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Improving the Performance of the Biocomposites Based on Starch and Rubber by Reinforcing with Cellulose Nanofiber

Md. Shamim Reza[1]*, Pran Krishna Das[2], Md. Minhaz- Ul Haque[2]
[1]*Dept. of Civil Engineering, North Western University, Khulna-9100, Bangladesh.*
[2]*Dept. of Applied Chemistry and Chemical Engineering, Islamic University, Bangladesh*

Abstract
Sustainable biodegradable material development has received great attention as an alternative to the petroleum-based plastic. The films of the biocomposite material were fabricated by solution casting technique in this study and the reinforcement was cellulose nanofibers (CNFs). The structural, mechanical, thermal, optical and moisture-related properties of the CNF loaded films were studied as a function of the different levels of loading (1–5 wt%). The tensile strength and Young's modulus were found to be 3.65MPa and 149.35MPa for starch/NR blend, respectively, and 5.42MPa and 180.05MPa for the composite of starch and 5 wt% CNF, respectively, with a tensile testing machine. The TGA suggested that with increasing CNF content, there was an increase in thermal stability. The moisture absorption decreased and optical transparency decreased due to the increase in the light scattering of the CNFs. The findings show that starch/NR biocomposites using CNFs as reinforcement are potential candidates for biodegradable packaging and sustainable applications, since it enhances the performance of the biocomposites.
Keywords: *Cellulose nanofibers, Starch/Rubber composites, Biodegradable film, Sustainable packaging matrials.*

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I. Introduction

The widespread use of petroleum-based plastics in packaging, agriculture and consumer products has raised significant environmental issues because they do not decompose easily and tend to persist in the environment for long periods [1]. Pollution and waste management problems are created by conventional synthetic polymers which take decades or even centuries to break down. Due to this, development of biodegradable, renewable, and sustainable alternatives that are not dependent on fossil fuel-based materials has come under the focus [2]. The biopolymer-based composites are being identified as the potential candidates for eco-friendly materials due to their biodegradability, renewability and possibility to adjust the material properties for various uses. Starch is one of the most abundant, cheap and available natural polymers in the world. It can be derived from many types of agricultural products such as corn, potato, rice, wheat and cassava [3]. Starch has been of great interest as a matrix material for the production of eco-friendly packaging films due to its biodegradability, non-toxicity, and film-forming property, as well as the production of biodegradable composites. Native starch has, however, some definite disadvantages such as low mechanical strength, brittleness, high moisture sensitivity, and low water resistance [4,5]. The disadvantages make it impractical to use directly in numerous industrial applications, so it must be modified or mixed with other materials to improve the way it performs. A good method to enhance starch-based materials is to blend starch with natural rubber. Natural rubber is a renewable elastomer consisting mainly of cis-1,4-polyisoprene, which is well-known for its properties of excellent elasticity, flexibility, resilience and mechanical properties [6,7]. The natural rubber incorporation in starch matrices can have a great effect on flexibility, toughness and elongation properties, keeping biodegradability of the material. Moreover, natural rubber latex is easily dispersed in aqueous starch solutions, so it can be easily processed to make homogeneous composite films by simple and cheap means [8,9]. Thus, starch-natural rubber blends have become more and more the subject of interest as promising biodegradable packaging materials, agricultural films and for other sustainable applications. Starch/rubber blends can have better flexibility than starch films, but the mechanical and thermal properties do not always meet the requirements for their practical application [10]. These constraints have led to the use of reinforcing fillers as a common technique to address them. CNFs are particularly appealing due to their superior mechanical strength, light weight, high aspect ratio, biodegradability and renewability, which make them of special interest for both scientific and industrial applications [11]. CNFs are obtained from lignocellulosic biomass and are highly crystalline, and can act as an efficient reinforcement of polymer matrices. They have a large number of hydroxyl groups which can form strong hydrogen-bonding interactions with starch molecules, which leads to better adhesion between the starch and the composite and better performance of the composite. Cellulose nanofiber use as reinforcing materials in biodegradable polymer systems is beneficial in several ways [12]. Small amounts of CNFs can enhance barrier properties, thermal stability, stiffness and tensile strength, and decrease water sensitivity and dimensional instability. In addition, by utilizing the agricultural residues or food processing waste as the sources for cellulose nanofibers, waste valorization and promotion of circular bioeconomy strategies can be achieved [13]. Nanocellulose derived from agricultural waste is also a more environmentally friendly material and is part of the current development trend towards more efficient use of resources in material production. Good dispersion of CNFs in the rubber matrix and their interactions with starch and rubber strongly affect the performance of starch/rubber/CNF biocomposites [14]. The uniform dispersion of cellulose nanofibers can achieve the effective transfer of stress between the matrix and reinforcement, and the mechanical properties can be improved significantly. Also, the thermal degradation behaviour, moisture absorption behaviour and water uptake properties of the resulting composites can be affected by the presence of nanocellulose [15]. Hence, it is important to know the correlation between composition, morphology and material property of biodegradable nanocomposite films to maximize the performance of the material. Many studies have reported in recent years the preparation of starch-based nanocomposites filled with cellulose nanomaterials [16]. However, studies on the starch/natural rubber-based composite systems filled with cellulose nanofibers are still relatively scarce. Furthermore, the synergistic effect of natural rubber and cellulose nanofibers on the morphology, thermal stability, moisture resistance and mechanical properties of biodegradable starch-based film is worth investigating. Developing high performance bio-based materials that can be used in real-world applications with a range of flexibility and mechanical strength makes these studies valuable. Hence, in the present study, the focus is on the preparation of biodegradable starch/natural rubber biocomposite films reinforced with cellulose nanofibers and assessment of their structural, mechanical, thermal and physicochemical characteristics. Simple solution-casting technique was used to prepare the composite films containing different concentrations of cellulose nanofibers using starch, natural rubber latex, glycerol and cellulose nanofibers. Scanning electron microscopy (SEM) was used to examine the morphology of the prepared films, and thermogravimetric analysis (TGA) was used to assess thermal stability. Mechanical properties were evaluated through tensile testing and moisture absorption, water uptake and optical transparency properties were also tested. The results of this research offer useful information on the performance of the starch/NR biocomposite reinforced by the addition of CNFs and promote the development of sustainable materials for future biodegradable packaging and other related applications.

