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Drying Effect on Mechanical Properties of Bio-nanocomposite Films Fabricated from Self-assembled Cellulose Nanocrystals into Potato Starch

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ABSTRACT

Composite films with higher mechanical properties from naturally occurring degradable materials are of present demand to achieve goals of sustainable development. Interaction within composite constituents during drying controls mechanical properties. Here, bio-nanocomposite films were first prepared from cellulose nanocrystals synthesized from jute fibres and extracted potato starch with the same chemical formulations. The filler, nanocrystals consist of nanorod-like cellulose particles obtained as an aqueous suspension by sulfuric acid (H₂SO₄) hydrolysis of jute fibres and the matrix was prepared by plasticization of potato starch after disruption of starch granules with water and glycerin. Nanocomposite films were obtained by casting the homogeneous aqueous suspension at 95°C and followed by natural drying (atmospheric drying, 25°C) and oven drying at 40°C. The thickness of the bio-nanocomposites film about 250 μm was controlled by using a 2 mm thick structural glass frame. It is revealed that with increasing the percentage of cellulose nanocrystals in composite films, mechanical properties corresponding to tensile strength and Young's modulus was increased significantly. The film containing the highest quantity of cellulose nanocrystals (20% w/w of starch) revealed better properties in case of natural drying (tensile strength 84.2 MPa, Young's modulus 0.563 GPa, elongation at break 27%) than the film properties (tensile strength 35.2 MPa, Young's modulus 0.423 GPa, elongation at break 20%) of oven drying.

Keywords: Bio-nanocomposites films, cellulose nanocrystals, starch, self-assembly, potato starch, and drying.

INTRODUCTION:

Development and engineering of bio-based products and innovative processing technologies which offer sustainability and mitigation of the dependence on fossil fuel are essential for sustainable civilization. Among biopolymers, starch is the most promising renewable bioresource due to its versatility, competitiveness in price, and applicability to various Industries (Yang *et al.*, 2007; Tianyu *et al.*, 2020; Pandey *et al.*, 2020; John and Thomas, 2008). Through destruct-

urization by the introduction of mechanical and heat energy or by incorporation of a plasticizing agent (e.g., water, amide, and/or polyols), starch can be processed into thermoplastic materials. Currently, biodegradable plastics are primarily used in food packaging films, shopping bags, and flushable sanitary product backing material (Bertoft, 2017; Rhim, 2007; Beytullah and Fatih, 2012). The use of starch-based materials is greatly hindered by its intractable nature, brittleness, water-sensitivity, and poor mechanical

strength (Alexander *et al.*, 2013; Kumar *et al.*, 2008; Liu *et al.*, 2009). It has been found that using reinforcing materials in a starch matrix is an effective method to obtain high-performance starch-based biocomposites (Cao *et al.*, 2008). Various types of cellulose fillers such as jute fibers, eucalyptus pulp fibers, flax fiber, tunicin, and ramie fibers, have been prepared and used as reinforcing agents in biocomposites (Chakraborty *et al.*, 2008; John *et al.*, 2007). Natural cellulosic nanocrystals with high aspect ratio have been gaining considerable interest because of their unique and attractive features in terms of functionality, cost effectiveness, and light weight.

Nevertheless, physical properties of various cellulose nanocrystals are significantly identical depending to their distinguished structural compactness, sizes, crystal alignment and overall morphologies. Processing and chemical treatment for fiber extraction involves removing of adhered surface wax, foreign non-cellulosic substances, and chemically broken cellulosic compounds due to the action of harsh chemical. Cellulosic fibers from different sources like cornstalks, rice, and wheat straw are composed of single cells and are about 0.5-3.0 mm in length, whereas flax fiber is as long as 77 mm reported by different research groups (Reddy and Yang, 2006; Reddy and Yang, 2007). Besides, cellulosic nanocrystals have a size of 2-50 nm in one dimension (Dufresne, 2008; Kennedy *et al.*, 2007). However, due to the interactive behavior of cellulose nanocrystals, it is difficult to control uniform dispersion in a matrix or into a solution because aggregation and agglomeration occurs within the nano-phases to form larger structures.

