

# DEVELOPMENT OF BIODEGRADABLE FILM FROM ACETYLATED CELLULOSE OF COTTON STALK

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## Abstract

There has been increased concern on the deterioration of the environment due to solid waste pollution from petrochemical based polymers which are non-biodegradable and harmful to both sea life and humans, when dispersed in nature, creating a global waste disposal problem.

In this work, the possibility of developing cellulose acetate film; a biodegradable polymer, as an alternative replacement for petrochemical based polymer, a non-biodegradable polymer was carried out. This was achieved by casting acetylated cellulose into film (cellulose acetate film) with varying amounts of Dimethyl Phthalate, DMP (0 wt%, 10wt%, 20 wt%, 30 wt%, 40 wt%, 50 wt%,) plasticizer to improve the mechanical properties of the film. The film with 30 wt% DMP exhibited the best tensile strength and elongation at break with values of 0.077MPa and 17.20% respectively.

The biodegradability of the cellulose acetate film was studied by soil burial test in compost soil for 90days and the result revealed that the tensile strength and percentage elongation of cellulose acetate film produced gradually reduced with time in the soil burial test. This indicates that a biodegradable polymer can be obtained from the source material; cotton stalk for different applications. Percentage weight loss analysis of the samples suggests that cellulose acetate film without plasticizer had highest weight loss of 17.8%. The surface microstructure of the film before and after burial was examined by Scanning Electron Microscopy (SEM) which revealed a physical deterioration of the film due to microorganisms attack present in the compost soil.

Keywords: Biodegradable, polymer, cellulose, cellulose acetate, tensile test, plasticizer, film.

## 1 INTRODUCTION

The potential of biodegradable polymers has been recognized for a long time, since they could be an interesting way to overcome the limitation of the petrochemical resources in the future [2]. Persistent polymers constitute significant sources of environmental pollution, harming wildlife when they are dispersed in nature [1]. The fossil fuel and gas could be partially or completely replaced by green agricultural resources, which would also enhance the reduction of CO<sub>2</sub> emissions [2]. For example, the disposal of non-degradable plastic bags adversely affects aquatic life. It is widely accepted that the use of long-lasting polymers in products with a short life-span, such as engineering applications, packaging, catering, surgery, and hygiene, is not appropriate. Moreover, incineration of plastic waste

presents environmental issues as well, since it yields toxic emissions (e.g., dioxin) [3].

Cellulose acetate is one of the oldest manmade macromolecules used extensively in the textile and polymer industries. It has an inherent advantage in that the starting material, cellulose, is a renewable natural resource. With cotton production standing at  $1.4789 \times 10^6$  MT in 2012 and more than 60% of this grown in northern part of Nigeria (United State Department for Agriculture, 2012), and cotton stalk being a renewable agricultural waste and constituting about 58.5 wt% of cellulose [4], it can adequately serve in the production of cellulose acetate when cellulose is extracted. The current applications of cellulose acetate include textiles, cigarette tow, lacquers, cellulose films, and packaging. Since it is nontoxic, cellulose acetate is widely used in food packaging [5]. The biodegradability properties of cellulose

acetate film make it a better alternative in packaging when compared to the common synthetic polymers often used which are produced from petrochemicals and are rarely biodegraded.

## 2.0 EXPERIMENTAL

### 2.1 Materials and reagents

The cotton stalk samples obtained from Institute of Agricultural Research (IAR) Farm, ABU Zaria was pretreated and acetylated to cellulose and cellulose acetate flakes respectively using the method by Bello et al., [3]. The reagents used are among others; 98% Acetone, Dimethyl Phthalate (DMP) plasticizer, 98.5% acetic anhydride, 99.5% glacial acetic acid, 98% H<sub>2</sub>SO<sub>4</sub>, all of which were of analytical grades manufactured by BDH Chemicals, Poole, England and purchased from Haddis Chemicals, Samaru Zaria. Fourier transform Infrared (FTIR) spectrometer was carried out at National Research Institute for Chemical Technology (NARICT) Zaria for functional group study, XRD analysis was carried out at Nigerian Geological Survey Agency, Kaduna for morphological characterization. The mechanical test of the film was carried out using the tensile testing machine at the Centre for Energy Research and Development (CERD), OAU, Ile-Ife.

