

Chemical Hydrolysis-Gamma Irradiation Processes for Cellulose Nanofibers Isolation from Rice Straw

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DOI: <https://doi.org/10.30880/ijie.2024.16.01.002>

Article Info

Received: 13 November 2023

Accepted: 11 January 2024

Available online: 25 April 2024

Keywords

Rice straw, cellulose nanofibers, acid hydrolysis, gamma irradiation

Abstract

Utilization of agriculture biomass waste into high value product is still potential to develop. In this study, cellulose nanofibers (CNF) as valuable materials were isolated from rice straw. Sodium hydroxide pulping and hydrogen peroxide bleaching were conducted as cellulose pretreatment. The CNF was prepared using 1 M hydrochloric acid hydrolysis followed gamma irradiation with various radiation doses, i.e. 40, 60, 80, 100 and 120 kGy. Compositions of α -cellulose and water retention of each stage processes increased inversely with hemicellulose, lignin and degrees of polymerization which had been strengthened by Fourier transform infrared spectroscopy (FTIR) spectra. X-ray Diffraction (XRD) analysis revealed that crystallinity index was more intense than rice straw, i.e. 80.85 %. Particle Size Analyzer (PSA) studies described the isolation process of CNF succeed with the smallest size 77.8 nm. The CNF functional groups of hydrophilic both hydroxyl and carboxylate slightly diminished with increasing of radiation doses followed water retention reducing, however the crystallinity had not changed significantly. Increasing radiation doses also could improve the thermal stability with the highest Tg 153.1 °C therefore the obtained CNF could be used as suitable nanocomposite.

1. Introduction

The excessive use of synthetic polymers is very dangerous for the environmental preservation because its properties are difficult to decompose naturally. Nowadays, much research has been conducted to explore polymer from renewable biomass. One of abundant biopolymers is cellulose and its chemical properties such as fibrous, physical, and chemical strength, biodegradable can be modified to replace synthetic polymers. However, the hydrophilic properties of cellulose and its poor compatibility with hydrophobic materials have limited application of unmodified cellulose. Many innovation studies have been carried out to overcome them including the development cellulose fibers into nanoscale. Cellulose nanofibers (CNF) which have fibres structure with a width scale around 5-100 nm and 1-5 μ m length were able to improve the mechanical and functional properties

of nanocomposites, such as biodegradability, transparency, gas barrier properties, specific surface area, and heat stability. Many studies reported widely application for CNF in industries such as waste adsorbent [1,2], food stabilizing [3], bio nanocomposites [4-6], medical materials [7] and packaging [8].

Biomass derived CNF from agricultural waste material is widely used in current research for enhance value added. Many researchers have studied on synthesis and characterization of CNF from different renewable biomass sources including cotton [1], softwood [9,10], olive trees [11], algae [3], banana peel [5,6,8], cassava biomass [12], and sugar pulp [4]. According to the most food crops especially in Indonesia, rice (*Oryza sativa sp.*) is commonly cultivated; therefore, rice straw is predominant generated as agricultural biomass waste and its produced averaged 20 million tons each year. However, rice straw utilization has not been optimized, for example, as feedstock for cattle, biochar fuel, and the other only burned in the field. In this research, rice straw will be used as valuable source for producing CNF as high-performance materials candidate.

Various methods for CNF production have been developed such as mechanical fibrillation processes [13,14], ultrasound fibrillation [15], electrospinning [16], acid hydrolysis [1,7,17], ozone oxidation [9], TEMPO oxidation [3,6,10,11], and enzymatic processes [18]. Currently, combining methods for CNF isolation have been carried out generally using acid hydrolysis-ultrasound fibrillation [5,12], acid hydrolysis-mechanical pressure [8], temperature-steam pressure [19], enzyme-mechanical processes [20] and TEMPO-mechanical fibrillation [21]. Based on the previous studies, the CNF products showed different properties which depend on the biomass source due to chemical composition, and degrees of polymerization also the synthesis method.

Commonly method for synthesis CNF that has been widely used is acid hydrolysis using HCl or H₂SO₄ solution. This method required a lot of chemical agents, a long time for synthesis and generated large liquid waste in manufacture scale. Generally, ionizing radiations initiate hydroxyl radical's productions which have a stronger oxidation that could enhance cellulose fibers scission. The degradation of cellulose structures using gamma irradiation were largely studied such as radiolytic degradation of cellulosic materials in nuclear waste [22], ruminal straw degradation using gamma irradiation [23], cellulose isolation from bacterial by gamma irradiation [24] and gamma irradiation treatment on chemical structure of soybean hulls [25].

Therefore, the purpose of this research is to modify acid hydrolysis with gamma irradiation as a new method for CNF isolation from rice straw, hence until now no one study has conducted. In our previous research, gamma irradiation was able to degrade cotton cellulose into microcrystalline cellulose [26]. Generally, ionizing radiations initiate hydroxyl radical's productions which have a stronger oxidation that could enhance cellulose fibers scission. In this present research, rice straw processed with alkali pulping and hydrogen peroxide bleaching to isolating the fibers. Synthesis of CNF was conducted by hydrolysis using HCl solution followed gamma irradiation at various radiation doses. The properties of obtained CNF were analysed including chemical functionality, crystalline structure, particle size, and thermal analysis.

