

PREPARATION AND CHARACTERIZATION OF BIODEGRADABLE CHITOSAN-CORNCOB-GLUTARALDEHYDE BIOCOMPOSITE FILM

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Abstract

This research work is concerned with the study on the properties of biodegradable composite film derived from chitosan, corncob and glutaraldehyde (used as cross-linking agent). Biodegradable composite films were prepared by using optimum composition of 0.2 g corncob and 1.5 % chitosan with different concentrations of cross-linking agent. The effect of cross-linking agent was studied over a range of concentrations from 6 % to 30 % (total solid weight). The comparative study of cross-linking agent and its concentrations incorporated into biodegradable films was investigated based on thickness, tensile strength, elongation at break and tear strength. From the results of physicochemical parameters, the tensile strength of biodegradable films incorporated with glutaraldehyde was higher than that of chitosan-corn cob biocomposite film. The physicochemical properties of biodegradable composite films such as degree of swelling was also determined. The degree of swelling increases with increasing immersion time. With increasing glutaraldehyde concentration, the ultimate degree of swelling after 4 days of immersion time of composite films was found to decrease. Comparative characterization of biodegradable composite films with corncob powder and glutaraldehyde were determined by SEM and TG-DTA analyses. From SEM analysis, the prepared chitosan film and chitosan-corn cob biocomposite film had distinct phase structures whereas chitosan-corn cob-glutaraldehyde biocomposite film showed homogeneous and smooth surface. In TG-DTA analysis, only exothermic peaks appeared in chitosan film but both endothermic and exothermic peaks were observed in chitosan-corn cob and chitosan-corn cob-glutaraldehyde biocomposite films. The biodegradable nature of prepared composite films was also studied according to the soil burial techniques.

Keywords: biodegradable composite film, cross-linking agent, degree of swelling, chitosan-corn cob-glutaraldehyde biocomposite

Introduction

Chitosan is a biopolymer that is derived from chitin or by-products of seafood processing such as crab shell, lobster shell, and prawn shell waste (Mathew and Abraham 2008; Park *et al.* , 2004; Garcia *et al.*, 2006). Among biopolymers, chitosan has been considered as one of the most promising materials for future applications on account of its excellent biocompatibility, biodegradability, anti-microbial activity, non-toxicity and its economic advantages (Rutnakornpituk *et al.* , 2006; Singh *et al.* , 2009; Dutta *et al.*, 2004). Generally, chitosan was used in producing plastic film due to its unique structure, multidimensional properties, and wide applications in biomedical and other industrial area (Ambri *et al.*, 2013; Aranaz *et al.*, 2009; Srinivasa *et al.*, 2007).

Corn cob is a major part of the corn's waste, problematic waste is now being converted into useful industrial materials (Inglett, 1970). Powdered corn cob is a naturally occurring composite material and thus does not have a specific molecular weight, molecular formula and structural formula. Corncobs contain a considerable reservoir of carbohydrates, most of which are of a polysaccharide nature. Corncobs are lignocellulosic materials composed of cellulose,

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hemicellulose and lignin. Corncobs contain approximately 39.1 % cellulose, 42.1 % hemicellulose, 9.1 % lignin, 1.7 % protein, and 1.2 % ash (Beall and Ingram, 1992).

Glutaraldehyde, a linear, 5-carbon dialdehyde, is a clear, colorless to pale straw-colored, pungent oily liquid that is soluble in all proportions in water and alcohol, as well as in organic solvents. Glutaraldehyde can react with several functional groups of proteins, such as amine, thiol, phenol, and imidazole (Habeeb, and Hiramoto, 1968) because the most reactive amino acid side chains are nucleophile. Glutaraldehyde is commonly used as a cross-linking agent for collagen-based biomaterials. Among cross-linking agents, glutaraldehyde is the most commonly used in chitosan (Uragami *et al.*, 1994).

A composite is a structural material that consists of two or more combined constituents that are combined at a macroscopic level and are not soluble in each other. One constituent is called reinforcing phase and one in which it is embedded is called the matrix. Composites may be classified into three types which are green composites, hybrid composites, and textile composites. These composites can be easily disposed of or composed without harming the environment.

If the films consist of two organic materials that can be easily biodegradable, it is called biocomposite degradable films (Ting Liu, 2008). The production of biodegradable and edible films from carbohydrates and proteins adds value to low-cost raw materials and can play an important role in food preservation (Nasir *et al.*, 2005; Peesan *et al.*, 2003).

The aim of this research work was to study the preparation and characterization of the biodegradable chitosan-corn-cob-glutaraldehyde composite film using natural waste (corn-cob). Corncobs were used as filler in composites because it is naturally abundant in Myanmar, and low cost. Chitosan-corn-cob-glutaraldehyde biocomposite films were prepared by solvent evaporating method.

