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Jeya Jeevahan, M. Chandrasekaran, and Akshay Sethu

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Influence of Nanocellulose Addition on the Film Properties of the Bionanocomposite Edible Films Prepared from Maize, Rice, Wheat, and Potato Starches

Jeya Jeevahan[1,b)], M.Chandrasekaran[2,a)] and Akshay Sethu[3,c)]

[1] *Research Scholar and Assistant Professor, School of Mechanical Engineering, Sathyabama Institute of Science and Technology, Chennai, India.*
[2] *Director, School of Mechanical Engineering, Vels Institute of Science, Technology and Advanced Studies, Chennai, India.*
[3] *UG Scholar, School of Mechanical Engineering, Sathyabama Institute of Science and Technology, Chennai, India.*

a)Corresponding author: chandrasekar2007@gmail.com
b)jeya.jeevahan@gmail.com
c)akshay.s.nambiar1998@gmail.com

Abstract. The starch based edible films have attracted much attention as the alternative food packaging materials to synthetic plastics due to their properties, such as, high transparency, odorless, biodegradability, tasteless, and semi-permeability to gases and food additives. However, the starch based edible films produced from native starch exhibit poor water resistance and poor mechanical strength. Incorporation of nanomaterials in starch films is considered as an effective method in recent times in order to improve the mechanical and barrier properties. Present investigation focused on the effects of nanocellulose on the film properties of edible films produced from different starch sources, such as, maize starch, rice starch, wheat starch and potato starch. The starch edible films were solution cast by adding 5g/100 ml water, 30 wt% of solution and varying concentrations of nanocellulose (0%, 2%, 5%, 10% & 15% of solution). Water vapor transmission rate (WVTR), mechanical strength (tensile strength, Young's modulus and percentage of elongation) and film color were determined for the films. The results showed that the addition of nanocellulose had improved the film properties in all the cases, and it could be used as a promising choice for food packaging applications. Among the four starch types, the nanocellulose incorporated edible films produced from potato starch and rice starch showed better film properties compared to those produced from maize starch and wheat starch.

INTRODUCTION

The term 'edible packaging' refers to a thin layer either formed directly on food surface or formed separately for wrapping over the food surface later. When the thin layer formed directly on the food surface, it is called edible coating. When the thin layer is formed separately and wrapped later over the food surface, it is called edible film [1]. Because these edible films and coatings are produced from the edible polymeric materials, the edible packaging can be consumed by human or living organisms along with the food items or beverages. Even when the edible packaging is not consumed, the degradation is much faster as compared to synthetic or biodegradable plastics that seriously caused waste disposal problems and the related health issues. Therefore, edible packaging films and coatings have attracted much attention as an alternative to synthetic and biodegradable plastics [2]. Edible films and coatings are produced from any of the type of edible polymers or their composites. The edible polymers are categorized into three types, such as polysaccharides, proteins, and lipids. The type and origin of the edible polymers have great influence on the film properties. Polysaccharides have good barrier properties to gases, but they have poor resistance to water vapors. Proteins have good mechanical strength, but they also have poor resistance to water vapors. Lipids, on the other hand, have good resistance to water vapors, but they do not form self-supporting films. Hence they are

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used as additives along with either polysaccharides or proteins in order to improve the resistance to water vapours and to provide glossy appearance [3].

Starch is a major energy source that constitutes about 50–70% of the human diet. The world production of starch is about 60 million tons of which 60% of starch is used for food applications and the remaining is used for non-edible and pharmaceutical applications [4]. Among various polysaccharides, starch based edible films have many advantages, such as, transparent, colorless, tasteless, odorless and non-toxic, which make them potential candidates for making edible films. However, the edible films produced from the native starches offered poor resistance to water vapors, poor solubility, and poor mechanical properties [5-7]. Polymer blending or multi-layers are the general options to improve the barrier properties and mechanical properties associated with high production cost, materials cost and difficult recycling process. However, with the used of nanotechnology, efficient monolayer edible films can be produced with less production cost, materials costs and easy recycling [8-10].

