



IMPROVED PROPERTIES OF CASSAVA STARCH-BASED FILM WITH CARBOXYMETHYL CELLULOSE FROM PLANTAIN PSEUDO-STEM

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Abstract: Films made from cassava starch are brittle and weak, leading to inadequate mechanical properties, thereby limiting its application. The properties of this starch film were modified by adding plasticizers and blending with carboxymethyl cellulose (CMC) biopolymers. The CMC was synthesized from cellulose extracted from plantain pseudo-stem through alkali hydrolysis followed by bleaching with sodium hypochlorite. A freshly prepared cassava starch (10% w/w) based films, plasticized with sorbitol (30 g/100 g starch) and various concentration of CMC (0, 10, 20, and 30% of the starch) were used to determine the effects of CMC on the properties of the films. The FT-IR spectrum confirmed the conversion of cellulose to CMC. Additionally, the FTIR confirms the intermolecular interactions between the cassava starch and CMC. The results showed a decrease in the water vapour permeation (WVP) and a reduction in the elongation at break (EB) of the film with increase in percentage of CMC. However, the tensile strength (TS) of the film increases as the amount of CMC increases.

Keywords: Starch, Carboxymethyl cellulose, elongation at break, tensile strength, water vapour permeation.

Introduction

In recent years, there has been an increase in the level of research on the development of new biopolymer-based materials for use in packaging, agriculture, medicine and other areas. Most plastic films used in packaging are derived from nonrenewable sources and are not degradable in the environment (Ma *et al.*, 2008). Research in this field has enjoyed a wide attention because biopolymer-based films could replace the use of some synthetic polymers in the food packaging industry and reduce the negative environmental impact associated with packages from non-renewable and non-recyclable resources (Tongdeesontorn *et al.*, 2011). Additionally, the buildup of plastics in our society is of major concern because of their harmful effects on the health (Proshad, *et al.*, 2018; Alabi *et al.*, 2019). A more sustainable approach to plastics is to make them from renewable materials such as plants. These bio-plastics can often be designed to be biodegradable, thus solving the environmental problems associated with synthetic films of petroleum products. The interest in biopolymer-based materials is further encouraged due to their low cost, availability of raw material and biodegradability (Rhim and Ng, 2007; Zhou *et al.*, 2008). Biopolymer films are generally produced from renewable natural and abundant biodegradable polymeric materials such as polysaccharides, proteins, lipids, or the combination of these components. Some films and coatings have been widely used for fresh fruits, vegetables, confectioneries, frozen foods, and meat products (Pavlat & Orts 2009; Ramos *et al.*, 2016; Swathi *et al.*, 2017). However, many films have limitations in mechanical or barrier properties or are prohibitively expensive. Biopolymer films formed from polysaccharide of starch often have good barrier properties to oxygen, carbon dioxide, and lipids and can protect products from lipid oxidation (Cutter, 2006; Hassan *et al.*, 2018).

Cassava starch (*Manihot esculenta* Crantz) or tapioca is one of the economically important crops in Nigeria and is the cheapest raw material for starch production (Jackson *et al.*, 2014). Structurally, cassava starch consists of 17% amylose content, and this is responsible for its strong film-forming characteristics (Bangyelean *et al.*, 2006). Cassava starch can readily be cast into films. However, the cassava starch film is brittle and weak, leading to inadequate mechanical properties. Overcoming the brittleness of the film can be accomplished by adding plasticizers (Vieira, *et al.*, 2011). Common plasticizers used in the production of starch films are water, glycerol, sorbitol, and other low-molecular weight polyhydroxyl compounds (Pouplin *et al.*, 1999; Laohakunjit & Noomhorm, 2004). Glycerol and sorbitol are widely used as

plasticizers because of their stability and edibility. The addition of plasticizers makes the brittle films more flexible, but also less strong (Suppakul *et al.*, 2006; Miller *et al.*, 2021) and results in higher moisture permeability (Kester *et al.*, 1986). This problem must be addressed to improve the functional properties of cassava starch films. Blending (Chandra *et al.*, 1986) or laminating (Coffin *et al.*, 1993) with other polysaccharide materials could improve cassava starch film mechanical functionality.

