Digest Journal of Nanomaterials and Biostructures Vol. 16, No. 2, April - June 2021, p. 697 - 704

Influence of rice starch nanocrystals on the film properties of the bionanocomposite edible films produced from native rice starch

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In the present investigation, the bionanocomposite films were prepared by incorporating rice starch nanocrystals with rice starch based edible films through solution casting method. SNC were obtained from rice starch granules through acid hydrolysis followed by ultrasonication. Rice starch based edible films were prepared by incorporating the rice SNC at varying concentrations (0, 5, 10, 15, 20and25% of rice starch) through solution casting. The film properties, such as water vapour transmission rate (WVTR), mechanical properties, and film colour were determined. From the results, it was observed that the addition of rice SNC did not affect the film colour but significantly improved the mechanical strength and water resistance.

(Received February 24, 2021, Accepted May 31, 2021)

Keywords: Rice starch, Bionanocomposite, Starch nanocrystals, WVTR, Mechanical properties, Film colour

1. Introduction

The food and beverage industry shows much interest on the use of nanotechnology and has already started focussing on the effective use of nano-materials in order to produce a lightweight packaging material with stronger barrier and mechanical properties. Polymer blending or the use of multi-layers could conventionally improve the barrier and mechanical properties of the edible films and coatings. However, they pose problems like high production costs, high material use, costs related to additives and adhesives, and difficult recycling. On the other hand, with nanotechnology, more efficient monolayer films can be produced with low manufacturing costs, less material use, low material costs, and easy recycling [1-9].The first work on isolation of SNC was found in 1996. Dufresne et al. [10] isolated SNC from potato starch granules through acid hydrolysis. The SNC produced was so called then 'microcrystals' even though they had dimensions a few tens of nanometers. Isolation of SNC were reported on literatures from various starch sources, such as maize starch [11], tapioca starch [12], mungbean starch [12], potato starch [12], wheat starch [13], barley [12], oats starch [14], taro starch [15], pea starch [12], and rice starch [16].SNC were produced using various isolation methods like acid hydrolysis [17-18], enzymatic hydrolysis [19], high pressure homogenization [20], ultrasonication [21] and nanoprecipitation [22].

The incorporation of SNC on the bionanocomposite films have been investigated by few researchers. Li et al. [23] produced the pea starch bionanocomposite films reinforced with waxy

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maize SNC (1-9%). The addition of SNC up to 5% decreased WVP significantly, while TS and YM increased. The surface of these films was smooth and dense. However, when the SNC were more than 7%, the SNC started to aggregate. Dai et al. [15] produced the bionanocomposite films from corn starch reinforced with taro starch nanoparticles (0.5%-15%). The addition of taro starch nanoparticles decreased WVP, increased TS from 1.11 MPa to 2.87 MPa, and increased the thermal stability of the films. González and Igarzabal [24] prepared the bionanocomposite films by incorporating corn SNC (5-40%) with soy protein isolate edible films. The addition of nanostarch improved the water solubility, swellability, WVP and TS. These films were found to be more transparent and homogeneous, while the opacity and degree of crystallinity were slightly increased at higher starch nanocrystal concentrations. Condés et al. [25]produced the bionanocomposite films by incorporating the maize SNC with amarnath protein. The resulting nano edible films showed lower WVP,increased surface hydrophobicity, improved optical properties and increased the mechanical properties. The films were homogeneous and translucent than the neat protein film. Jiang et al. [26] produced the bionanocomposite films by incorporating the potato starch nanoparticles with pea starch. The addition of nanoparticles increased TS from 8.8 MPa to 15.0 MPa, good water resistance and thermal stability than control films.