Formation of the nanocomposites is a promising method to improve the mechanical and barrier properties of the polymers. When the nano sized fillers or additives are dispersed into polymer matrix, the resulting films are termed as nanocomposite films. These nanocomposite films display great mechanical performance and strong barrier properties [11-13]. Addition of nanofillers can also perform other food packaging functions, such as antimicrobial activity, oxygen scavenger, and biosensor, etc. [14-16]. Nanocellulose is kind of biopolymeric nanomaterials with amazing physicochemical properties, such as, highly transparent, low density (1.6 g/cm^3) and high strength (TS = 10 GPa and YM = 220 GPa). Its stiffness is higher than Kevlar fiber, whereas its tensile strength is higher than cast iron. It also has 8 times higher high strength to weight ratio than that of stainless steel. Hence, incorporation of nanocellulose can greatly influence the mechanical properties and barrier properties of starch based edible films [17-20].

Incorporation of nanocellulose in starch based edible films has been investigated by researchers. In the work of Silva et al. [21], the bionanocomposite films were produced from cellulose nanocrystals cassava starch based films. The addition of a small amount of cellulose nanocrystals (0.2 wt%) had improved the mechanical properties significantly (TS by 92% and YM by 400% as compared to control films). In the work of Ghanbari et al. [22], the nanocellulose reinforced corn starch bionanocomposite were produced through extrusion processing. The addition of nanocellulose improved the water absorption, thermal, and mechanical properties. In the work of Savadekar and Mhaske [23], the addition of cotton nanocellulose increased TS, reduced EAB and reduced WVTR and oxygen transmission rates of the thermoplastic starch films. In the work of Soykeabkaew et al. [24], corn starch-based bionanocomposite was prepared with the nano-sized bacterial cellulose. The bionanocomposite reinforced with >40 wt% nanocellulose had significantly increased the mechanical properties (YM by 106 times and TS by 20 times compared to the control films). In the work of Zhao et al. [25], out of three kinds of chitason edible films reinforced with carboxymethyl cellulose, nanocellulose and nanocellulose with microcalcium carbonate, the edible films reinforced with nanocellulose could produce the lowest WVP and lowest film solubility in water as compared to other films. Although the above-mentioned research findings showed that the incorporation of nanocellulose increased the mechanical performance and barrier properties, the research work is limited to determine the effects of nanocellulose on various starch sources. Starch type of starch and sources significantly affect the film properties. For example, in the work of Tang et al. [26], the nanocomposite films were produced by incorporating montmorillonite clays (0–21 wt%) with three different starch sources, such as, corn, wheat, and potato. and). The results showed that the bionanocomposite showed 15–92% higher TS and 22–67% lower WVP as compared to the control films. However, such comparative analysis is not found on nanocellulose addition. This article attempts to provide a comparative analysis of the effects of nanocellulose on the film properties of four different starch sources, such as, maize, potato, rice, and wheat.

MATERIALS AND METHODS

Materials

Banana pseudo stems were obtained from the local banana farms, Chennai. Starch powders (maize starch, potato starch, rice starch and wheat starch), glycerol (plasticizer), and other chemicals (sodium hydroxide, sodium hypochlorite and sulphuric acid) were purchased the local chemical shops, Park Town, Chennai.

Isolation of Cellulose

Cellulose was isolated through the isolation method described in Elanthikkal et al. [27] and Mueller et al. [28] from banana pseudo stems. The banana pseudo stems were first cut into small pieces for drying in sunlight. After drying for about a week, the dried pieces were pretreated using a miller, alkaline solution treatment and bleaching treatment. The small pieces of banana pseudo stems were milled in order to obtain fine powder of about 500 µm. Alkaline treatment is required in order to remove pectin and the hemicellulose, where the milled banana stem powder was poured into an aqueous sodium hydroxide (NaOH) solution (3 M, i.e. 120 g per 1 liter of water) kept at 80 °C for about 4 h. The alkaline treated banana powder was subsequently washed with de-ionized water to remove the unwanted components. After repeating the alkaline treatment and washing cycle twice, the banana stem pulp was bleached to remove lignin. In this process, the banana stem pulp was poured in sodium hypochlorite (5 wt% of NaOCl per 100 mL volume of water) solution for at 45 °C for about 2 h so as to precipitate lignin. The resulting cellulose pulp was washed in de-ionized water until the pH value reached 7 and could be used for nanocellulose extraction.

