Characterization of cellulose nanocrystal with cellulose II polymorph from primary sludge and its application to PVA nanocomposites

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Wood Sci Technol (2018) 52:555–565
https://doi.org/10.1007/s00226-017-0976-8

ORIGINAL

Characterization of cellulose nanocrystal with cellulose II polymorph from primary sludge and its application to PVA nanocomposites

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Received: 31 March 2017 / Published online: 24 November 2017

Abstract Cellulose nanocrystals with cellulose II polymorph (CNC-II) were obtained from primary sludge fiber (PSF) in a pulp and paper mill by sulfuric acid hydrolysis after purification to remove inorganic materials and lignin. The CNC-IIs obtained were applied as reinforcing fillers for polyvinyl alcohol (PVA). Characterization of CNC-IIs was performed using FTIR spectroscopy and X-ray diffraction. The morphology, conductivity, and tensile properties of CNC-IIs-reinforced PVA nanocomposites were also investigated. Purification treatment effectively reduced non-cellulosic material in the sludge, increasing the cellulose content from 39.87 to 76.34%. The conductivity and tensile properties of the PVA/CNC-II nanocomposite was better than those of neat PVA and PVA/PSF composite.

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Introduction

Utilization of agricultural and forestry wastes for the production of cellulose resources has increased because of its abundant availability and low cost (Brinchi et al. 2013; Neto et al. 2013; Johar et al. 2012; Dai et al. 2013; Fahma et al. 2010). Sludge from pulp and paper mills also has the potential to be used as an alternative cellulosic resource. It is mainly composed of cellulose fibers (50–60%) and inorganic materials such as minerals and ashes (Mehmood et al. 2010), which are difficult to dispose of. Efficient recycling of the sludge into value-added products can lead to environmental and economic benefits.

Cellulose fiber from sludge has been used to prepare cellulose nanofiber (CNF). Jonoobi et al. (2012) have produced low-cost CNF from sludge by dissolving cellulose produced by ultrafine grinding. The defibrillation efficiency of sludge fiber was found to be better than that of dissolving cellulose. The CNF obtained had a diameter less than 100 nm and a specific surface area of 112 m^2/g. They insisted that the preparation of CNFs from sludge could be considered as an economic, energy efficient, and viable approach to generating value-added products from cellulose sludge while minimizing sludge disposal issues. Leao et al. (2012) have reported on the preparation of cellulose nanofibers from primary sludge and its effectiveness as reinforcement fillers for polymeric matrices to improve the mechanical, optical, and dielectric properties of matrix polymers.

Polyvinyl alcohol (PVA) is one of the most popular biodegradable and hydrophilic polymers and is known to have strong interactions with cellulose by forming hydrogen bonds. This strong interaction between cellulose and PVA results in miscible blends (Nishio and Manley 1988; Nishio et al. 1989). PVA has been widely used as the polymer matrix in the manufacture of composite films because of its many advantages including flexibility, resistance to solvents, and the wide spectrum of applications that can be improved by incorporating fibers (Khiar and Arof 2009). Roohani et al. (2008) have prepared and characterized cellulose nanocrystal (CNC)-reinforced PVA nanocomposites. The results obtained showed that the higher hydrolyzing degree of polyvinyl acetate caused the generation of more hydroxyl groups, resulting in stronger filler/matrix interactions compared to partially hydrolyzed samples. The reinforcing effect was found to be higher as the hydroxyl group content of the matrix increased. Peresin et al. (2010) used CNC to reinforce electrospun PVA nanofibers with different acetyl group contents. The elastic modulus of the nanocomposite mats increased significantly as a consequence of strong interactions due to the hydrogen bonds between CNC and fully hydrolyzed PVA electrospun fibers.

In the present study, CNC-IIs from primary sludge fibers (PSFs) were prepared using the sulfuric acid hydrolysis process combined with ultrasonication after the purification of primary sludge. PVA–PSF composite and PVA–CNC-IIs nanocomposites were prepared, and their morphology, transparency, conductivity, and tensile properties are compared.

Materials and methods

Materials

Primary sludge was obtained from PT. Indah Kiat Pulp and Paper Products in Serang, Indonesia. Sulfuric acid (95%) and other chemicals such as ethanol, benzene, sodium chlorite ($NaClO_2$), acetic acid, sodium hydroxide, and hydrochloric acid were purchased from Sigma-Aldrich.

Methods

Purification of primary sludge

Purification treatment was conducted using the method proposed by Fahma et al. (2010) with some modification to isolate the cellulose fiber from primary sludge prior to acid hydrolysis. The primary sludge was extracted with ethanol/benzene (1:2 v/v) for 8 h to remove the extractive components (resins, oils, fats, and waxes) and then washed with ethanol to remove benzene, followed by washing with distilled water. The extractive-free fibers were bleached with 1.25 wt% sodium chlorite solution in acetate buffer (pH = 4–5) at 70 °C for 4 h, and washed repeatedly with distilled water until neutral. To remove inorganic materials, the fibers were treated with 1 M aqueous hydrochloric acid solution followed by repeated washing with distilled water until neutral. Subsequently, they were oven-dried at 50 °C. The resultant cellulose fibers (1 g) were then treated with a 17.5% sodium hydroxide aqueous solution (w/w) (25 mL) for 2 h at 20 °C and washed several times with distilled water until neutral. During this solid-state process, cellulose fibers were swollen in alkali and recrystallized into cellulose II polymorph.