The use of starch nanofillers and matrix of same botanical origin is rarely investigated for making bionanocomposite films. The incorporation of SNC could exhibit strong reinforcing properties when they are dispersed in starch films of the same botanical origin(Viguié et al. 2007). The reason for strong reinforcing effect of SNC on the starch matrix of the same botanical origin is the strong filler-filler interactions as well as filler-matrix interactions due to their chemical and dimensional similarities. The addition of SNC also slows down the matrix re-crystallization during aging in humid atmosphere[11,27]. Angellier et al. [11]produced the bionanocomposite films by waxy maize SNC with waxy maize starch and the effects of aging were investigated. Aging improved the mechanical properties. However, the mechanical properties of non-aged starch films with up to 10 wt % SNC were very close to those of the aged starch films. When the concentration of nanocrystals was above 10 wt%, the resulting films showed increased YM and TS (after aging) without significantly affecting EAB. Liu et al. [28] prepared the bionanocomposite films from waxy corn starch reinforced with corn starch nanoparticles at varying concentrations (0–25%). The addition of corn starch nanoparticles below 15%increased TS from 1.40 to 2.35 MPa, reduced WVP from $5.89\text{x}10^{-12}$ to $3.08\text{x}10^{-12}$ g/(m s Pa) and improved the thermal stability as compared to the control films. Oliveira et al. [29]prepared the bionanocomposite films by incorporating mango SNC (0–10 wt%) with mango starch (both obtained from mango kernals). The addition of up to 7.5% starch nanoparticles showed increased TS (90%) and YM (120%), while reducing WVP (15%). These research findings showed that the bionanocomposite films produced from starch nanoparticles filler and starch matrix of the same botanical origin offered higher mechanical performance and barrier properties. However, from the literatures, the use of both nanofillers and matrix of the same starch source is very limited and there is a research perspective in this area. In the present work, the effects of the addition of rice SNCon the film properties were investigated on the bionanocomposite films produced from rice starch.One such work was carried out by Piyada et al. [30], in which the rice starch based bionanocomposite films were prepared by incorporating rice SNC (0-30%) and sorbitol as plasticizer. The mechanical properties, WVP and thermal stability were found to be improved with the addition of rice SNC. However, no research is found on rice starch nanofillers and rice starch matrix with glycerol, the most widely used plasticizer in the preparation of edible films.In our investigation, glycerol was used as the plasticizer. The rice starch based bionanocomposite films were produced by adding the rice SNC at varying concentrations (0, 5, 10, 15, 20 and 25% of rice starch). WVTR, mechanical properties, and film colour were determined for the resulting films and discussed.

2. Materials and methods

2.1. Materials

Rice starch, glycerol (plasticizer), and sulphuric acid (for acid hydrolysis) were purchased the local chemical shops, Chennai.

2.2. Synthesis of Rice SNC

Rice SNC were produced through acid hydrolysis method based on the procedures described by Angellier et al. [31] with slight changes. 36.5 g of rice starchgranules (14.6 wt%) were mixed in 250 mL of 3.16 M sulfuric acid solution (178 mL of concentrated sulphuric acid and 822 mL of distilled water) kept in a 500 mL Erlenmeyer flask. The suspension was stirred at 100 rpm and maintained at 40°C for 5 days in an environmental chamber. After 5 days of acid hydrolysis reaction, the suspension was centrifuged (at 5000 rpm for 25 min) and washed several times by successive centrifugations in distilled water till the solution reached the pH value of 7 (neutral). The resulting suspension of concentrated rice SNCwas freeze-dried in order to obtain solid rice SNC.

2.3. Film preparation

Edible films were then produced through solution casting method. The aqueous starch solution of 5 g of rice starch granule in 100 mL distilled water was first prepared. 1.5 g of glycerol and a measured amount ofrice SNC (0, 5, 10, 15, 20 and 25 w/v) were added into the initial aqueous starch solution and mixed well in order to make the film forming solution. The film forming solution was then transferred into a beaker and heated in magnetic stirrer with hot plate(at 90 °C and 500 rpm) for about 20-25 min until the gel-like was formed. This gel-like solution was cooled to about 40 °C, spread onto flat Teflon-coated plates, and allowed to dry for about 5-6 days. After drying, the resulting dried films were peeled off from the Teflon-coated plates manually and stored in the environmental chamber (at 23°C and 50% RH).

2.4. Film thickness

The film thickness of the bionanocomposite films was measured using a digital micrometer (Yuzuku digital micrometer, with the sensitivity of 0.001 mm). The film thickness was measured at least in 3 random locations for every film, and averaged.