Synthesis of Nanoellulose

Acid hydrolysis method is used to extract nanocellulose from banana pseudo stem pulp. The procedure, described by Beck-Candanedo et al. [29] with slight modifications, was used to isolate the nanocellulose. The cellulose pulp (acid-to-pulp ratio of 8.75 mL/g) is mixed in the sulfuric acid (64 wt%) solution at 45 ◦C for 30 min. After acid hydrolysis reaction was complete, the acid hydrolyzed suspension was diluted and washed in ice in distilled water. The resulting nanocellulose suspension was centrifuged at about 5000 rpm for 25 min at 10 °C. After a final wash with ultra-pure water to reach a pH value of about 7, the concentrated nanocellulose suspension was ultrasonicated for about 5 min in order to obtain the colloidal cellulose nanoparticles, which was filtered using filter papers of about 20 µm pore size. The resulting nanocellulose suspension was freeze-dried for about 1 day and stored before use.

Film Preparation

The aqueous solution of starch (5 g of starch per 100 mL distilled water) was first prepared. Food grade glycerol (30g of glycerol/100 mL of starch solution) and varying concentrations of nanocellulose (0%, 2%, 5%, 10% and 15%) were added into the starch solution and mixed well with a hand stirrer. Edible films were then produced through solution casting method. In this method, the prepared solution of starch, glycerol, and nanocellulose was poured in a beaker and heated in a water bath maintained at 90 °C while stirred continuously using a mechanical stirrer. The continuous heating and stirring for about 30 min creates the gel-like solution due to gelatinization process. This film forming solution was cooled to about 40 °C and poured onto flat Teflon-coated plates. The solution was spread evenly using a glass roller, and dried in sunlight for about 3 days. After drying for 3 days, the produced films were peeled off from the Teflon-coated plates manually and stored at 23°C and 50% RH in environmental chamber before use.

Film Thickness

The thickness of the bionanocomposite films was measured with the help of a digital micrometer (Yuzuku brand with sensitivity = 0.001 mm). The film thickness was measured at 5 random locations in every film, and the average thickness is calculated. The average film thickness (in mm) was considered for determining barrier and tensile properties.

Water Vapor Permeability

The WVTR, of the bionanocomposite edible films, was determined using gravimetric method (upright dry cup method) based on ASTM E96 standard (Standard Test Methods for Water Vapor Transmission of Materials) with slight modifications. In this method, the test specimens were cut into circular pieces of 7.5 cm diameter. Each test specimen was then placed over the metal cup (brass) containing 100 ml distilled water in it and sealed with a gasket and clamped. Before the WVTR measurement, the initial weight and time were recorded. The weight of the test cup was determined with the help of a weighting balance (sensitivity = 0.001 g). The test cup was then placed in an environmental chamber at 50±3 %RH and 23±1 °C. The subsequent weight loss and recording time were periodically observed for every 3 hours. The number readings should be repeated unless the curve representing weight loss versus time becomes linear. WVTR was calculated from the slope of the straight line in the plot of weight loss versus time (Δm/Δt). The WVTR is calculated from the following formula:

$$\mathrm{WVTR} = \frac{\left(\Delta m / \Delta t\right)}{A} \tag{1}$$

where, WVTR is the water vapor transmission rate (g/m^2/day), Δm is the weight loss (g), Δt is the duration of weight loss and A is the exposed area of the test film (m^2) to the environment. At least 3 replicates were considered and the averaged WVP values were considered for the analysis.