II. Materials and methods

Materials

The polymer matrix was mainly starch and the elastomeric component was natural rubber (NR) to enhance film flexibility and toughness. High aspect ratio and high mechanical strength were the properties that made CNFs obtained from the lignocellulosic biomass suitable as reinforcing nanofillers. Glycerol was used as a plasticizer in order to increase the flexibility and decrease the brittleness of the film. To facilitate starch gelatinization and enhance the film forming ability, acetic acid was used. Distilled water was used as the solvent for preparing all film-forming solutions. All chemicals and materials were used as received without further purification.

Methods

Solution-casting method is used to prepare starch-based nanocomposite films. First, the starch was dispersed in distilled water and then stirred and heated at 80-90 °C until complete gelatinization was completed to obtain a homogeneous solution. A plasticizer, glycerol, was then added, followed by the addition of acetic acid for better dispersion of starch and film formation. Natural rubber latex was slowly added to the gelatinized starch solution and the mixture was stirred to ensure a uniform starch-rubber mixture. The cellulose nanofibers were first dispersed individually in distilled water and then vigorously stirred to reduce agglomeration. The CNFs were then incorporated into the starch/NRL mixture at various loadings (1 to 5 wt%) and stirred constantly in the mixture to ensure uniform distribution in the matrix. The obtained film forming solution was poured on to clean Petri dishes and dried at room temperature to get flexible films. SEM was used to study the morphology of the films. Thermal stability was analyzed by TGA and tensile properties were determined by universal testing machine. The effects of CNF incorporation on the morphological, mechanical and thermal properties of the starch/natural rubber biocomposite film were systematically investigated.

III. Result and Discussions

The TG and DTG curves of starch, starch/NR (60/40), and CNF-reinforced composites are presented in Figure 1, which shows an initial evaporation of moisture for all the samples at temperatures below 150 °C. The major degradation happened in the temperature range from 200 °C to 400 °C, which is the starch, glycerol, natural rubber, and cellulose components decomposition temperature. The composites showed higher thermal stability with the incorporation of the CNFs due to the shifting of degradation to higher temperature. The thermal stability and char residue of the composite with 5 wt% CNF was the highest, suggesting a greater resistance to thermal degradation than that of the neat starch and starch/NR blend films, and indicating strong interfacial interactions between the CNF and the starch matrix.

