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Effect of Adding Carrageenan Processing Solid Waste from Eucheuma cottonii to Art Paper Manufacture from Water Hyacinth

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ABSTRACT The carrageenan industry produces solid waste *E. cottonii* of 65% to 70%, so it had the potential to be used as a paper material. The aimed of this study was to analyze the effect of the concentration of *E. cottonii* waste which was added in the production of art paper from water hyacinth stems on the quality of the paper. The experimental design used was Complete Randomized Design with 1 factor, namely the difference of *Eucheuma cottonii* waste concentration (0%, 10%, 15%, and 20% (w/w)). The results showed that the addition of carrageenan processing from *E. cottonii* with difference concentration gave significantly different (p<0.05) on the value of grammage, tensile strength, tear resistance, density, and lightness of the paper. The best concentration of *E. cottonii* was 15% because it had the highest tear resistance value (p<0.05) and higher of lightness than the concentration of 0% and 10% (p<0.05). Paper with waste *E. cottonii* 15% had a grammage 148.93 g/m²; tensile resistance 1.28 kN/m; tear resistance 2497.37 mN; density 1.52 g/cm³; and lightness L* 80.19. The results of microstructure observations using Scanning Electron Microscopy (SEM) were the paper with *E. cottonii* waste 15% seen less free space (tighter) than other papers. Based on the grammage and tensile resistance value, paper with *E. cottonii* waste 10% according to Indonesian National Standard 8218:2015 regarding paper and cardboard for food packaging requirements.

Keywords: Art paper; E. cottonii; tear resistance; water hyacinth

INTRODUCTION

As one of the most popular fisheries commodities, seaweed has been utilized as raw material for multiple industries, ranged from household to large scale industries. The majority of seaweed processing industries in Indonesia in hydrocolloids were carrageenan, jelly, and alginate. The extraction of carrageenan from E. cottonii was only used by 30 to 30%, and 65 to 70% became solid waste which tent to be thrown away and become organic garbage. According to Sintaria (2017), seaweed waste of E. cottonii contained 17.47% cellulose and 21.16% hemicellulose. Those components can be used as raw materials for paper production. Cellulose was a supporting component in the production of paper pulp. Cellulose in seaweed could become an innovation of composite application as an amplifier factor (Singh et al., 2017). Therefore, seaweed waste was potentially utilized as material for paper production. However, composite was a collection of materials from various ingredients, that the paper production from seaweed waste still needs other components.

Water hyacinth (*Eichhornia crassipes*) was a weed that had rapid growth so that it could interrupt the water ecosystem. Water hyacinths had natural fiber, i.e., cellulose and hemicellulose, that can be utilized for supporting paper production. SEM analysis from water hyacinth fiber showed that it had a lot of perforated pores (Bhuvaneswari & Sangeetha, 2016). Based on that case, the increment of *E. cottonii* waste expected could fill the pores on the water hyacinth to increase the quality of the paper produced.

Art paper production commonly used some non-wood fiber sources. Cellulose fiber from agriculture waste had a huge

potential as art paper material to produce rough texture paper. Art paper production was one of the waste treatment alternatives to reduces the usage of wood fiber as paper material. Paper from water hyacinth material known as a very convenient art paper material. Examples of art paper usage were tissue boxes, picture frames, invitation card, and others with a high economic value (Nata *et al.*, 2013).

Research on kraft paper production from seaweed material that was red algae *E. cottonii* made into kraft paper bags had been done. *E. cottonii* at 20% produces betterquality paper than 15% and 25% (Ariefta *et al.*, 2019). Previous research on paper production using water hyacinth fiber with 70% concentration had also been done (Tamrin *et al.*, 2013). Nevertheless, no one had reported a study on paper from water hyacinth combined with *E. cottonii* seaweed dregs, a waste of the carrageenan processing industry. Therefore, this research aimed to study the effect of added *E. cottonii* waste concentration on paper production from water hyacinth pulp towards quality and grade of the art paper.