Tensile Properties

The test specimens of 100 mm x 25.4 mm were cut from the bionanocomposite edible films and the mechanical properties were determined based on ASTM D882 standard (Standard Test Method for Tensile Properties of Thin Plastic Sheeting). Texture analyzer was used to determine the mechanical properties (TS, EAB and YM). Initial grip separation of 50 mm and the crosshead speed of 500 mm/min were set for the test. The microcomputer attached to it recorded the stress–strain curves. TS, YM and E were plotted by the computer based on the following formulas:

$$\mathrm{T}S = \frac{\text{Max. load before failure}}{\text{Initial area of cross section}} \tag{2}$$

$$\mathrm{YM} = \frac{\text{Change in stress}}{\text{Change in strain}} \tag{3}$$

$$EAB = \frac{\text{Change in length at break}}{\text{Original length}} x100 \tag{4}$$

Film Color

The film color is described by three color parameters, L, a, and b, where "L" represents the darkness-lightness (0 for black to 100 for white), "a" represents redness-greenness (-80 for greenness to 100 for redness) and "b" represents the blueness-yellowness (-80 for blueness to 70 for yellowness). The film color was determined using the digital photography method explained by Afshari-Jouybari and Farahnaky [30] and Farahnaky et al. [31]. The high resolution images of the test specimens were captured using a high resolution digital camera. The camera was set to 1000 dpi of resolution, 62 % of contrast and 62 % lightness, respectively. These digital images (in JPEG file format) were then opened in the Adobe Photoshop software (Version 8), where the color parameters (L, a and b) were observed in the 'LAB' mode of the Adobe Photoshop software. The white color (L*=96.74, a*=0.09, b*=2.20) was set as the standard background color so that the comparison can be made for all the bionanocomposite test films. The test procedure was repeated for at least 3 replicates and the averaged color parameters were considered for the comparative analysis.

RESULTS AND DISCUSSION

Water Vapor Permeability

The WVP of the edible films produced from rice starch, wheat starch, maize starch and potato starch are presented in Fig. 1. The results of plain edible films showed that WVTR of potato starch based edible films, followed by rice starch based edible films, were lower than those of wheat starch and maize starch based edible films. This trend was in good agreement with the results of Basiak et al. [32] in which the WVP of potato starch film (0.01x10^{-10} g/(m s Pa)) were found to be the least among other two edible films, namely, corn starch film (0.79x10^{-10} g/(m s Pa)) and wheat starch film (0.52x10^{-10} g/(m s Pa)). When nanocellulose was added, the starch based edible films dispersed with nanocellulose showed better water vapor barrier properties (lower WVP) as compared to that of the plain films. It was also observed that the WVTR decreased as the percentage of nanocellulose increased from 2% to 15%. Similar reduction of WVTR due to the addition of nanocellulose were noticed in the literatures about edible films produced from alginate [33], sugarcane baggasse [34], cassava starch [35], whey protein [36] and chitosan [37]. The reasons for the reduction of WVTR are as follows: Inclusion of nanocellulose act as obstacles to the diffusion of water vapors. Hence, the water vapor takes a long travel path due to increased number of blockings delaying the flow of water vapors. Due to the presence of hydroxyl group in starches, strong interaction of nanocellulose and the hydroxyl group was predominant. In addition, the structural and dimensional similarities of starch and nanocellulose make the edible films with lower defects and strong adhesion, leading to high resistance to diffusion of water vapors. Therefore, nanocellulose acts as the barrier to the flow of water molecules. Due to the incorporation of nanocellulose, WVP was reduced up to 68% in potato films, 40.1% in rice films, 16.6% in maize films, and 19.3% in wheat films. From the results, it can clearly be said that the addition of nanocellulose significantly improved the WVTR in starch based edible films.

