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Eny Kusrini
Department of Chemical Engineering, Faculty of Engineering, Universitas Indonesia

Pael Desen Thesa Lonika
Department of Chemical Engineering, Faculty of Engineering, Universitas Indonesia, Kampus

Denisa Restu Erminda
Department of Chemical Engineering, Faculty of Engineering, Universitas Indonesia, Kampus

Anwar Usman
Department of Chemistry, Faculty of Science, Universiti Brunei Darussalam,

他

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Synthesis of Biodegradable Tissue Paper Comprised of Cellulose Isolated from Rice Straw Waste and Chitosan: Characterizations and The Effect of Chitosan Concentrations

Eny Kusrini[1,2,3,*], Pael Desen Thesa Lonika[1], Denisa Restu Erminda[1], Anwar Usman[4], Lee D. Wilson[5], Ponky Ivo[6], Suyanti[7]

[1]Department of Chemical Engineering, Faculty of Engineering, Universitas Indonesia, Kampus Baru UI, Depok 16424, Indonesia
[2]Research Group of Green Product and Fine Chemical Engineering, Laboratory of Chemical Product Engineering, Department of Chemical Engineering, Universitas Indonesia, Kampus Baru UI, Depok, 16424, Indonesia
[3]Tropical Renewable Energy Research Center, Faculty of Engineering, Universitas Indonesia, Kampus Baru UI, Depok, 16424, Indonesia
[4]Department of Chemistry, Faculty of Science, Universiti Brunei Darussalam, Gadong BE1410, Brunei Darussalam
[5]Department of Chemistry, University of Saskatchewan 110 Science Place, Room 156 Thorvaldson Building, Saskatoon, SK S7N 5C9, Canada
[6]Research Center for Advanced Material, Badan Riset dan Inovasi Nasional Serpong, KST B.J. Habibie Puspiptek, Setu, Tangerang Selatan, Banten, 15314, Indonesia
[7]Research Center for Mining Technology, National Research and Innovation Agency, Jl. Ir. Sutami KM. 15, Tanjung Bintang, Lampung Selatan, Lampung 35361, Indonesia

*Author to whom correspondence should be addressed:
E-mail: eny.k@ui.ac.id

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Abstract: In this study, environmentally friendly biodegradable tissue paper (BTP) based on commercial chitosan and cellulose from rice straw waste (RSW) has been prepared. The chitosan composition was varied of 2-5% (w/v), resulting in BTP with properties which fullfil the requirements for the standardized tissue papers SNI 0103:2008. The appearance of BTP is clean, soft, and not perforated, but its color is slightly yellow and does not fade. The BTP products have high water absorption properties in the range of 68–72 mm, respectively, which is much higher than the standard value (30 mm) of tissue paper.

Keywords: biodegradable tissue paper; cellulose; Chitosan; rice straw waste; SNI 0103:2008 method

1. Introduction

Increasing consumer awareness of environmentally friendly products has encouraged industries to develop eco-friendly consumable products. One of them is tissue paper which is a practical item, essential daily commodities and most popular paper grades[1)] that is often carried everywhere. Usually, tissue papers are used for toilet paper, napkins, facial tissue, and paper towels[1)]. There are various conveniences of using tissue paper in everyday life, ranging from personal, household, cooking and toilet needs. To meet the demand of environmentally friendly products, it is necessary to develop a biodegradable tissue paper (BTP) which can be decomposed by living organisms or can be destroyed in water.

A healthy and clean lifestyle continues to develop in Indonesia in line with better economic conditions. This encourages higher consumption of tissue papers, as reflected by the increasing demand every year. Based on the statistical data, for instance, the volume of production of tissue papers in Indonesia has consistently increased by 4.76% from 2011 to 2015[2)]. As compared with printing paper industry, tissue papers industry is growing faster around with the population and sanitary awareness, especially during COVID-19 crisis in 2020[3)].