Materials and Methods

Sample Collection

The chemicals used in this research work were procured from British Drug House (BDH) Chemicals Ltd., England. Commercial chitosan was purchased from Shwe Poe Company, Hlaingtharyar Township, Yangon Region, Myanmar. Maize (*Zea mays*) sample was purchased from Thirimingalar Market, Kyeemyindaing Township, Yangon Region, Myanmar.

Preparation of Corn-cob Powder and Base Activated Corn-cob Powder

The collected raw corncobs were cut into pieces and washed with water (three times) to remove dust and impurities. Then they were directly dried in sunshine and dried in an oven at 80 °C for 24 h. The dried corncobs were obtained. They were made into powder form with blender. Corn-cob powders were sieved by a sieve (80 mesh size).

The 30 g of corn-cob powder was chemically treated with 300 mL NaOH solution having concentration of 0.1 M. The mixture was placed in an autoclave at 121 °C for 20 min to obtain homogeneous solution. Then, it was washed with distilled water until pH 7.0. After that it was dried in an oven at 120 °C for 24 h. The dried sample was ground in mortar and pestle to obtain fine particle size. Finally, base activated corn-cob powders were sieved by a sieve (80 mesh size).

Preparation of Chitosan-Corncob Biocomposite Films

Chitosan solution (1.5 % w/v) was prepared by dissolving 1.5 g of chitosan in 100 mL of 2 % v/v acetic acid with often stirring and heating for 30 min at 100 °C to get a clear solution. After the chitosan was dissolved completely, the solution was filtered by a filter paper to remove any small lumps in the solution. Then 0.1 g to 0.5 g of base activated corncob powder was added to the above chitosan solution. The mixture was stirred and heated for 30 min at 100 °C to get a homogeneous solution. The mixture solutions were then made into films by pouring onto a melamine shallow plates.

The melamine plates containing the mixture solution were left for about three days at room temperature. Then they were heated at 50 °C in an oven for about 6 h. All of the prepared chitosan-corn-cob biocomposite films were kept under dry conditions before further use.

Preparation of Chitosan-Corncob-Glutaraldehyde Biocomposite Films

Chitosan solution (1.5 % w/v) was prepared by dissolving 1.5 g of chitosan in 100 mL of 2 % v/v acetic acid with often stirring and heating for 30 min at 100 °C to get a clear solution. After the chitosan was dissolved completely, the solution was filtered by a filter paper to remove any small lumps in the solution. Then 0.2 g of base activated corncob powder was added to the above chitosan solution. Various concentrations of glutaraldehyde (6, 12, 18, 24 and 30 % of the total solid weight) were added to the prepared chitosan-corn-cob solutions. The mixture solutions were stirred and heated for 30 min at 100 °C to get a homogeneous solution. These solutions were kept for sufficient time to remove any bubble formation and were cast onto a cleaned and dried melamine plate at room temperature.

The melamine plates containing the mixture solution were left for about three days at room temperature to obtain chitosan-corn-cob-glutaraldehyde biocomposite films. Then they were heated at 50 °C in an oven for about 6 h. All of the prepared chitosan-corn-cob-glutaraldehyde biocomposite films were kept under dry conditions before further use.

Characterization of the Prepared Samples

The physicochemical properties (pH, moisture, specific gravity and bulk density) of chitosan and base activated corncob powder were determined. The morphological structures of the prepared biocomposite films were characterized by SEM. Thermal properties of the biocomposite films were evaluated by TG-DTA.

The scanning electron microscopy (SEM) images of raw corncob powder, base activated corncob powder, chitosan film, chitosan-corn-cob biocomposite film, and chitosan-corn-cob-glutaraldehyde biocomposite film were obtained using JSM-5610 Model SEM, JEOL-Ltd., Japan.

Thermogravimetric analyses of samples were performed using TG-DTA instrument, (Hi-TGA 2950 model). The temperature range between 0 °C and 600 °C under nitrogen gas (at a rate of 50 mL/min).

Determination of Biodegradation

Soil burial test

The films were cut $1" \times 1"$ dimension. The films were then accurately weighed and buried in soil at the depth of 5 cm. They were taken out from the soil at an interval of one week. Sample geometry on degradation was also recorded with photos and they are presented in Figure 10.

Results and Discussion

Sample Preparation

The corns were collected from local market. The seeds were removed from corns. The obtained corncobs were cut into small pieces and washed with water (three times). They were directly dried in sunshine and dried in an oven at $80\text{ }^{\circ}\text{C}$ for 24 h. Then they were made into powder form with blender. Finally, the obtained corncob powder were sieved by a sieve (80 mesh).