2.5. Water vapour transmission rate

WVTR was determined using gravimetric method (upright dry cup method) according to the ASTM E96 standard [32]. The procedure was followed as described by Hu et al. [33], Huang and Qian (2008) and McCullough et al. (2003) with slight modifications. The test films were cut into circular shape of 7.5 cm diameter. Test filmswere placed over the metal cups (brass) having 100 ml of distilled water, sealed with a gasket and clamped tight enough. The initial weights of the test cup and time of taking readings were recorded using a weighting balance (with sensitivity of 0.001 g). The test cups were then placed in an environmental chamber (50±3 %RH and 23±1 °C). The weight of the test cups and time of readings were periodically measured for every 4 hours. The weight measurements were continued until the weight loss versus time plots became linear. The slope ($\Delta m/\Delta t$) was calculated from the weight loss versus time plots. WVTR is calculated from the following formula:

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

where, WVTR is the water vapour transmission rate (g/m^2/day), $\Delta m/\Delta t$ is the slope obtained from the weight loss versus time plot (g/s), and A is the exposed area of the film (m^2). A minimum of 3 replicates were recorded and the averaged for the analysis.

2.6. Tensile properties

The mechanical properties of the test films were determined according to the ASTM D882 standard [36]. The test procedure was followed as described by Beruzzi et al. [37] and Beruzzi et al. [38] with minor modifications. The test films were cut into rectangular shape of 100 mm x 25.4 mm dimensions. Texture analyzer was used to determine the mechanical properties, such as TS, YM and EAB). Initial grip separation and the crosshead speed were set as 50 mm and 500 mm/min respectively. The microcomputer attached to the texture analyzer recorded the stress–strain curves, from which TS, YM and E were determined by the computer. The following formulas were used for the calculations:

$$\text{TS} = \frac{\text{Maximum load before failure}}{\text{Initial area of cross section}} \tag{2}$$

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

$$\text{E} = \frac{\text{Change in length}}{\text{Original length}} \text{ x } 100 \tag{4}$$

2.7. Film colour

Film colour was determined through the digital photography method as described by Afshari-Jouybari and Farahnaky [39] and Farahnaky et al. [40] with minor modifications. A high resolution digital camera was used for this purpose. The camera was initially set to 1000 dpi of resolution, 62 % of contrast and 62 % lightness, respectively. The digital images (in JPEG file format) of the test films were captured in the camera and transferred to a computer with Adobe Photoshop (Version 8) package. Before taking the images of test films, the image of white colour standard was taken and the colour parameters (L*, a* and b*) were recorded. The digital images were opened in the package and the colour parameters (L, a and b) were noted from the ‘LAB’ mode in the package. L, a and b colour parameters were measured at 3 random locations in each image and the average values were considered for the analysis. The colour change index (ΔE) was calculated from the following equation:

$$\Delta\text{E} = \sqrt{(L^{*} - L)^2 + (a^{*} - a)^2 + (b^{*} - b)^2} \tag{5}$$

where L*, a* and b* are the standard colour parameters of the white colour, and L, a and b are the sample colour parameters of the test films.

3. Results and discussion

3.1. Water vapour transmission rate (WVTR)

Fig. 1 shows the WVTR of the prepared bionanocomposite films. The control films exhibited the WVTR of about 143 g-mm/(m^2-day), which is close to the WVTR value of the rice starch film (of 156 g-mm/(m^2-day)) produced by Laohakunjit and Noomhorm [41]. The WVTR of the rice starch films reinforced with the rice SNC varied from 121 to 105g-mm/(m^2-day). As the amount of the rice SNC increased, the WVTR of the bionanocomposite films reduced. It should be noted that the SNC are suspected to create the tortuosity in the diffusion path for water or gas molecules due to platelet morphology of SNC [42]. In addition, due to high density of OH groups in starch nanofillers, they tend to form aggregates and strongly associate with each other through strong hydrogen bonding and create the preferential paths for allowing water vapours to diffuse through. Therefore, the permeability of the films increases. For example, in the work of Garcia et al. [43], the WVP of thermoplastic films showed 79% higher WVP with the addition of only 2.5% wt% waxy maize nanocrystals. Similar findings were reported by Kristo and Biliaderis [44], where the addition SNC did not significantly improve WVP of pullulan films with the addition of up to 20% (w/w) SNC. However, significant reduction of WVP in pullulan films was observed in films reinforced with more than 20% (w/w) nanoparticles. This could be because of high filler addition that produced the tortuous path for water molecules to pass through. However, as both SNC and starch matrix were derived from the same botanical origin, they have similar polarity and the nanofiller-matrix interactions were stronger, which weakens the matrix-water interactions. Therefore a slight decrease in WVTR could be observed in starch films reinforced with SNC(2017).The WVTR results also indicated that the rate at which the WVTR reduced was about

15%, 7%, 4%, 1%, 0% and 0% respectively for the addition of each 5% of SNC. This meant that the WVTR of the bionanocomposite films was reduced up to 15% of SNC addition and it was not significantly increased afterwards. Similar result was obtained by Liu et al. [28]where the WVP was increased when the amount of the SNC were above 20%. This was attributed to the formation of aggregates at higher addition of starch nanoparticles. Similar effects were also reported by Piyada et al. [30] and Condes et al. [25]. However, the WVP was found to be reduced when SNC of up to 15% were added.