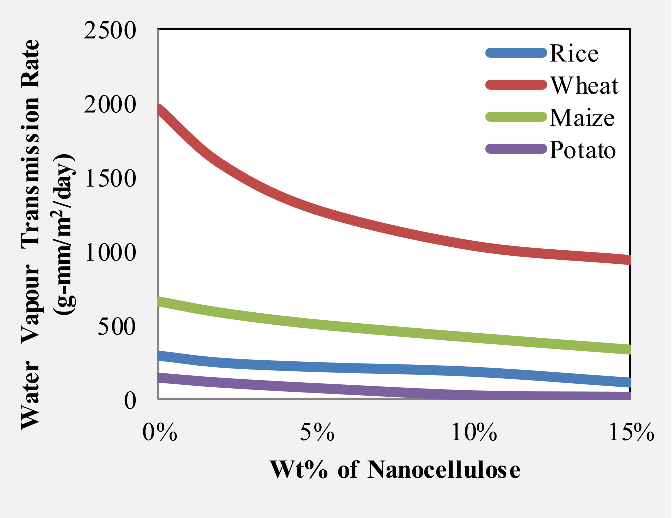

FIGURE 1. Effects of Nanocellulose on WVTR of the Starch Films

Tensile Properties

The tensile properties of the edible films (TS, YM and EAB) are presented in Fig 2, 3 and 4. Plain edible films of rice starch, wheat starch, maize starch and potato starch, showed TS of 9.1 MPa, 8 MPa, 15 MPa and 17 MPa with YM of 216 MPa, 227 MPa, 308 MPa and 343 MPa respectively, while EAB values were found to be 49%, 34%, 31% and 36% respectively. These values indicated that the films made from potato starch, followed by maize starch, showed higher mechanical strength as compared those made from wheat starch and rice starch. This trend was in good agreement with the work of Basiak et al. [32], in which the mechanical strength of potato starch film (TS = 6.56 MPa) were found to be higher than those of corn starch film (TS = 3.72 MPa) and wheat starch film (TS = 3.29 MPa). The addition of nanocellulose had increased both TS and YM, while reducing EAB. When the percentage of nanocellulose increased from 2% to 15%, the mechanical strength increased in all starch types. However, the increase in mechanical strength was not significant when the percentage of nanocellulose inclusion was 15%. The increase of mechanical strength due to the addition of nanocellulose was reported in literatures, where nanocellulose was dispersed in other polymer matrices, such as cassava starch [38], sodium caseinate [39], sugarcane baggasse [40], potato starch [41] and chitosan [42]. Therefore the results are in good agreement with the previously reported results. Due to similar chemical structure and dimensional similarity, strong interaction of nanocellulose with starch sources was achieved, which in turn increased the YM. Stiffness is defined the ratio of stress required to cause strain. As nanocellulose makes strong bonding, the stress required to cause deformation in the prepared edible films was higher. The surface area-to-volume of the nanoparticles is larger as compared to the microparticles or bulk material of the same material. Due to the high surface area-to-volume ratio of the nanoparticles, the interfacial regions are available for the matrix-filler interactions. Hence, the addition of nanocellulose increased the interfacial regions, which contribute to more stress transfer. As a result, the stress required to cause deformation in the prepared edible films was higher and hence the nanocellulose dispersed starch films showed higher TS and YM [43]. On the other hand, the elongation at break (EAB) decreased as the percentage of nanocellulose in the edible films increased. This was due to the brittleness caused with the addition of nanocellulose. Among the starch based edible films made, with nanocellulose addition, potato starch based edible films showed the highest mechanical strength (TS = 34.4 MPa and YM = 724 MPa), followed by maize films (TS = 29.1 MPa and YM = 601 MPa) and rice films (TS = 18.9 MPa and YM = 512 MPa), while the least mechanical strength was found in wheat based edible films (TS = 14.2 MPa and YM = 388 MPa).