MATERIALS AND METHODS

Materials

The raw material used in this research was seaweed waste processing carrageenan from *E. cottonii* from CV. Karagen Indonesia Factory, Semarang City, Indonesia and water hyacinth from Rawa Pening Lake, Semarang Regency, Indonesia. The chemical material used in this research was from Merck (Germany), which were NaOH, Na₂CO₃, dan H_2O_2 . The equipment used for paper quality examination covers texture analyzer (TA Plus Ametek Lloyd Instrument Ltd., England), micrometer (Mitutoyo, Japan), Chromameter

(Minolta CR-200, Japan), and Scanning Electron Microscopy (SEM) (Jeol JSM 6510LA, Japan).

Methods

Materials preparation

E. cottonii waste was obtained in a semi-wet solid state and dried by sunlight for one day or until the water content was about 5%. Sample waste of *E. cottonii* which had been dried, then mashed using a blender and sifted with strainsized 80 mesh. At the same time, water hyacinth was obtained in the form of a fresh stem with a length between 60 to 90 cm. Before processing, the water hyacinth stalk was cleaned and then cut to 1 cm size.

Paper production

Paper production started by making water hyacinth pulp by extraction using alkali liquid (75 g NaOH and 25 g Na₂CO₃ in 1 L distilled water) for 1 h at 85 to 95 °C with material and solvent ratio 1:2 (w/v). The following process was the extraction of neutralization pulp with running water and filtering. The whitening process was carried out using H_2O_2 2% solution for 30 min and followed by neutralization with running water and filtering. Water hyacinth pulp was mixed with *E. cottonii* waste with different concentrations of 0%, 10%, 15%, and 20%. Paper dough was processed using a cloth screen, followed by dried until the paper was dry.

Grammage test

Grammage value was determined with calculation of mass and area of 5x5 cm paper (ISO, 1995). Grammage value was obtained through the equation.

Grammage
$$\left(\frac{g}{m^2}\right) = \frac{\text{test sample mass (g)}}{\text{test sample area (m2)}}$$

Tensile and tear resistant tests

Tensile and tear resistance tests on paper were determined by pulling 1x8 cm paper using a texture analyzer. Tensile resistance calculated in kN/m, while tear resistance was in mN.

Table 1. Characteristics of art paper.

Density test

According to SNI 140702, density was a quantity that expresses the ratio between mass and volume expressed in g/cm3 (BSN, 1989).

Degree of lightness test

The degree of Lightness test was performing using the CIELAB Method Chromameter, which was a color space that includes all colors the eye could see. The degree of lightness was the L* value read through the chromameter where 0 = black and 100 = white.

Microstructural observations

Paper microstructure was coated by platinum before observed by SEM. The sample paper was observed with 15 kV and 5000x magnification.

Statistical analysis

All data (except microstructural observations) obtained were analyzed to determine the variance and the difference in the effect of each treatment, and Duncan's test was carried out. The presented data was an average of three three replications. The application used in data analysis was SPSS version 20 (International Business Machines Corporation, USA).

RESULTS AND DISCUSSION

Grammage

The results of the grammage test of the art paper by *E. cottonii* and water hyacinth shown in Table 1.

The research results showed that different concentrations of *E. cottonii* waste significantly affected paper grammage value (p<0.05). Subtracting water hyacinth pulp and replacing it with *Echeuma* sp. waste by as much as 10% could increase the grammage value of the paper. The highest grammage value obtained by treatment with a 10% concentration was 189.07 g/m2. It showed that grammage

	Parameters				
Artpaper	Grammage (g/m²)	Tensile resistance (kN/m)	Tear resistance (mN)	Density (g/cm²)	Degree of lightness
E. cottonii waste 0%	100.13±23.81ª	2.49±0.90 ^b	1523.90±0.75°	0.75±0.51ª	64.98±1.67ª
E. cottonii waste 10%	189.07±18.60°	1.67±0.30 ^{ab}	1092.70±0.72ª	1.49±0.44 ^{ab}	75.33±2.43 ^b
E. cottonii waste 15%	148.93±30.62b	1.28±0.15 ^{ab}	2497.37±0.66d	1.52±0.12ab	80.19±0.70°
E. cottonii waste 20%	121.73±22.71ª	0.64±0.21ª	1234.80±0.96 ^b	1.68±0.14 ^b	84.09±2.13°
Kraft paper	73.09	5.54	2260.00	-	54.99
Printed paper A (SNI 7274:2008) [BSN, 2008]	50 to 100	Min. 2.0	-	-	-
Plastic laminated wrapping paper (SNI 6519:2016) [BSN, 2016]	70	Min. 1.65	-	-	-
Paper and cardboard for food packaging (SNI 8218:2015) [BSN, 2015]	26 to 210 (Low grammage) 225 to 500 (High grammage)	Min. 1.6	-	-	-

