CELLULOSE NANOCRYSTALS: SYNTHESIS, CHARACTERISTICS AND EFFECT ON HYDROXYPROPYL METHYLCELLULOSE-BASED COMPOSITE FILMS AND COATINGS

Nguyen Thi Minh Nguyet[*], Nguyen Duy Lam, Pham Anh Tuan

*Vietnam Institute of Agricultural Engineering and Postharvest Technology,
60 Trung Kinh, Trung Hoa, Cau Giay, Ha Noi*

[*]Email: *minhnguyet.viaep@gmail.com*

ABSTRACT

This study aimed to synthesize, determine characteristics of cellulose nanocrystals such as morphology, size particle, zeta potential, Fourier transform infrared spectroscopy (FT-IR) and transmission electron microscopy (TEM). The study also evaluated effect of concentration of added cellulose nanocrystals to hydroxypropyl methylcellulose-base films and coatings containing carnauba Nano emulsion and chitosan nanoparticles. Results showed the shape and size of the nanoparticle generally depend on hydrolysis condition of microcrystalline cellulose. Cellulose nanocrystals obtained from processing hydrolyzed microcrystalline cellulose in range from 47 wt. % to 55 wt. % had length range of from 160 to 196 nm and diameter from 9 to 11 nm. Cellulose nanocrystals were added into nanocomposite HPMC that incorporated with both of carnauba Nano emulsion and chitosan nanoparticles with concentration of cellulose nanocrystals at levels: 0.2, 0.5, 0.8 and 1.1 %. Scanning electron microscopy (SEM) images revealed that nanocomposite films become more compact and dense due to the cellulose nanocrystals occupy empty spaces of porous of matrix nanocomposite HPMC increasing the collapse of the pores in the films. The concentration of cellulose nanocrystals at 0.5 % was added to Nanocomposite HPMC including both of carnauba nano emulsion and chitosan nanoparticles improved the physical strength of film with the best quality compared with other films as: film solubility was raised up 16.65 %, film drying time is faster than others (51.33 minutes). Assessement of the effect of cellulose nanocrystals added into the nanocomposite films coated on fruit bananas showed that respiration of bananas were reduced from 57.08 ml CO_2/kg.h to 30.17 ml CO_2/kg.h; weight loss was declined from 4.0 % to 1.82 % thanks to the limitation of the films gas and moisture permeability.

Keywords: carnauba nano emulsion, chitosan nanoparticles, cellulose nanocrystals, HPMC, banana.

1. INTRODUCTION

Nanocomposite of bio-polymer combined natural nanoparticles was appreciated having a

great applicable potential in food packaging due to their excellent properties as high barrier properties against the diffusion of oxygen, carbon dioxide, flavor compounds, water vapor, intensively mechanical stability and environmentally friendly [1]. Among various bio-polymers, hydroxypropyl methylcellulose (HPMC) has been paid great attention in the area of packaging applications. So far, the most studied bio-nanocomposites as a potential material source for edible films and coatings are chitosan nanoparticles and cellulose nanocrystal [2]. Cellulose nanocrystals have attracted a great deal of interest in the nanocomposites field due to their appealing intrinsic properties such as nano scale dimensions, high surface area, unique morphology, low density renewability, biodegradability and high mechanical strength. Therefore, cellulose nanocrystals dispersed in polymer matrix facilely and ability to form interconnected network structures through hydrogen bonding which increases filler-filler interactions therefore films were enhanced mechanical properties as also increased barrier properties [3]. These types of matrix-filler interactions are more prominent in water soluble and other hydrophilic polymer, where the nanoparticles can attain a high level of dispersion in polymer matrix [4**Error! Reference source not found.**]. Acording to researchs, the effect of reiforcing depend on their nano dimensions and shape [5]. Cellulose nanoparticles are generally obtained by the acid hydrolysis of cellulose micro fibrils or microcrystals cellulose of plant origin. However, the geometric properties of the nano-cellulose structures (shape, length and diameter) depend mainly on the origin of the cellulose and the extraction process [6]. Recently, the publications about effect of cellulose nanocrystals in HPMC polymer containing several nanoparticles (carnauba nanoemusion, chitosan nanoparticle) have not done yet.