CNC-II preparation

The cellulose fibers obtained above were hydrolyzed with sulfuric acid solution (50% v/v) at 45 °C for 30 min (denoted as CNC-II-30) and 60 min (CNC-II-60) with constant agitation. The cellulose fiber–sulfuric acid solution ratio was 1:20. Distilled water was added and centrifuged. The supernatant was removed and the precipitated reactant was diluted with distilled water, followed by centrifugation. This process was repeated until a constant pH (6.5) was obtained for the precipitated reactant. Subsequently, the reactant was sonicated for 60 min to improve the dispersion in water and then stored in a refrigerator at 4 °C.

Chemical composition analysis

The chemical composition of the primary sludge before and after purification was determined in accordance with TAPPI standards (TAPPI T204 om-88 1988a; TAPPI T222 om-88 1988b; TAPPI T211 om-93 1993). The holocellulose content was measured by the acid chlorite method (Browning 1967), and the α-cellulose content was determined by treating the holocellulose with 5 wt% potassium hydroxide solutions (Browning 1967).

The elemental compositions of the primary sludge before and after purification were determined by energy-dispersive X-ray spectroscopy (EDS; JEOL JED 2300/2300F, Japan).

Characterization of purified primary sludge and CNC-II

Fourier transform infrared (FTIR) spectra were acquired using an FTIR (ABB MB 3000, Canada). Samples were vacuum-dried at 80 °C for 1 h, and the standard KBr pellet method was employed for all measurements. Wide-angle X-ray diffraction (WAXD) was measured in reflection mode (Shimadzu XRD-7000 MaximaX, Japan). Nickel-filtered Cu-K_α radiation (wavelength of 0.154 nm) was used at 40 kV and 30 mA. The diffraction intensity profiles were collected in the 2θ range 10°–40°.

Preparation of nanocomposite films

CNC-II and PSF-reinforced PVA nanocomposite films with contents of 1, 3, and 5% CNC-IIs and PSFs were prepared using the casting method. A suspension of CNC-II and PVA obtained by stirring at 800 rpm and 80 °C for 1 h was casted on Teflon. The obtained films were then vacuum-dried at 40 °C for 24 h.

Characterization of nanocomposite films

The transmittances of the nanocomposite films were measured from 250 to 850 nm using a UV–Vis Spectrophotometer (Ocean Optic 2000, USA). The electrical conductivity was measured with an LCR Hi-Tester (Hioki 3522-50, Japan) in the frequency range 1–1000 kHz. The nanocomposite film was placed between the capacitor plates and then the conductance (G) was measured. The electrical conductivity value was determined from the following equation (Khiar and Arof 2009):

$$\sigma = \frac{l}{A} \times G$$

where σ is the electrical conductivity (S/cm), l is the sample thickness (cm), A is the sample area, and G is the conductance (S). Tensile properties of ten specimens were measured with a Shimadzu Autograph AG-IS 1 kN (Japan) at crosshead speed of 5 mm/min, according to ASTM D882-75b.

Results and discussion

Chemical composition of primary sludge before and after purification

The chemical compositions of the primary sludge before and after purification are shown in Table 1. The cellulose content after purification increased from 39.87 to 76.34% because the non-cellulosic components (i.e., extractives, hemicellulose, lignin, and ash) were removed during the purification process. The lignin and ash contents decreased remarkably. Primary sludge generally contains inorganic materials such as kaolin, clay, and calcium carbonate (Girones et al. 2010; Gomez and de Alda 2008). Table 2 shows the elemental analysis results of the primary sludge before and after purification by EDS. The mass of C and O increased by 6.42 and 3.32% after purification, respectively. The mass of Ca decreased remarkably, whereas the masses of Al, Si, S, and Zr did not change significantly.

FTIR spectroscopy

FTIR spectra of the primary sludge, purified primary sludge, and CNC-IIs are shown in Fig. 1. The peaks near 3500 cm^{-1} in all spectra are representative of OH groups and suggest the presence of cellulose (Johar et al. 2012). The peak intensity at 1420 cm^{-1} due to lignin decreased in the purified sludge, CNC-II-30, and CNC-II-60, which might be due to the removal of lignin during the purification process (Fahma et al. 2011). In typical FTIR spectra of cellulose, peaks at 750 and

Table 1 Chemical composition of primary sludge before and after purification

Component	Mass (%)	
	Before purification	After purification
Extractives	4.49	0.61
Hemicellulose	20.49	12.88
Cellulose	39.87	76.34
Lignin	21.62	2.76
Ash	33.47	4.96

Table 2 EDS data of primary sludge before and after purification

Element	Mass (%)	
	Before purification	After purification
C	38.58	45.00
O	43.65	46.97
Al	1.11	1.29
Si	1.50	1.32
S	0.41	0.31
Ca	10.63	0.25
Zr	4.12	4.86