### 2.2 Preparation of cellulose acetate film and casting

Films were produced by the method of Hyppola *et al.* [5]. Films were developed by dissolving 5 g of acetylated cellulose powders in 100ml acetone and stirred continuously for three hours at 25 °C, the clear portion was carefully decanted after the solution was allowed to settle for 60 mins. To the clear portion, a varying amount of Dimethyl Phthalate, DMP (0 wt%, 10 wt%, 20 wt%, 30 wt%, 40 wt%, 50 wt%) plasticizer was added to the solution to increase the plasticity of the film and the solution was stirred for 30 mins. Cellulose acetate film was formed by pouring the solution obtained onto a glass plate covered with polyethylene film at room temperature.

## 2.3 CHARACTERIZATION OF CELLULOSE ACETATE

### 2.3.1. Fourier transform infrared spectroscopy (FTIR)

The chemical investigation of functional groups in acetylated cellulose (cellulose acetate) were carried out using Fourier Transform Infra-Red Spectroscopy (Spectrum Perkin Elmer). The sample disc was prepared by mixing and compressing the sample and KBr at a 1:1 ratio. FTIR spectra were produced by scanning at a 4 cm<sup>-1</sup> resolution for transmission wavelength range 300 to 4500 cm<sup>-1</sup>.

### 2.3.2 X-ray diffraction (XRD) analysis of samples

The structural changes of polymers can be evaluated using XRD curves. So the XRD patterns of the pretreated cotton stalk and acetylated cotton stalk were obtained at room temperature with a scan speed of 4°/min for 2θ ranging from 3° to 80° at a voltage of 40kV and current of 40mA.

## 2.4 Mechanical Properties of films developed

### 2.4.1 Tensile properties

The cellulose acetate film was conditioned in a standard testing atmosphere of 21 °C and 65% relative humidity for 24 hrs before performing the tensile tests. The test was performed on single film of thickness 0.32 mm and gauge length of 20 mm to obtain breaking load, percentage elongation at break and Young modulus of the film.

### 2.5 Biodegradability Test

The biodegradability test was conducted on the produced cellulose acetate film by the soil burial standard test method (ASTM D882), to determine the weight difference in the film before and after burial and further examine the biodegradability of the sample using SEM [7].

Compost soil obtained from oversea area in Nasarawa, Nasarawa State was collected in a nursery pot and strips of film of approximately same size (20 mm) and weight (0.5 g) cut from the raw film without plasticizer, one with plasticizer and that of conventional polyethylene film

(control) were buried individually in the nursery pots and incubated for 90 days at ambient temperature ;7 & 8]

2.5.1 Weight loss measurement

The films were removed from the nursery pots after 90 days, cleansed thoroughly and then dried until constant weight was obtained. The corresponding percentage weight loss was calculated using Equation 1 [9]

Percentage weight loss

$$= \frac{w_i - w_f}{w_i} \times 100 \dots \dots \dots 1$$

Where  $w_i$  is weight of film before burial,  $w_f$  is the weight of film after burial.

2.5.2 Morphological studies

The morphology of the untreated cotton stalk, fibers obtained from the stalk (cellulose acetate fiber) and

degraded material obtained from the fibers was observed using scanning electron microscopy (SEM). Thus, SEM images of the pretreated cotton stalk and cellulose acetate (acetylated) were recorded at 100 x and 2,000x magnification using an acceleration voltage of 20 kV.

3 RESULTS AND DISCUSSION

3.1 Characterization of cellulose acetate

3.1.1 FT-IR Analysis of Cellulose Acetate

The assignment of IR bands for different functional groups present in the acetylated cellulose was investigated and is seen to be in agreement with what was reported by the author in [25]. The absorption band at 3409.34  $\text{cm}^{-1}$  is an indication of a decrease in the intensity of the -OH absorption band which conforms to what was reported for acetylated cellulose as given by Hu et al. [10].