The aim of the present study was to systhesize and characterize and investigate the effect of cellulose nanocrystal in nanocomposite HPMC containing carnauba nanoemulsion with chitosan nanoparticles on films properties and capability of moisture and gas barrier in the coated banana.

2. MATERIAL AND METHODS

2.1. Preparation of cellulose nanocrystals

Cellulose nanocrystal (CNC) was prepared from Microcrystalline cellulose with average particles size of 1.4 μm (Avicel 101, FMC BioPolymer, US) sources by acid-catalyzed hydrolysis using procedure described in Ioelovich (2012) study [7]. Acid concentration was 47, 55 and 64 % sulfuric acid. Briefly, the calculated amount of material was mixed with measured amount of sulfuric acid and vigorously stirred at 60 ∘C for 2 h. An acidic suspension of cellulose nanoparticles resulted. The resulted suspension was centrifuged and washed with deionized water several times to reduce acid con- centration. The suspension was finally neutralized with 0.5 N NaOH solutions and again washed with distilled water until neutrality by successive centrifugations at 10,000 rpm for 30 minutes. The prepared nano-cellulose suspension was freeze-dried to get cellulose nanoparticle powder.

2.2. Particle size analysis

FT-IR analysis: The FT-IR spectra of MCC and CNC were recorded with a Paragon 1000 Perkin Elmer Spectrum (Perkin Elmer Life and Analytical Sciences, Inc., Waltham, Mass., U.S.A.) in the range from 4000 to 400 cm−1. The FT-IR spectra were used to characterize the nanoparticle properties. Powdered sample was prepared using KBr to form pellets. *Particle analysis*: The particle size distribution of solutions was measured in a Zetasizer Nano ZS (Malvern Instruments Inc., Irvine, Calif., USA) using laser diffraction to measure particle size of

suspensions. All analysis was performed by triplicate. *Transmission Electron Microscope (TEM) analysis*: The CNC was characterized with Transmission Electron Microscope Philips CM200 (Philips Electronic Instruments, Mahwah, NJ, USA). TEM was used to observe the morphology of the nanoparticles. The CNC solutions were sonicated for 1 min to produce better particle dispersion and to prevent nanoparticles agglomeration on the copper grid. One drop of the nanoparticles solution was spread onto a grid that was then dried at room temperature for TEM analysis.

2.3. Nanocomposite preparation

Nanocomposite HPMC-Carnauba nano emulsion-chitosan nanoparticles (HPMC-CNe-ChNp) was described in detail in our previous publication (Nguyen Thi Minh Nguyet and others [8]). *Nanocomposite HPMC-Carnauba nano emulsion-chitosan nanoparticles -cellulose nanocrystal (HPMC-CNe-ChNp-CNC)*: HPMC (Methocel®, Dow Chemical Co., USA) was prepared in hot water at 80 °C. The control film forming solution was prepared by dissolving 3 % HPMC in distilled water (w/w) using a hot/cold technique. The powder was first dispersed by mixing thoroughly with one-fifth to one-third of the total volume of water heated to above 90 °C until HPMC was thoroughly hydrated. The balance of water was added as cold water to lower the dispersion temperature. Once the dispersion reaches 70 °C, HPMC becomes completely water solubilized. Carnauba nano emulsion and chitosan nanoparticles were added to HPMC solution to get final HPMC/Carnauba/CHNP ratios in dried films of 3:6:1 in stirring condition at 15.000 rpm for 5 min (Ultra-Turax model T25, IKA, Germany). The nanocomposite forming solutions were prepared by adding cellulose nanocrystals (CNC was prepared by acid-catalyzed hydrolysis at sulfuric acid concentration was 55 wt.%) to HPMC based composite solution at levels: 0.0, 0.2; 0.5, 0.8 and 1.1 % corresponding CP1, CP2, CP3, CP4, CP5. The solutions were homogenized using a Ultra-Turax model T25 (IKA, Germany) at 15.000 rpm for 5 min. Vacuum was applied to degas the film forming solutions to prevent microbubble formation in the films.