Physicochemical Analysis of Chitosan and Base Activated Corncob Powder

Table 1 shows the physicochemical properties of chitosan and base activated corncob powder. From the physicochemical properties, the moisture content of chitosan is higher than the base activated corncob powder. However, the pH value of chitosan is lower than the base activated corncob powder.

Table 1 Physicochemical Properties of Chitosan and Base Activated Corncob Powder

Physicochemical properties	Chitosan	Base Activated Corncob Powder
pH	4.10	7.49
Moisture (%)	13.050	10.1549
Specific gravity (gmL^{-1})	1.0205	-
Bulk density (gcm^{-3})	-	0.2609

Characterization of Corncob Powder

SEM analysis

The scanning electron micrographs of raw corncob powder (RCP) and base activated corncob powder (BACP) are shown in Figure 1. From SEM analysis, the base activated corncob powder (BACP) had more distinct features than the raw corncob powder (RCP).

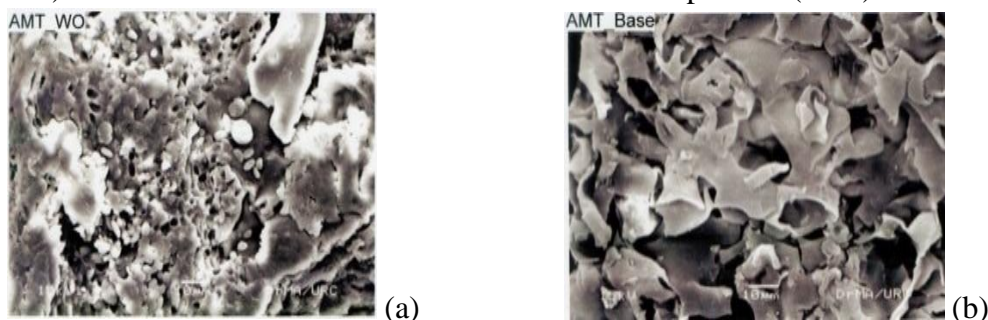


Figure 1 Scanning electron micrographs of (a) raw corncob powder (RCP) and (b) base activated corncob powder (BACP)

Preparation of Different Types of Chitosan-Corncob Biocomposite Films

The various amount of base activated corncob powder (0.1, 0.2, 0.3, 0.4, and 0.5 g) were added to the chitosan solution (1.5 % w/v). The mixture solutions were stirred and heated for 30 min at 100 °C to get a homogeneous solution. These solutions were made into films by pouring onto a melamine shallow plates. These melamine plates containing the mixture solution were left for about three days at room temperature to obtain different types of chitosan-corncob biocomposite films. Then they were heated at 50 °C in an oven for about 6 h. All of the different types of chitosan-corncob biocomposite films were kept under dry conditions before further use.

Physicomechanical Properties of Chitosan-Corncob Biocomposite Films

The mechanical properties of chitosan-corncob biocomposite films are presented in Table 2 and Figure 2. The thicknesses of CC biocomposite films are approximately 0.20 to 0.34 mm. The composition of chitosan for preparing the biocomposite films was 1.5 % w/v and different weights of base activated corncob powder. In the concentration range of base activated corncob powder 0.1 g to 0.5 g, the tensile strength decreased slightly, but the maximum tensile strength was found to be 15.7 MPa for CC-2 film with CC content 0.2 g.

The more the elongation at break of a certain film, the higher the stability of the film. Therefore, based on the data of elongation at break, CC-2 film was the most suitable for the preparation of film. The elongation at break of corncob content 0.2 g was 5.2 %. The tear strength of chitosan-corncob CC biocomposite film increased significantly with progressively increase of corncob concentration up to 0.2 g and then it decreased in CC-3, CC-4 and CC-5 biocomposite films. The tear strength of CC-2 was 48.5 kN/m.

Table 2 Physicomechanical Properties of Chitosan Film and Chitosan-Corncob Biocomposite Films

Biocomposite Films	Weight of corncob powder (g)	Thickness (mm)	Tensile strength (MPa)	Elongation at Break (%)	Tear strength (kN/m)	Tensile strength/elongation
C	-	0.17	18.8	6.0	51.5	3.13
CC-1	0.1	0.20	10.4	3.1	40.6	3.35
CC-2	0.2	0.21	15.7	5.2	48.5	3.02
CC-3	0.3	0.25	10.1	2.9	39.4	3.48
CC-4	0.4	0.31	6.28	2.3	35.7	2.51
CC-5	0.5	0.34	4.39	1.8	30.6	2.44