Note:

- Data ± standard deviation. Values followed by a different superscript showed significantly differences at 5%.

SNI = Standar Nasional Indonesia (Indonesian National Standard)

- BSN = Badan Standardisasi Nasional (National Standardization Agency)

value was affected by the material used in paper production. The greater the mass or weight of the paper the greater the grammage value (Adamopoulos *et al.*, 2013). Based on that case, this research concluded that subtracting water hyacinth pulp and replacing it with *E. cottonii* waste could reduced the mass of paper. It was due to the water hyacinth pulp had more water content than dry *E. cottonii* waste.

An increment of E. cottonii waste concentration of more than 10% generates paper with lower grammage value, even at 20% concentration, produced no significant grammage value (p>0.05) compared with paper without E. cottonii waste (0%). The higher the grammage value, the thicker the paper (Nurhayati et al., 2021). It was suspected because the grammage value of a paper was also affected by the mass of each solid material of paper, that were, cellulose and lignin. Water hyacinth stem contain 66.6% and 7.42% lignin (wb) (Hartono et al., 2010), while the carrageenan processing waste contains 0.96% cellulose, 7.12% hemicellulose, and 8.26% lignin (wb) (Yuliani et al., 2020). An increment of E. cottonii waste could enhance the solidity of paper because it had a soft fiber that could fill the space of paper. Therefore, the density of paper increased. High density on paper could cause thinner paper (Sukaryono & Loupatty, 2018).

The grammage value of art paper in this research were between 100.13 to 148.93 g/m². These value were higher than the grammage value of art paper from *E. cottonii*, 73.09 g/m² (Ariefta *et al.*, 2019). The grammage value of the paper in this research had met the requirement of SNI 8218:2015 about the grammage value of paper and cardboard for food packaging (BSN, 2015). The higher the grammage value, the thicker the paper (Nurhayati *et al.*, 2021).

Tensile resistance

Based on table 1 noted that different concentrations of *E. cottonii* waste significantly impacted paper tear resistance (p<0.05). Tensile resistance value dropped along with the increment of *E. cottonii* waste concentration in paper production. It was affected by the bond between paper that impacted the tensile resistance of the paper. Higher cellulose concentration could generate better and stronger paper fiber, made the paper a more excellent tensile resistance (Pratiwi & Cahya, 2015). The higher the resistance or strength of the paper tensile, the better quality of the paper (Andarini, 2013). In this case, water hyacinths had higher cellulose content than *E. cottonii* waste. Therefore, an increment of the amount of *E. cottonii* waste and the subtraction of water hyacinth pulp caused the degradation of paper tensile resistance in this research.

The tensile resistance value of paper from *E. cottonii* waste and water hyacinth ranged between 0.64 to 1.67 kN/m. The result of this research was almost the same as the tensile strength of kraft paper made from mixed extract cellulose of oil palm and kraft paper from cement bag that was 0.99 kN/m (Nasution, 2019). However, the tensile strength of the paper in this research was much lower than kraft paper made from *E. cottonii* seaweed, 5.54 kN/m (Ariefta *et al.*, 2019). The tensile resistance value of paper from *E. cottonii* waste was 10%, and water hyacinth had met the requirement of SNI 8218:2015 about

tensile resistance value for paper and cardboard for food packaging (BSN, 2015) and SNI 6519:2016 about plastic laminated wrapping paper (BSN, 2016). Paper from water hyacinth without the addition of *E. cottonii* waste also met the requirement of SNI 7274:2008 about tensile resistance value for printing paper A (BSN, 2008).