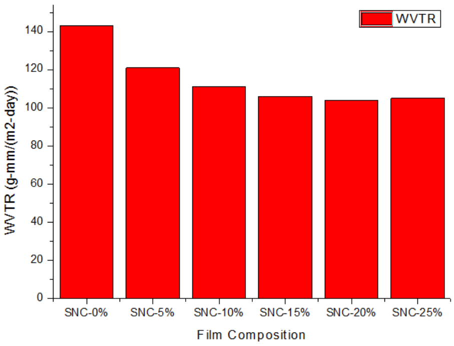

Fig. 1. Effect of the rice SNC on WVTR of the rice starch films.

3.2. Tensile properties

Fig. 2 shows the tensile properties of the rice starch films. Plain edible films plasticized with glycerol showed TS, YM and E of 2.1 MPa, 21 MPa and 59% respectively, which are close to the results of rice starch films produced by Dias et al. [45] (where TS, YM and E were reported as 1.6 MPa, 59 MPa and 21.3 % respectively), by Slavutsky & Bertuzzi [46] (where TS, YM and E were reported as 2.8 MPa, 112 MPa and 44.9 % respectively), and by Ashwar et al. [47], where TS and E were 1.94 MPa and 14.59 % respectively. The addition of rice SNC in rice starch films increased the mechanical strength. The incorporation of rice SNC resulted in the increase of TS and YM by 38-171% and 62-400% respectively, while reducing EABby17-53 %. Similar results on the improved mechanical properties (i.e., increase in TS and YM with the reduced EAB) due to the addition of SNC were also reported by Liu et al. [28], Angellier et al. [11], Zheng et al. [48], Da-Silva et al. [49] and Ren et al. [50]. Some literatures showed that, if the concentration of starch nanoparticles is above 7%, starch nanoparticles starts to aggregate, which might weakens the adhesion between the nanofiller-matrix interfaces. Therefore, for higher nanofiller addition, the SNC self-aggregate to destroy the structure orderness leading to lower mechanical strength[26,51-52].However, the produced rice starch films did not show any reduction of mechanical strength as the amount of rice SNC increased.As the nanoparticles have high surface area to volume ratio, the mechanical properites of the bionanocomposite films are strongly influenced by the nanofiller–matrix interfacial interactions. Uniformly distributed nanoparticles with strong interfacial bonding with the matrix offer the effective stress transfer from matrix to nanoparticles, thus increasing the modulus and tensile strength of the bionanocomposites [44].

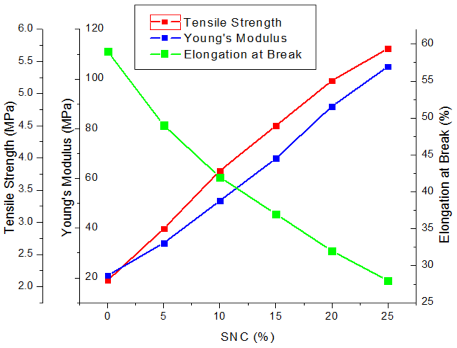

Fig. 2. Effects of the rice SNC on the tensile properties of the rice starch films.