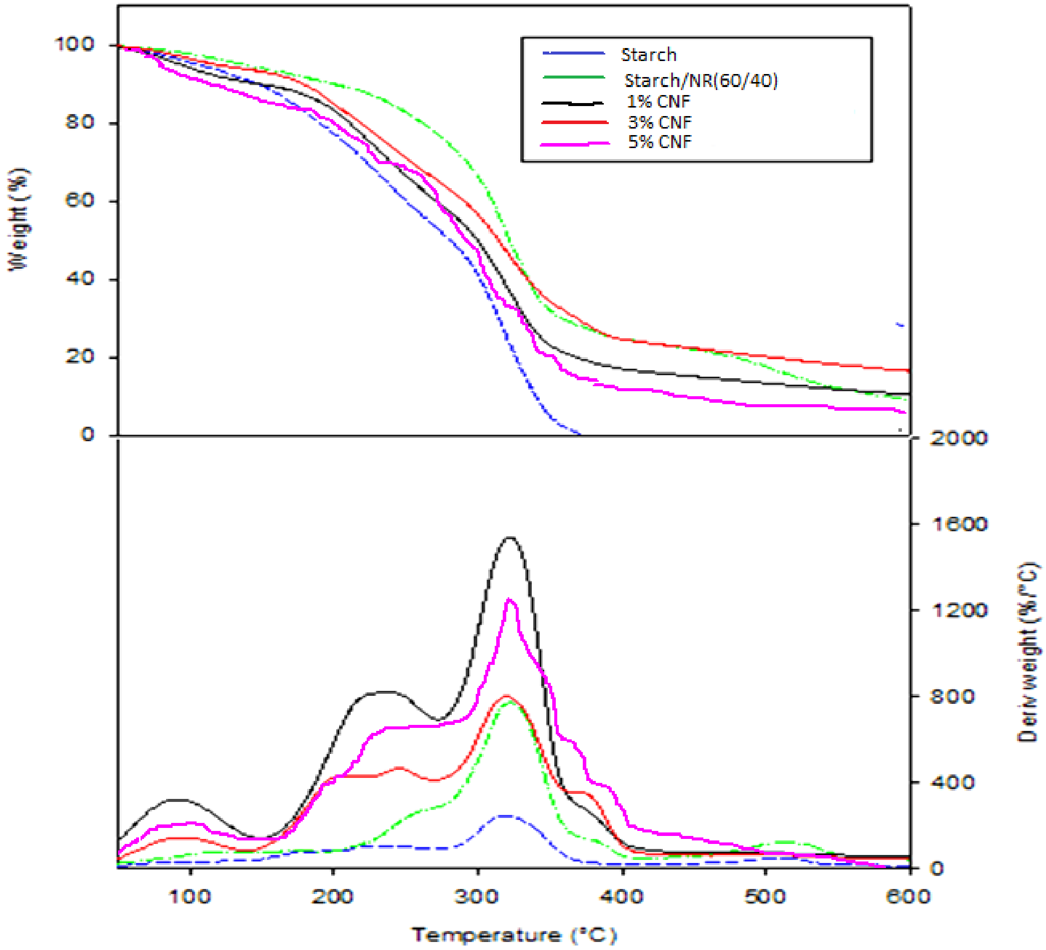

Figure 1: TG and DTG curve of Starch, Starch/rubber film and starch/rubber/CNF based composites.

Tensile mechanical test was carried out of the dried starch film, starch/NR blend films and its composites with cellulose nano-fibers. The relatively large aspect ratio and high elastic modulus of CNF should have a significant reinforcing impact on the mechanical properties of the polymer. [17] The tensile properties of the neat starch, starch/NR blend and starch/NR/CNF films were investigated by tensile tests and the obtained stress-strain curves are presented in Figure 2. From the stress-strain curves, the elastic modulus (Young's modulus), tensile strength and elongation at break of the films were extracted and presented in Table 1.

Table 1: Tensile modulus, tensile strength and elongation at break % of starch, starch/NR and starch/NR/CNF composites.