Carboxymethyl cellulose (CMC), xanthan, guar, and arabic gum, which are water-soluble heteropolysaccharides with high molecular weights, are often used together with starches to provide desirable texture, control moisture and water mobility, and improve overall product quality and/or stability (Li *et al.*, 2008). CMC is an anionic linear polysaccharide derived from cellulose. It is an important industrial biopolymer with a wide range of applications in flocculation, drug reduction, detergents, textiles, papers, foods, and drugs (Bisawal *et al.*, 2004). CMC is used primarily because it has high viscosity, is non-toxic, and is non-allergenic. The numerous hydroxyl and carboxylic groups in CMC enable water binding and moisture sorption properties (Kumsah, 1976; Tongdeesontorn *et al.*, 2011; De Wever, 2021). CMC hydrogel has a high-water content, good biodegradability and a wide range of applications due to its low cost (Nie *et al.*, 2004). The molecular weight and structure arrangement of CMC enhances its filling properties and thus it is been employed as filler in bio-composite films (Almasi *et al.*, 2010). Siró *et al.* (2011) study the effects of multiple homogenization steps on the properties of carboxymethylated softwood pulp. The morphological analysis of the study exhibited a diminution in fiber aggregates by increasing homogenization steps. Two or three additional homogenization increased elasticity, tensile strength and modulus of elasticity of films whereas oxygen permeability did not change significantly. They summarized that carboxymethylated cellulose gels had the potentiality to convert extremely transparent and oxygen barrier films.

CMC is able to improve the mechanical and barrier properties of pea starch-based films (Ma *et al.*, 2008). Some films based on mixtures of multiple polysaccharides such as starch-methylcellulose (Arvantoyannis *et al.*, 1999), pullulan-starch (Billaderis *et al.*, 1999), chitosan-starch and chitosan-pullulan (Lazaridou *et al.*, 2002), CMC-rice starch (Li *et al.*, 2008), and CMC-corn/cassava starch (Tavares, 2019) have been investigated. Plantain (*Musa paradisiaca*), a non-wood source of fiber, is an important food crop in the humid forest and mid-altitude agro ecologies of sub-Saharan Africa. This crop provides more than 25% of carbohydrate for about 70

million people and generates mega tonnes of agricultural wastes (Odum, 2021). The waste is a major source of fiber. Plantain pseudo-stem (PPS) a waste from plantain farm is a rich source of lignocellulose (Sunday, 2017) and it can be profitably utilized for numerous applications. PPS cellulose content is moderately high and tolerable, low lignin content and high percentage of ash and extractive content (Akpabio, 2012). PPS offer the advantages of being a cheap and readily available source of cellulose from which carboxyl methyl cellulose (CMC) can be synthesized.

In this study, CMC obtained from waste plantain pseudo-stem cellulose was integrated into the cassava starch films at various concentrations. The influence of varying concentration of CMC on the mechanical and water vapour permeability properties of the starch were investigated.

Materials and Methods

Materials

Plantain pseudo-stems and cassava starch were obtained from Tobstyl farms, Lagos and was identified at the Department of Plant Science, Olabisi Onabanjo University Ago-Iwoye. All chemicals for the preparation and characterization of CMC were used as received without further treatment.

Extraction of Cellulose

Cellulose was isolated from PPS using the method of Elanthikkal *et al.* (2010) and Alemdar *et al.* (2008) with some modifications. The PPS was cleaned and sun-dried for two days. Dried PPS was cut into small pieces. The delignification of cellulosic material was carried out by boiling 360 g of PPS with 500 ml of 10% sodium hydroxide (NaOH) in a reaction vessel for 2½ hrs at temperature of 95°C to 110°C. The residue was washed severally with distilled water until the filtrate becomes neutral to litmus paper. The final stage of the isolation involves bleaching of the cellulose obtained with 400 ml of 3.5% sodium hypochlorite at room temperature for 24 hrs.

Synthesis of CMC from PPS Cellulose

20 g of cellulose was weighed and added to 400 ml of isopropanol with continuous stirring. 100 ml of 18% w/v NaOH was added drop-wise into the mixture and further stirred for an hour at room temperature. The carboxymethylation was initiated when 12 g of monochloroacetic acid (MCA) was added with continuous stirring for another 2 hrs on hot plate at 50°C. The slurry was soaked in 300 ml of methanol overnight. The next day, the slurry was neutralized with 90% of acetic acid to pH 7 and then filtered using sintered funnel. The final product was washed twice by soaking in 300 ml of 99% ethanol for 10 mins to remove undesirable by-products and then washed again with absolute methanol. The obtained CMC was filtered and dried at 60°C to constant weight and kept in a dry place.