2. Materials and Methods

Rice straw was procured from rice field in Yogyakarta, Indonesia. The raw material compositions were 33.7 ± 1.4 α -cellulose, 23.2 ± 1.1 hemicellulose, 16.9 ± 1.3 lignin which was analyzed using Chesson-Datta method and degrees of polymerization (DP) was 1506 ± 3.9 according to Mark-Houwink using cupriethylenediamine. Analytical grade sodium hydroxide, hydrogen peroxide, and hydrochloric acid used Merck products. The research was conducted in the radiation chemistry laboratory at Polytechnic Institute of Nuclear Technology.

2.1 Rice Straw Preparation

The rice straw was cut into small sizes, washed with water, and rinsed with distilled water. Then, the cleaned straws were dried using oven at 55 °C for 24 h. The fine micro straws with 100 mesh size have been found by conventional blender and sieve screener. The first stage of cellulose fibers isolation was conducted by sodium hydroxide pulping. During this process, fifty g dried rice straw powders placed into a 2 L reflux flash then 500 ml NaOH 5 % (v/v) added. Digestion pulping was carried out for 3 h at 80 °C and the obtained fibers were separated using Whatman filter paper No 40. The neutralized fibers were obtained by washing with demineralized water. Treated pulping products were bleached in order to remove remained lignin, and hemicellulose. Total 50 g dried rice straw pulp have been bleached using 750 ml H₂O₂ 5 % (v/v) and stirred with speed 100 rpm for 2.5 h at 75 °C. Bleached products were separated with Whatman filter paper No 40 and the white bleached fibers were rinsed using demineralized water.

2.2 Cellulose Nanofibers Isolation

In this research, CNF were isolated based on acid hydrolysis followed by gamma irradiation. Firstly, 40 g bleached cellulose were mixed with 400 ml of 1 M HCl at a continuous speed of 150 rpm for 1 h at 100 °C. The suspension fibers were rinsed with demineralized water. The neutral slurry was transferred into aluminum vial and irradiated using Gamma Cell with Cobalt-60 sources (Institute of Isotopes Co. Ltd., Hungary) at Polytechnic

Institute of Nuclear Technology for various doses i.e. 40, 60, 80, 100 and 120 kGy with dose rate 7.4 kGy/hr in room condition. The resulting CNF was separated by centrifugation at 1000 rpm.

2.3 Characterization

The composition yield of α -cellulose, lignin, and hemicellulose for rice straw, treated pulping fibers, bleached fibers, and the CNF were analyzed by Chesson-Datta method which had been detail described by Sarto et al [23]. The degrees of polymerization (DP) quantification for each stage process were conducted using an Ostwald viscometer procedure [28,29]. The IR spectrums of cellulose fibers were measured using Shimadzu prestige 21 FTIR. The characterization was carried out to follow the change in the functionality of cellulose as a result of the processes. The fiber samples were scanned in transmittance measurement between 300 and 4000 cm^{-1} with resolution 7.7 cm^{-1} . Crystallinity changes for each stage process of fibers were carried out using XRD with D2 Phaser Bruker. Each fibers sample was measured from 2θ between 5 and 100° with scanning resolution 0.02° . Crystallinity index (C.I.) was determined by Segal formula where the calculations were based on crystalline peak intensity (I_{002}) at $2\theta = 22.5^\circ$ amorphous peak (I_{am}) at $2\theta = 18.5^\circ$ which has accounted for baseline correction as shown in Eq. 1.

$$C.I. = \frac{I_{002} - I_{am}}{I_{002}} \times 100 \% \quad (1)$$

The crystalline sizes (D) of the fibers were referred to Scherrer's equation as can be seen in Eq. 2.

$$D = \frac{k \lambda}{\beta \cos \theta} \quad (2)$$

Where $k=0.9$ is the Scherrer factor, λ is the X-ray radiation wavelength, β is the full width at half maximum (FWHM) and θ is the Bragg angle for crystalline peak.

Swelling property of the obtained cellulose fibers was analyzed using water retention (WR) experiment according to other researches [11,21]. The isolated CNF sizes were evaluated by Particle Size Analyzer (PSA) Horiba SZ-100. Thermal stability for all obtained cellulose fibers was carried out with differential scanning calorimeter (DSC) Netzsch DSC 214 with software to calculate the glass transition temperature (Tg).