Film preparation was followed the method of Gontard *et al.* [9]. The mixtures were cast at a wet thickness of 0.5 mm onto plates that have diameter are 15 cm, after which the plates were placed on a leveled surface at room temperature and allowed to dry for 24 h. After drying, the films were removed from the plate and conditioned for 3 days in plastic bags at room conditions: 25 ± 1 °C and 34 ± 2 % relative humidity (RH).

2.4. Film characterization

Film thickness: Film thickness was measured using a digital micrometer (7221, Mitutoyo Manufacturing, Tokyo, Japan) at 4-6 random positions in the film. *Water solubility:* In order to determine the water solubility of the films, a modification of the procedure proposed by Gontard *et al.* was used [9]. Approximately 100 mg of film sample was weighed and dried in an oven (80 ± 2 °C, 24 h) to obtain the initial dry matter weight of the films. The dried films were immersed in 30 ml of deionized water added 0.02 % sodium-azide (25 ± 2 °C, 24 h). Following, the insolubilized film was taken out of the water and dried (75 ± 2 °C, 24 h) to determine the weight of the dry matter that was not solubilized in water. The estimated water solubility percentage of the films was calculated using the formula given below:

$$W_s (\%) = (W_o - W) \times 100 / W_o$$

where W_s is the water solubility, W_o is the weight of initial dry matter, and W is the weight of dry matter not solubilized. *Drying time was determined following* Gontard *et al.* [9]. Collect 06

plastic sheets in size of 10×10 cm, marked in order, then coating/dipping nanocomposite. Place these sheets on table in room condition until their surfaces are non-adhesive. Weight plastic sheets interval and stop when plastic sheets attain constant weight.

2.4. Plant material and analysis methods

Mature green bananas (*Musa AAA*) cultivated in Gia Lam (Ha Noi), were harvested at maturity stage 2 (still green). The bananas were transferred to the laboratory and treated within 24 hours after harvest. Fruits were treated with sodium hypochlorite solution 0.05 ppm. Coating banana with nanocomposite CP1, CP2, CP3, CP4 and CP5 corresponding to CT1, CT2, CT3, CT4 and CT5, respectively. Each experiment was done with 30 fruits and replicated three times at temperature 20 ^{o}C for 16 days. *Moisture barrier* was determined based on the weight loss of banana fruits. Weight loss was calculated according to the formula: weight loss (%) = $(W_i-W_f)\times100/W_i$, in there, W_i is initial weight (gram), W_f is final weight (gram). *Gas permeability* was determined by via respiration rates. Closed system experiments (Haggar et al., 1992) were used to measure the respiration rates as a function of CO_2 concentrations at temperatures 20 ^{o}C. The experiments were replicated three times. The experimental respiration rates were calculated using the method of Gong and Corey (1994):

$$RCO_2 = ([CO_2]^{t+1} - [CO_2]^t) \times V/(100 \times W \times \Delta t)$$

where: V is the free volume of container in ml; W is the weight of banana fruits in kg, $[CO_2]_t$, $[CO_2]_{t+1}$ are concentrations of CO_2 (t) and (t+1) respectively in %; Δt is the time interval between (t) and (t+1) in hour. Gas samples from the closed container were taken periodically through an airtight septum. The samples were analyzed for CO_2 concentrations by a CheckPoint portable analyzer. The free volume of closed container was 5554 ml and weight of banana fruits inside the container was 1000 g. The respiration rates of banana fruits was calculated and converted to mg/or ml of CO_2 released by 1 kg of banana fruits in an hour (ml CO_2/kg/h).

Statistical analysis was applied by using Paswstat 18 to determine significance of differences between means.