The cellulosic crystal structure, morphology, and crystallinity are mainly varied depending on the crystal sources its processing parameters e.g. pre-treatments chemicals, intensity of temperatures, types of solvents, suspension or dry state, and other related conditions. Like in Bangladesh, jute is a renewable and easily growing natural bioresource abundant in many parts of the world. It has several advantages including small environment load, rapid growth, renewability, relatively high strength, and good flexibility. Cellulosic nanocrystals from jute fiber may play an important role in forming new organic

structural nanocomposites, and is recognized as an attractive candidate for reinforcing agent. However, research in manufacture and use of cellulose nanocrystals from jute is not still up to the mark. Understanding of the structural feature and morphology of jute cellulosic crystals could lead to more and efficient utilization of this bioresource (Wei-ming *et al.*, 2009). In this study, hydrolysis was carried out to synthesize nanocrystals from jute fibers digestion using 64 % w/v sulfuric acid at 60 °C. The objective of the study was set to investigate the drying effect on mechanical properties of the cellulose nanocrystals assisted potato starch bio-nanocomposite films. The results of the research work would play vital role to direct the right pathway for the fabrication of environment friendly films for food packaging.

MATERIALS AND METHODS:

Materials

Jute, potato and bleaching powder (CaOCl_2) were collected from local market (Dhaka, Bangladesh). Sulfuric acid (98%, H_2SO_4) and glycerin were purchased from Sigma-Aldrich, and were used as received. Pre-designed glass frame was used to control the size and shape of the films.

Methods

Preparation of Cellulose Nanocrystals - At first 64% w/v sulfuric acid was prepared (Revol *et al.*, 1994; Carla and Ticiane, 2019). For this 35 mL of sulfuric acid (98%, specific gravity 1.85) was added slowly into 65 mL of distilled water in a 250 mL beaker to prepare 100 mL of aqueous acid solution. Later 2.765 g of cellulose (jute fibres) was then immersed into acid solution for hydrolysis. This solution was then mechanically stirred at 60°C for 35 minutes. The aqueous black suspension was subsequently diluted with an equal part of water (cold) and washed by successive centrifugation at 10,000 rpm and at 25°C (three times) (**Fig 1**). Dialysis against distilled water was performed to remove free acid in dispersion. After this we obtained the grey like substance which was subjected to bleach.

Bleaching - An amount of 40 g bleaching powder was dissolved in 400 mL water to make a bleaching solution. The solution prepared from commercially

available bleaching powder was mechanically stirred at 40°C for 25 minutes and removed undissolved impurities by decantation. The collected bleaching solution of about 100 mL was first added to the hydrolyzed cellulose suspension, which was mechanically stirred for 30 minutes at 40°C. After 10 minutes of stirring the suspension was kept in rest without disturbance, clear brownish solution was decanted again. Similarly, decolorization treatment with the bleaching solution was carried out three times. Finally, a white suspended jelly like material was washed by cold water several times to remove free bleaching agents from cellulose nanocrystals. The nanocrystals were then recovered by drying from an atmospheric dryer at 105°C.

Extraction of starch from potato - Extraction of starch from potato was carried out according to the procedure as described. At first about 100 g cleaned potato but without peeling was grated and charged into a blending machine with addition of about 100 mL distilled water. After 10 minutes blending, the mass of potato was carefully transferred and diluted with more 100 mL water to settle into the beaker. Mass of residual potato from the beaker, leaving behind the white starch (settled at the bottom) was separated. The mass of the beaker was washed several times with copious water and filtered using a cloth to collect granular starch (**Fig 2**).



Fig 1: Hydrolysis of jute using 64% w/v H₂SO₄ for preparation of cellulose nanocrystals (left: initial stage, right: after hydrolysis).