3. Results and Discussion

3.1 Chemical Composition

The main goal of sodium hydroxide pulping and bleaching process was to eliminate hemicellulose and lignin in order to isolate cellulose fibers. The cellulose composition yield for each step of isolation processes is summarized in Table 1. Visual image of fibers product for each stage treatment is presented in Fig. 1. The experimental data revealed that pulping treatment using sodium hydroxide could increase α -cellulose yield otherwise both hemicellulose and lignin yields reduced. Sodium hydroxide can partially dissolve hemicellulose and lignin because of the ester-linked chain scission during pulping processes. The pulping product has dark yellow color as delignification process (Fig. 1). According to Table 1, bleaching process using hydrogen peroxide significantly reduced lignin yield content hence hemicellulose slightly decreased therefore cellulose content enhanced. At the time of the bleaching process, hydrogen peroxide would oxidize lignin in order to breaks the linked of hemicellulose-lignin. Berglund et al., Leticia et al., and Fillat et al. stated that bleaching could completely eliminate lignin [11,12,31]. In this research, there is still a small amount of lignin after bleaching caused by the use of dilute hydrogen peroxide in this research. Along bleaching treatment, the fibers color turn from dark yellow to whitey because of a little lignin remained as presented in Fig. 1. Obtained CNF show that α -cellulose slightly increased while hemicellulose significantly decreased and the remaining lignin dissolved completely during acid hydrolysis followed by gamma irradiation. Hydrochloric acid could cleavage hemicellulose structure and residual lignin degraded into phenolic derived therefore these compounds would dissolved in water. In addition, gamma irradiation might form hydroxyl radical as strong oxidizer that enhanced hydrolysis processes. The final CNF product was shaped similar to sponge with white color (Fig. 1). Coincided result had been found from our previous result regarding the synergetic effect on waste cotton degradation [22]. The similar observations also had been reported for wheat straw hydrolysis using enzymatic-gamma irradiation [28] and ion beam irradiation assisted sorghum straw hydrolysis [33].

As long as pulping, bleaching and hydrolysis, the degrees of polymerization of cellulose decreased as shown in Table 1. The highest DP reduction was found in CNF as acid-gamma irradiation hydrolysis product and the final DP value was 278 ± 0.8 . Pulping and bleaching processes promoted hemicellulose and lignin degradation as mentioned above therefore it enhanced depolymerization of cellulose fibers structure. During acid-gamma

irradiation hydrolysis, cellulose depolymerization increased due to almost completely hemicellulose and lignin breakdown from the fibers structures then the DP of CNF was the lowest. Consistence DP result below 300 were found in CNF from olive tree biomass using TEMPO oxidation [11], softwood using acid-TEMPO hydrolysis [29] and aspen wood using mechanical-temperature processes [19].