3. RESULT AND DISCUSSION

3.1. TEM image of cellulose nanocrystals

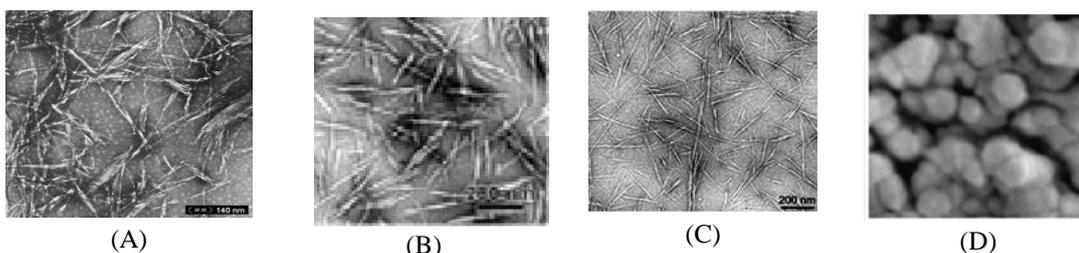(A)   (B)   (C)   (D)

Figure 1. TEM image of structure of cellulose particles obtained at different sulfuric acid concentration. (A)- MCC; (B) – sulfuric acid 47 %; (C) - sulfuric acid 55 %; (D) - sulfuric acid 64 %.

Cellulose nanocrystals (CNC) obtained from acid-catalyzed hydrolysis microcrystalline cellulose (MCC) at three sulfuric acid concentration was 47, 55 and 64 %. TEM images of structure of cellulose particles are shown in Figure 1. The result indicated that when MCC was

hydrolyzed in condition of concentration of sulfuric acid in range from 47 to 55 wt. %, structure of CPs had rod-like shape of about 9×196 nm. According to other researchers disclosed that treatment of the cellulose in low concentration of sulfuric acid, the obtained particles retain crystallinity that is similar to the crystallinity of the initial MCC sample. Therefore, in order to obtain the nano-scale particles, the concentration of sulfuric acid should be higher than 50 wt. %. However, in this case, CNC obtained due to initial cellulose source is MCC with small size (about 1.4 µm), so MCC easy to solve in the acid solution with its concentration is 47 wt. % at 60 °C for 2 hours. The nanocrystalline particles isolated by treatment of initial MCC sample with about 55 wt. % SA, have a rod-like shape of about 160 × 11 nm. The result gained similar to other researchers [7]. When hydrolysis MCC with concentration of SA is 64 wt. %, MCC fully dissolves; moreover the dissolving process is accompanied by de-polymerization of cellulose macromolecules in the hot acid and falling of the yield. After diluting of the solution with water, flocks of the low-molecular amorphous cellulose are precipitated. Under the high-power disintegration in water medium these flocks turn into amorphous nanoparticles; as a result instead of nanocrystalline particles the amorphous particles are formed with wide size distribution (from 42 - 62.7 nm) are observed. The amorphous particles have poor mechanical characteristics 0.5 - 0.7 GPa; TS = 40 - 50 MPa) and therefore these particles cannot be suitable as a reinforcing nano-filler [7]. Overall, to obtain CNC order to reinforcing material, MCC should be hydrolyzed in the acid range from 47 to 55 wt. % at 60 °C for 2 hours.