- C = chitosan film with 1.5 % chitosan and 2 % acetic acid
 CC-1 = composite film with 1.5 % chitosan + 2% acetic acid+ 0.1 g corncob
 CC-2 = composite film with 1.5 % chitosan + 2% acetic acid+ 0.2 g corncob
 CC-3 = composite film with 1.5 % chitosan + 2% acetic acid +0.3 g corncob
 CC-4 = composite film with 1.5 % chitosan + 2% acetic acid +0.4 g corncob
 CC-5 = composite film with 1,5 % chitosan + 2% acetic acid +0.5 g corncob

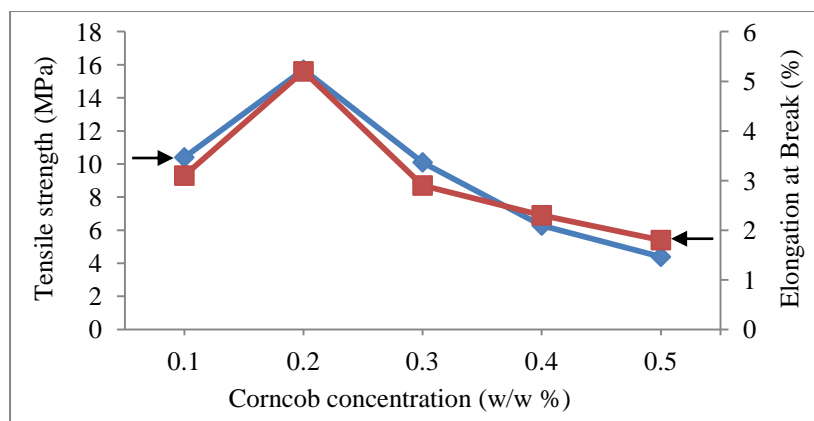


Figure 2 Tensile strength and elongation at break of chitosan-corncob biocomposite films as a function of different corncob concentrations

Preparation of Different Types of Chitosan-Corncob-Glutaraldehyde Biocomposite Films

Base activated corncob powder (0.2 g) was added to the chitosan solution (1.5 % w/v). The various concentrations of glutaraldehyde (6, 12, 18, 24, and 30 % of the total solid weight) were added to the above chitosan-corncob solutions. The mixture solutions were stirred and heated for 30 min at 100 °C to get a homogeneous solution. These solutions were kept for sufficient time to remove any bubble formation and were cast onto a cleaned and dried melamine plate at room temperature. The melamine plates containing the mixture solution were left for about three days at room temperature to obtain different types of chitosan-corncob-glutaraldehyde biocomposite films. Then they were heated at 50 °C in an oven for about 6 h. All of the different types of chitosan-corncob-glutaraldehyde biocomposite films were kept under dry conditions before further use.

Physicomechanical Properties of Chitosan-Corncob-Glutaraldehyde Biocomposite Films

The mechanical properties such as tensile strength, elongation at break and tear strength are important parameters which revealed the nature of the film. The mechanical properties of chitosan-corncob-glutaraldehyde biocomposite films are presented in Table 3 and Figure 3. The thicknesses of CCG biocomposite films are approximately 0.18 mm to 0.23 mm.

The tensile strength of prepared CCG biocomposite films decreased with increasing the amount of cross-linked glutaraldehyde beyond CCG-3. The value of tensile strength decreased from 19.5 MPa to 8.5 MPa when the amount of glutaraldehyde increased from 6 % to 30 %. The maximum tensile strength of 35.2 MPa was found at CCG-3 biocomposite film.

The elongation at break of CCG biocomposite films decreased from 5.4 % to 2.8 %. The percent elongation at break was found to be increased significantly at CCG-3 (ca 8.3 %) among the prepared CCG biocomposite films. The tensile strength and the percent elongation at break of CCG-3 biocomposite film were found to be higher than those of the other film. The tear strength of CCG-3 was found to be 59.8 kN/m.

Table 3 Physicomechanical Properties of Chitosan-Corn-cob-Glutaraldehyde Biocomposite Films

Biocomposite Films	Amount of glutaraldehyde (drops)	Thickness (mm)	Tensile strength (MPa)	Elongation at Break (%)	Tear strength (kN/m)	Tensile strength/elongation
CCG-1	2	0.18	19.5	5.4	50.3	3.61
CCG-2	4	0.16	24.8	6.1	55.4	4.07
CCG-3	6	0.15	35.2	8.3	59.8	4.24
CCG-4	8	0.21	10.2	3.4	45.6	3.00
CCG-5	10	0.23	8.5	2.8	40.3	3.04