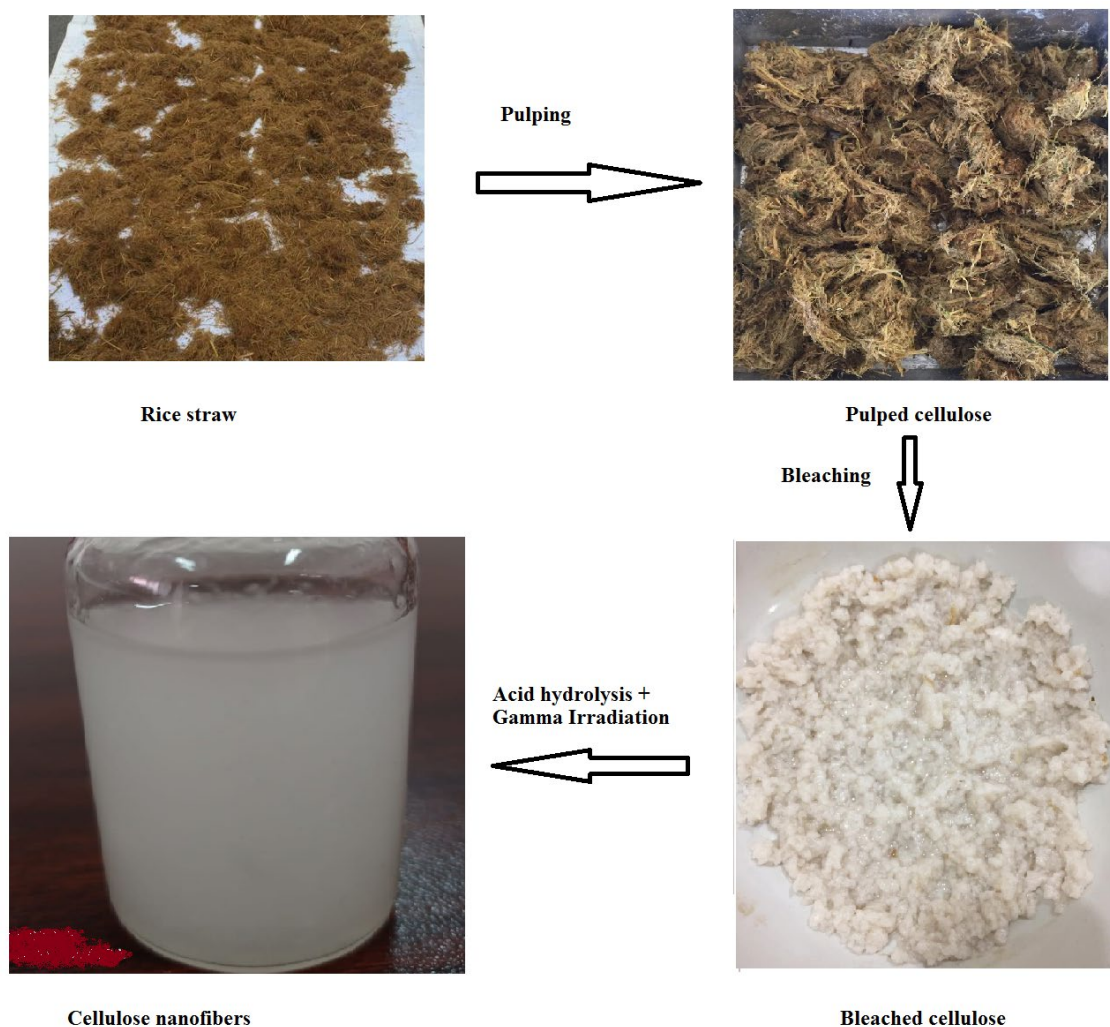


Fig. 1 Visual image of CNF isolation processes

Table 1 Cellulose composition yield and degrees of polymerization

Samples	α -cellulose (%)	Hemicellulose (%)	Lignin (%)	Degrees of polymerization
Rice straw	33.7 \pm 1.4	23.2 \pm 1.1	16.9 \pm 1.3	1506 \pm 3.9
Treated pulping fibers	73.3 \pm 2.7	11.9 \pm 1.7	12.1 \pm 2.2	772 \pm 1.4
Bleached fibers	87.1 \pm 0.9	9.1 \pm 1.1	2.1 \pm 0.8	582 \pm 2.2
Cellulose nanofibers	98.9 \pm 0.6	1.2 \pm 0.6	-	278 \pm 0.8

3.2 FTIR Analysis

FTIR analysis was carried out to determine functional groups especially the presence of lignin, hemicellulose and cellulose compounds in the fibers and the changes that occurred during each stage isolation process. The IR spectra of rice straw, pulped, bleached and CNF products are shown in Fig. 2. The spectra of all fibers samples revealed a broadened band around 3400 cm^{-1} indicating to hydroxyl groups stretching. Among the obtained fibers, the CNF has increasing peak intensity at 3410 cm^{-1} that is due to hydrogen bonds enhancing as amorphous lignin dissolution during hydrolysis. The appearance of a slight peak at 1867 cm^{-1} in pulped, bleached and the CNF indicates C = O from phenolic compound because of lignin degradation. A decrease in the peak spectra for carbonyl groups at 1651 cm^{-1} during pulping and bleaching treatment is caused by chain scission in hemicellulose structures. The CNF spectra also show peak at 1651 cm^{-1} with shifted to 1733 cm^{-1} .

According to Yang et al. [34] and Bakkari et al. [21], this result verify carboxylates formed as hydrolysis reaction products. Cellulose has specific peak spectra at $800 - 1400 \text{ cm}^{-1}$ as mentioned in other researches [2,6,12] which consist peak around 880 cm^{-1} for C-H vibration of β -glycosidic, 1090 cm^{-1} for C-O-C stretching and 1420 cm^{-1} for C-H deformation of crystalline cellulose. These specific bands had increased after pulping; bleaching and hydrolysis treatment therefore the cellulose isolation was successfully carried out and it were affirmed by yield composition data as shown in Table 1.