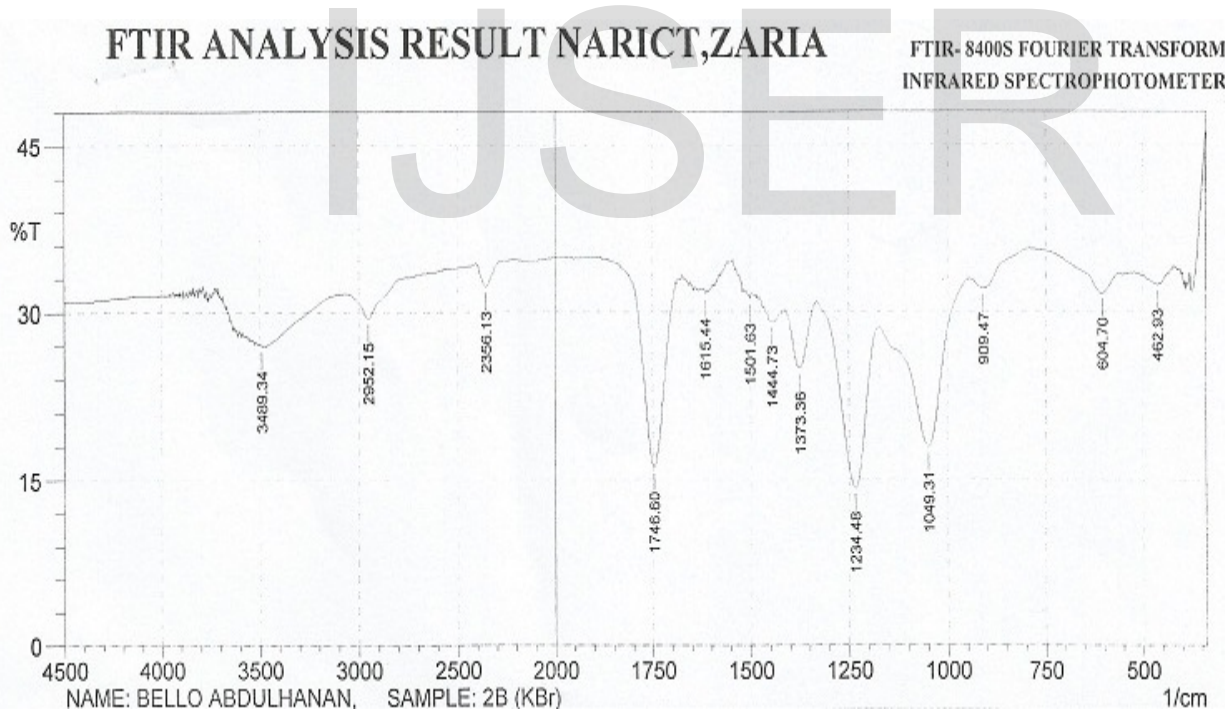


Figure 1: FT-IR Analysis of Acetylated Cotton Stalk.

Figures 1 represent the FT-IR spectroscopy of acetylated cotton stalk. As seen on the figure, the shoulder near the -OH stretching vibrations, 2921  $\text{cm}^{-1}$  is attributed to C-H

stretch and corresponds to the aliphatic groups in polysaccharides (cellulose). The ester carbonyl absorption peaks at 1746.60  $\text{cm}^{-1}$ , carbonyl hydrogen (C-H) peak at

1374.36  $\text{cm}^{-1}$  in acetyl group and 1230  $\text{cm}^{-1}$  absorption (C-O) in O-C=O group verified ester bond have been formed in the acetylated cotton stalk and their relative intensity is enhanced. The characteristic peaks developed confirmed the acetylation of cellulose extracted from cotton stalk. The absence of development of peaks or stretching at 1840 – 1760  $\text{cm}^{-1}$  is an indication of the absence of free acetic anhydride during acetylation of cellulose. This is in agreement with the authors' work in [10] where absence of free acetic anhydride

was observed from the FT-IR spectra of modified rice straw for thermoplastic application.

### 3.1.2 X-ray Diffraction of Cellulose acetate

The crystallinity of the acetylated cellulose was analyzed. There were diffraction patterns which represented that of a typical cellulose acetate as affirmed by Diana et al. [12] who presented the XRD patterns of cellulose after acetylation as seen in Figure 2.