Materials	Tensile strength (MPa)	Young modulus (MPa)	Elongation at break (%)
Starch film	2.85	141.87	35.08
S/NR blend	3.65	149.35	31.11
1% CNF	3.89	156.27	28.52
3% CNF	4.42	169.26	26.24
5% CNF	5.42	180.05	23.65

According to these results, it is clear that the selected tensile properties of starch were affected by its blending with NR and then by the addition of different contents of CNF. The neat starch film exhibited an elastic modulus of 141.87 MPa, a tensile stress of 2.85 MPa and an elongation at break of 35.08%. When starch was blended with NR, tensile strength and the elastic modulus were decreased from 2.85 to 3.65 MPa and from 141.87 to 149.35 MPa, respectively. The elongation at break of starch was not affected in the starch/NR blend (Table 1). This variation in the tensile properties of starch was not surprising because of the lower tensile properties of NR biopolymer with respect to the starch biopolymer. With respect to the starch/NR blend film, the elastic modulus and tensile strength of the starch/NR/CNF bio-nanocomposites film increased gradually with increasing of CNF content from 1 to 5 wt%. When 5.0 wt% CNF was added (Starch/NR/CNF-5.0), the elastic modulus and tensile strength were increased. Such improvements confirmed that Starch/NR/CNF bio-nanocomposites film is mechanically strong materials. The elongation at break of Starch/NR was not largely affected by the addition of CNF and decreased, indicating that the Starch/NR/CNF bio-nanocomposites film are mechanically flexible materials. The main reason for the improvement of the mechanical properties is the strong interfacial interactions between the Starch/NR blend matrix and CNF, which was due to the high contact area exposed on the CNF and their functionalized surfaces.

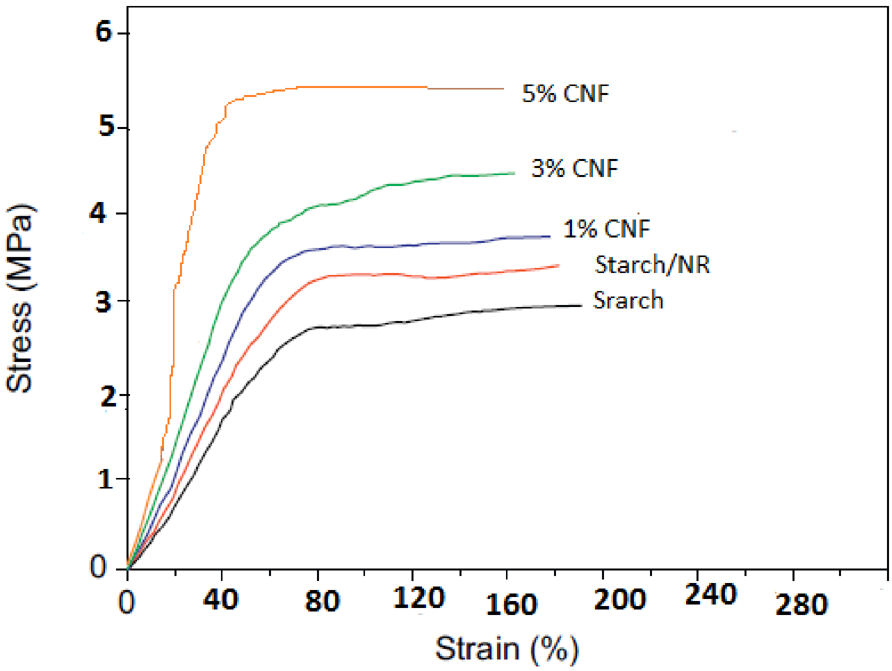

Figure 2: Stress-strain curves of starch, starch/NR blend and CNF-reinforced biocomposite film with improved strength and stiffness.

Both in the figure 2 and in the table 1, it was seen that the strength and modulus of Starch/NR blend films were improved by the addition of cellulose nanofibers. On the other hand, elongation at break % decreased as compared to starch film. The young's modulus of elasticity increased with the addition of cellulose nanofibers content due to the higher modulus of cellulose nanofibers [18,19]. The composite containing 5 wt %

cellulose nanofibers had the highest modulus of elasticity. The modulus of elasticity increased from 149.35 to 180.05 MPa in composites.