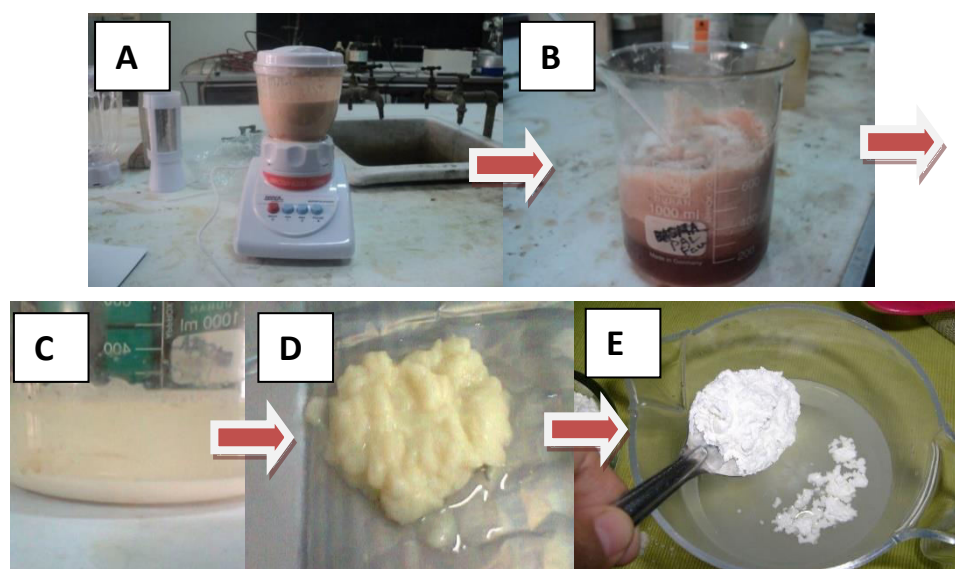


Fig 2: Extraction of starch from potato (A) potato chopped was blended, (B) settling, (C) washing, (D) screening, and (E) dried starch powder.

Neat Film Preparation - A quantity (3 g of mixture) of dried starch and glycerol (2:1) was first dispersed into 35 mL of distilled water. Then the mixture was subjected to heat at 95 °C for 15 minutes with mechanical stirring. Gelatinized starch was cast and stored in a non-ventilated oven at 40 °C for 24 h.

Composites Film Preparation - Starch (2 g) was first dispersed in distilled water (35 mL). Besides, cellulose nanocrystals (5%, 10%, 15% and 20% w/w of starch) were homogeneously dispersed in 1 g of glycerol, so that the agglomeration was prevented by sonication for 10 minutes. Then the two mixtures were added together and stirred mechanically while the solution was heated at 95°C for 15 minutes. The mixture was then stirred and sonicated for 10 min to remove micro-bubbles before casting onto the surface of glass wrapped with Teflon sheet. The film was dried by atmospheric evaporation or in an oven at 40°C for 24 h.

Tensile properties - Tensile strength, elongation at break and Young's modulus of the films with dimensions of 7×4× 0.25 mm (length × width × thickness), conditioned at 75% RH, were measured using a universal testing machine (CMT6503, Shenzhen SANS Test Machine Co. Ltd., Shenzhen,

China). Initial gap of the jaw was fixed at 10 mm. Dried films with an average thickness of around 250 µm were obtained by measuring with Phynix digital micrometer.

RESULTS AND DISCUSSION:

Cellulose nanocrystals with some microfibrils from jute were prepared by hydrolysis in presence of 64% H₂SO₄ at 60 °C. The product obtained from reactor was deep brown due to presence of some unknown coloring entities in the processed jute and may be some undesirable reaction with oxygen occurred in the reactor. Aqueous solution (10% w/v) of commercial bleaching powder was used to purify the nanocrystals. It was noteworthy to mention that the purification was not complete after bleaching at room temperature (25 °C) and then the product still contained little brown color. Whereas, when bleaching was carried out at 50 °C, all of the coloring materials were removed leaving a white cellulose nanocrystals as shown in the **Fig 3A**. Besides, the yield of extracted starch from potato was about 18%. The higher yield of starch and low cost of potato indicate that it can be a potential source of industrial raw material as shown in **Fig 3B**. A white cotton fabric (cloth) was used for separation of starch by filtration.



Fig 3: Shows the optical images of (A) cellulose nanocrystal, (B) potato starch, and (C) bio-nanocomposite film.

Therefore, a portion of starch was trapped into the blended potato mass. The yield of starch may be increased by using an improved sieving technique. Biopolymers are usually hydrophilic and thus, water is the solvent used most often to dissolve to obtain film forming solutions. Instead of water some other

solvents with or without water can be used to dissolve biopolymers. Usually, heating with solvent is needed to disrupt the native structure of the biopolymer to obtain a film forming solution. Plasticizer is added to the film forming solution at a convenient stage of the process to obtain flexible and elastic films which are

often desired. There are various biomaterial film forming processes such as casting, spraying, extrusion and thermo-molding. The most common process to produce films on a laboratory scale is casting, which was used to produce films for testing. In this process, a film forming solution was cast on a non-adhesive surface of glass covered with Teflon ribbon. Water or solvent is evaporated from the solution in order to form the film. As a result of solvent evaporation, biopolymer was left onto the Teflon surface and the basic film structure was developed by hydrogen bonding as shown in **Fig 3C**. Environmental parameters, such as temperature and a relative humidity, during the evaporation stage could be used to control some of the film properties.