Key properties of the tissue papers are the high absorbency, low density, and its low basis weight. It must be hygienic, and has high water absorption, high tensile strength, and good softness[1]. However, the use of tissue papers can cause irritation, especially for sensitive skins if they are used for long term, unclean, and unhealthy, as tissue papers have preservatives or perfumes that can cause allergic skin reactions. In addition, the use of toilet papers can cause vulvitis, which is an inflammatory condition in the female genitalia which is characterized by itching, burning, redness, and swelling. Therefore, it is necessary to add chitin or chitosan as an antibacterial agent to tissue paper[4-6]. In ambient condition, chitosan has a positively charged amine functional group that can bind electrostatically with negatively charged functional groups. In a previous report, various advantages of chitosan related to its abundant hydroxyl and amine groups was demonstrated[7-11]. Unique antibacterial properties imbued by these functional groups have also been reported[5-6]. Chitosan can be used as the scaffold material in hydrogels[12]. This natural polymer has properties such as biodegradability, biocompatibility, and low toxicity[12-13]. Chitosan with diverse various grades can be used for many applications in food, industrial, and pharmaceutical sectors, depending on the purity of the product.

Generally, the main raw material for commercial tissue paper production is pulp combined with synthetic fibers. The fibrous cellulose is the major component for all hygiene tissue papers[14]. It is usually originated from tree. The use of pulp derived from wood causes the shrinkage of forests. Globally, the World Wide Fund for Nature estimates that every day, about 270,000 trees that are cut down and end up in landfills. In addition, the synthetic fibers in tissue paper are difficult to be broken down by the soil over relatively short time intervals, thus accumulating and causing environmental problems. Furthermore, the use of tissue as toilet papers which contain fibers is difficult to disintegrate in water, causing blockages in the drain.

On the other hand, rice straw waste (RSW) is one of agricultural waste[15] and can be used as the alternative raw materials to replace wood cellulose in tissue paper production. RSW represents one of the main cereal straws that is produced in large quantities each year globally[16], and is one of the most widely available agro-industrial wastes. For instance, in 2015, Indonesia produced 99,617 kilotons of RSW[17]. Usually, RSW is used for animal feed and the rest is left to rot or burn that generate pollutant greenhouse gases such as CO_x, NO_X, SO_X, which contribute to pollution and negative climate change effects. RSW has also been reported to produce porous carbon material by carbonization process at temperature ranged from 400 – 600°C and used as adsorbent for removal of organic dyes, such as methylene blue and methyl orange, as well as metal ions such as Pb^{2+}, Cu^{2+}, Ni^{2+}, and Cd^{2+} [15]. In general, adsorption capability of the porous carbon materials that derived from RSW is lower than activated carbon (AC), but those obtained by carbonization at 400°C is potential for purification and treatment of wastewater[15]. On the other hand, AC which can be used not only as an adsorbent in wastewater treatment, but also as an electrode in electric-double layer capacitance-type supercapacitors[18].

In this study, utilization of RSW as raw material for cellulose production can reduce the use of wood as a basic material for preparation of tissue papers, and also address the challenges of tissue paper waste. The utilization of RSW requires a strategy to valorize the agricultural waste which contains 30% cellulose, 25% hemicellulose and 12% lignin[19]. The relatively high cellulose content in RSW allows its utility as a raw material of cellulose for tissue paper production, while RSW cellulose can be decomposed via natural processes.

Cellulose is a colorless natural biopolymer, belonging to polysaccharides, that consists of linearly bound β-1,4-linked D-glucose. This biopolymer is widely used in the manufacture of paper, foods, textiles, adhesives, and pharmaceuticals[20], and can be obtained from rice straw and many other agricultural waste. On the other hand, chitosan is a natural amino-polysaccharide polymer that derived from chitin[21], which can be found in shrimp shell and other seafood waste[4,6,22,23]. Many researchers reported that chitosan has excellent mechanical properties, in addition to its high adsorption capability[5,24,25]. On the other hand, Limam et al.[26] reported that it can be found as commercial food waste products from the marine bioprocessing industry. As biopolymers, cellulose and chitosan are non-toxic, biodegradable, biocompatible[12-13].

In this study, biodegradable tissue paper (BTP) products were prepared using commercial chitosan and natural cellulose from RSW. The effects of chitosan mass ranged from 2 to 5 % in the formation of BTP were also evaluated in details. The physical properties, including (i) crushability, (ii) absorption, (iii) appearance and (iv) color of the resulting BTP were analyzed according to the technical standard for toilet tissue papers. A significant outcome of this study is expected to provide an alternative solution for RSW for BTP production in Indonesia, including the technical feasibility for the development of new industrial for BTP production.