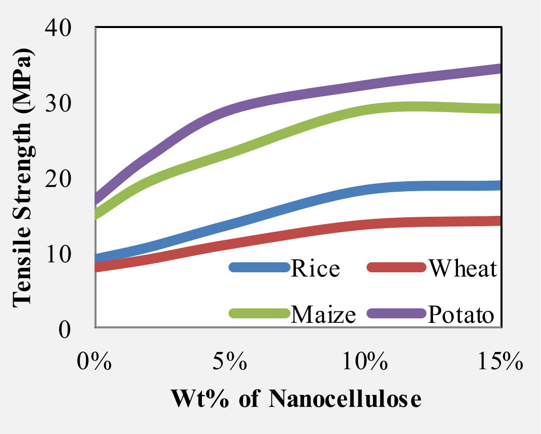

FIGURE 2. Effects of Nanocellulose on TS of the Starch Films

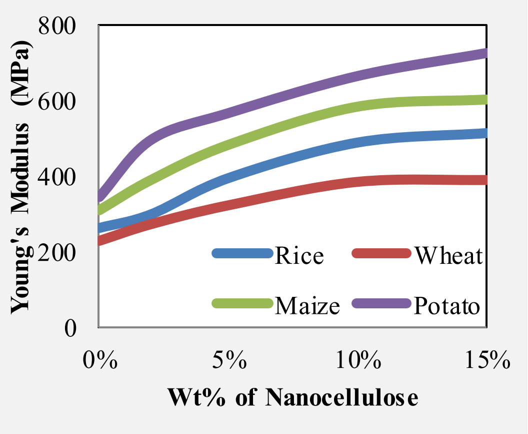

FIGURE 3. Effects of Nanocellulose on YM of the Starch Films

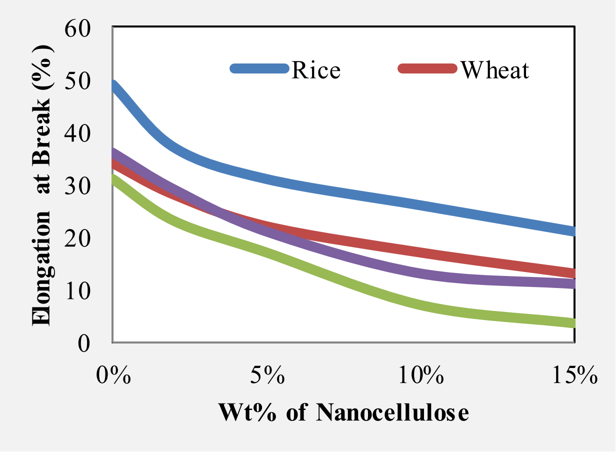

FIGURE 4. Effects of Nanocellulose on EAB of the Starch Films

Film Color

The variations in the color parameters (L, a, and b), for the prepared starch based edible films, are shown in Fig 5, 6 and 7. Micro-scale particles in a polymer matrix composite scatter more light, reducing the light transmittance as well as the optical clarity. On the other hand, when nanoscale particles with good polymer-nanofillers adhesion are added to the composite, the composite could eliminate the scattering effect. This can improve the transparency of the starch based edible films and coatings [44-46]. For instance, in the work of Yano et al. [47], the transparent thermoset polymer sheets were produced from different polymers epoxy, acrylic and phenol formaldehyde) with the reinforcement of 60-70 wt% nanocellulose. Even though high fiber content was added, a high degree of transparency was observed in the composite sheets. Similarly, the color parameters were not varied much due to nanocellulose addition in the film color of the starch films. However, there were variations due to the starch source type. In nanocellulose incorporated starch edible films, the ‘L’ & ‘b’ color parameters increased slightly, while the ‘a’ values slightly reduced. The increase of ‘L’ and ‘b’ values and reduction of ‘a’ values have been reported by Yu et al. [48], Reddy & Rhim [49], Shankar & Rhim [50], and Li et al. [51]. Hence, the values are in accordance with the literature data. The increased values of ‘L’ showed that the addition of nanocellulose increased the whiteness of the prepared films as compared to the plain films. The increase in ‘a’ indicated the greenness of the edible film, while the decrease ‘b’ indicated the increase of yellowness. However, the change in film color did not significantly vary. Therefore, the addition of nanocellulose improved the WVTR and mechanical strength of the file without affecting the film color.