SEM of Cellulose nanocrystals - Scanning electron microscopy (SEM) observation was performed using Philips CM200 microscope (Eindhoven, The Netherlands) with a 20 KV accelerating voltage. A drop of a dilute cellulose nanocrystals and starch suspensions were deposited on a glow disc-graded carbon-coated microscopy grid. After one minute, the liquid in excess was blotted with filter paper and the remaining film was allowed to dry. Because of atmospheric drying, an agglomeration of crystals was observed from SEM images. Since we have followed an established procedure to synthesize nanocrystals from jute, the synthesized nanocrystals having a 100-150 nm and 5-10 nm diameter were maintained which is available in literature (Carla and Ticiane, 2019). **Fig 4A**, and **B** shows the SEM images of cellulose nanocrystals processed in 64% w/v H₂SO₄ and starch extracted from local potato.

Grain size of potato starch was also checked by SEM and a range of 10-40 μm was found (**Fig 4**) which is also complied by literature survey (Daniel *et al.*, 2019), particle shapes of the potatoes starch were mainly spherical and polyhedral. This is in conformity with common starch.

Mechanical properties - Humidity is one of the most important factors affecting the mechanical properties of thermoplastic starch. For example, the mechanical strength of starch composites can reach 20 MPa in 0%RH; however, at high moisture conditions, the tensile strength may be below 1 MPa due to water plasticization (Glenn *et al.*, 2007). Cellulose nanocrystals are reported to have a high modulus, up to 134 GPa, and strong tensile strength of more than 4 GPa (Sakurada *et al.*, 1962).

Determination of Tensile strength and Young's modulus - It was possible that varying amounts of cellulose nanocrystals might affect the film thickness and consequent the mechanical properties. Film thickness measurement was, therefore, carried out using Phynix digital micrometer. It was found that film thickness of films containing 5, 10, 15 and 20 wt% cellulose nanocrystals around 250 μm. These results indicated that the dry film thickness was not significantly varied with different amounts of added nanocrystals. The resulting mechanical properties of film therefore were reasonably comparable. The results show that the preparation of starch-based composite with cellulose nanocrystals could improve the mechanical properties. The addition of cellulose nanocrystals increases the composite film strength and stiffness.

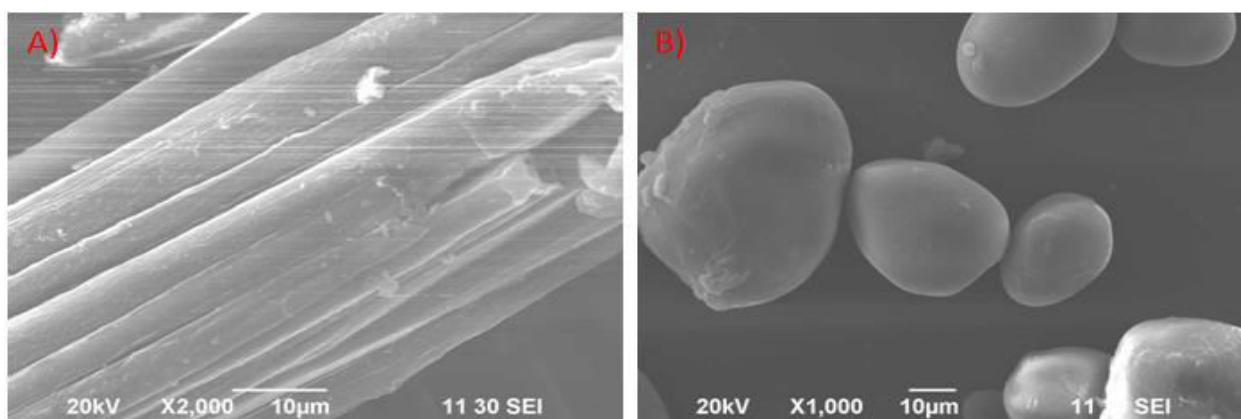


Fig 4: Shows the SEM images of (A) cellulose nanocrystals, and (B) potato starch granules.