2. Experimental

2.1. Materials

All materials used in this study are commercially available. Chitosan powder with the degree of deacetylation 90.77% was purchased from PT. Biotech Surindo (Cirebon, Indonesia), while RSW was obtained from the I-AG Shop through Tokopedia (Indonesia). The chemicals, including hydrochloric acid (HCl 37%), sulfuric acid (H_2SO_4 98%), n-hexane, acetic acid (CH_3COOH 96%), and methanol were purchased from Merck KGaA (Germany), while sodium hydroxide (NaOH) was obtained from Asahimas Chemical. All the chemicals are analytical grade.

2.2. Methods

2.2.1. Preparation of Cellulose from Rice Straw Waste

Isolation of natural cellulose from RSW was carried out by chemical methods using the acid and alkaline processes. RSW was washed in a 30 L bucket three times to remove dirt and dust. The RSW was dried for 8 h under the sun to reduce the water content, followed by chopping to the sizes of 1 cm. The RSW was then weighed and further dried at 80 °C in an oven to ensure its minimum water content to be less than 10%. The dried RSW was then ground using a blender, followed by sieving to achieve a particle diameter less than 1 mm to enhance the surface-to-volume ratio of RSW particles to facilitate the dissolution process of extractive substances during the cellulose isolation.

2.3. Isolation of Cellulose

The cellulose was isolated from RSW by placing 50 g of dried RSW powder in a filter paper which was then fastened using a thread. In the first process, the extraction cellulose was carried out using a Soxhlet set-up with a solvent mixture of n-hexane:methanol (2:1 v/v) for 6 h. The solvent mixture also extracts other substances, such as lignin and hemicellulose. The temperature used in the extraction process of cellulose was 80 °C.

After the extraction, the spent RSW powder was collected and dried for further isolation process using alkaline media. In this case, 1500 mL of 7% NaOH added into the solution of the spent RSW powder, followed by heating at 80°C and stirring for 5 h on a hotplate stirrer. The cellulose yield obtained was 33.63% with residual lignin levels of 2.42%. The mixture was cooled at room temperature and acidified using 10% H_2SO_4 until pH of the mixture was in the range of pH 3–4. The mixture was then separated by filtration using a Buchner funnel and a vacuum pump to allow removal of the air bubbles and dirt trapped in the solution. The filtrate was collected. The bleaching process of cellulose was carried out using 2% H_2O_2 at 50°C for 5 h. The cellulose was then isolated from the filtrate using a Buchner funnel under vacuum and rinsed using water. After the bleaching process, the cellulose was dried at room temperature for one week. The cellulose was collected and stored for further use. Overall isolation process of cellulose using alkaline solution is illustrated in Figure 1.

2.4. Moulding of Tissue Paper

The moulding process of BTP was carried out by varying the composition of commercial chitosan to be 2–5 w/v% with respect to the RSW cellulose, as shown in Figure 2. Chitosan was dissolved using 1% acetic acid for 2 h at 50 °C. The solution was then filtered, and it was then mixed into the cellulose suspension. Then, 5 g of talcum was added into chitosan-cellulose suspension. The resulting tissue is softened with the addition of talcum.

For the preparation of 1 sheet of BTP with a size of 10 cm × 10 cm, 50 mL of water was added into the mixture of commercial chitosan, cellulose, and talcum. The mixture was thoroughly stirred, and was then put into a printing tool in the form of a filter with a 50 mesh size. The printing tool was slowly lifted. Finally, BTP was dried for 2 days at room temperature.

2.5. Characterization

The chemical structures of cellulose from rice straw, chitosan, and BTP were analysed based on their Fourier transform infrared (FTIR) spectra, which were recorded using the ATR mode on a FTIR spectrophotometer in the range of 400–4000 cm^{-1} and a resolution of 3.2 cm^{-1}. Each spectrum was recorded over 16 scans.