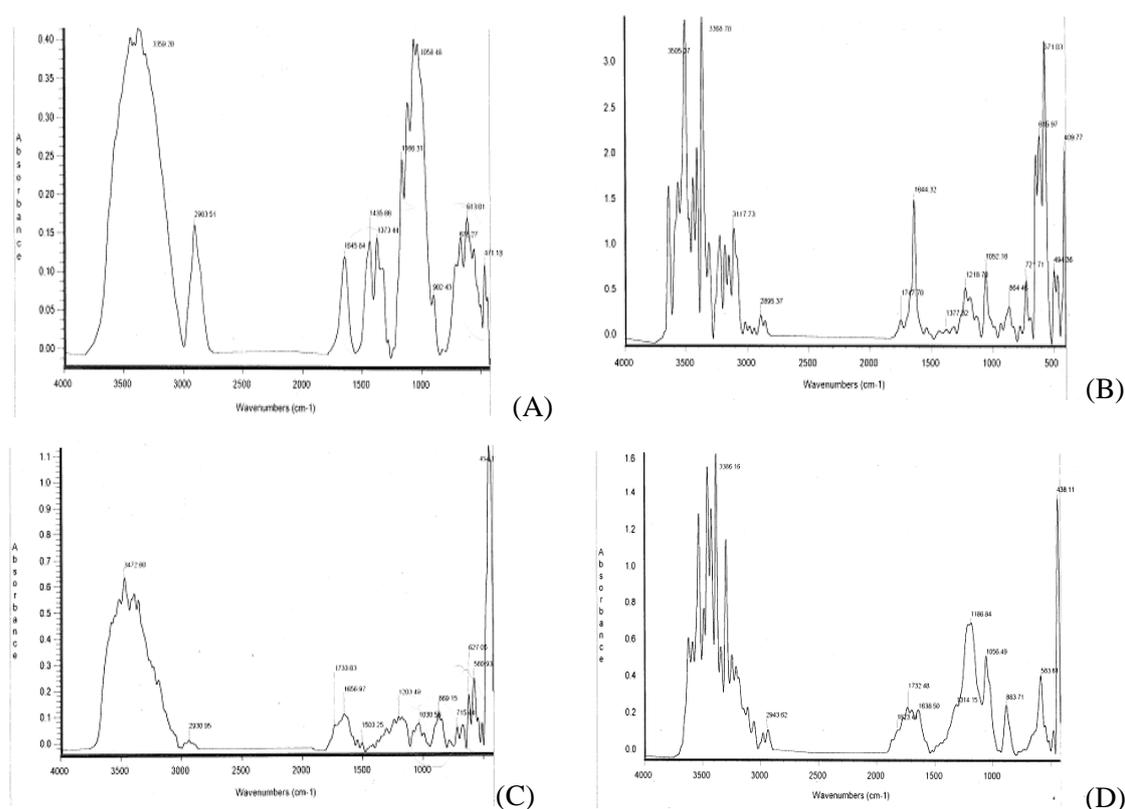## 3.2. Fourier transform infrared (FTIR) analysis

Figure 2. FT-IR diagram of celluloses nanocrystals obtained at different sulfuric acid concentration.
(A)- MCC; (B) – sulfuric acid 47 %; (C) - sulfuric acid 55 %; (D) - sulfuric acid 64 %.

Figure 2 displayed FTIR spectra of precusor and CNC got from three different concentration of acid: 47, 55 and 64 wt. %. The spectra of the precursor and the corresponding cellulose nanoparticles exhibited a broad band in the region 3500 - 3200 cm^{-1} that indicates the free O-H stretching vibration of the OH groups in cellulose molecules. Moreover, the spectra of all samples showed the characteristic C-H stretching vibration around 2900 cm^{-1} [10]. Besides, the transmittance peaks observed in the spectra of nano-cellulose obtained after chemo-mechanical treatments in the region 1649 - 1641 cm^{-1} are attributed to the O-H bending of the adsorbed water [10]. The peak observed in the spectra of the all samples at 1030 - 1058 cm^{-1} is due to the C-O-C pyranose ring (antisymmetric in phase ring) stretching vibration. The most significant absorption band which continually prominent on acid hydrolysis, respectively, of all the cellulose nanoparticles is that in the range from 864 to 883 cm^{-1} (associated with the glycosidic linkages between glucose units in cellulose) which stands for cellulose, the content of which increases progressively from precursor to cellulose nanoparticles. The C-C ring breathing band at 1150 cm^{-1} and the C-O-C glycosidic ether band at 1100 cm^{-1} both of which arise from the polysaccharide component is getting gradually lost or merge with 1050 cm^{-1} peak in cellulose nanoparticles because of hydrolysis and reduction in molecular weight [10].

3.3. Particles size and Zeta potential of the synthesized nanoparticles

The results of size and zeta potential were shown in Table 1. The nanocrystalline particles isolated by treatment of initial MCC sample with 47 wt. % SA, CNC had diameter and length of 196×9 nm with zeta potential is -34.1 mV. When the acid concentration of 55 wt. %, the length of CNC had a rod-like shape of 160×11 nm with zeta potential is -32.1 mV. CNC obtained from processing hydrolysis MCC in the acid 64 wt. % SA had a ovan shape with medium size around 67 nm and zeta potential is -29.9 mV. In this way, with a zeta potential ≤ -32.5 mV, CNC obtained at 47 wt. % and 55 wt. % have rich mechanical characteristics.