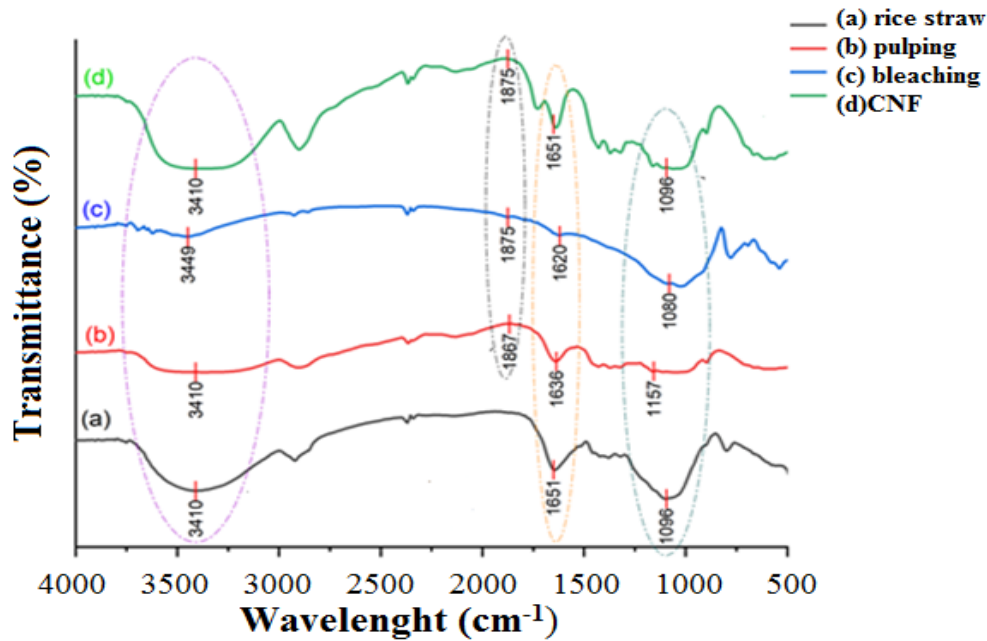


Fig. 2 FTIR spectra of rice straw, pulped, bleached and CNF products

The influence of gamma irradiation doses on FTIR spectra of the CNF is displayed in Fig. 3. The spectra of all CNF were observed similar pattern with slightly different in peak intensity that indicated small alteration along increasing irradiation doses. The hydroxyl group broad band at 3440 cm^{-1} was slightly reduced in case of intermolecular hydrogen bond breaking and hydrolysis reaction. The similar result was also observed in biomass cellulose irradiated with electron beam [35]. A slight decrease in peak intensity of carbonyl groups at 1650 cm^{-1} was also found. Increasing irradiation doses would enhance chain scission of acetyl function in hemicellulose structure. Kubovský et al. [36] obtained consistent results for laser irradiation on hardwoods. The other peak spectra did not change significantly. A good agreement results also could be obtained in our previous research regarding gamma irradiation for cotton cellulose degradation [26]

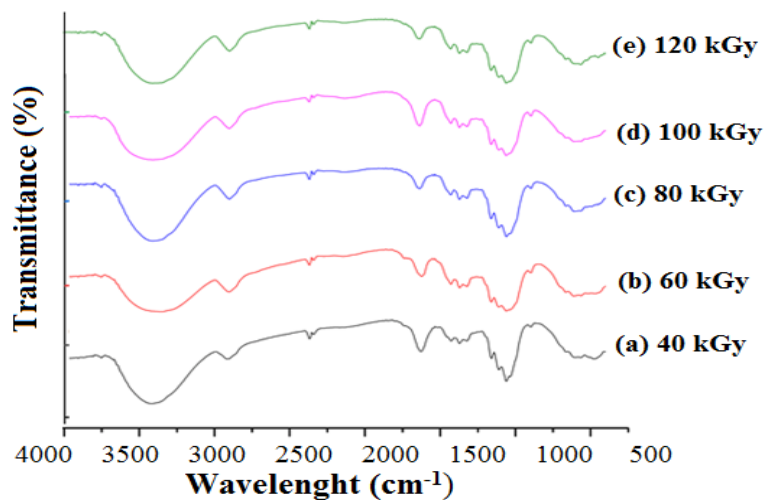


Fig. 3 FTIR spectra of the CNF at various radiation doses

3.3 XRD Analysis

XRD analysis was conducted to determine the structure of cellulose crystallinity in the fibers as well as the changes that happened during the treatment process. The diffraction pattern of cellulose before and after treatment is explained in Fig. 4 hence the C.I. and crystalline sizes is summarized in Table 2. The specific XRD patterns of cellulose I typical structures were obtained for all fibers which appear diffraction peaks at 2θ around 22° , 18° and 16° . The consistency of the diffraction pattern shows that along isolation step treatment does not change the structure. Characteristic crystalline peak of cellulose was found at 22° whereas amorphous peak appeared at 18° . The crystalline peak of cellulose fibers produced from pulping, bleaching and hydrolysis has a higher peak than rice straw as shown in Fig. 4. The CNF main peaks exhibit a highest peak for crystalline area at 22.4° and 16.1° which is much enhanced cellulose I structure. All these results imply that each stage of isolation could increase cellulose crystallinity as shown in Table 2.

In rice straw structure, cellulose crystalline fibers are surrounded by amorphous networks of hemicellulose and lignin. Utilization of NaOH in pulping and H_2O_2 in bleaching processes might break and dissolve partly of lignin and hemicellulose therefore the C.I. would increase. The larger enhancement of C.I. was occurred in the CNF due to further the removal processes. These results are consistent with cellulose composition yield and FTIR analysis that showed increased cellulose content in each treatment process. A good agreement results also could be found in CNF from carrot waste using mechanical treatment [31], banana peel with chemical-ultrasonication treatment [5] and oil pulp biomass using mechanical grinding [37]. The opposite result was mentioned by Fillat et al. [11] which found the C.I. reduction of CNF from eucalyptus pulp because of worse fibrillation.

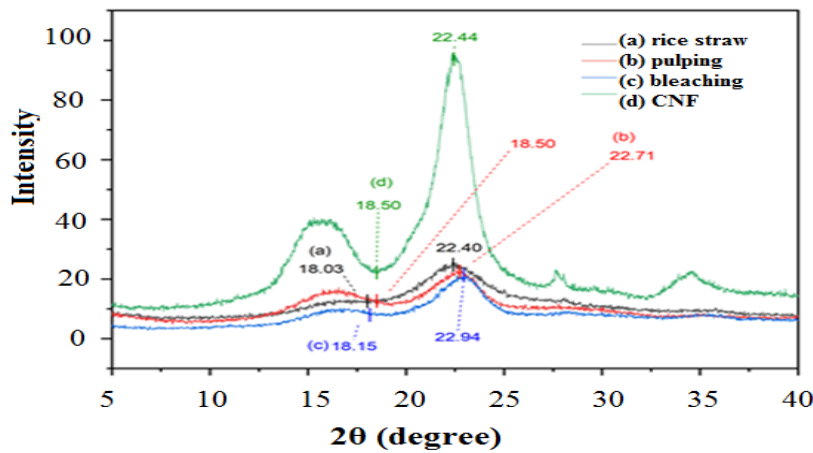


Fig. 4 XRD of rice straw, pulped, bleached and CNF products

Table 2 Crystallinity index and crystalline sizes of rice straw, pulped, bleached and CNF products