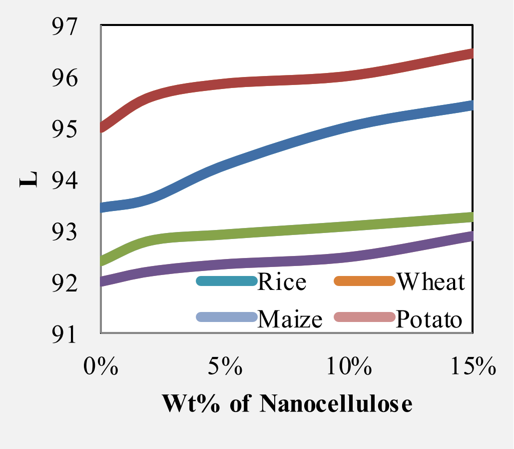

FIGURE 5. Effects of Nanocellulose on “L” values

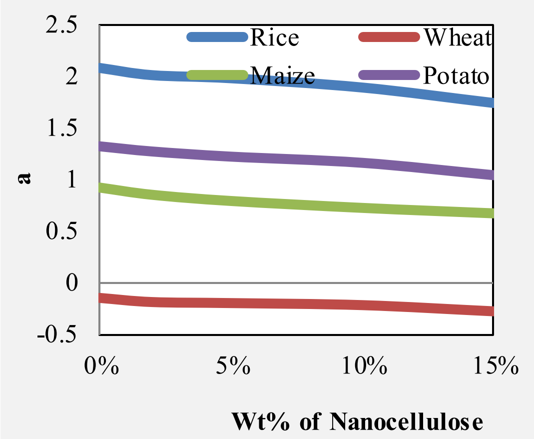

FIGURE 6. Effects of Nanocellulose on “a” values

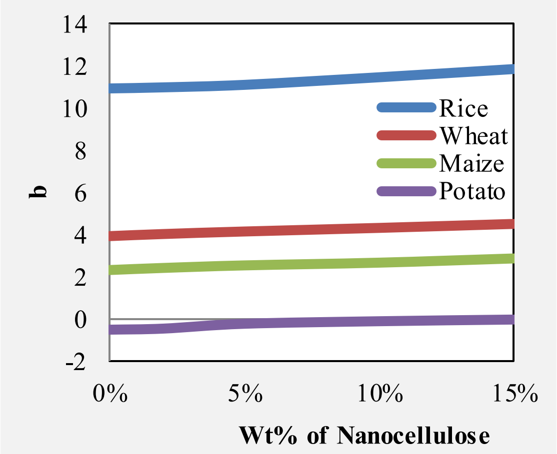

FIGURE 7. Effects of Nanocellulose on “b” of the Starch Films

CONCLUSION

In this work, nanocellulose was extracted from banana pseudo stems and the effects of nanocellulose addition on the starch based edible films were investigated. The starch based edible films were solution cast by adding starch solution from different sources, such as, rice, wheat, maize and potato, with varying concentrations of nanocellulose. WVTR, mechanical strength (TS, YM and EAB) and film color were determined for the films. From the results, the following observations were made: nanocellulose incorporated with starch films reduction the WVP of the starch films. Incorporation had increased the TS and YM of the starch edible films, while reducing EAB. Incorporation of nanocellulose had a slight effect on the film color of the starch based edible films. Among the four starch sources, potato and rice films had shown the highest strength and barrier properties compared to the maize and wheat starch films. On the whole, from the results, it was clear that the addition of nanocellulose had improved the WVP, tensile strength, and film color, making it a promising additive for making the starch based edible films suitable for food packaging applications.

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