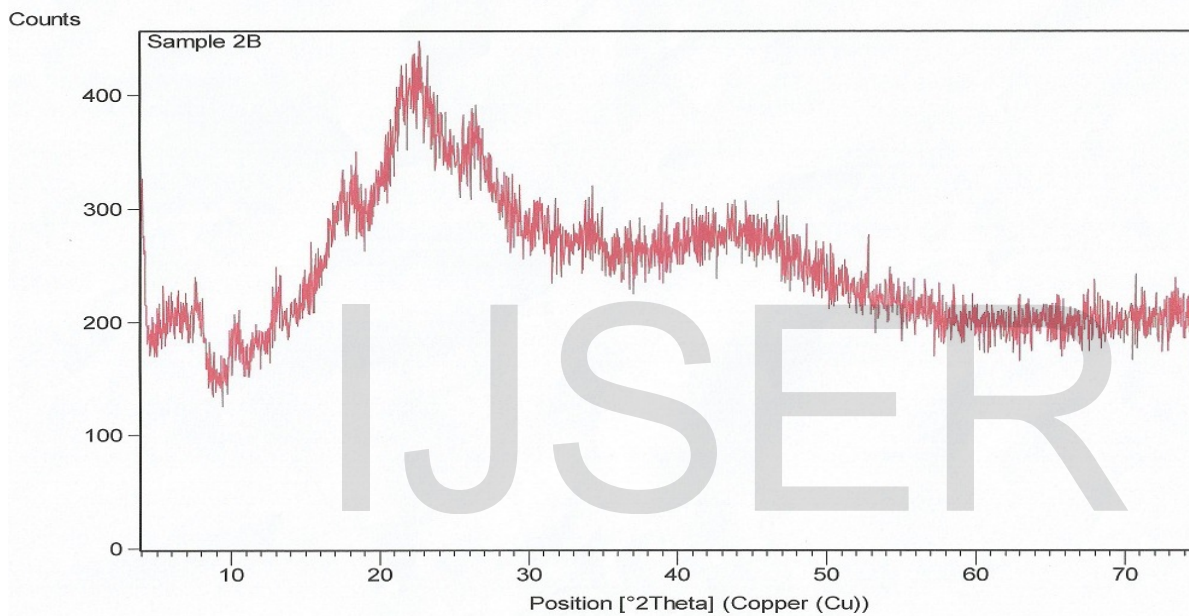


Figure 2: XRD Pattern of Acetylated Cotton Stalk

The decrease in crystallinity is expected due to the degradation of the crystal structure of cellulose during acetylation [13], this is because the number of hydrogen bonds is decreased due to the substitution of hydroxyl groups by acetyl groups that have a larger volume and this confirmed why the acetylated sample is amorphous. As the cellulose chains at the surface are acetylated, they become soluble and removed, further exposing the cellulose crystals, which therefore reduces the crystallinity of remaining

cellulose chains within the cellulose films as the acetylation continues [14].

### 3.2 Tensile Properties of Cellulose Acetate Film

#### 3.2.1 Effect of dimethyl phthalate (plasticizer) on tensile strength of film.

Figure 3 presents the effect of varying plasticizer weight on the tensile strength of cellulose acetate film.

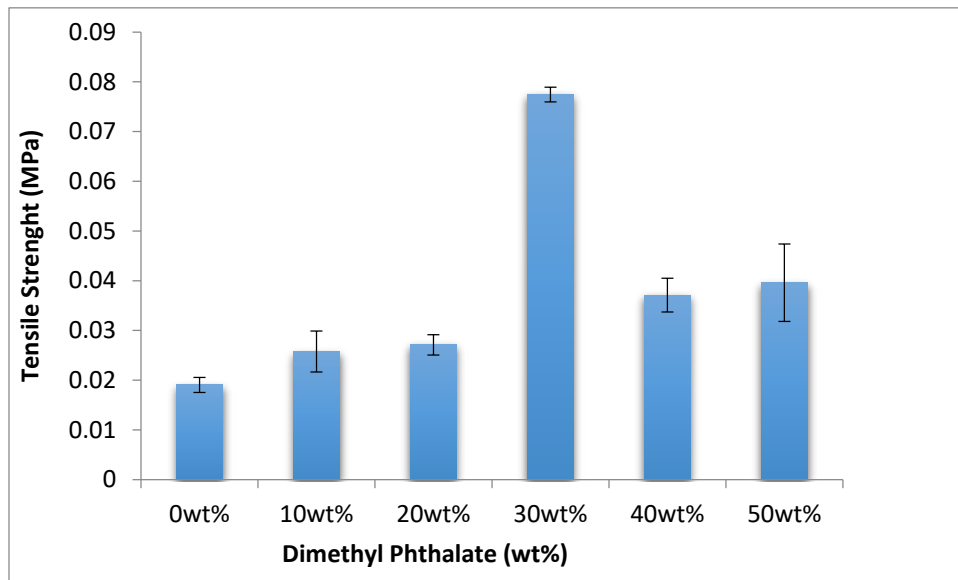


Figure 3: Effect of Dimethyl Phthalate Plasticizer on the Tensile Strength of Cellulose Acetate Film.

As observed from the plot, the result of the test showed that the tensile strength values of the raw cellulose acetate film and that of varying plasticizer loading increased to a maximum peak at 30 wt% and then a downward trend was observed. The observed decrease results from the softening of the film which could be attributed to the reduction in intermolecular forces of the film due to the addition of plasticizer. The 30 wt% plasticizer loading has the highest average tensile strength of 0.077 MPa as compared to other cellulose acetate film with plasticizer loading of 10 wt%, 20 wt%, 40 wt% and 50 wt%. The 30 wt% obtained using dimethyl phthalate plasticizer is low when compared with optimal value of 1.11 MPa at 20 wt% diethyl phthalate plasticizer loading for films produced from rice straw [13]. Plasticizer added to polymer always plays an important role in its tensile strength [14, 15]. It is clear that the plasticizer influences the tensile strength of the cellulose acetate film significantly with the films becoming weaker beyond 30 wt% DMP added. This is expected because the plasticizer increases the mobility of the polymer chain, so that the mechanical strength decreases [16]. Hence, the variation

observed in the tensile strength of the film obtained when compared with that of [13] could be as a result of the difference in the plasticizer used, the strength of the films obtained from two difference sources and the weight of the plasticizer.