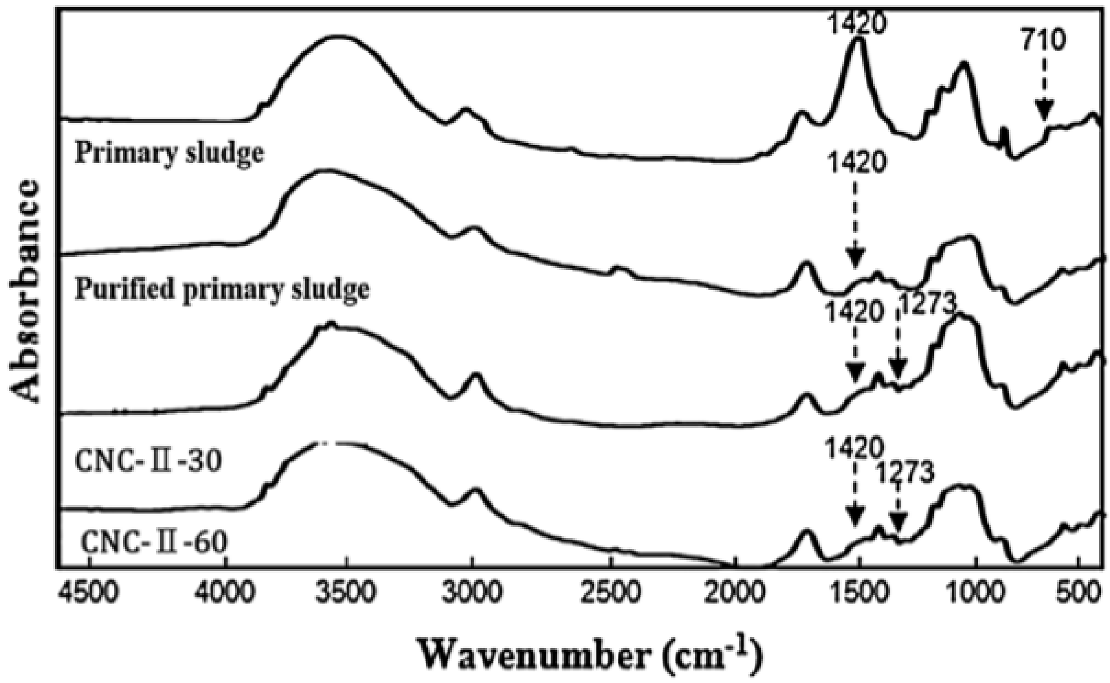

Fig. 1 FTIR spectra of primary sludge, purified primary sludge, and CNC-IIs

710 cm^{-1} are representative of cellulose Iα and Iβ, respectively, but no peak at 750 cm^{-1} was detected in any FTIR spectrum. Meanwhile, only the peak at 710 cm^{-1} was seen in the sludge spectra, indicating that the Iβ crystalline polymorph of cellulose was dominant in the sludge (Zuluaga et al. 2009; Fahma et al. 2011). The peak at 710 cm^{-1} was not detectable in the spectra of CNC-II-30 and CNC-II-60, suggesting transformation from cellulose I to cellulose II.

X-ray diffraction analysis

The X-ray diffraction patterns in Fig. 2 show that the sludge was mainly composed of cellulose and calcium carbonate phases. This finding agrees with the finding of Girones et al. (2010) that sludge is primarily composed of cellulose fibers and inorganic materials. The calcium carbonate peak disappeared after the purification treatment. The three peaks at $2\theta = 14.80°$ (110), 16.20° ($1\bar{1}0$), and 22.70° (200) confirmed that only cellulose I was present in the primary sludge sample. Purification treatment changed the three peaks to 12.34°(110), 19.95° ($1\bar{1}0$), and 21.85° (200), indicating the formation of the cellulose II structure (Yue 2011; Neto et al. 2013). This change occurred because of alkaline treatment during purification. As reported by Kim et al. (1990) and Zugenmaier (2008), the structure of cellulose I can be converted to cellulose II when treated with a high concentration of sodium hydroxide. The X-ray diffraction patterns of CNC-II-30 and CNC-II-60 also showed the structure of cellulose II.