Paper tensile strength was affected by the concentration of bleach and another paper materials. Escalation of the bleaching agent would decrease the strength paper; otherwise, an increment in concentration from the adhesive would increase the paper's strength. Tapioca was an adhesive capable of fixing the bond between fiber from paper, like internal and surface strength (Sintaria, 2017). Meanwhile, this research was not using adhesive on paper production from water hyacinth and *E. cottonii* waste. Therefore, it still needs further research regarding art paper from water hyacinth and *E. cottonii* waste to know the bleach concentration and the adhesive that was proper for paper production.

Tear resistance

The treatment given to *E. cottonii* waste that affected toward tear resistance of paper, which the highest tear resistance value was found in paper with *E. cottonii* waste at 15%, while the lowest value on concentration at 10% (p<0.05) (Table 1). It was affected by bond between paper fiber that had an impact on tear resistance of paper. In this research, it was suspected that with the content of *E. cottonii* waste 15% produce stronger bond with cellulose on water hyacinth pulp. Higher cellulose concentration indicating that the created fiber would be better and stronger, so it produced better tear resistance (Syamsu *et al.*, 2014). However, usage of *E. cottoni* waste on 20% concentration cause decrease of tear resistance value of paper. Higher cellulose cause hard or brittle on paper and that condition would make the paper easy to tear (Yosephine *et al.*, 2012).

The extraction process of water hyacinth pulp production with chemical material also affected paper strength. Using lye with NaOH and Na₂CO₃ in the extraction process worked to dissolve the remaining lignin in the material. Lignin could interrupt the formation of fiber bonds. The delignification process, besides aiming to dissolve lignin, also had a side effect, the degradation of cellulose on the material that caused fiber would be damaged and not bond perfectly. Damaged fiber would affect the bond between fibers, so it would also affect the tear resistance on paper. Tumolva et al. (2013) explained that chemical material on water hyacinth fiber could affect the mechanical properties of the fiber. The results of the SEM test on water hyacinth fiber with alkaline treatment showed protrusions of short fibers from the cracked samples and a few fibers that came out. The usage of modification chemicals on nature fiber improved the fiber's ability to had better adhesion but caused the fiber surface to had holes due to the delignification process. Therefore, adding E. cottonii waste with the right concentration could fill the gaps and strengthen the fiber binding on paper.

The other factor that affected paper tear resistance was the fiber on each material. Material with fiber bonding that was long and solid caused high tear resistance as well. The content of more short cellulose fiber bonds would cause the paper to be more complex and more brittle. Bhuvaneshwari and Sangeetha (Bhuvaneswari & Sangeetha, 2016) inform that water hyacinths had a long fiber, 15 to 20 cm, and 320 µm diameter. In comparison, the fiber on *E. cottonii* waste tend to be shorter because the content of materials was reduced while processing and could also be seen from the form that was like powder and easily brittle.

The highest tear resistance value of art paper from water hyacinth and *E. cottonii* waste in this research was 2497.37 mN. The tear resistance value was higher than that of paper made from mixed extract palm tree cellulose with cement bag paper 20%, which was 2304.65 mN and paper from *E. cottonii*, which was 2260.00 mN (Ariefta *et al.*, 2019).

Density

Table 1 showed that the increment of concentration of *E.* cottonii waste positively correlated with the enhancement of paper density (p<0.05). It was because one of the functions of *E.* cottonii waste was as composite filler. Paper with high quality had a high density. The higher density of the paper mass, the paper would be more vital because the fiber arrangement and the structure on that paper were getting denser and closing the pores (Nata *et al.*, 2013).

The factor that affected the density of paper was fiber on the materials. Solid pulp sheets would increase the thickness of the paper. Water hyacinth included material with thin-walled fibers. Thin-walled fibers were easy to flatten, producing pulp sheets with more excellent crack resistance than thick-walled fibers. Otherwise, thick-walled fiber would generate sheets with high tear resistance but poor crack resistance (Sugesty, 2015). Therefore, the density value of paper increased along with the increment of *E. cottonii* waste concentration in this research.