Film preparation.

Solutions used to prepare films were prepared by dispersing cassava starch (10% w/v) and CMC at different concentrations (0%, 10%, 20% and 30% CMC) in distilled water. Sorbitol (30% w/w solid) was added as the plasticizer. The film solutions were heated to 80°C with constant stirring to obtain starch gelatinization for 10 mins. The blend solution was evaporated at room temperature for 36 hrs and the films were obtained. The films were then peeled and kept in desiccator before characterization.

Fourier transform infrared (FTIR) spectroscopy

The functional groups of the present in cellulose, synthesized CMC and CMC/cassava starch blends were determined using Perkin Elmer Spectrum RXI Fourier Transform Infrared Spectrophotometer. The samples were dried in an oven at 60 °C to remove the moisture. About 0.2 mg of each sample and 2 mg of potassium bromide were mixed and ground finely before the mixture was compressed to form a transparent

pellet. The infrared spectra of these samples were measured in the transmission of a wavelength number range between 4000 and 500 cm⁻¹

Determination of Mechanical properties of Film

The mechanical properties examined included tensile strength (TS) and elongation at break (EB). The mechanical properties were determined using a Tinius Olsen H10KS-0748 light weight tester with the procedure according to the ASTM D638M (ASTM, 2002). The film samples were cut into 30 mm × 80 mm rectangular strips to be used for tensile testing. All CMC containing films strips were preconditioned for 48 hrs to 0 % relative humidity (RH) in a desiccator using silica gel at room temperature (25 ± 2°C) before testing. The thickness of each strip was measured using a micrometer with sensitivity 1 µm at five randomly selected points, which are the center and four corners. The initial grip separation and test speed was set at 100 mm/min. The tensile strength (MPa) was calculated by dividing the maximum load by cross-sectional area of the film. The percent elongation at break was express as percentage of change of the original length of a samples between grips at the break.

Determination of Water vapor permeability (WVP)

WVP tests were carried out using the standard ASTM D1653 (1995) with some modifications according to Mali *et al.* (2006). Special cups, with an average diameter of 4 cm and a depth of 5 cm were used to determine WVP of films. Films were cut into discs with a diameter slightly larger than the diameter of the cup. After placing 3 g of silica gel in each cup, they were covered with films of varying composition. Relative humidity (RH) was maintained using silica gel in the cup. Each cup was placed in a desiccator containing saturated NH₄NO₃ solution in a small beaker at the bottom. A small amount of solid NH₄NO₃ was left at the bottom of the saturated solution to ensure that the solution remained saturated at all times. Saturated NH₄NO₃ solution in the desiccator provides a constant RH of 97% at 25 °C. The desiccator was kept in an incubator at 25.0±0.1°C. Cups were weighed every 8 hrs and water vapor transport was determined by the weight gain of the cup. Changes in the weight of the cup were recorded as a function of time. The water vapor transmission rate (WVTR) was defined as the slope (g/h) divided by the transfer area (m²). WVP (g m⁻¹ h⁻¹ Pa⁻¹) was calculated as:

$$WVP = \frac{WVTR}{P(R_1 - R_2)} X \quad (1)$$

where P is the saturated vapor pressure of water (Pa) at the test temperature 25°C; R₁ = RH in the desiccator; R₂ = RH in the cup and X is the film thickness (m). Under these conditions, the driving force [P (R₁-R₂)] is 3073.93 Pa.