The indicated error bars show that the 30 wt% plasticizer loading has the least errors. This suggests that minimum variations existed within the films. The non-overlapping of the error bars is an indication that the tensile strength of the film for the entire respective plasticizer loading was significantly different from one another but not for 40 wt% and 50 wt% indicating they may exhibit the same properties.

### 3.2.2 Effect of dimethyl phthalate on elongation at break of film

The variation of the elongation at break of cellulose acetate film to the percentage weight of dimethyl phthalate is presented in Figure 4. It was observed that there was an improvement in the elongation at break of the plasticized film with their respective amount of plasticizer compared to the unplasticized cellulose acetate film. The 30 wt% plasticized cellulose acetate film exhibited the highest

extension at break of 3.4195 mm corresponding to 17.10% elongation. This extension is within the acceptable limit on the strength – elongation chart for polymer material [24] which indicates that the polymer is more ductile than the

unplasticised cellulose acetate film with just 1.1844 mm extension at break corresponding to just 5.59% elongation which suggest a weak and brittle polymer on the strength – elongation chart.

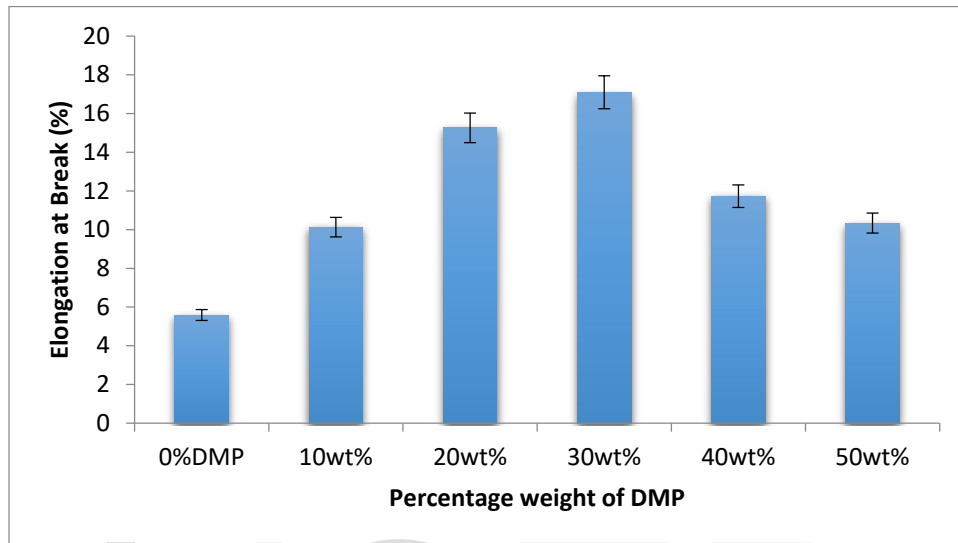


Figure 4: Effect of DMP on Elongation at Break of Film

The percentage elongation obtained for 30 wt% is higher than the values obtained for the 10 wt%, 20 wt%, 40 wt% and 50wt% of plasticized cellulose acetate film by factors of 59, 89, 68 and 60 respectively. The 10wt% plasticized film has the least extension at break which corresponds to 10.13%

elongation. The brittle nature of the film may have been responsible for this observation. The 17.10% elongation at break observed with the 30 wt% cellulose acetate film is higher than that recorded for rice straw [13].