Samples	Crystallinity index (%)	Crystalline sizes (nm)
Rice straw	65.96	58.5
Treated pulping fibers	66.33	39.3
Bleached fibers	72.77	10.7
Cellulose nanofibers	80.85	5.1

From Table 2, the crystalline sizes had been reduced after each isolation step with the lowest size was 5.1 nm for CNF. As stated above, pulping and bleaching would increase chain scission both lignin and hemicellulose from the fiber structures. Hence, hydrolysis with HCl following gamma irradiation could pull out individual crystalline cellulose and attributed to smaller crystal size. A study on the influence of gamma irradiation dose on the CNF crystallinity was carried out and the results are presented in Fig. 5 and Table 3. The XRD spectra patterns weren't found differences for all various doses. This indicated that increasing radiation doses up to 120 kGy did not affect the CNF crystallinity as shown in Table 3. A coincided studies was also obtained in research of gamma irradiated cellulose paper [37]. However, the studies which conducted by Tarrsini & Teoh [35] regarding electron beam irradiation on biomass cellulose and Kapoor et al. [39] concerning gamma irradiation on bagasse cellulose observed crystalline reduction during irradiation exceeding 200 kGy. They reported that higher irradiation doses could degrade crystalline structure into amorphous structure.

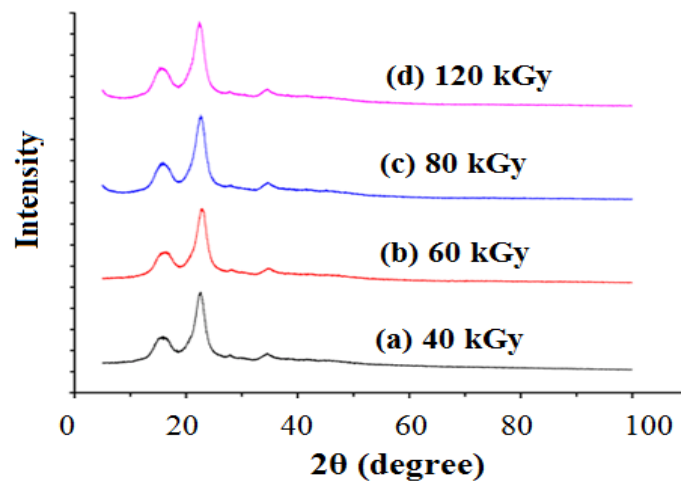


Fig. 5 XRD of the CNF at various radiation doses

Table 3 Crystallinity index and crystalline sizes of the CNF at various radiation doses

Radiation dose (kGy)	Crystallinity index (%)	Crystalline sizes (nm)
40	80.85	5.1
60	80.62	4.5
80	81.19	3.1
120	81.27	3.0

3.4 Water Retention

Water retention is primary property regarding cellulose capacity for absorbing and retaining water. The experimental result of water retention at each stage isolation process is presented in Table 4. Water retention increased as the result of pulping, bleaching and hydrolysis from 2.2 to 11.7 g/g. As mentioned earlier, each step cellulose isolation treatment from rice straw could enhance cellulose content and hydrophilic groups both hydroxyl and carboxylate functional structure. Moreover, the larger fiber surface area due to fibrillation during the process might increase WR value. These results are also consistent with other research, such as CNF from pulping wood using TEMPO oxidation [11] and CNF from softwood using TEMPO-mechanical fibrillation [21]. Water retention of CNF for various gamma irradiation doses is summarized in Table 5. A slightly water retention reduction was found as increasing irradiation doses. This result was caused decreasing hydroxyl and carboxylate groups due to higher radiation doses which were confirmed by FTIR spectra at Fig. 3.

Table 4 Water retention of rice straw, pulped, bleached and CNF products

Samples	Water retention (g water/g fiber)
Rice straw	2.2 ± 0.1
Treated pulping fibers	3.9 ± 0.1
Bleached fibers	7.4 ± 0.3
Cellulose nanofibers	11.7 ± 0.2

Table 5 Water retention of the CNF at various radiation doses

Samples	Water retention (g water/g fiber)
40	11.7 ± 0.2
60	10.4 ± 0.1
80	9.1 ± 0.2
100	8.5 ± 0.2
120	8.5 ± 0.1

3.5 Particle Size Analysis

In the interest of influence gamma irradiation doses on the sizing of CNF, PSA analyses were done and the result is described in Fig. 6. The CNF which was isolated with higher radiation doses from 40 to 80 kGy showed a smaller average diameter size from 361.1 to 77.8 nm. These trends show good agreement with other research such as radiolytic degradation of cellulosic materials in nuclear waste [22] and cellulose degradation from soybean hulls [25]. An increase in the total dose enhances the formation of radicals. As mentioned earlier, higher radicals formed would enhance degradation of cellulose fibers, therefore the fiber sizes should decrease. More homogeneous size distributions of the CNF were also obtained with higher radiation doses as seen in narrower peak widths. These indicated that isolations of the homogeneous CNF from rice straw using gamma irradiation were successfully obtained.

