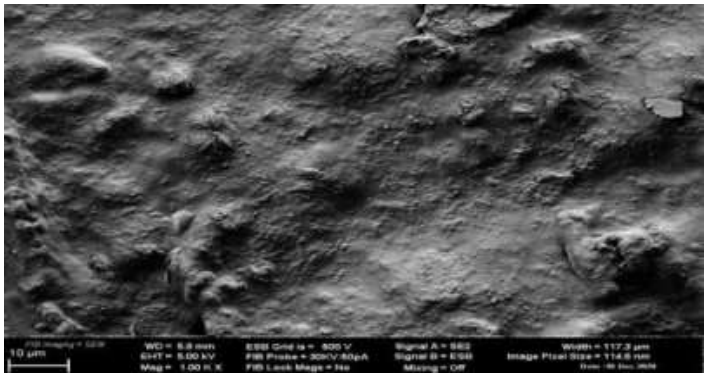

Figure 13 SEM photomicrograph of SCB 3 bioplastic

Thermal stability of SCB 3 bioplastic (Figure 14) and the interpretation (Table 10) are performed. The data show three stages of distinct weight losses. The first stage dehydration of water occurred at 280.44 ºC with weight loss of 13.05 %. The second stage started at about 348.86 ºC with weight loss of 63.24 %, which is attributed to the depolymerization with volatilization. In the third stage, the exothermic peak was found at 479.35 ºC with the weight loss percent about 22.40 % which is corresponded due to the carbonization. The TG-DTA analysis of SCB 3 bioplastic suggested that the degradation of total mass loss is 98.69 %.

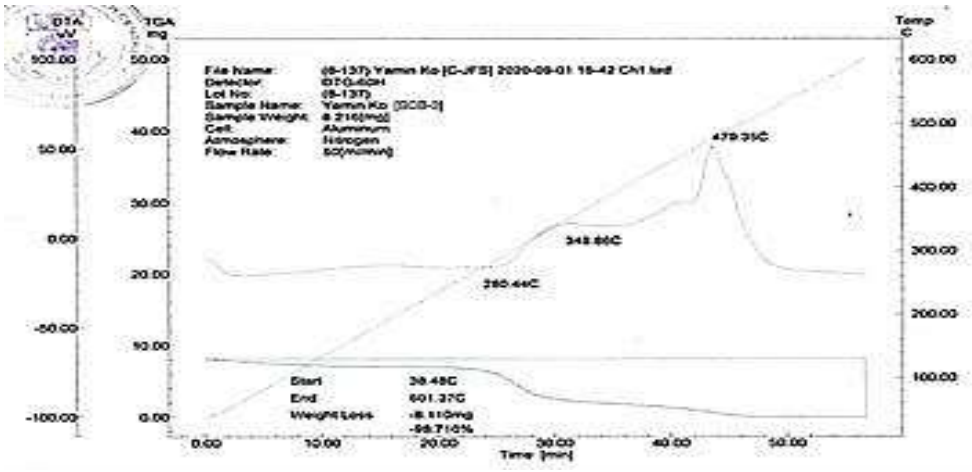

Figure 14 TG-DTA thermogram of SCB-3 bioplastic

Table 10 TG-DTA Data of SCB-3 Bioplastic

Sample	TG		DTA		Remarks
	Temperature Range (°C)	Weight Loss (%)	Peak Temperature (°C)	Nature of Peak	
SCB-3 Bioplastic	38.48-300	13.05	280.44	endothermic	Dehydration of water
	300-400	63.24	348.86	exothermic	Depolymerization with volatilization
	400-500	22.40 98.69	479.35	exothermic	Carbonization

Biodegradation of the Prepared SCB Bioplastics

These bioplastics clearly showed a slight deformation, after two days. Degradability of bioplastics is important when a polymeric system is applied in daily lives as its weight loss degree has a direct influence on the environment. The effect of different amounts of cellulose on bioplastic weight loss rate was conducted via soil burial test. Figure 15 shows the biodegradation nature of SCB 3 bioplastic for 2 days interval. The degradation rate (%) of the prepared bioplastics are presented in Table 11. It was found slightly degradation, after 2 days.

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

Figure 15 The physical appearances of SCB 3 bioplastic; (a) before burial test, (b) after two days, (c) after four days, (d) after six days and (e) after eight days burial

Table 11 Degradation Rate of Prepared Bioplastics by Soil Burial Test

Bioplastics / Time (day)	Degradation Rate (%)				
	0	**2**	**4**	**6**	**8**
SCB-1	0	4.008	12.84	24.65	40.98
SCB-2	0	9.65	18.46	30.78	49.26
SCB-3	0	15.2	24.58	36.14	59.62
SCB-4	0	20.96	31.62	42.27	68.46
SCB-5	0	26.02	37.02	49.18	76.35
SCB-6	0	34.02	40.28	56.58	82.48

Some Possible Application of Prepared the SCB Bioplastic

Bioplastic packaging options include bags for compost, agricultural foils, horticultural products, nursery products, toys, textiles, disposable cups, salad bowls, plates and food containers. The photographs of SCB bioplastic are presented (Figure 16) and the cup for food made from

prepared bioplastic is shown in Figure 17. Bioplastics last for 6 months at room temperature. But after 6 months, fungi are found on the surface. So, its shelf life is 6 months.

Figure 16 Photograph of SCB 3 bioplastic

Figure 17 Cup made of bioplastic

Conclusion

The physico-mechanical properties of SCB bioplastics such as thickness, tensile strength, elongation at break and tear strength were investigated. The effect of cellulose content on tensile strength and elongation at break of the bioplastics were also studied. Among them, SCB 3 bioplastic has the highest tensile strength, elongation at break and tear strength. It possesses 0.18 mm of thickness, 9.40 MPa of tensile strength, 45.00 % of elongation at break and 56.70 kN/m of tear strength respectively. All prepared bioplastics showed plain, smooth surface, flexible and pale-yellow colour. Among them, SCB 3 bioplastic is better than the other bioplastics. The prepared SCB bioplastics will be widely used in food packaging and making utensils. The use of cellulose-based bioplastic materials in the production of eco-friendly and less expensive utensils when compared to conventionally synthesized polymers.

Acknowledgments

The authors would like to express their deepest gratitude to for the provision of opportunity to do this research the Rubber Research and Development Centre for the necessary research testing and Universities' Research Center (URC) for TG-TDA, SEM, XRD and thanks are extended to Department of Chemistry, University of Yangon for FT IR.

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