### 3.3 Biodegradability Study of Cellulose Acetate Film

### 3.3.1 Weight loss

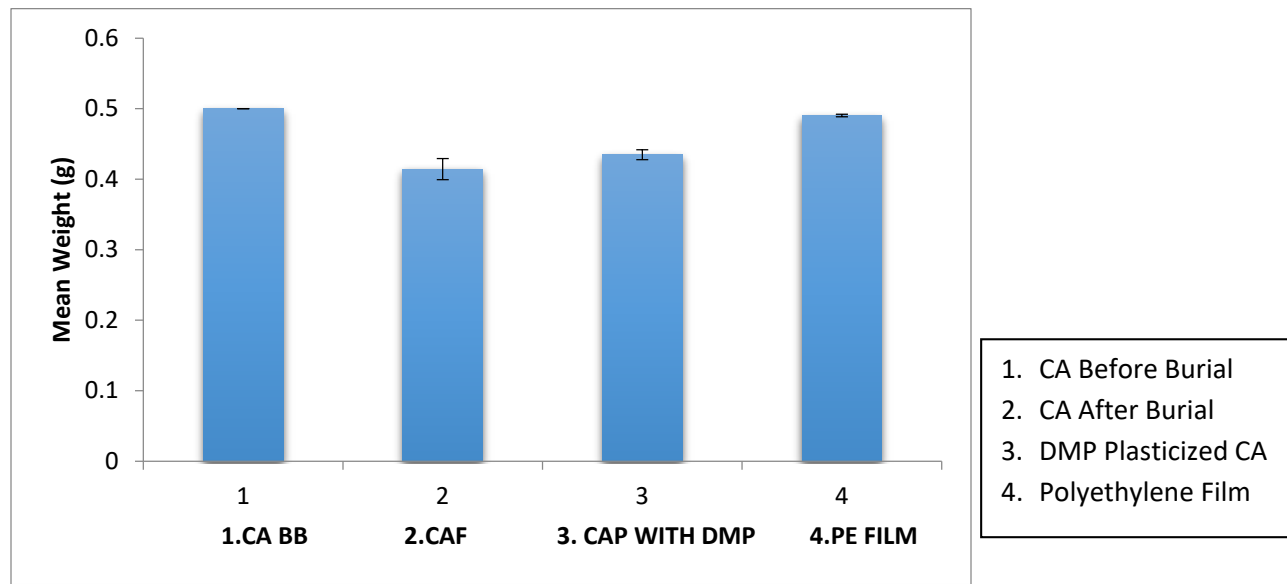


Figure 5: Mean Weight of Film after Degradation

Figure 5 presents the weight loss over time during burial of the film and the effect of weight loss on the film after burial in soil respectively. The result reveals a positive degradation for films with and without plasticizer as compared to that of polyethylene film. The percentage weight loss indicated that unplasticized cellulose acetate film had the percentage weight loss of 17.8% and that with plasticizer of 13% as compared to that of polyethylene film of 1.8% weight loss. The presence of the plasticizer may be responsible for the different rate of degradation in the cellulose acetate films. The plasticized film with higher tensile strength had lower rate of degradation which could be as a result of the plasticizer (dimethyl phthalate) type used not being biodegradable itself. Microorganisms naturally attacks polymers in the soil particularly on the surface of the polymer where they digest cellulose and leave a porous, sponge-like structure with high interfacial area and low structural strength [17]. Then the polymer matrix is degraded by enzymatic attack by soil microorganisms,

causing scission of molecules leading to reduction of weight of polymer.

### 3.3.2 Scanning electron microscope (SEM) examination

Effects used to describe degradation include roughening of the surface, formation of holes or cracks, de-fragmentation, changes in color, or formation of bio-films on the surface. These changes do not prove the presence of a biodegradation process in terms of metabolism, but the parameter of visual changes can be used as a first indication of any microbial attack. To obtain information about the degradation mechanism, more sophisticated observations can be made using scanning electron microscopy (SEM) [18].

After soil burial of the cellulose acetate film, the samples were examined using SEM machine and the micrograms are displayed in Figures 6,7,8 for cellulose acetate film without plasticizer, cellulose acetate film with plasticizer and polyethylene film respectively.