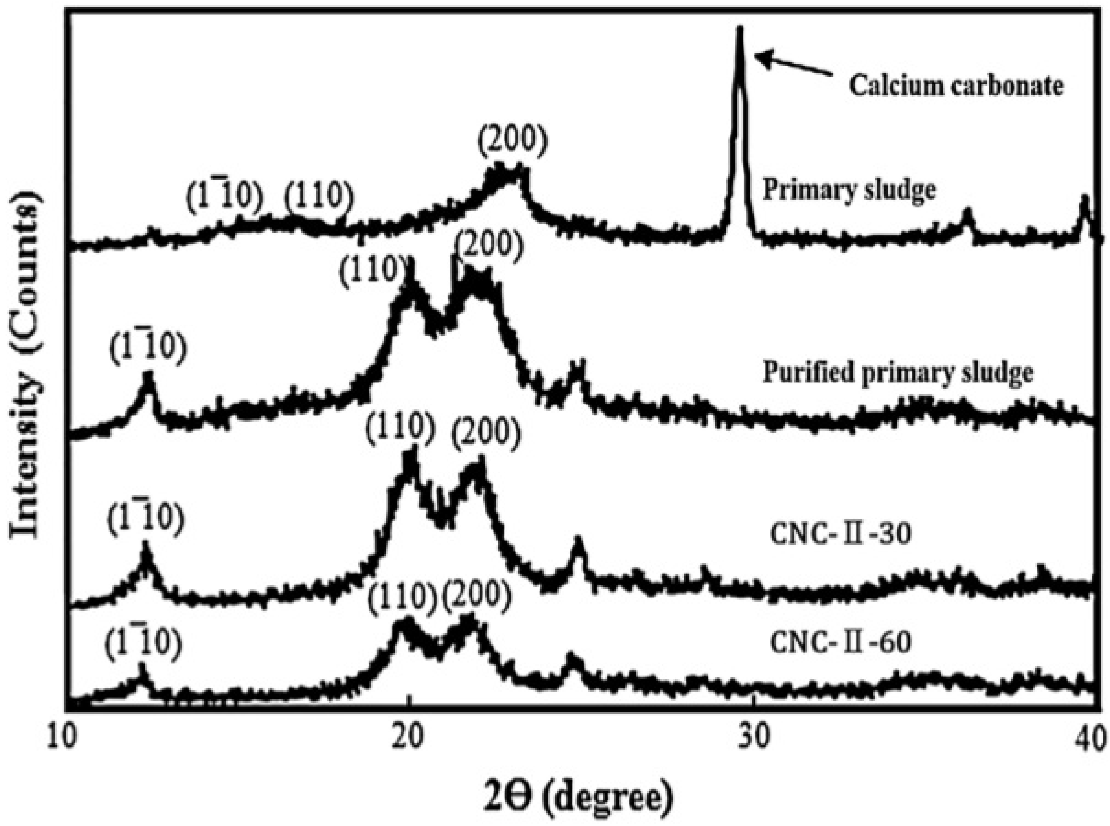

Fig. 2 WAXD profiles of primary sludge, purified primary sludge, and CNC-IIs

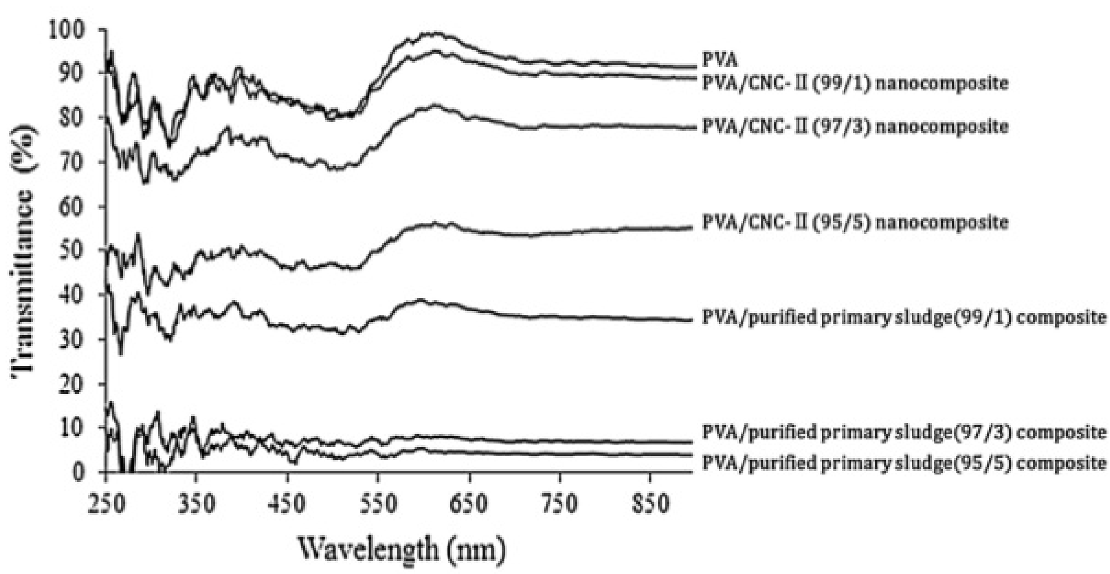

Fig. 3 UV–Vis spectra of neat PVA film and composites with purified primary sludge and CNC-IIs

Physical and tensile properties of CNC-II-reinforced PVA nanocomposite

Figure 3 shows the UV–Vis transmittance spectra of the nanocomposites. PVA film is a transparent polymer with a transmittance of 93% at 600 nm (wavelength). The 1 and 3% CNC-II-reinforced PVA nanocomposites had transmittances of 80%. On the other hand, the nanocomposite reinforced with 5% CNC-IIs had a transmittance of 54%. Uniform distribution of the CNC-IIs in the PVA matrix brings about the transparent nature of the nanocomposites (Fortunatia et al. 2013a). The composite films reinforced with 1, 3, and 5% fiber of the purified sludge had low transparent values, namely 35, 6, and 4%, respectively. This may be due to poor distribution of the fibers in purified sludge in the PVA matrix.