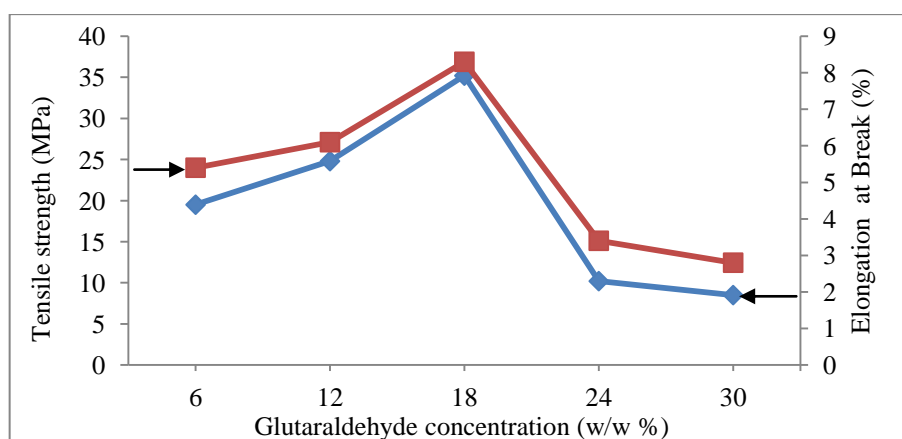
CCG-1 = chitosan-corn-cob biocomposite film with 6 % glutaraldehyde

CCG-2 = chitosan-corn-cob biocomposite film with 12 % glutaraldehyde

CCG-3 = chitosan-corn-cob biocomposite film with 18 % glutaraldehyde

CCG-4 = chitosan-corn-cob biocomposite film with 24 % glutaraldehyde

CCG-5 = chitosan-corn-cob biocomposite film with 30 % glutaraldehyde

**Figure 3** Tensile strength and elongation at break of chitosan-corn-cob-glutaraldehyde biocomposite films as a function of different glutaraldehyde concentrations

Degree of Swelling (%) of Chitosan-Corn-cob and Chitosan-Corn-cob-Glutaraldehyde Biocomposite Films

The degree of swelling of a series of chitosan-corn-cob (CC) biocomposite films as a function of immersion time in distilled water at room temperature are shown in Table 4 and Figure 4. According to the experimental data, the degree of swelling reached a maximum after 4 days. After 4 days of immersion time, the degree of swelling of CC biocomposite films decreased.

The degree of swelling of different chitosan-corn-cob-glutaraldehyde biocomposite (CCG) films with as a function of immersion time are also shown in Table 5 and Figure 5. For a given composition, mostly the degree of swelling increased with increasing immersion time. With increasing glutaraldehyde contents, the ultimate degree of swelling of CCG biocomposite films were found to decrease CCG-1 to CCG-5. Among them the degree of swelling of CCG-3 was found to be higher than that of others with respect to their immersion time. The CCG-3 biocomposite film had the most degree of swelling compared with the other CCG biocomposite films as a function of time and glutaraldehyde content. For CCG biocomposite film, the equilibrium degree of swelling was found to decrease from 85.77 to 62.33, as a function of

glutaraldehyde content. The degree of swelling was one of the essential parameters for determining the degradable biocomposite film. The values of degree of swelling of CCG biocomposite films were more than CC biocomposite films.

Table 4 Degree of Swelling (%) of Chitosan-Corncob (CC) Biocomposite Films Under Different Contact Time

No.	Biocomposite Films	Degree of Swelling (%)				
		1 h	2 h	3 h	4 h	4 days
1	CC-1	46.67	52.94	57.89	60.01	71.43
2	CC-2	50.01	68.33	71.54	94.29	105.25
3	CC-3	37.50	44.44	48.01	55.33	62.67
4	CC-4	30.52	39.15	45.37	53.65	59.43
5	CC-5	22.37	35.45	39.63	45.27	49.56

Table 5 Degree of Swelling (%) of Chitosan-Corncob-Glutaraldehyde (CCG) Biocomposite Films Under Different Contact Time

No.	Biocomposite Films	Degree of Swelling (%)				
		1 h	2 h	3 h	4 h	4 days
1	CCG-1	70.83	73.08	75.86	78.13	85.77
2	CCG-2	72.55	77.42	80.56	82.28	94.12
3	CCG-3	78.45	93.52	117.68	173.14	198.25
4	CCG-4	50.62	62.75	68.12	75.34	81.22
5	CCG-5	39.17	45.27	49.81	57.65	62.33