Four types of nanocomposite films and a film of starch were prepared to study the effects of nano-crystals of jute fiber attributes and matrix compatibility on several end-use properties. During film preparation of the composite, we have considered some factors so that an identical film could be produced. Therefore, a glass sheet with 2 mm thick was chosen and cut in a rectangular shape. This glass sheet was used to produce a rectangular pan using another glass boundary and the mixture of mass was poured in the void and open space of rectangular pan. The bubble of the suspension was removed by 10 minutes sonication. Then the film was dried in atmosphere as well as in an atmospheric dryer at 40°C separately. An increase in the amount of cellulose nanocrystals in the film a gradual increase in both tensile strength and Young's modulus was found. For example, considering the tensile strength values of starch films containing 0, 5, 10, 15, and 20 wt% cellulose nanocrystals are varies from 58 to 84 MPa for atmospheric drying and 10 to 35 MPa for oven drying films respectively as shown in **Fig 5A**, and **B**. Structurally, cellulose nanocrystals are very strong, as a result, the incorporation of nanocrystals into the starch composite filmed to an

improvement in both tensile strength and Young's modulus due to the reinforcement effect.

It is revealed from **Fig 5A**, the film of starch only shows tensile strength about 58 MPa, but after 5% addition of cellulose nanocrystals gives higher tensile strength than the starch film. The increment of strength was remained steady until 15% of nanocrystals added. With increasing the quantity of cellulose nanocrystals, the tensile strength (MPa) was increased linearly. It is clear that the value of tensile strength of the film containing 15% of nanocrystals is closer to the tensile strength of the film containing 20 % of nanocrystals. This phenomenon can be explained as the crystal amount increased excessively; the binding capacity of matrix was not working significantly. This naturally drying starch film is actually strong due to homogeneous drying. In case of oven drying, the film property, in terms of tensile strength is similar but the values of tensile strength were found below half of natural drying as shown in **Fig 5B**. **Fig 6** shows the comparison of tensile strength between natural drying and oven drying systems.

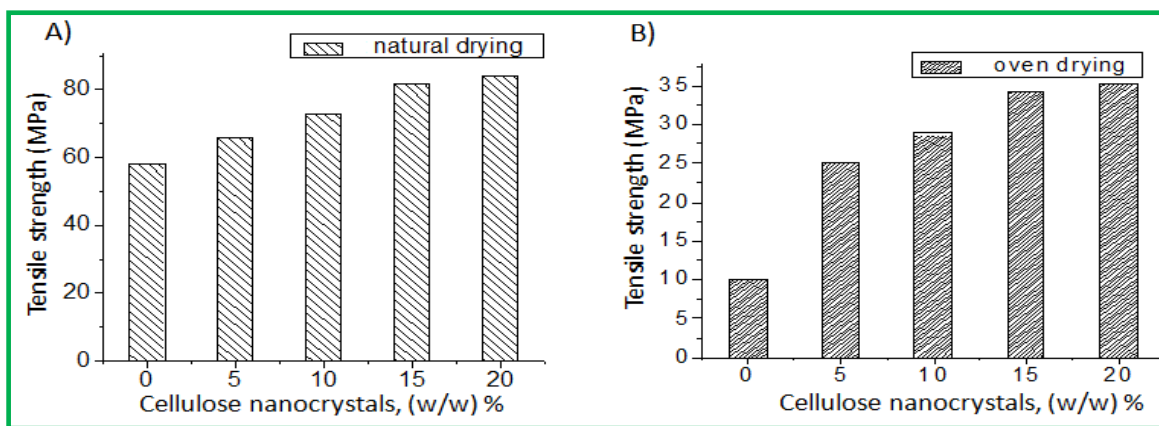


Fig 5: Shows the tensile strength of different composite films at natural drying (A), and oven drying (B).