Figure 4 shows the conductivity values of the PVA film and the nanocomposite films with purified primary sludge and CNC-IIs. The conductivities of the composites with CNC-II were higher than that of neat PVA, which was higher than those of the purified sludge embedded in PVA (at different levels). The conductivity increased with increasing fiber content in CNC-II-reinforced nanocomposites, but decreased in the composite with cellulose fiber of the purified sludge.

Figure 5 shows the tensile properties of the composites. The tensile strength and tensile modulus of the 1 and 3% CNC-II-reinforced nanocomposites were higher than those of the neat PVA films. In particular, 3% CNC-II addition remarkably increased the elongation at break. This indicates that the homogeneous dispersion of CNC-II enhanced the formation of hydrogen bonds between PVA and CNC-II (Fortunatia et al. 2013a). Yue (2011) reported that the change of cellulose I to cellulose II can increase tensile properties. The tensile properties of the PVA/PSF composite were lower than those of the nanocomposites with CNC-IIs, indicating that the larger surface area and reduced size might be the cause for the beneficial effects of CNC-IIs on tensile properties. Fortunati et al. (2013b) reported the PVA/CNC nanocomposite with CNC extracted from Okra fibers. Young's modulus and elongation at break of the PVA/CNC nanocomposite with 1, 2, and 5% CNC ranged from 700 to 120 MPa and 10–130%, respectively. They suggested that 5 wt% CNC addition is ideal to improve mechanical interaction between the PVA and cellulose structures.

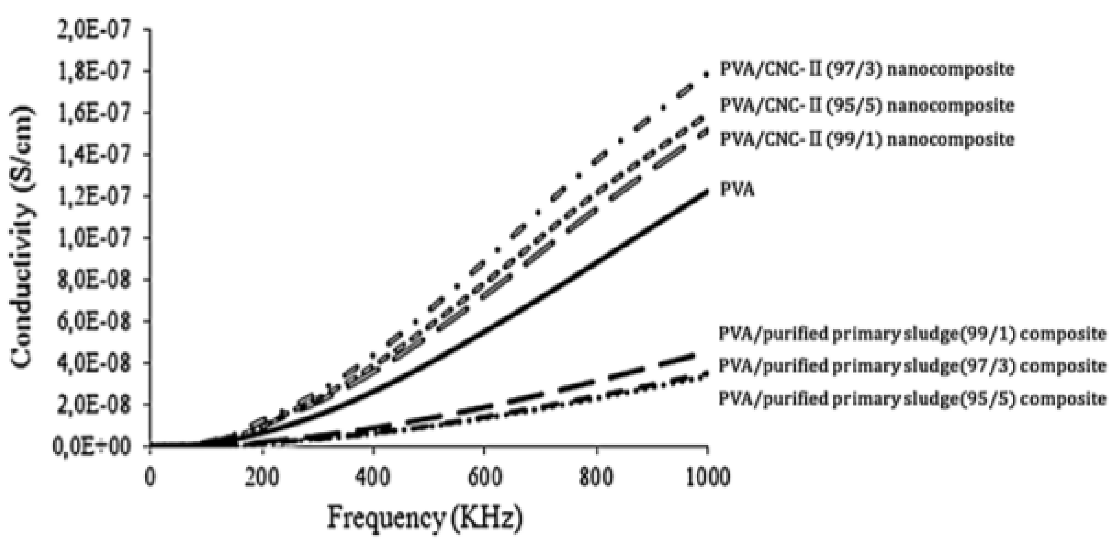

Fig. 4 Conductivity values of neat PVA film and composites with purified primary sludge and CNC-IIs