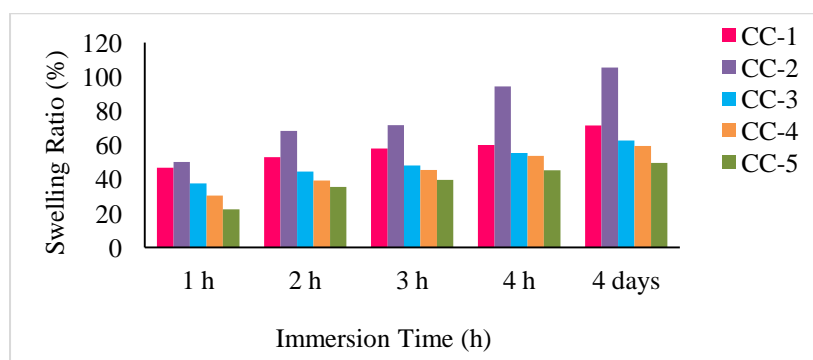


Figure 4 The effect of time on degree of swelling of chitosan-corncob (CC) biocomposite films

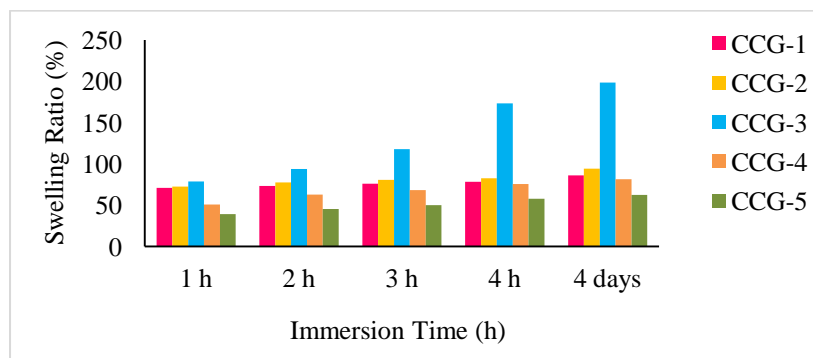


Figure 5 The effect of time on degree of swelling of chitosan-corncob-glutaraldehyde (CCG) biocomposite films

Characterization of Chitosan Film, Chitosan-Corncob Biocomposite Film and Chitosan-Corncob-Glutaraldehyde Biocomposite Film

SEM analysis

The scanning electron micrographs of chitosan (C) film, chitosan-corncob (CC-2) biocomposite film and chitosan-corncob-glutaraldehyde (CCG-3) biocomposite film are shown in Figures 6 (a), (b) and (c). The chitosan (C) film and chitosan-corncob (CC) biocomposite film have distinct phase structure. The chitosan-corncob-glutaraldehyde (CCG) biocomposite film had relatively smooth, homogeneous and continuous matrix without cracks with good structural integrity.



Figure 6 Surface morphologies of (a) C film (b) CC biocomposite film and (c) CCG biocomposite film

TG-DTA analysis

According to the TG-DTA thermogram profiles, the thermogram of chitosan (C) film is shown in Figure 7, having a weight loss in two stages. In the first stage, the temperatures range from 120 °C to 310 °C and the weight loss was 30.03 % due to the vaporization and elimination of volatile products. In the second stage, the weight loss started at 310 °C and that continued up to 600 °C where the weight loss was 19.42 % due to the degradation and decomposition of cellulose of chitosan polymer.

TG-DTA analysis also confirmed the chitosan-corncob (CC) biocomposite film in Figure 8. The first stage ranges between 39 °C and 120 °C with 11.31 % of weight loss due to the evaporation of absorbed water. The second stage of weight loss started at 120 °C and that continued up to 320 °C during which there was 36.36 % of weight loss due to the decomposition of cellulose and hemicellulose. The third stage in the loss in weight of about 30.68 % was actually observed from 320 °C to 600 °C due to the decomposition of lignin in corncob sample.

The nature regarding the thermogram profile of chitosan-corncob-glutaraldehyde (CCG) biocomposite film is presented in Figure 9. It indicated the two stages in weight loss. The percentage of weight loss was found to be 50.25 % at the temperature range between 120 °C and 320 °C. This weight loss was probably due to the vaporization and evaporation of volatile materials. The second stage in the loss in weight of about 33.52 % was observed within the temperature range of 320 °C up to 600 °C due to the decomposition of chitosan polymer.