Neat starch, starch/NR blends and starch/NR/CNF biocomposite films were studied by UV-Vis spectroscopic analysis in the range of 200–800 nm shown in figure 3. The absorbance of all the samples decreased with an increasing wavelength. The use of natural rubber (NR) slightly reduced transparency and raised absorbance of the films compared with neat starch. The CNFs also improved the absorbance and decreased transmittance, primarily because of higher light scattering of the cellulose nanofibers dispersed in the matrix. The transmittance values dropped from 71% to 47% for starch/NR blend and to 33%, 18%, and 12% for composites with 1, 3, and 5 wt% of CNF, respectively. The decrease of the transparency at higher CNF loading levels suggests the presence of localized CNF agglomeration or non-uniform dispersion of the CNFs in the starch/NR matrix, as the higher loading of CNFs causes stronger light scattering effects.

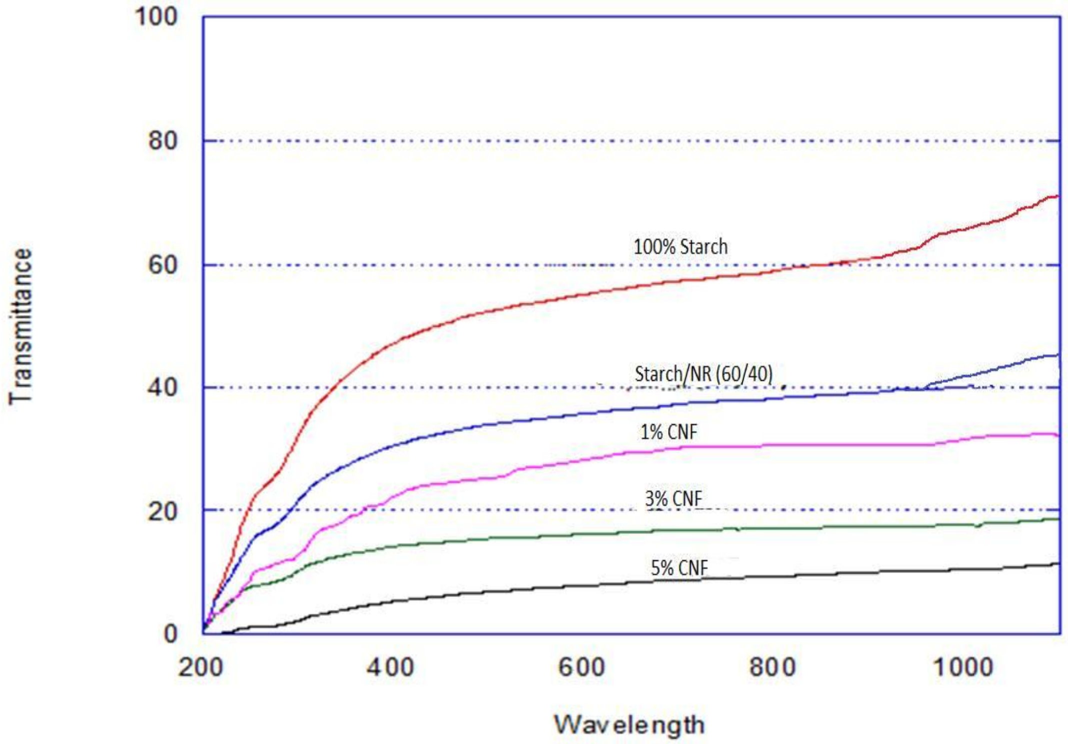

Figure 3: The transmittance spectra of starch, starch/NR blend and CNF-reinforced biocomposite films with reduced transparency in the UV-Vis region.

The moisture absorption and water uptake of Starch, Starch/NR blend and Starch/NR/CNF biocomposite films are summarized in Table 2. The moisture absorption value for neat starch film was 2.253% and the water uptake value was 1.0523%. Water absorption was slightly decreased (2.125%) and water uptake was slightly increased (1.2742%) when starch was blended with NR. The moisture absorption reduced gradually from 1.852% to 1.372% with the increase in the CNF content from 1 to 5 wt%, which showed that the moisture absorption resistance of the materials gradually increased. The water uptake of the composites, on the other hand, rose with the increasing CNF loading from 1.4826% to 1.6354%. The drop in moisture absorption could be due to a higher interaction at interfaces between CNF and the starch/NR matrix that hinder moisture diffusion. But the hydrophilic characteristic and the hydroxyl groups of CNFs facilitate the permeation of water, with the water uptake increasing with increased CNFs content.

Table 2: Moisture absorption and water uptake (%) of Starch and Starch/NR based composites.