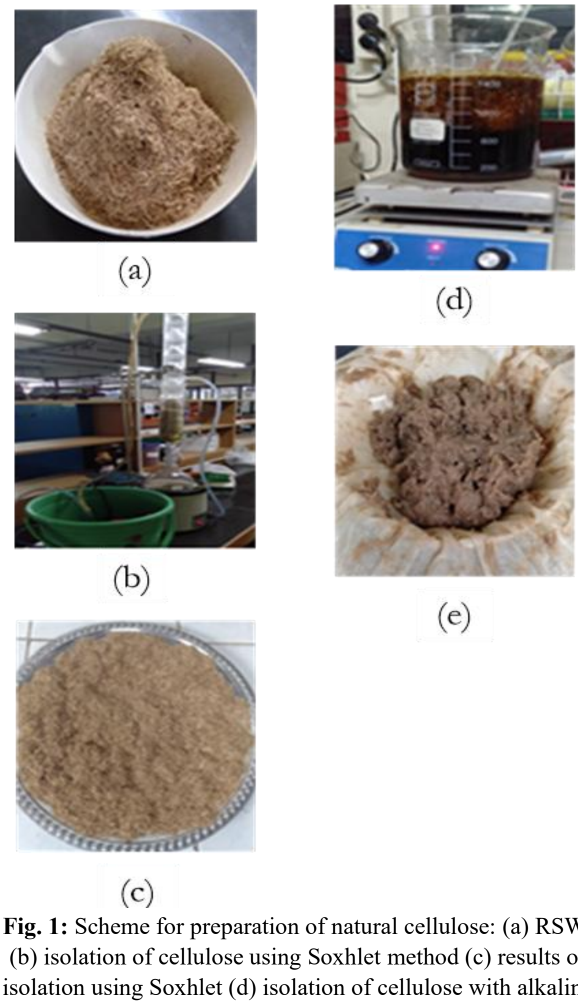


Fig. 1: Scheme for preparation of natural cellulose: (a) RSW, (b) isolation of cellulose using Soxhlet method (c) results of isolation using Soxhlet (d) isolation of cellulose with alkaline solution, (e) cellulose product

2.6. Toilet Tissue Paper Quality Standard

The resulting BTP was analysed and compared with toilet tissue paper standard, in terms of grammature, crushability, water absorbency, appearance, and color.

2.6.1. Grammature Test

The tissue paper grammar test is a tissue weight test, where the toilet tissue paper should have a standard minimum weight of 14 g/m^2.

2.6.2. Crushability Test

In this test, the tissue paper was soaked into water under stirring for 60 seconds, if the tissue breaks down, it means that the tissue has properties that are favorable for degradation.

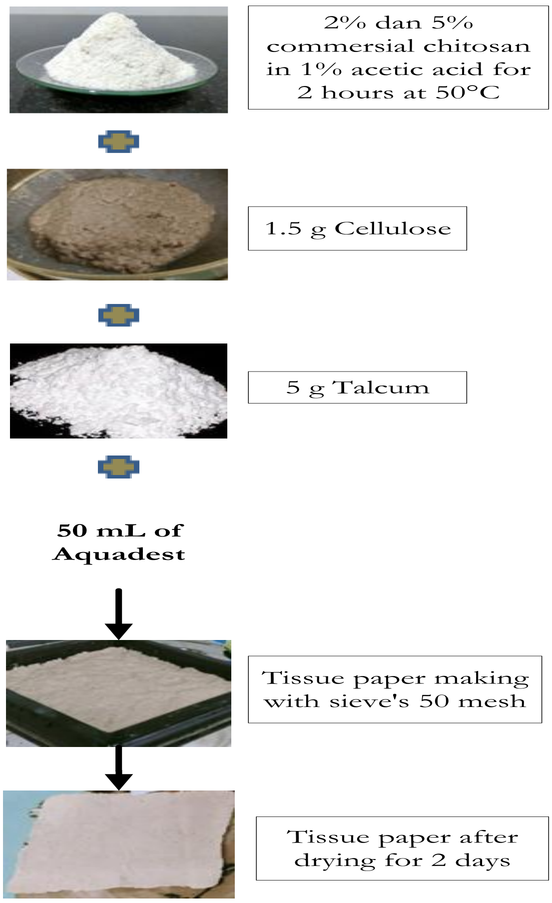


Fig. 2: Illustration of molding process for production of BTP

2.6.3. Water Absorbency Test

The water absorbency test was carried out on a 20 cm × 1.5 cm tissue paper. The tissue paper was hanged perpendicular to the surface of water with one end immersed as deep as 1 cm into the water for 10 minutes. The height of the water absorbed by the tissue paper was recorded.

2.6.4. Appearance Test

The tissue paper appearance test in this study refers to SNI 0103:2008. Tissue that complies the standard is a tissue that has a clean, soft appearance, and does not have holes.