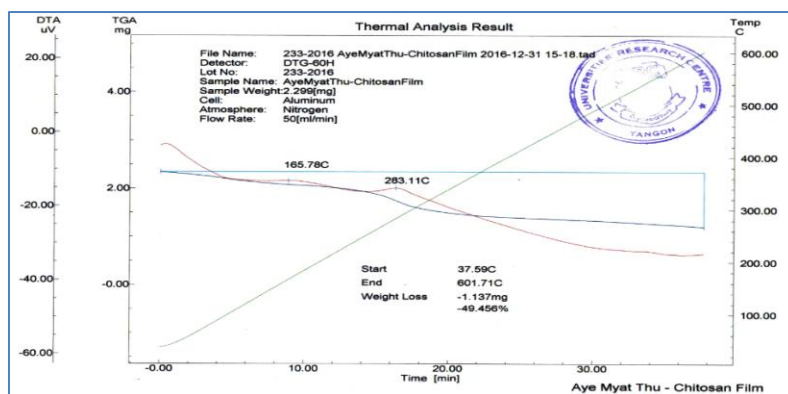


Figure 7 Thermal degradation analysis of pure chitosan film by TG-DTA analysis

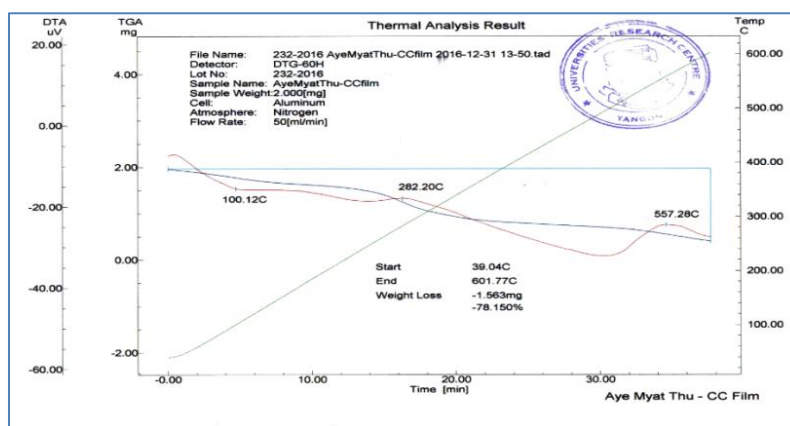


Figure 8 Thermal degradation analysis of chitosan-corn cob biocomposite film by TG-DTA analysis

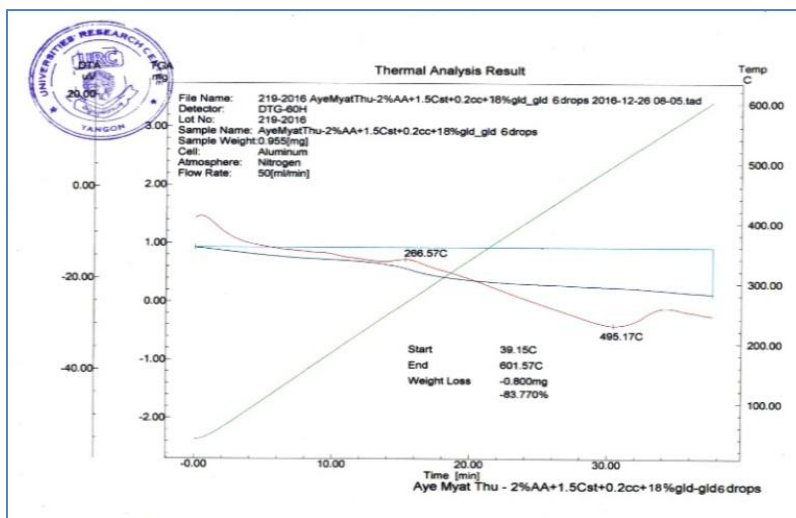


Figure 9 Thermal degradation analysis of chitosan-corn cob-glutaraldehyde biocomposite film by TG-DTA analysis

Biodegradation of Biocomposite Films

In this work, biodegradation of chitosan (C) film, chitosan-corn cob (CC) biocomposite film and chitosan-corn cob-glutaraldehyde (CCG) biocomposite film were tested by soil burial method. Soil burial is a traditional way to test samples for degradation because of its similarity to actual condition of waste disposal. Uniformly sized samples were buried in the soil from

waste disposal. The physical parameters of the soil to be tested are presented in Table 6. From the physical parameters, the pH value of the tested soil is slightly greater than 7 and moisture content is 2.18.

Figure 10 shows biodegradation nature of C film, CC biocomposite film and CCG biocomposite film. These figures clearly show that significant deformation of C film, CC biocomposite film and CCG biocomposite film was found after two weeks although there was significant degradation of these films. However, quite deformation of C film, CC biocomposite film and CCG biocomposite film appeared after four weeks. Therefore, the prepared CC and CCG biocomposite film may be disappeared after short time duration by using burial method.