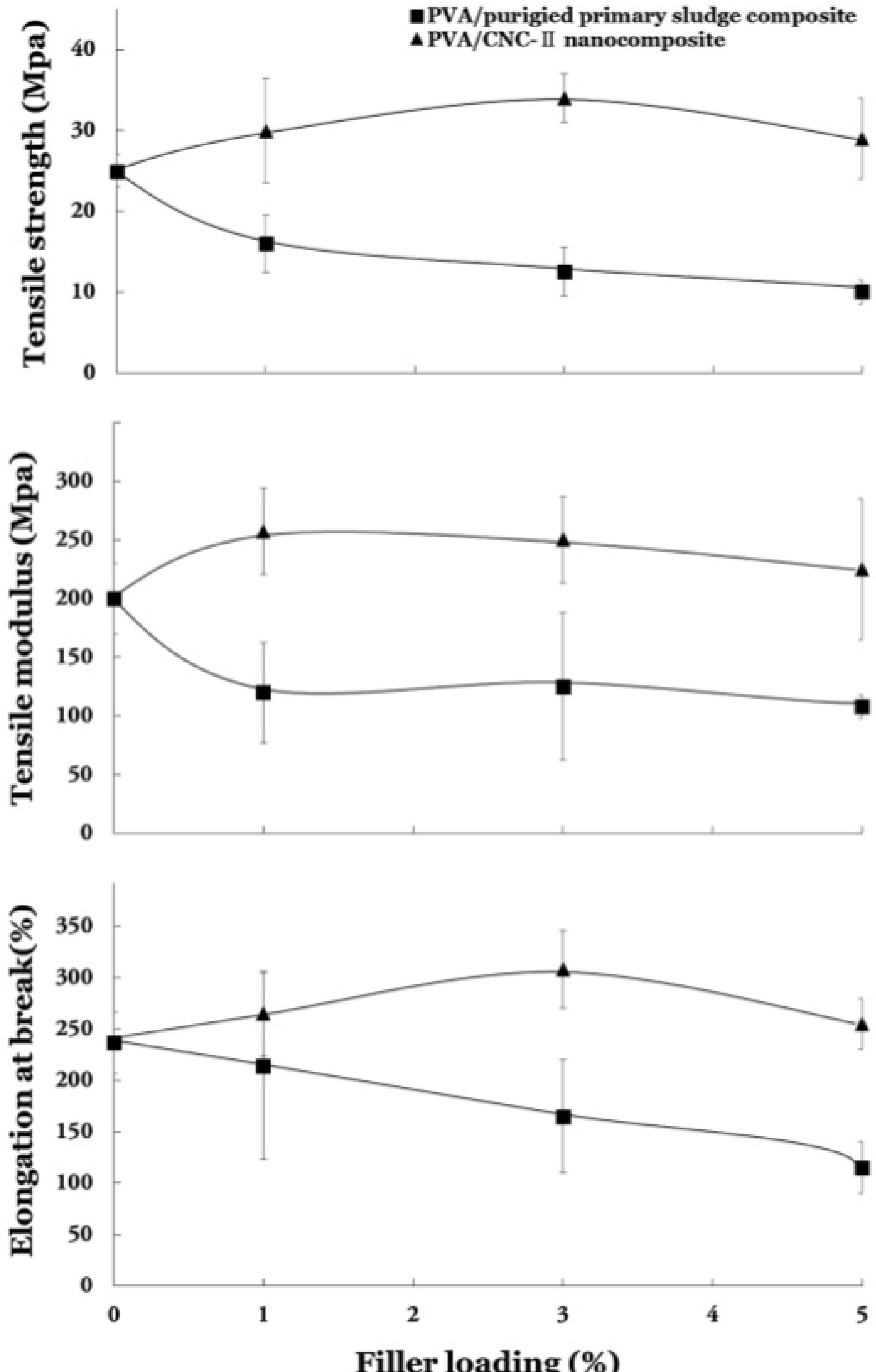

Fig. 5 Tensile strength, tensile modulus, and elongation at break of PVA film and composites with purified primary sludge and CNC-IIs

Conclusion

CNC-IIs were successfully prepared by the sulfuric acid hydrolysis of cellulose fiber in primary sludge after purification. The non-cellulosic materials such as lignin, hemicellulose, and inorganic materials in primary sludge were remarkably reduced after purification treatment. During alkali treatment in the purification process, crystalline polymorphs of cellulose I changed to cellulose II, which is more stable and less reactive than cellulose I. The physical and tensile properties of CNC-II-reinforced nanocomposites were improved relative to those of the neat PVA film and the composite reinforced with PSFs.

Acknowledgements The authors would like to thank the Korean Forest Service for supporting this study.