2.6.5. Color Test

A good tissue paper has a white color and does not wear off. For this purpose, the tissue paper was immersed in water for approximately 60 seconds. If the water is colorless, it means the tissue paper will not wear off.

3. Results and Discussion

3.1. Isolation of Cellulose from Rice Straw Waste

RSW consists of cellulose as main fibers, hemicellulose as inter-connected branch, and lignin as a binder[27)]. To isolate cellulose, hemicellulose, lignin, and removal of other components from RSW, various physical or chemical methods can be employed to isolate cellulose[28)]. Physical methods include laser treatment, ionized gas (plasma or corona) treatment, or thermal steam explosion[29,30)].

The isolation of cellulose from lignin and other compounds was performed in alkaline condition. The presence of lignin in the sample is indicated by the presence of a blackish brown solution upon dissolution in alkaline media. Lignin removal is necessary because lignin causes stiffness in the resulting tissue paper. Lignin is difficult to decompose, thus, it can influence the biodegradability of the tissue paper product.

The addition of sulfuric acid facilitates isolation of cellulose through dissolution of the hemicellulose. It is comparable with procedure that reported by Kusrini et al.[31)], that the isolation of lignin from oil palm shell using polyaluminum chloride (PAC) and sulfuric acid, where 800 mL of black liquor can produce lignin up to around 15 g.

Removal of hemicellulose from RSW samples is important as hemicellulose forms amorphous branching structures[32)], which causes low mechanical strength of the fibers. This is not expected for tissue papers. Nevertheless, the precipitate obtained from treatment using 7% NaOH is still brown, suggesting the presence of lignin or other phenolic compounds. Therefore, a bleaching process is necessary to obtain a cellulose with a whiter color appearance. The bleaching process was carried out to remove the lignin and other phenolic compounds. The bleached cellulose from RSW is ready to use as precursor for preparation of BTP.

3.2. FTIR studies

FTIR spectra of cellulose, chitosan, and BTP with 2% and 5% (w/v) of chitosan are depicted in Figure 3. The presence of talcum and chitosan has altered the vibrational peaks of cellulose. For instance, the vibrational band of the hydroxyl (OH) group of cellulose shifted from 3335 cm^{-1} to 3095 and 3669 cm^{-1} in BTP 2% (w/v) of chitosan, and to 3275 cm^{-1} in BTP 5% (w/v) of chitosan. These spectral shifts suggest that the OH groups of cellulose are bound to magnesium ions of talcum and the chitosan amine (NH_2) groups. The vibrational band of chitosan NH_2 groups at 1588 cm^{-1} is covered by the intense peak of the vibrational bands of hydroxyl group of cellulose in BTP.

In addition, the vibrational band of OH group of chitosan also shifted from 3288 and 3355 cm^{-1} to 3095 and 3669 cm^{-1} in BTP 2% (w/v) of chitosan, and to 3275 cm^{-1} in BTP 5% (w/v) of chitosan. This indicated that the OH group of chitosan also interacts with magnesium ions of talcum and the OH groups of cellulose.

The FTIR spectrum of cellulose from RSW showed a vibrational band at 3335 cm^{-1} which indicated the OH bond stretching (Figure 4a). The CH stretching and bending vibrations appeared at 2913 cm^{-1}, 1429 cm^{-1}, and 1322 cm^{-1}. The OH and CH bonds are originated from lignin, cellulose, and hemicellulose.

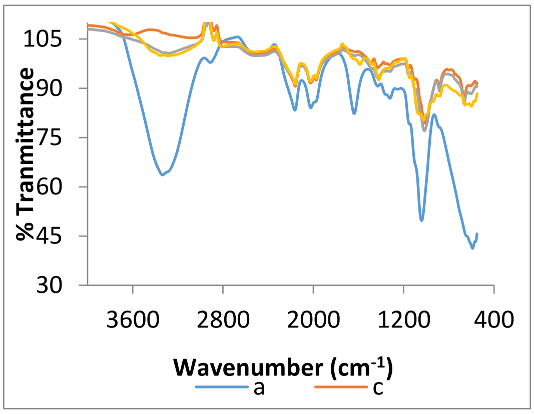
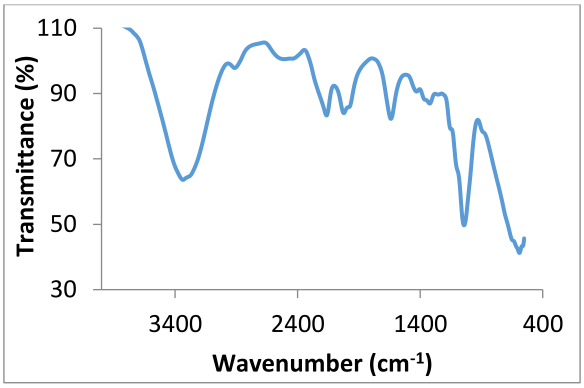