Solubility in water

Solubility in water was defined as the percentage of the dry matter of film which is solubilized after 24 h immersion in water (Gontard *et al.*, 1994). Varying mass of each film samples were immersed in beakers containing 50 ml of distilled water at room temperature for 24 h with periodical gentle manual agitation. The films were removed from the water and were placed back in the desiccator until they reached a constant weight to obtain the final dry weight of the film. The percentage of the total soluble matter (%TSM) of the films was calculated using the following equation:

$$\%TSM = \frac{\text{initial dry weight} - \text{final dry weight}}{\text{initial dry weight}} \times 100 \quad (2)$$

Results and Discussion

Yield of Cellulose & CMC

The obtained cellulose is white in colour and its texture is like that of wool. The yield of cellulose isolated from Plantain pseudo-stem (PPS) and the yield of CMC synthesized from the cellulose was measured based on dry weight basis. The yield of cellulose, expressed in percentage, was calculated

based on the amount of Plantain pseudo-stem used as stated in equation (3)

$$\text{Yield of cellulose (\%)} = \frac{WC_o(g)}{WPPS(g)} \times 100\% \quad (3)$$

Where WC_o and $WPPS$ are the weights of the extracted cellulose and weight of plantain pseudo-stem samples respectively.

The average percentage yield of cellulose obtained from the alkaline hydrolysis of plantain pseudo-stem in study is 45.7%. The percentage yield of CMC obtained in this study is 175%. The reaction of cellulose with monochloroacetic acid in alkaline condition led to the substitution of hydroxyl group of the cellulose molecule with carboxymethyl group and this is responsible for increase in mass after modification (Selke *et al.*, 2004). The result obtained are similar to previous studies on banana peel extraction (Yuliasmi *et al.*, 2019), sugar cane bagasse (Koh., 2013) and rice husk (Gupta *et al.*, 2020).

FTIR analysis.

FTIR spectroscopy technique provides information on chemical changes that occur during various chemical treatments of cellulose. The FTIR spectrum of the dried native cellulose, carboxyl methyl cellulose (CMC), starch film and cassava starch/CMC film blends are shown in Fig. 1

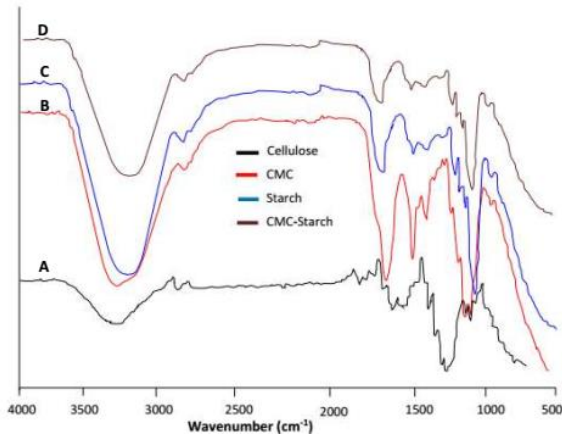


Fig. 1: FTIR spectra of (A) native cellulose (B) CMC; (C) cassava starch film; (D) CMC-cassava starch film.

For the native cellulose a broad absorption band at 3268 cm^{-1} is due to the stretching frequency of the OH-group, and a band at 2900 cm^{-1} is attributed to C-H stretching vibration (Biswal *et al.*, 2004). The bands around 1412 cm^{-1} and 1345 cm^{-1} are assigned to $-\text{CH}_2$ bending in plane and C-OH bending vibration respectively. Similarly, in the FTIR spectrum of CMC, the broad and intense absorption band at 3354 cm^{-1} is due to O-H stretching vibration of carboxyl group overlapping with $-\text{COO}$ group stretching (Tongdeesoontorn *et al.*, 2011; Su *et al.*, 2010). The band at 2917 cm^{-1} is due C-H stretching of the CH_2 group. The absorption band at 1584 cm^{-1} is assigned to asymmetrical COO^- stretching of CMC (Chen *et al.*, 2002; Yang *et al.*, 2009). The FTIR of CMC/cassava starch film with are shown display a similar characteristic to that that of the starch films. The importance of FTIR characterization is to confirm the homogeneity of the cassava starch and CMC films which is evident in the result obtained. The broad OH absorption band ranges between $3315\text{--}3286\text{ cm}^{-1}$ is present in all the films. The band attributed to COO^- group at 1584 cm^{-1} , water soluble group of CMC disappears in all films. Some of the bands in CMC have lower intensity in the blend due to interaction with the starch bands, (Tavares, 2019; Ma *et al.*, 2017; Tongdeesoontorn *et al.*, 2011). These FTIR results are in agreement with those reported for CMC-rice starch

(Suryiatem *et al.*, 2018), Chitosan corn starch gelation fthermoplastic (Mendes *et al.*, 2016), cellulose nanocrystals-cassava starch films (Ma *et al.*, 2017) and CMC-rice starch (Li *et al.*, 2008).