Weight (%)	Moisture absorbance %	Water uptake %
Starch film	2.253%	1.0523%
Starch/NR film	2.125%	1.2742%
1 wt % CNF composites	1.852%	1.4826%
3 wt % CNF composites	1.643%	1.5647%
5 wt % CNF composites	1.372%	1.6354%

From the visual observation of films, it was noted that film of 100 wt % Starch was cracked and not continuous due to break down of glycoside bond of starch, but films of starch and acetic acid were less cracked than film of 100 wt % starch. For the film of 95 wt % starch and 5 wt % acetic acid, continuous with little cracked films were found in figure 4. For the films of starch, glycerol and acetic acid, continuous with little cracked films were also found. Continuous and uncracked films were obtained when NR and cellulose nano-fiber was added. Because, cellulose-nano-fiber binds starch and reduces free volume of starch. But films of Starch/NR blend were continuous but less transparent than film of 100 wt % Starch. For the film of Starch/NR/CNF in 1, 3, 5 wt % composition was also obtained continuous films.

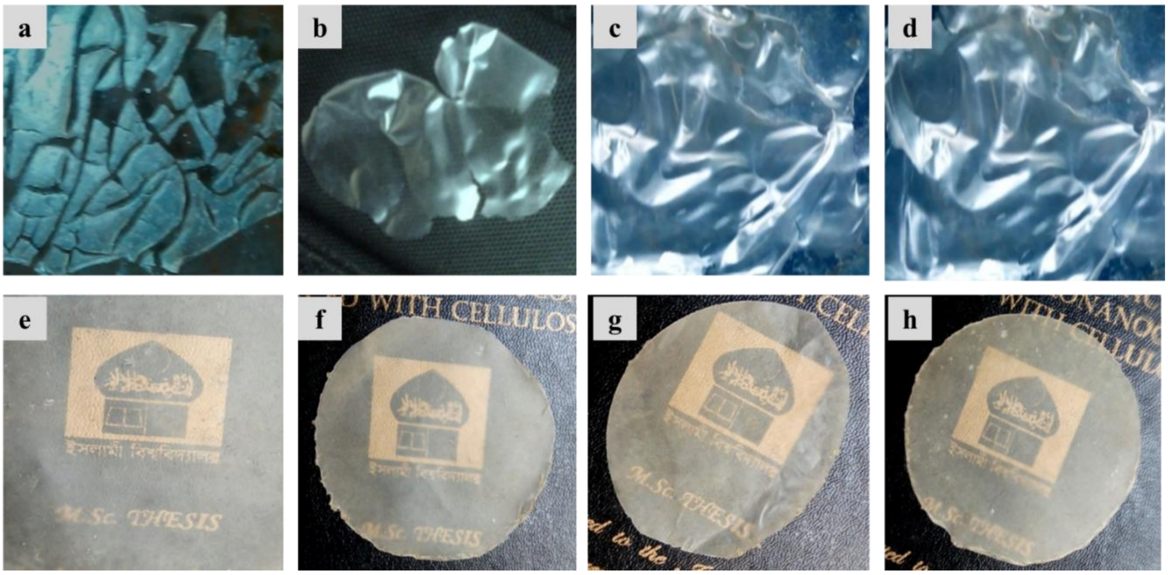

Figure 4: Appearance of the prepared films: (A) starch film, (B) 90% starch/10% acetic acid film, (C) 95% starch/5% acetic acid film, (D) starch–glycerol–acetic acid film, (E) starch–NR blend film, (F) starch/NR/1% CNF film, (G) starch/NR/3% CNF film and (H) starch/NR/5% CNF film.

IV. Conclusions

Starch/NR bio-composite films reinforced with cellulose nanofibers were successfully developed using solution casting method. The addition of cellulose nanofibers had greatly enhanced the mechanical and thermal properties of the matrix. With the increase in cellulose nanofiber contents, tensile strength and modulus were also found to be significantly improved, indicating the effectiveness of reinforcing cellulose nanofibers. Increased thermal stability was also achieved by adding cellulose nanofibers. Moisture uptake was reduced because of strong interaction between cellulose nanofibers and polymer matrix. While there was a decrease in film transparency with the increase in CNF contents, still it showed good film-forming property. Based on the properties evaluated, 5% CNF added film showed the best property among all the samples tested. Therefore, it could be concluded that the CNF reinforced starch/NR bio-composite is promising materials for eco-friendly packaging.

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