Bảng 1. The properties of CNC at different concentration of sulfuric acid.

Concentration of H_2SO_4 (wt. %)	Size particles (nm)	Zeta potential (mV)
47	9×196 (± 4)	-34,1
55	11×160 (± 4)	-32,5
64	67 (± 4)	-29,9

3.4. The effect of cellulose nanocrystals in the HPMC based composite

The structure of HPMC-CNe-ChNp nanocomposite when added CNC at different levels was displayed in Scanning electron microscopy (SEM) images (Fig. 3). SEM images reveal that structure of nanocomposite was different when CNC was added at different levels. The nanocomposite became more compact and dense when cellulose nanocrystals were added at levels 0.2 and 0.5 wt. %. The cellulose nanocrystals occupy empty spaces of porous of matrix nanocomposite HPMC increasing the collapse of the pores in the films. Thus water solubility of film decreased significantly and thickness increased lightly. The results also indicated that when CNC was added in to the composite at 0.5 wt. %, water solubility of HPMC based composite film is the smallest. However, when CNC concentration increased 0.8 and 1.1 wt. %, both of values of water solubility and thickness of film were upward trend.
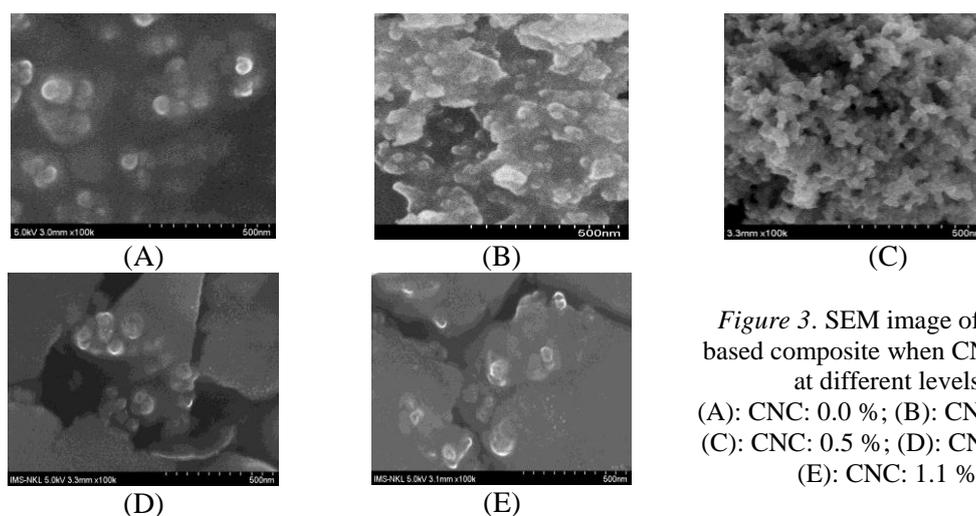
(A)   (B)   (C)

Figure 3. SEM image of HPMC based composite when CNC added at different levels.
(A): CNC: 0.0 %; (B): CNC: 0.2 %; (C): CNC: 0.5 %; (D): CNC: 0.8 % (E): CNC: 1.1 %.

(D)   (E)

3.5. The effects of CNC on properties of HPMC - based composite film

Table 2. The effect of CNC on properties of HPMC-based composite film.

Formula	Concentration of CNC (%)	Thickness (µm)	Drying time (min)
CT1	0.0	21.3[a]	53.17[a]
CT2	0.2	22.3[b]	47.33[b]
CT3	0.5	23.3[c]	51.33[c]
CT4	0.8	27.3[d]	53.67[ad]
CT5	1.1	31.3[e]	55.33[e]

Note: Values in the same column having at least a same letter are not significantly different at $\alpha \leq 0,05$
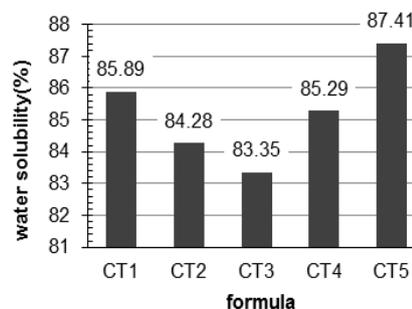

Figure 4. The effect of CNC on water solubility of the films of HPMC based composite films.