Fig. 3: FTIR spectra of cellulose from RSW (a), BTP with 2 % (w/v) of chitosan (b), BTP with 5% (w/v) of chitosan (c), and commercial chitosan (d)

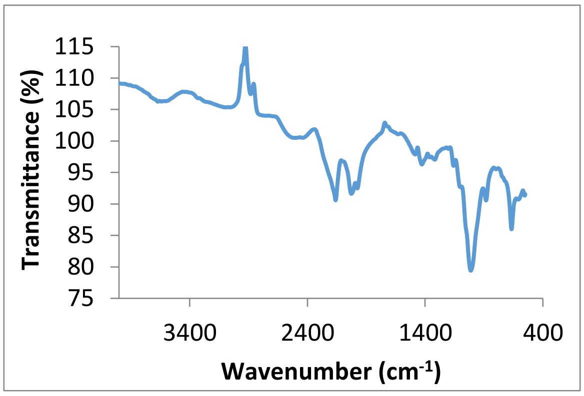
The C–O functional group of cellulose is observed at 1041 cm^{-1}. In addition, the vibrational band at 1638 cm^{-1} indicates the presence of carbon double bonds (C=C) due to lignin components. The presence of lignin in the cellulose product was also noted by the yellow colored appearance of the cellulose product obtained from RSW.

As shown in Figure 4b, chitosan showed two vibrational bands at 3288 and 3355 cm^{-1}, suggesting the NH and OH groups. The vibrational band at 2881 cm^{-1} is due to CH stretching and those at 1420 cm^{-1} and 1479 cm^{-1} are due to CH bending vibrations. The stretching vibration at 1588 cm^{-1} indicates the NH groups of chitosan. The spectral bands at 1026 cm^{-1} and 1147 cm^{-1} indicate the C–O bond stretching, while the glycosidic bonds of the biopolymer was observed by the stretching vibration at 892 cm^{-1}. As shown in Figure 4c and 4d, the absence of vibrational bands associated to new functional groups in BTP 2 w/v% and 5w/v%, after cellulose, talcum, and chitosan were mixed, indicates that the chemical structures of the biopolymers in BTP are not disturbed.

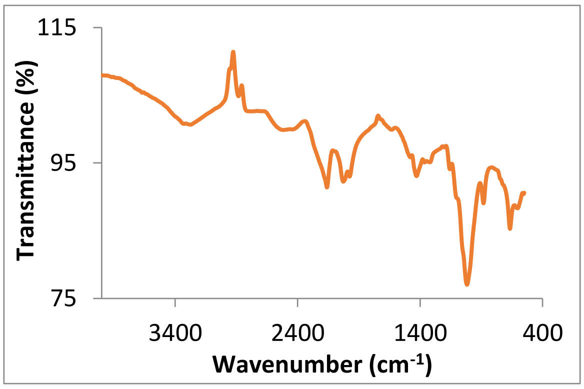
The FTIR spectrum of commercial chitosan from this study is comparable with that reported by Kusrini et al.[33]. The peaks at 3404, 2903, 1603, 1361, and 1032 cm^{-1} were assigned to the vibrational bands of OH, C-H, N-H, and C-N groups, respectively[33].