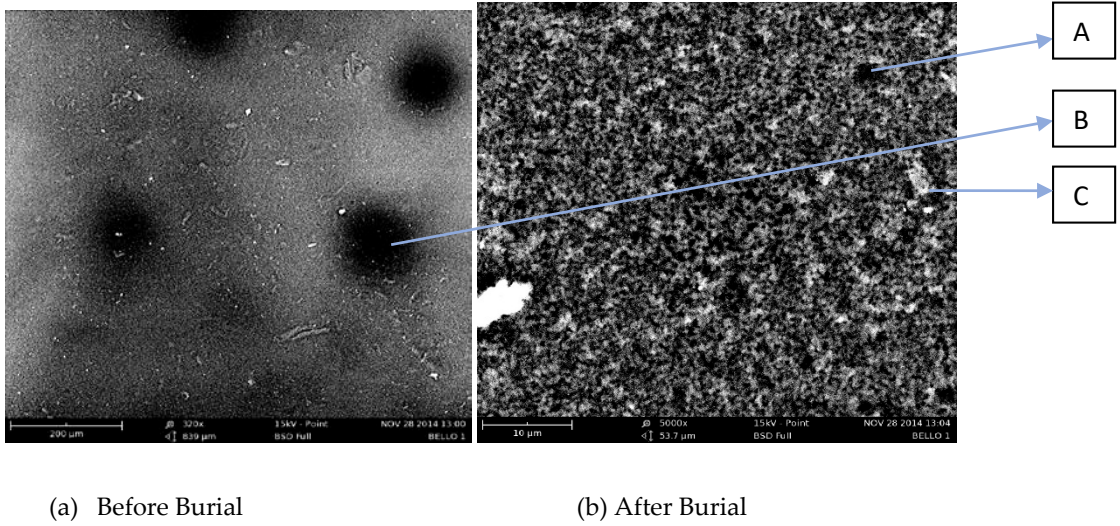


Figure 6: SEM images of Cellulose Acetate Film without Plactisizer

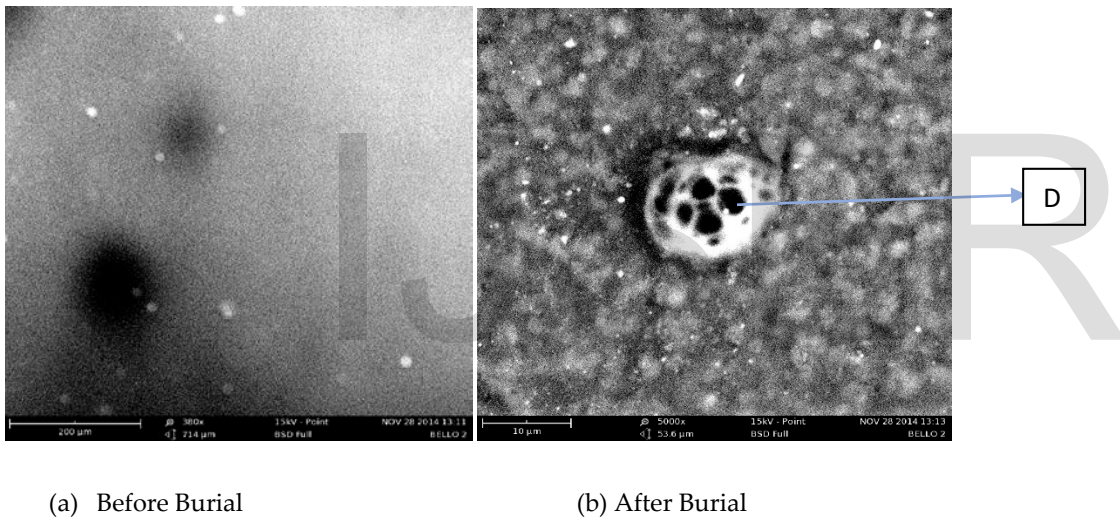


Figure 7: SEM images of Cellulose Acetate Film with Plactisizer (DMP)