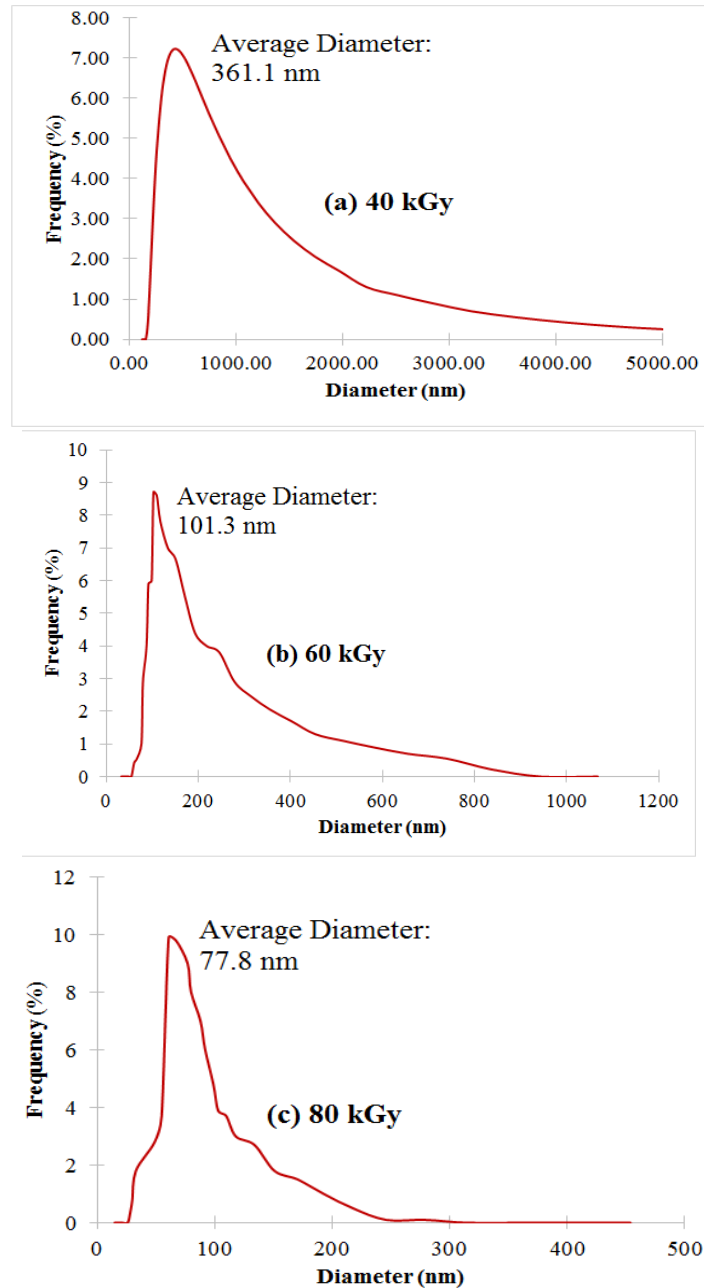


Fig. 6 PSA of the CNF at various radiation doses

3.6 DSC Analysis

In order to study thermal property especially the glass transition temperature (T_g), DSC analysis of the CNF was conducted for various gamma irradiation doses and the result is shown in Fig. 7. By increasing radiation dose from 40 to 80 kGy, T_g switched to higher temperatures from 129.5 to 153.1 °C. This result indicated that higher

radiation doses could increase the thermal stability of CNF. Decreasing fiber size as a result of higher irradiation doses would enhance surface area due to the densely cellulose structures therefore the higher Tg could be achieved. Davoudpour et al. [40] also obtained that the Tg of CNF would increase as a result of smaller fiber during increasing mechanical pressure. The CNF which was isolated from rice straw using gamma irradiation has a great opportunity as strengthening filler for nanocomposites.