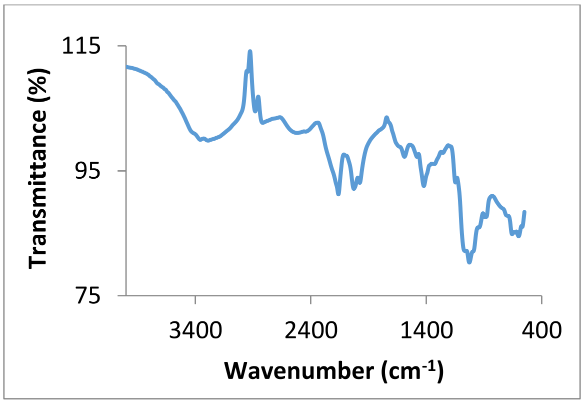
(a)



(b)



(c)



(d)

Fig. 4: FTIR spectra of (a) cellulose isolated from RSW, (b) BTP with 2 % (w/v) of chitosan, (c) BTP with 5% (w/v) of chitosan, and (d) chitosan

On the other hand, the modifications of pH level in chitosan solution can produce the protonated amine groups, which further affect both electronic charge and the solvability of chitosan solution in the formation of BTP. Thus, various weights of chitosan should result in different properties of the resulting BTP. Chitosan presented good antimicrobial activities[34] including antibacterial and antifungal[33], and an excellent mechanical strength. Thus, chitosan and its derivatives can be used for bio-drug and/or medicines[34].

3.3. Moulding process for preparing of BTP

The tissue paper was produced by varying the composition of chitosan 2% (w/v) and 5% (w/v) against cellulose of rice straw. The BTP products were evaluated according to the standardized toilet tissue paper protocols, namely SNI 0103:2008. The results showed that BTP for both variations of chitosan could be degraded in water. However, the degradation tends to decrease with the chitosan content in BTP. The ability to absorb water increases with greater chitosan content in the tissue paper. The resulting BTP is still thick and slightly yellowish in color. Based on the test results, the tissue paper produced nearly meets the requirements as tissue toilet paper. As summarized in Table 1, the BTP fullfil several technical requirements; e.g. crushability, absorption, and appearance. The quality of the BTP is similar to that reported by Ghasimi et al.[14].

In comparison, toilet papers available in the market are different in quality depending on their compositions, materials, characteristics, and source of cellulose[35,36]. Generally, they are smooth and can be embossed, unprinted or patterned, tinted, purely white or off-white depending on the materials used. The type of pulp and paper chemicals used have significant effects on the final quality of the tissue papers, namely softness, strength, absorbency and appearance[14].

The ability of chitosan to induce enzymatic degradation to be one favor of choice in the formulation of BTP. Chitosan can be disintegrated within the range of hydrolytic enzymes that are commonly found in the natural environment through biodegradation process. It is a crucial property for toilet papers to ascertain them after their intended use for household and daily usage. BTP can naturally break down and return to the environment, without contributing to the accumulation of municipal solid waste.

For the case of greater chitosan content of the tissue paper had the lower capacity to be degraded in water. The degradability of BTP with 2% (w/v) of chitosan was better than that of 5% (w/v) of chitosan. This is related to the relative insolubility of chitosan in water above the pK_a of chitosan[24,37]. It is noted that increasing the weight of chitosan from 2 to 5 % (w/v) in the tissue paper will reduce degradation of BTP.

Cellulose from RSW can absorb water because of the hydroxyl groups on cellulose which interact with water. The presence of chitosan in tissue paper shows an increase in the absorption capacity as the amount of chitosan content increases in the tissue paper. This is due to the presence of more pores in the tissue paper with higher composition of chitosan. The BTP containing 5% (w/v) of chitosan had improved absorption capacity over that found in BTP containing 2% (w/v) of chitosan. In this study, water absorbency of the resulting BTP was as high as 68 mm and 72 mm for BTP with chitosan 2% and 5% (w/v), respectively. These results are in accordance with study evaluating the antibacterial and mechanical properties of cellulose-chitosan composites for packaging applications reported by Tanpichai et al.[38]. On the other hand, Deng et al.[39] found that, upon mixing with chitosan, swelling and the water diffusion through the cellulose film are reduced, resulting in lower water vapor permeability. In addition, Reis and coworkers[40] reported that, upon coating with chitosan, water absorption and moisture permeability of Kraft paper were decreased by 35% and 44%, respectively, due to that chitosan acts as a filler to fill the pores of the Kraft paper, reducing its hydrophilicity.

Talcum as an additive was added to produce a softer tissue paper. The presence of magnesium and multiple OH groups in talcum can interact through Mg...O and O...N interactions with the hydroxyl groups of cellulose and the amine groups of chitosan.