Tensile strength (TS) and Elongation at Break (EB)

The effect of CMC content on tensile strength (TS) and elongation at break (EB) of CMC/cassava starch blend films are shown in Figs.2 and 3, respectively. Fig. 2 shows that the tensile strength of cassava starch films was improved with the increase of CMC content. With increasing CMC concentration from 0 - 30%, the TS increased significantly from $110.0 - 178.5\text{ kgcm}^{-2}$. This was probably due to the formation of intermolecular interaction between the hydroxyl group of the cassava starch and the carboxyl group of CMC (Gupta *et al.*, 2020; Tongdeesoontorn *et al.*, 2011; Babak *et al.*, 2010).

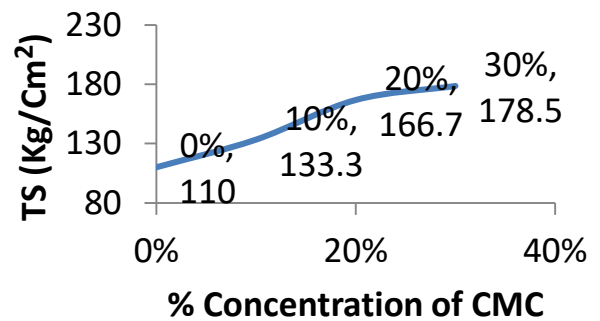


Fig. 2: Effect of CMC Concentration on Tensile Strength (TS)

According to Ghanbarzadeh *et al.*, (2010), during the process of blending and drying of the films, the hydrogen bond between starch molecules was replaced by the new hydrogen bonds formed between the hydroxyl groups in starch molecules and the carboxyl groups in CMC. Furthermore, other intermolecular interactions between cassava starch and CMC including dipole-dipole and charge effect resulted in a more compact molecular structure which causes an increased in TS (Li *et al.*, 2008). These results were consistent with cassava starch film (Tongdeesoontorn *et al.*, 2011), pea starch film (Ma *et al.*, 2008) and corn starch films (Ghanbarzadeh *et al.*, 2010), in which TS was improved as the concentration of CMC added in starch film formulation increases. Besides, the flexibility of the films was also affected by the blending composition. As shown in Fig. 3., the elongation at break (EB) of the film decreases as the CMC content in the cassava starch film increases.

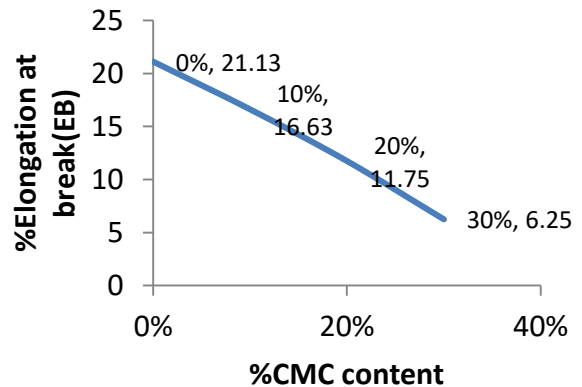


Fig. 3: Effect of CMC concentration on elongation at break (EB)

These could be related to structural modification of the starch network by water and CMC content, which causes a greater

flexibility in polymer structure (Rachtanapun 2009; Tongdeesontorn *et al.*, 2011). Aguirre-Cruz, *et al.* (2005) reported that CMC increases the viscosity of corn starch, due to the formation of three-dimensional network through CMC-starch association.

Water Vapour Permeability (WVP)

The effect of CMC content on the WVP of CMC/cassava starch films was investigated and the result is shown in Fig. 4. Film for food packaging is often required to have at least moisture transfer between the food and the surrounding atmosphere, thus WVP should be as low as possible (Ma *et al.*, 2008).