The overall all strength of composite is stronger for natural drying (filled circle), whereas the oven drying (open circle) produced a hetero system in the film by evaporating the solvents from surface of film rapidly, but a soft interior remain in the film leads to weaken the film property. In 1994, Bader and Göritz (1994) reported that a starch films swelled at high relative vapor pressure (RVP) due to high water content of

film. During swelling, number of hydrogen bonds between starch-polyol and starch-starch decreases and number of hydrogen bonds between starch-water and polyol-water increases resulting in weakened structure of the film which do not last remarkable deformations. This was observed at high RVP as elongation of film was found to decrease instead of increase which was expected to occur with increasing plasticizer content

and RVP as reviewed, *e.g.*, by Krochta, (2002). Moreover, the Young's modulus and tensile strength of glycerol plasticized potato starch films increased with increasing the amount of crystalline cellulose nanocrystals (**Fig 6**). Crystallization of starch components occurred during film formation increasing

Effect of cellulose nanocrystals on % Elongation at break - The maximum % elongation at break was found from the film of starch and glycerol. Here, also the elongation values of the films in case of natural drying showed higher than the values of oven drying.

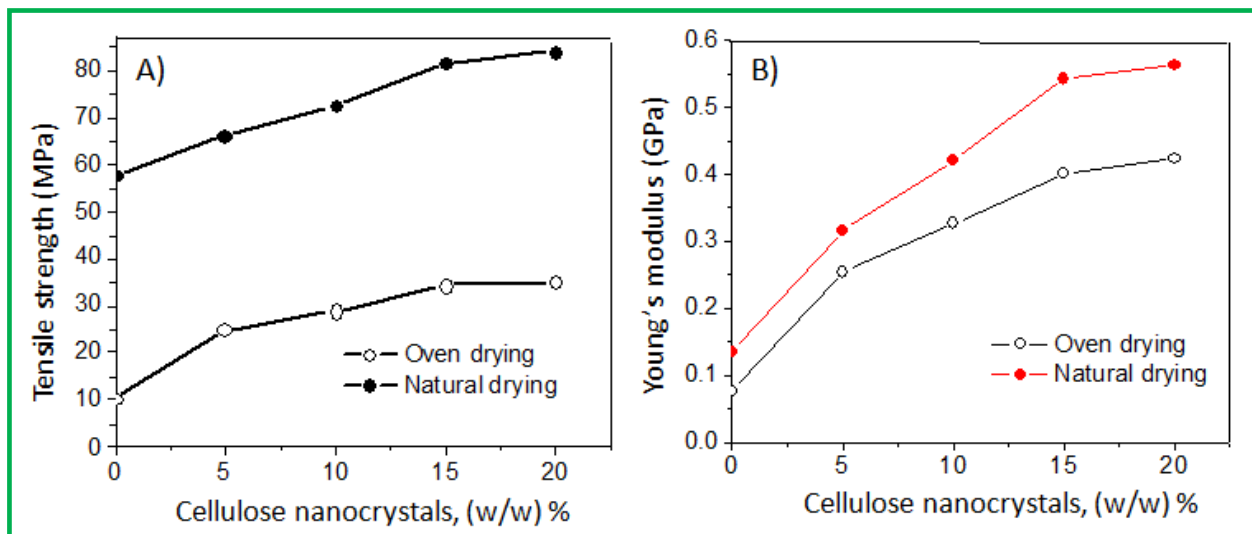


Fig 6: Shows a comparison of natural drying and oven drying of composite film in terms of (A) tensile strength, and (B) Young's modulus.

Young's modulus and tensile strength which could be observed from increased rigidity of the starch film. The Young's modulus and tensile strength of glycerol plasticized cassava starch films have been reported to increase with increasing amount of added amylose due to a denser polymer network (Alves *et al.*, 2007).

The maximum decreasing of the value is observed when 5% w/w of cellulose nanocrystals was added. After sharp decreasing, the elongation at break decreased very slowly. On the other hand, this can be explained as the addition of cellulose nanocrystals proceeded, the expansion of the film was reduced and

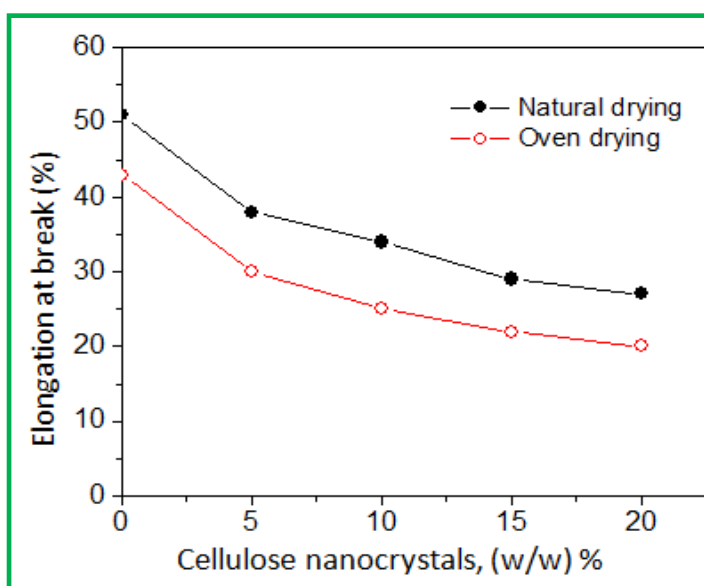


Fig 7: Shows the percentage elongation at break of different composite films prepared at natural drying and oven drying.