Table 1: Quality and parameters of BTP according to SNI standard of toilet tissue paper 0103:2008

Parameter	Requirements	Results	
		Chitosan 2% (w/v)	Chitosan 5% (w/v)
Crushability	max. 60	Destroyed well	Destroyed pretty well
Absorption	min. 30	68	72
Appearance	Clean and soft	Clean and soft	Clean and soft
Color	White & does not fade	Slightly yellow and not fade	Slightly yellow and not fade

Some parameters according the SNI standards, namely (i) crushability, (ii) absorption, (iii) appearance and (iv) color of the BTP properties were analyzed. Overall, the crushability of BTP product showed it can be destroyed in water for 60 s. The standardized tests, suggested that the BTP products are nearly satisfied those of toilet tissue papers. However, the BTP is still thick and slightly yellowish in color (Figure 5). The yellowish color of BTP product is due to the cellulose from RSW which is brownish yellow in color, suggesting that the bleaching process in this study was not completely successful to produce white cellulose. On the other hand, the dried cellulose undergoes agglomeration because the natural hydrophilic nature of cellulose tends to undergo agglomeration during drying[41]. It is very difficult to break down the cellulose into a suspension solution. As the result, BTP after printed using a 50 mesh filter is also still thicker as compared with commercial tissue paper.

It is important to note that cellulose has a strong affinity for itself and hydroxyl (-OH) containing materials, for example water[42]. Cellulose shows the amphiphilic properties, thus it can extent the extensive intra- and inter-molecular hydrogen bonds as well as simultaneous hydrophobic molecular interactions. Thus, it gives significant impacts for its solubility in water and most organic solvents[43].

The biodegradability of cellulose is depending on its fibrous content and its crystallinity[14], as it consists of powdered cellulose and microcrystalline cellulose[14]. The maximum biodegradability can be achieved when no fibers are present in the toilet papers. For instance, Guan and coworkers[37] reported that the use of cellulose nanofiber can improve mechanical property and water absorption capability of the resulting tissue paper. This finding can be rationalized by considering that the cellulose nanofiber has larger specific surface area, more OH groups exposed to the water, higher hydrophilicity as compared with the granulated cellulose.

(a)

(b)

Fig. 5: Comparison color and appearance of biodegradable tissue paper with variable content of commercial chitosan (a) 2% (w/v) and (b) 5% (w/v)

It is noted that the abundance of small-sized pores allows the capillary effect and facilitates more hydrogen bonds linked to the cellulose nanofiber, thus enhancing water absorption and improving the strength properties of tissue paper reported in an earlier study[44]. Nanocellulose is light weight, biodegradable, renewable, and readily available, and has superior mechanical properties[45,46].

The resulting tissue paper (Figure 5) still needs further improvements for applications. Furthermore, it is necessary to evaluate a different solvent, such as H_2O_2 for the bleaching process of cellulose from RSW to produce whiter cellulose and the resulting BTP.

Chemical functionality related to surface chemistry, properties of fiber, and sustainable treatment process for natural fibers such as rice straw[47,48], bamboo[36,49] and flax fibers[50] as cellulose resources are of interest to explore. Based on this study, cellulose can be produced from biomass such as rice straw, pineapple fiber and others. This research also support the mitigation of climate change with development of an environmental friendly form of tissue paper that utilizes biomass waste from RSW. On the other hand, cellulose also can be used as matrix for the formation of new adsorbents with physical and or chemical modifier for the separation of oil-water mixtures.

4. Conclusion

Rice straw waste (RSW) is one of the alternative raw materials to produce cellulose and serve as an alternative material to replace wood cellulose for tissue paper production. Potential RSW for production of cellulose in the preparation of biodegradable tissue paper (BTP) according to the SNI 0103:2008 has been revealed as reflected by its properties that fullfil the standardized properties of tissue papers. The resulting BTP decomposes readily in water and is expected to be decomposed by living organisms. BTP products with high water absorption properties were obtained. The impact for production of BTP especially in Indonesia are to minimize the RSW waste, increasing the value of RSW. In future, the production of BTP can fulfil the demand of industries and house daily consumption, hospital and also others. It is also recommended that conversion of cellulose into nanometer sized of cellulose particles for further studies to produce a thinner tissue and comparable with commercial tissue toilet papers grades.

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