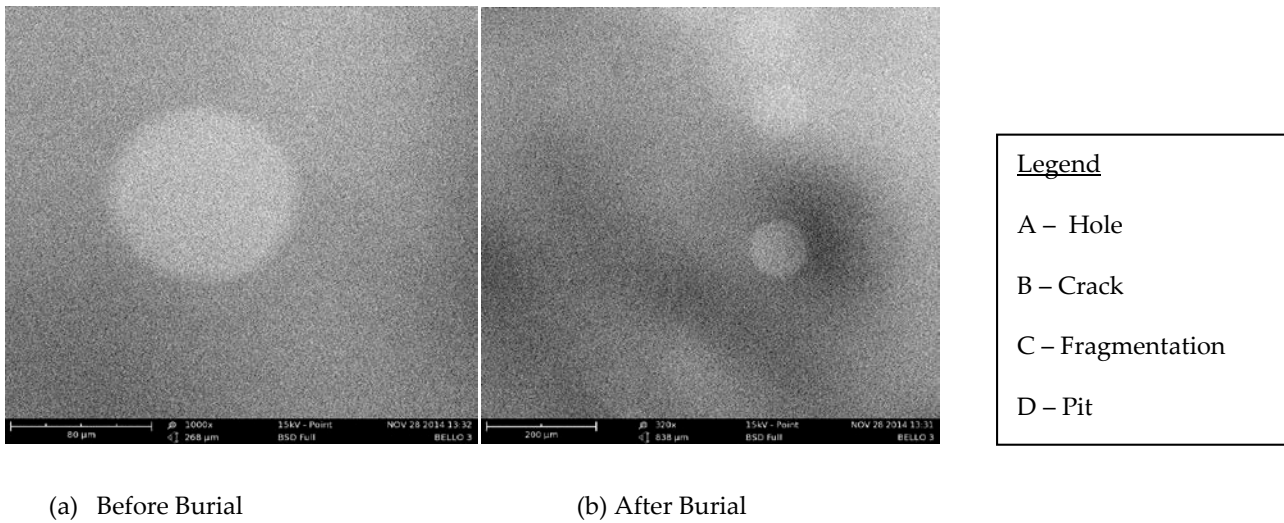




Figure 8: SEM images of Polyethylene Film

SEM was employed to analyze the surface changes in the cellulose acetate film samples. Figures 6,7,8 reveals the micrograph of cellulose acetate film without plasticizer, cellulose acetate film with 30 wt% Dimethyl Phthalate and Polyethylene film before and after burial respectively. A variety of degradation pattern resulting from structural changes were observed.

Figures 6a and b shows the microstructures of cellulose acetate film without dimethyl phthalate plasticizer. The micrograph reveals a relatively smooth surface in (a). It is suggested that smooth surfaces correspond to small vascular bundles embedded in the subepidermal sclerenchyma [19]. An irregular surface observed on the epidermis of the film (b) from the SEM images indicates that all cell of surface of the film have been completely destroyed with relative development of bacterial cells which adheres to the surface of the film and an attendant formation of pit as seen. The appearance of dark spot on the surface and formation of pit during examination is an indication of post molding molecular degradation taking place that is associated with microorganisms attack. This result is in agreement with the findings of Ohtaki *et al.* [7] and Milstein *et al.* [20] where prominent cracks were observed on the surface of samples when compost burial was performed on biodegradable and polyurethane plastics, indicating physical deterioration of the samples. Figure 7 shows the surface microstructures of cellulose acetate film with 30 wt% DMP plasticizer before and after burial. The image reveals that before burial in soil (a) a relatively smooth surface and some isolated areas that tend to suggest degradation as observed in the epidermis of the film. The observed morphology suggested a selective degradation of the film domain. When compared to Figure 7(b) a development of cracks which are indications of

environmental stress cracking and the examination revealed features that are characteristic of a brittle fracture but not as degrading when compared to Figure 6(b) which suggests a selective degradation of the film. This could be as a result of the DMP added which in its self is not biodegradable, thus it tends to inhibit the degradation tendency of the film. The absence of cracks or pit formation on Figure 8a and b reveals a non-microorganisms attack on the film after burial as the surface microstructure relatively remain smooth as it was before soil burial. This is an indication of a non-biodegradability of polyethylene film when compared to that of cellulose acetate film which is degraded over time due to microorganism attack on the morphology of the film. The non-degradation of the polyethylene film after burial is due to its extreme stable nature in any environment [21].

#### 4 CONCLUSION

Based on the results obtained from the study, the following conclusions are made:

- ❖ The results of FT-IR and XRD analysis revealed that a cellulose acetate of good characteristic properties can be developed by acetylation of cellulose obtained from cotton stalk. The development of peaks and spectra before modification and decrease in intensity of the peak and development of new spectra after modification from the XRD patterns and FTIR spectra respectively confirms the production of cellulose acetate which indicates a biodegradable polymer can be developed from cotton stalk.
- ❖ Plasticizer plays an important role in the mechanical properties of polymers as observed for cellulose acetate film with maximum values obtained at 30

wt% dimethyl phthalate. Within the limit of experimental error, the cellulose acetate film with 30 wt% DMP plasticizer was found to exhibit the highest mechanical properties with 0.077 MPa tensile strength and 17.2% elongation when compared with that of 1.11MPa for rice straw [7], thermoplastic starch (TPS) of 3.4 MPa [22] and corn gluten powder blended with LDPE of 6MPa [23], all used in packaging, is low which is an indication blending with either starch based biopolymers (PLA or PGA) or chitosan to enhance the mechanical properties of the film.

- ❖ The results of SEM images for the cellulose acetate film without plasticizer from the biodegradability study reveals an inexpensive and biodegradable cellulose acetate that can effectively replace raw materials from fossil fuels as starting material in polymer industries was developed due to the recorded 17.10% weight loss of film after burial when compared with that plasticized with 30 wt% DMP. The SEM examination reveals formation of cracks and pits which are indications of physical deterioration of film resulting from microorganisms attack from the soil on the unplasticized film.

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