The first investigation about the reinforced effect of cellulose nanocrystals on properties of nanocomposite HPMC that incorporated with both of CNe and ChNp with concentration of CNC at levels: 0.2, 0.5, 0.8 and 1.1 %, which were demonstrated in Table 2. The results displayed that thickness of the nanocomposite films were 21.3, 22.3 and 23.3 µm corresponding to concentration of CNC at levels 0.0, 0.2 and 0.5 wt. % added into nanocomposite. As can be seen clearly, the thickness of the film significant increased when CNC added into nanocomposite more than 0.8 wt. %, which may be lead to not enough levels of O_2 to create anaerobic conditions inside the coated fruit. Drying time of film was changed as CNC was added (Table 2). Films added with CNC of 0.2 % and 0.5 % had reduced drying time 11 % and 3.5 %, respectively compared to film without CNC. Drying time of 0.8 % added CNC film lightly increased about 53.67 minutes (significantly $\alpha \leq 0.05$). When concentration of CNC increased 1.1 %, drying time enhanced more 4.1 %.

The effect of CNC on water solubility of HPMC-CNe-ChNp film was shown in Fig. 4. In there, water solubility of films varried due to CNC added into nanocomposite at different levels.

Water solubility of films were 85.89, 84.28, 83.35, 85.29 and 87.41% corresponding to CNC added at levels 0.0, 0.2, 0.5, 0.8 and 1.1 %, respectively. Minimum water solubility obtained at film added CNC of 0.5 % (significantly $\alpha \le 0.05$).

3.6. The effect of CNC on gas and moisture barrier of the nanocomposite coating

3.6.1. The moisture barrier of coating

The water-loss is occurred in fresh commodities after harvesting due to the transpiration from fruit/vegetable to environment. The coating retarded the water loss rate of coated fruits, leading to weight loss reduction and thus resulting maintaining their postharvest quality. The effect of CNC mixing ratio on moisture barrier of the HPMC-CNe-ChNp coating was determined by weight loss and showed in the Fig. 5. The weight loss on all samples was significant differences ($\alpha \le 0.05$) at day 8, 12 and 16. Especially, at day 16 the weight loss percentage of coated banana using nanocomposite coating without CNC was increased in range 24 – 60 % compared to other samples. The higher CNC ratio using in coating leading to the lower mass loss because there were differences in the thickness and structure of coating thus resulting restricted water loss of banana during a prolonged storage period.
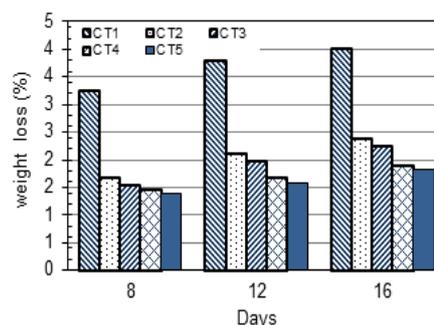

Figure 5. The effect of CNC on weight loss of banana during 16 days of storage at 20 °C.

3.6.2. The exchanged gas of coating

Nanocomposite coating not only limited water loss rate but also reduced the permeability of O_2 and CO_2. The respiration rate of coated fruit reduced depending on the gas barrier of coating. Figure 6 presented respiration rate of coated and uncoated banana during 16 days of storage. In comparison to sample coated by HPMC based composite coating without CNC, the respiration reduced 25.0, 34.6, 41.6 and 47 % at CNC concentration of 0.2, 0.5, 0.8 and 1.1 %, respectively. This means that the range of CNC concentration from 0.5 % to 1.1 %, the nanocomposite coatings were enhanced gas barrier consequently respiration rate of coated banana reduced.
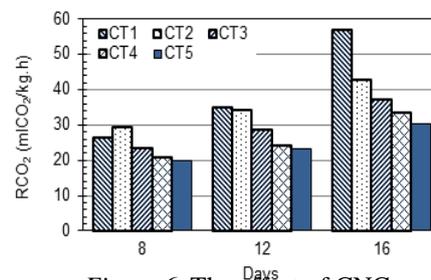

Figure 6. The effect of CNC on respiration rates of banana during 16 days of storage at 20 °C.