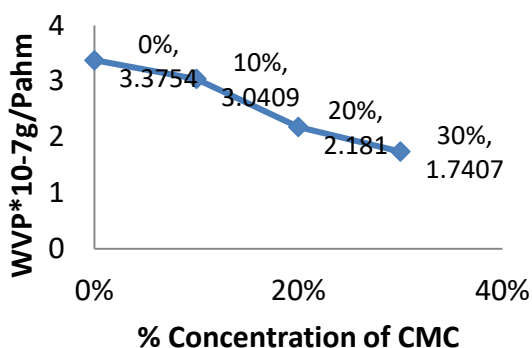


Fig. 4: Effect of CMC concentration on WVP.

WVP is a property that is directly associated to the hydrophilic nature of the film whereby more hydrophilic films will have greater WVP values, which mean lower water vapour barrier. The WVP for the cassava starch film was the highest with a value of $3.3754 \times 10^{-7} \text{gPa}^{-1} \text{h}^{-1} \text{m}^{-1}$, signifying that water vapour easily permeated the cassava starch. The WVP of CMC/cassava starch blend films decreased with increasing content up to 30% (w/w) of CMC from $3.3754 \times 10^{-7} \text{gPa}^{-1} \text{h}^{-1} \text{m}^{-1}$ - $1.7407 \times 10^{-7} \text{gPa}^{-1} \text{h}^{-1} \text{m}^{-1}$. This is because CMC acts as filler at low content, which disperse well in the cassava starch matrix and blocked the water vapor transmission. Chang *et al.* (2009), reported better water resistance of CMC than starch biopolymer. This could be due to highly crystalline and hydrophobic character of cellulose fiber in comparison to starch polymer. Reduction of the WVP with increasing CMC content results in an improvement of the functional properties of these films, considering the hydrophilic characteristics of the matrix (Arvanitoyannis & Biliaderis, 1999; Ma *et al.*, 2008).

Solubility in water

Solubility in water is an important property of starch-based films. Potential applications may require water insolubility to enhance product integrity, moisture barrier properties and shelf-life. The water solubility of cassava starch film blends depended on the CMC concentration (Fig. 5). The water solubility of the control cassava starch film was about 75%, and film solubility decreased with the increase concentration of CMC in the film. The decrease in solubility with addition of CMC indicates that intermolecular interaction occurred between starch and CMC in the cassava starch-CMC films. The hydroxyl group and carboxyl group of CMC can form strong hydrogen bonds (Tavares *et al.*, 2019) and ester bonds (Arvanitoyannis *et al.*, 1999) respectively, with the hydroxyl groups on starch. This results in the improvement in the cohesiveness of the biopolymer matrix, thereby decreasing the water solubility (Alves *et al.*, 2006). Similar findings were reported for corn starch-CMC films (Ghanbarzadeh *et al.*, 2010) and corn starch-CMC nano-clay films (Almasi *et al.*, 2010).

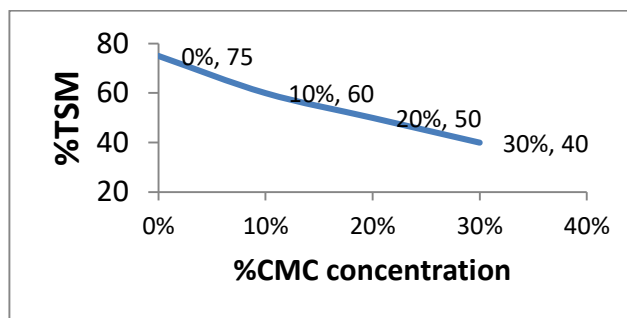


Fig. 5: Effect of CMC concentration on films % Total soluble matter (TSM).

Conclusion

45.83 % of cellulose was extracted from plantain pseudo-stem through alkali treatment followed by bleaching with sodium hypochlorite. The FTIR spectrum confirmed the conversion of cellulose to CMC by the presence of C=O group at wavenumber of 1584cm^{-1} . The CMC synthesized was blended with cassava starch film at various concentrations and the homogeneity of the samples was confirmed by the FTIR analysis. The water vapour permeation (WVP) decreases as the concentration of CMC increases in the film samples. The mechanical tests show that the Tensile strength (TS) increase across the samples as elongation at break (EB) reduces. This study has been able to utilize plantain pseudo stem, an agricultural waste as a useful source of cellulose derivative. In addition, the cassava starch-CMC composite film has the potential to replace conventional packaging films, thus reducing the environmental problem associated with synthetic films.

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