Table 6 Physical Parameters of the Tested Soil

Physical parameters	Soil
pH	7.64
Moisture (%)	2.18

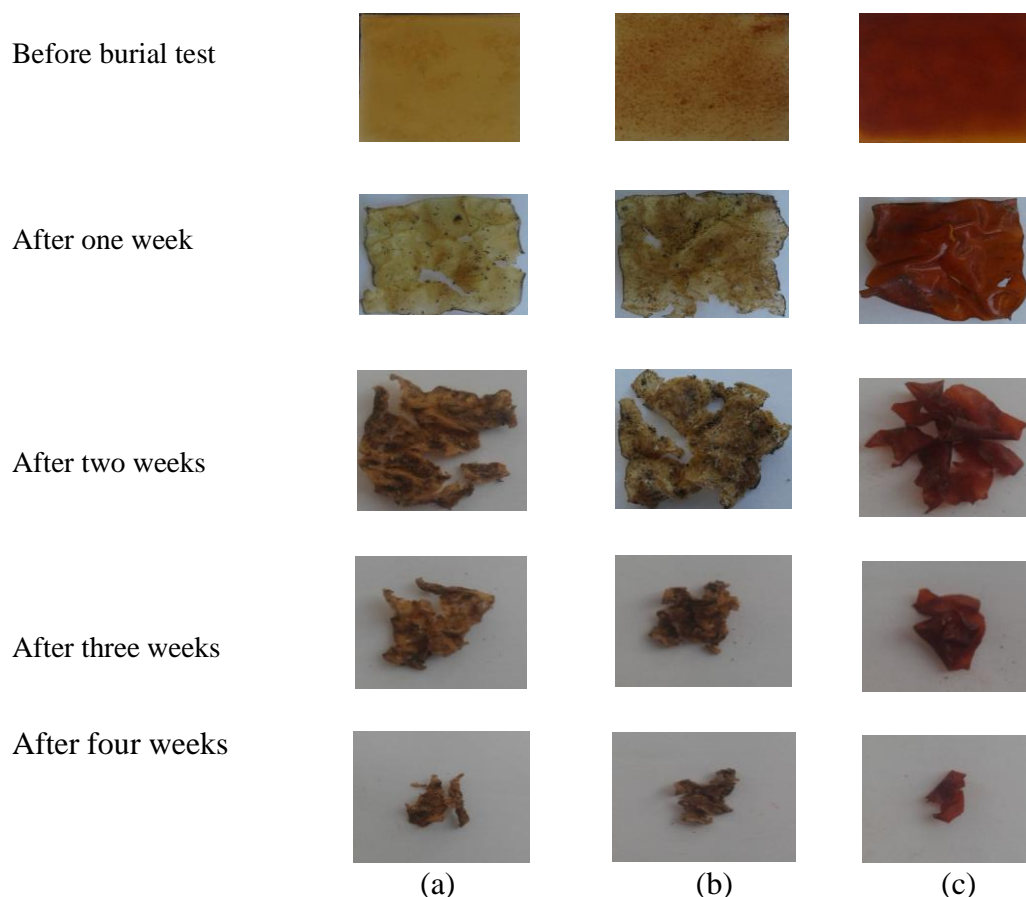


Figure 10 The physical appearances of (a) C film
(b) CC biocomposite film
(c) CCG biocomposite film

Conclusion

In this study, three types of films such as chitosan (C) film, chitosan-corncob (CC) biocomposite film and chitosan-corncob-glutaraldehyde (CCG) biocomposite film were prepared from the waste corncob with chitosan and various concentrations of cross-linking agent (glutaraldehyde) by solvent evaporating method. The effect of concentration of glutaraldehyde was studied over a range of concentrations from 6 % to 30 % (total solid weight). The optimum condition of CCG biocomposite film was achieved by using in the range of chitosan (1.5 %) and corncob (0.2 g) with glutaraldehyde (18 %). Based on the mechanical properties such as tensile strength (MPa), elongation at break (%) and tear strength (kN/m), the optimum condition was achieved by using in the range of 1.5 % chitosan and 0.2 g of corncob with different concentrations of glutaraldehyde. The CCG-3 biocomposite film provided the film with the highest mechanical strength (35.2 MPa), elongation at break (8.3 %), tear strength (59.8 kN/m) and film flexibility. The degree of swelling of biocomposite film had a lower degree of hydration, as measured by swelling ratio, which can be altered by varying weight percent of CC and CCG in the membrane matrix. From SEM analysis, the surface morphology of CC biocomposite film had distinct phase structure and CCG biocomposite film had homogeneous and smooth surface. From TG-DTA analysis, thermal stability of CC biocomposite film was found to be lower than that of CCG biocomposite film. According to the soil burial techniques, significant deformation and degradation of both CC biocomposite film and CCG biocomposite film occurred as the same as chitosan film. In addition, the prepared C film, CC biocomposite film and CCG biocomposite film can be disposed easily after using it, without environmental impact.

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