3.3. Film colour

Fig. 3 shows the colour variations of the prepared rice based edible films. Actually, the rice SNC is 45% crystalline and the addition of rice SNC can affect the film colour and light transmittance[42]. CIE colour parameters of the plain rice starch films were determined to be 93.44, 2.08 & 10.44. The colour parameters, L, a & b, of the glycerol plasticized rice starch films are somewhat close to those of the rice starch films produced by Thakur et al. [53], where colour parameters were found to be L, a & b or 95.52, -0.31 & 5.03 respectively, Wittaya et al. [54], where L, a & b values are approximately 88, -1 and 1.5 respectively, and Nawapat and Thawien [55], where L, a & b values are approximately 87, -1.3 and 2.5 respectively. The slight variations in the colour parameters could be seen as a result of the purity level, botanical origin of the species and/ or other factors, such as uncertainty due to the level of lighting and reflectivity. However, as these colour values of the produced control films were somewhat closer, the colour parameters were considered to be in good agreement with the published results. From the results, it was observed that the incorporation of rice SNC on rice starch films altered the colour parameters slightly. The maximum change of colour parameters (L, A and B) was found to be only 1%. As the variation of the parameters was less than 2%, the change in colour parameters is considered to be insignificant. While L and A slightly decreased and B slightly increased. Hence, the film colour changes to slightly whiter, greenish, and yellowish. The overall colour change (ΔE) however was found to be increasing as the amount of rice SNC increased. Similar observations on film colour were recorded by Li et al. [23] and Condes et al. [25] when SNC was added to other starch films.Therefore, it can be said that the addition of rice SNC had slightly affected film colour of the rice starch edible films.

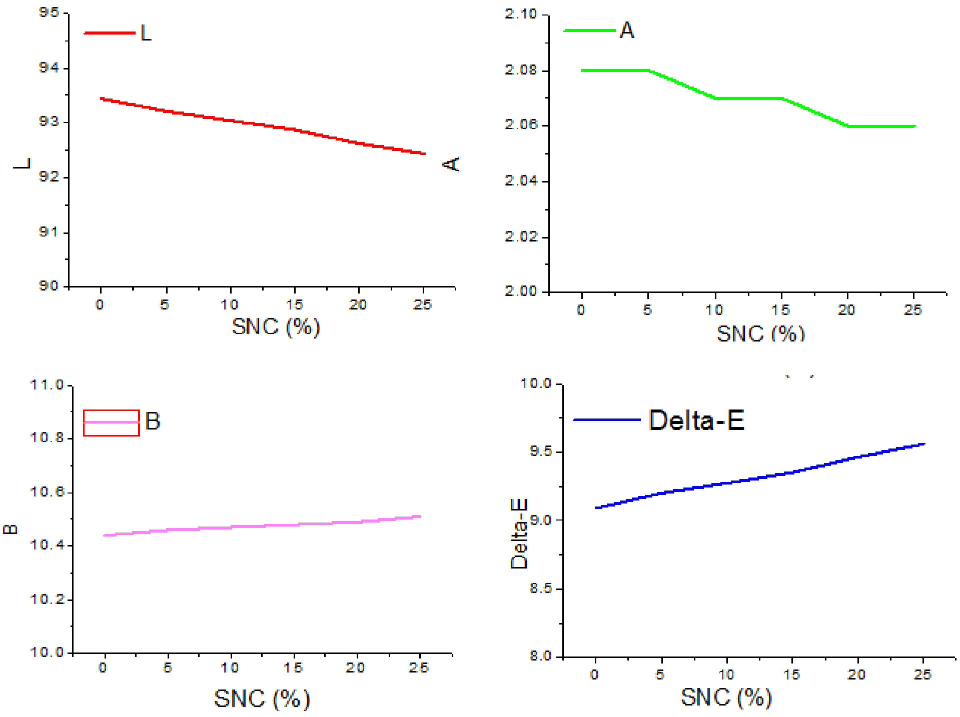

Fig.3. Effects of the rice SNC on the film colour of the rice starch films.

4. Conclusions

In this work, the effects of the rice SNC was investigated on the film properties of rice starch based edible films. The rice starch based nanocomposte films were prepared through solution casting. Film properties (WVP, tensile properties, and film colour) were determined and discussed. The addition of rice SNC in bionanocomposite films reduced the WVTR in the range of 15-27% as compared to that of the control film. The mechanical properties were increased with the addition of rice SNC. While TS and YM were increased in the range of 38-171% and 62-400% respectively, EAB was reduced in the range of 17-53%.The addition of rice SNC had a very little effect on the film colour of the rice starch based edible films.

he overall colour change of 1-5% was observed while individual parameter varied to about 1% only. From these results, it was concluded that the addition of rice SNC had improved the WVTR, tensile strength, and film colour acceptance. Thus, it is concluded that the addition of rice SNC could be considered a potential method for improving the film properties of the rice starch based edible films.

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