References

Brinchi L, Cotana F, Fortunati E, Kenny JM (2013) Production of nanocrystalline cellulose from lignocellulosic biomass: technology and applications. Carbohydr Polym 94:154–169
Browning BL (1967) Methods of wood chemistry, vol II. Wiley, New York
Dai D, Fan M, Collins P (2013) Fabrication of nanocelluloses from hemp fibers and their application for the reinforcement of hemp fiber. Ind Crops Prod 44:192–199
Fahma F, Hori N, Iwamoto S, Iwata T, Takemura A (2010) Isolation, preparation, and characterization of nanofibers from oil palm empty-fruit-bunch (OPEFB). Cellulose 17:977–985
Fahma F, Hori N, Iwamoto S, Iwata T, Takemura A (2011) Effect of pre-acid-hydrolysis treatment on morphology and properties of cellulose nanowhiskers from coconut husk. Cellulose 18:443–450
Fortunatia E, Puglia D, Luzi F, Santulli C, Kenny JM, Torrea L (2013a) Binary PVA bio-nanocomposites containing cellulose nanocrystals extracted from different natural sources: part I. Carbohydr Polym 97:825–836
Fortunatia E, Puglia D, Monti M, Santulli C, Maniruzzaman M, Kenny JM (2013b) Cellulose nanocrystals extracted from okar fibers in PVA nanocomposite. J Appl Polym Sci 128(5):3220–3230
Girones J, Pelach MA, Pardini G, Mutje P, Vilaseca F (2010) Recycling of paper mill sludge as filler/reinforcement in polypropylene composites. J Polym Environ 18:407–412
Gomez JA, de Alda O (2008) Feasibility of recycling pulp and paper mill sludge in the paper and board industries. Resour Conserv Recycl 52(7):965–972
Johar N, Ahmad I, Dufresne A (2012) Extraction, preparation and characterization of cellulose fibres and nanocrystals from rice husk. Ind Crops Prod 37:93–99
Jonoobi M, Mathew AP, Oksman K (2012) Producing low-cost cellulose nanofiber from sludge as new source of raw materials. Ind Crops Prod 40:232–238
Khiar ASA, Arof AK (2009) Conductivity studies of starch-based polymer electrolytes. Ionics 16(2):123–129
Kim NH, Sugiyama J, Okano T (1990) X-ray and electron diffraction study of Na-cellulose I: formation and its reconversion back to cellulose I. Mokuzai Gakkaishi 36(2):120–125
Leao AL, Cherian BM, Souza SFD, Sain M, Narine S, Caldeira MS, Toledo MAS (2012) Use of primary sludge from pulp and papermills for nanocomposites. Mol Cryst Liq Cryst 556:254–263
Mehmood S, Khaliq A, Ranjha SA (2010) The use of post consumer wood waste for the production of wood plastic composites: a review. In: Third international symposium on energy from biomass and waste, Venice, Italy, 8–11 Nov 2010
Neto WPF, Silverio HA, Dantas NO, Pasquini D (2013) Extraction and characterization of cellulose nanocrystals from agro-industrial residue–Soy hulls. Ind Crops Prod 42:480–488
Nishio Y, Manley RSJ (1988) Cellulose/poly(vinyl alcohol) blends prepared from solutions in *N*,*N*-dimethylacetamide-lithium chloride. Macromolecules 21(5):1270–1277
Nishio Y, Haratani T, Takahashi T (1989) Cellulose/poly(vinyl alcohol) blends: an estimation of thermodynamic polymer–polymer interaction by melting point depression analysis. Macromolecules 22(5):2547–2549

Peresin MS, Habibi Y, Zoppe HJ, Pawlak JJ, Rojas OJ (2010) Nanofiber composites of polyvinyl alcohol and cellulose nanocrystals: manufacture and characterization. Biomacromol 11:674–681
Roohani D, Habibi Y, Belgacem NM, Ebrahim G, Karimi AN, Dufresne A (2008) Cellulose whiskers reinforced polyvinyl alcohol copolymers nanocomposites. Eur Polym J 44(8):2489–2498
TAPPI Method T204 om-88 (1988a) Solvent extractives of wood and pulp. TAPPI Press, Atlanta
TAPPI Method T222 om-88 (1988b) Acid-insoluble lignin in wood and pulp. TAPPI Press, Atlanta
TAPPI Method T211 om-93 (1993) Ash in wood, pulp, paper and paperboard: combustion at 525°C. TAPPI Press, Atlanta
Yue Y (2011) A comparative study of cellulose I and II fibers and nanocrystals. Master thesis, School of Renewable Natural Resources, Louisiana State University, Louisiana, pp 24–61
Zugenmaier P (2008) Crystalline cellulose and derivatives: characterization and structures. Springer, Berlin
Zuluaga R, Putaux JL, Cruz J, Velez J, Mondragon I, Ganan P (2009) Cellulose microfibrils from banana rachis: effect of alkaline treatments on structural and morphological features. Carbohydr Polym 76(1):51–59

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10	Phanthong, Patchiya, Guoqing Guan, Yufei Ma, Xiaogang Hao, and Abuliti Abudula. "Effect of ball milling on the production of nanocellulose using mild acid hydrolysis method", Journal of the Taiwan Institute of Chemical Engineers, 2016. Crossref	13 words — < 1%

11 etd.aau.edu.et
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12 Keigo Mikame. "Polymer Structure of Lignophenol I —Structure and Function of Fractionated Lignophenol—", Polymer Journal, 2006
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13 words — < 1%

13 Shiyu Geng, Kun Yao, Qi Zhou, Kristiina Oksman. "High-Strength, High-Toughness Aligned Polymer-Based Nanocomposite Reinforced with Ultralow Weight Fraction of Functionalized Nanocellulose", Biomacromolecules, 2018
Crossref
13 words — < 1%

14 Yatim Lailun Ni'mah, Ming-Yao Cheng, Ju Hsiang Cheng, John Rick, Bing-Joe Hwang. "Solid-state polymer nanocomposite electrolyte of TiO2/PEO/NaClO4 for sodium ion batteries", Journal of Power Sources, 2015
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11 words — < 1%

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17 Munawar Khan, Ahmad Nawaz Khan, Abdul Saboor, Iftikhar Hussain Gul. "Investigating mechanical, dielectric, and electromagnetic interference shielding properties of polymer blends and three component hybrid composites based on polyvinyl alcohol, polyaniline, and few layer graphene", Polymer Composites, 2018
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18 academic.sun.ac.za
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19 Pereira, André Luís S., Diego M. do Nascimento, Men de sá M. Souza Filho, João Paulo S. Morais, Niedja F. Vasconcelos, Judith P.A. Feitosa, Ana Iraidy S. Brígida, and Morsyleide de F. Rosa. "Improvement of polyvinyl alcohol properties by adding nanocrystalline cellulose isolated from banana pseudostems", Carbohydrate Polymers, 2014.
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20 Matthew P. Orr, Meisha L. Shofner. "Processing strategies for cellulose nanocrystal/polyethylene- co -vinyl alcohol composites", Polymer, 2017
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21 biotechnologyforbiofuels.biomedcentral.com
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22 Sugimoto, M.. "Synthesis of acyl chitin derivatives and miscibility