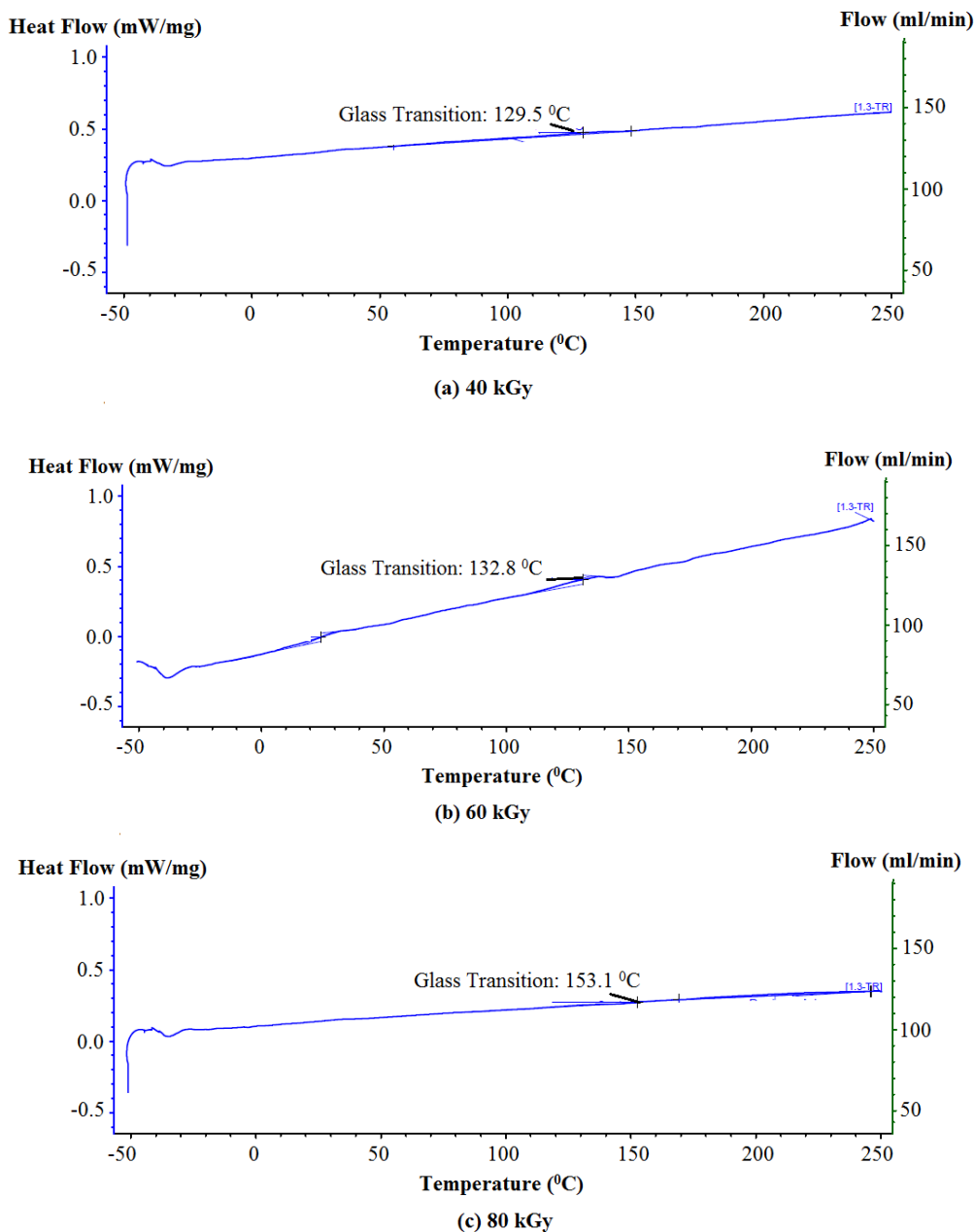


Fig. 7 DSC thermographs of the CNF at various radiation doses

4. Conclusions

Acid hydrolysis combined with gamma irradiation has been successfully used for cellulose nanofibers isolation from rice straw. Each stage of isolation treatment, i.e. pulping, bleaching and hydrolysis could enhance α -cellulose and water retention; however hemicellulose, lignin and degrees of polymerization had been obtained the opposite results and it was confirmed by FTIR results. In the present research, crystallinity index of the CNF reached higher than rice straw, i.e. 80.85 % as shown in XRD analysis. Based on PSA measurement, the existence of nanofibers could be proven with the smallest size 77.8 nm. Enhancement radiation doses would slightly reduce hydroxyl and carbonyl groups resulting water retention decreasing while the crystallinity of CNF was not affected. The average diameter size of CNF was smaller with higher radiation doses, whereas the glass transition

temperature increased with the highest Tg 153.1 °C. These cellulose nanofibers have a favorable application as a nanocomposite material in various industries.

Acknowledgement

The authors are grateful for the financial support of The Ministry of Research, Technology and Higher Education of the Republic of Indonesia through the INSINAS program with the contract number 06/INS-1/PPK/E4/2019. The author would like to thank Miss Mamluatul Faizah, Miss Winahyu Saputri and Miss Mutia Sari Sholikha for contribution help to this research activity.

Conflict of Interest

Authors declare that there is no conflict of interests regarding the publication of the paper.

Author Contribution

The authors confirm contribution to the paper as follows: **study conception and design:** Deni Swantomo, Kris Tri Basuki; **data collection:** Deni Swantomo, Haryanto, Saefurrochman; **analysis and interpretation of results:** Deni Swantomo, Kris Tri Basuki, Doonyapong Wongsawaeng; **draft manuscript preparation:** Deni Swantomo, Haryanto Doonyapong Wongsawaeng. All authors reviewed the results and approved the final version of the manuscript.

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