4. CONCLUSION

Characterization of cellulose nanocrystals such as particle size, zeta-potential, FT-IR and TEM were characterized. Beside that the reinforced effect of cellulose nanocrystals on properties of nanocomposite HPMC - based films or coatings that incorporated with both of carnauba nanoemulsion and chitocan nanoparticles was assessed. Results showed the shape and size of the nanocrystal generally depend on hydrolysis condition of microcrystalline cellulose. The morphology of CNC was the best when the MCC was hydrolysised in range of 47 – 55 % of sulfuric acid concentration at 60 °C for 2 hours. The Scanning electron microscopy (SEM)

images were demonstrated about the effect of CNC on nanocomposite films or coatings. The nanocomposite films contained 0.5 % CNC was improved the mechanical of film and was the best film compared with other samples such as: film solubility declined 16.65 %, film drying time is faster (51.33 minutes). Coating was enhanced gas and moisture barrier and hence respiration rate of coated bananas reduced from 57.08 ml CO_2/kg.h to 30.17 ml CO_2/kg.h while weight loss reduced by 54.5 %.

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TÓM TẮT

CELLULOSE NANOCRYSTALS: TỔNG HỢP, ĐẶC TÍNH VÀ TÁC DỤNG TỚI MÀNG COMPOSITE CHỨA HYDROXYPROPYL METYLCELLULOSE

Nguyễn Thị Minh Nguyệt[*], Nguyễn Duy Lâm, Phạm Anh Tuấn

Viện Cơ điện nông nghiệp và Công nghệ sau thu hoạch,
60 Trung Kính, Trung Hòa, Cầu Giấy, Hà Nội

[*]Email: *minhnguyet.viaep@gmail.com*

Mục đích của nghiên cứu là tổng hợp được cellulose nanocrystals, xác định được các đặc trưng cơ bản như kích thước hạt, phân bố hạt, thế zeta, phổ FT-IR và TEM, đồng thời có được đánh giá về tác dụng của loại nano này khi bổ sung vào composite HPMC có chứa nano nhũ tương sáp carnauba và nano chitosan tới một số đặc trưng của màng film và màng phủ. Kết quả cho thấy, hình dạng và kích thước hạt phụ thuộc nhiều vào điều kiện thủy phân. Ở nồng độ H_2SO_4 là 47 % và 55 % cho hạt cellulose nanocrystals có dạng hình que với kích thước chiều dài nằm trong khoảng 160 - 196 nm và chiều rộng đạt 9 - 11 nm. Cellulose nanocrystals đã được bổ sung vào nanocomposite HPMC có chứa đồng thời cả nano nhũ tương carnauba và nano chitosan với tỉ lệ (w/v) 0,0; 0,2; 0,5; 0,8 và 1,1 %. Hình ảnh TEM chụp cấu trúc bề mặt của nanocomposite cho thấy cấu trúc của nanocomposite đã trở lên chặt chẽ hơn nhờ CNC đã xen vào vị trí không gian trống trong cấu trúc của polymer làm. Cellulose nanocrystals được bổ sung vào nanocomposite HPMC ở nồng độ 0,5 % đã cải tiến chất lượng của màng đạt được đặc tính cơ lí tốt nhất như độ bền nước của màng tăng lên (16,65 %), thời gian khô nhanh hơn (51,33 phút). Đánh giá tác dụng bổ sung cellulose nanocrystals của màng phủ trên bề mặt quả chuối cho thấy màng được tăng cường khả năng cản trao đổi khí và trao hơi nước vì đã làm giảm cường độ hô hấp của quả từ 57,08 ml CO_2/kg.h xuống 30,17 ml CO_2/kg, giảm mức hao hụt khối lượng từ 4,0 % xuống còn 1,82 %.

Từ khóa: carnauba nanoemulsion, cellulose nanocrystals, HPMC, nano chitosan.