rigidity as well as toughness was increased as shown in **Fig 7**. In 1999, Biliaderis *et al.* (1999) reported that elongation of starch films increased remarkably as water content increased because of change from glassy to rubbery state, which supports the result oppositely. In the schematic **Fig 8A**, it is revealed that the surface of film was dried and became brittle when the film was

rapidly dried by oven drying. As a result, the core matrix of the film remained soft and wet which produce mechanically a weak film due to lack of interactive compactness. Besides, natural drying leads to produce a homogeneously dehydrated film which makes relatively compacted and mechanically stronger films as shown in **Fig 8B**.

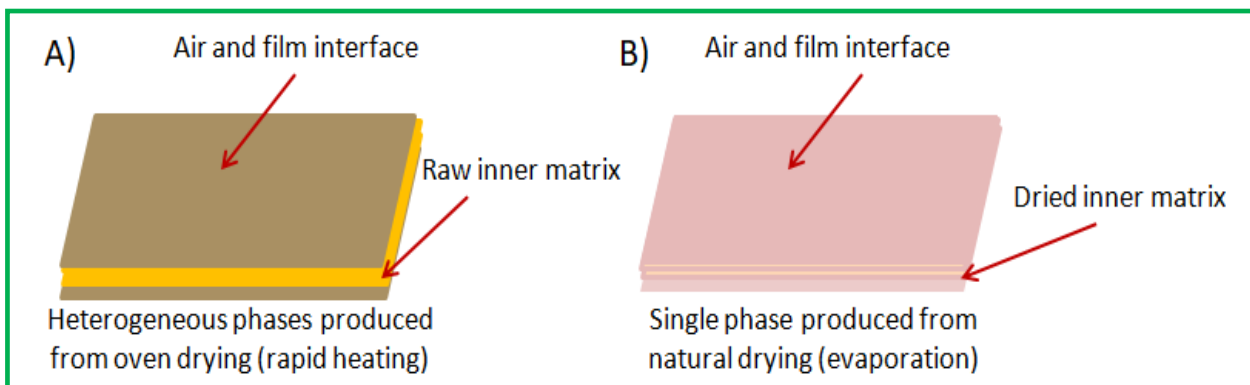


Fig 8: Shows schematically the mechanistic representation of the effect of (A) oven drying, and (B) natural drying on the surface of film.

CONCLUSION:

Environment friendly bio-nanocomposites films with 250 μm thicknesses were successfully fabricated using cellulose nanocrystals synthesized by sulfuric acid hydrolysis of jute fibres as reinforcing phase and potato starch plasticized with glycerol as matrix. A solution (10% w/v) of commercial bleaching powder was effectively used to remove undesirable color of nanocrystals. It is revealed that with the increase of cellulose nanocrystals in the composite film, a gradual and remarkable increase in both tensile strength and Young's modulus was found. Changing of drying techniques from atmospheric to dryer, deteriorate the mechanical property of films due to heterogeneous drying. Naturally atmospheric drying films showed 2.5 to 6 times MPa higher tensile strength than that found from oven dried films. Young's modulus of the films was also increased and found 0.563 GP in presence of 20% cellulose nanocrystals from naturally drying film. It is concluded that a drastic drying technique would create rapid removal of interlocked solvent and plasticizer after destruction of hydrogen bonds within the matrix. Natural drying technique would certainly provide a self-assembled and entropically stable composites film with higher mechanical properties from other nanoparticles and polymer matrix with

selective solvents. Since fabrication of nanocomposite films with homogeneous dispersion of particles are still challenging, this result will imply a pathway to fabricate desirable film for separation, biomedical and engineering advanced applications.

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CONFLICT OF INTERESTS:

The authors declared that there was no conflict of interest.

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