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24 Abdul Khalil, H.P.S.. "Green composites from sustainable cellulose nanofibrils: A review", Carbohydrate Polymers, 20120115
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25 Bavan, D, and G Kumar. "A View on Cellulosic Nanocomposites for Treatment of Wastewater", Nanocomposites in Wastewater Treatment, 2014.
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26 Anand Babu Perumal, Periyar Selvam Sellamuthu, Reshma B Nambiar, Emmanuel Rotimi Sadiku. "Development of polyvinyl alcohol/chitosan bio-nanocomposite films reinforced with cellulose nanocrystals isolated from rice straw", Applied Surface Science, 2018
Crossref
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27 "Handbook of Nanofibers", Springer Science and Business Media LLC, 2019
Crossref
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28 Yan Wu, Qinwen Tang, Feng Yang, Li Xu, Xuehua Wang, Jilei Zhang. "Mechanical and thermal properties of rice straw cellulose nanofibrils-enhanced polyvinyl alcohol films using freezing-and-thawing cycle method", Cellulose, 2019
Crossref
8 words — < 1%

29 Myriam Le Normand, Rosana Moriana, Monica Ek. "Isolation and characterization of cellulose nanocrystals from spruce bark in a biorefinery perspective", Carbohydrate Polymers, 2014
Crossref
8 words — < 1%

30 Bruna Grosch Schroeder, Patrícia Raquel Silva Zanoni, Washington Luiz Esteves Magalhães, Fabricio Augusto Hansel et al. "Evaluation of biotechnological processes to obtain ethanol from recycled paper sludge", Journal of Material Cycles and Waste Management, 2015
Crossref
8 words — < 1%

31 MIYAMOTO, Hitomi, Chihiro YAMANE, Masaharu SEGUCHI, and Kunihiko OKAJIMA. "Structure and Properties of Cellulose-Starch Blend Films Regenerated from Aqueous Sodium Hydroxide Solution", Food Science and Technology Research, 2009.
Crossref
8 words — < 1%

32 Wenshuai Chen, Haipeng Yu, Yixing Liu, Yunfei Hai, Mingxin Zhang, Peng Chen. "Isolation and characterization of cellulose nanofibers from four plant cellulose fibers using a chemical-ultrasonic process", Cellulose, 2011
Crossref
8 words — < 1%

---

33 Sujosh Nandi, Proshanta Guha. "A Review on Preparation and Properties of Cellulose Nanocrystal-Incorporated Natural Biopolymer", Journal of Packaging Technology and Research, 2018
Crossref
8 words — < 1%

---

34 Eve Saarikoski, Sami Lipponen, Marja Rissanen, Jukka Seppälä. "Blending cellulose with polyethylene-co-acrylic acid in alkaline water suspension", Cellulose, 2012
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---

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---

36 digilib.uin-suka.ac.id
Internet
8 words — < 1%

---

37 Karama Elfehri Borchani, Christian Carrot, Mohamed Jaziri. "Untreated and alkali treated fibers from Alfa stem: effect of alkali treatment on structural, morphological and thermal features", Cellulose, 2015
Crossref
7 words — < 1%

---

38 Shahab Kashani Rahimi, Robabeh Aeinehvand, Kyoungtae Kim, Joshua U Otaigbe. "Structure and Biocompatibility of Bioabsorbable Nanocomposites of Aliphatic-Aromatic Copolyester and Cellulose Nanocrystals", Biomacromolecules, 2017
Crossref
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---

39 Hanieh Kargarzadeh, Ishak Ahmad, Ibrahim Abdullah, Alain Dufresne, Siti Yasmine Zainudin, Rasha M. Sheltami. "Effects of hydrolysis conditions on the morphology, crystallinity, and thermal stability of cellulose nanocrystals extracted from kenaf bast fibers", Cellulose, 2012
Crossref
7 words — < 1%

---

40 Pratima Bajpai. "Chapter 6 Options for Utilization of Waste", Springer Science and Business Media LLC, 2015
Crossref
7 words — < 1%

---

41 Buaban, B.. "Bioethanol production from ball milled bagasse using an on-site produced fungal enzyme cocktail and xylose-fermenting Pichia stipitis", Journal of Bioscience and Bioengineering, 201007
Crossref
6 words — < 1%

---

42 H. Sehaqui, K. Kulasinski, N. Pfenninger, T. Zimmermann, P. Tingaut. "Highly Carboxylated Cellulose Nanofibers via Succinic Anhydride Esterification of Wheat Fibers and Facile Mechanical Disintegration", Biomacromolecules, 2016 — 6 words — < 1%
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