Low-density value of paper due to no additions of adhesive in this research. Density factor and adhesive effect toward bending strength between bonds where the higher density and more significant the adhesive, the bond strength increased. It showed that the interaction that happens between density and adhesive had a substantial effect on paper strength (Setyanto *et al.*, 2011).

Degree of lightness

The paper in this research was yellowish-white. The result of the degree of lightness test on art paper from *E. cottonii* waste and water hyacinth was presented in Table 1. The data above showed the L* parameter value of *E. cottonii* waste paper. and water hyacinth showed an increase with increasing concentration of *E. cottonii* waste. The higher L* value indicates that produced paper had a brighter and more transparent color. The increase in L* value proves that adding waste carrageenan processed of *E. cottonii* could improve paper quality in color parameters. Based on Indrayani (2016), when the value of L* showed 0, meaning dark or black, while the value showed 100, meaning bright or white so that the color would be brighter and vice versa.

The degree of lightness of the paper was affected by the content present in the material. The higher concentration of carrageenan processing of *E. cottonii* waste caused an increase in the degree of lightness L* value of paper; which happens due to characteristics of the waste that had a powder form with whiter color compared to the color of water hyacinth pulp. Therefore, subtracting water hyacinth waste and replacing it with *E. cottonii* waste caused whiter

paper production.

The pulp-making process with extraction method with alkali still leaves lignin content and lignin was the most affecting content of paper. The residual lignin on pulp had a poor influence on color, therefore it was necessary to continue the bleaching process with an H_2O_2 solution. This research uses H_2O_2 with a minor concentration of 2%. The higher of H_2O_2 concentration, the more hydroperoxide anions (HOO-) were formed, then could react with H_2O to produce hydroxyl radicals (HO*). The higher concentration caused condensation of free radicals, so it didn't have a practical impact during the bleaching process, which was marked by the color of the pulp that becomes dark (Widiastuti et al., 2016).

Microstructural paper

SEM analytics was used to find the morphology of art paper (Figure 1). The SEM results showed that the fiber of art paper from the water hyacinth had long and thin thread characteristics (Fig. 1A). Characteristics the fiber of art paper with addition *E. cottonii* waste shown in Figures 1B, 1C, and 1D had bigger sizes and flat shapes like white plates and fill the empty space on the water hyacinth fiber. The results of microstructural observations also showed that the paper with *E. cottonii* waste 15% look had less free space (denser) compared to the other paper.

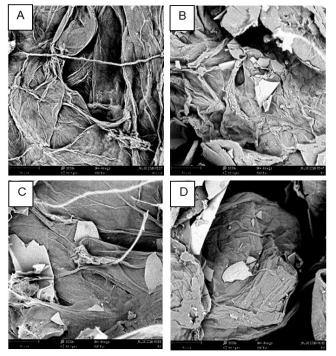


Figure 1. Microstructural of art paper from water hyacinth with *E. cottonii* waste addition (A) 0%, (B) 10%, (C) 15%, and (D) 20%.

Fiber density and properties of the composite were affected by the existing tiny space. Therefore, more concentration of *E. cottonii* waste would close that space, increasing the density value. It positively correlated with the density value, *E. cottonii* waste 20% and water hyacinth had the highest density value and were not significantly different with a concentration of 15%.

The fibers were arranged and bind to each other or fill so that they would have space that indicates the fiber had the strength to hold the liquid content and had good absorption. Therefore, *E. cottonii* waste and water hyacinth could be one of the papers with high absorption. Paper with high absorption can be used as oil-absorbent paper or tissue that quickly absorbs water.

E. cottonii waste paper and water hyacinth can be utilized as art paper, also called mixed fiber paper or composite paper. Hybrid fiber, or composite paper, was paper made from a mixture of two or more materials on paper pulp. The objective was to increase the paper use value. Hybrid fiber paper production was an alternative to produce paper that helps waste reduction (Yosephine *et al.*, 2012).

CONCLUSION AND RECOMMENDATION

Conclusion

This research showed that the increments of *E. cottonii* waste with different concentrations had a significantly different (p<0.05) on grammage value, tensile strength, tear resistance, density, and degree of lightness. Based on the grammage value, the paper with *E. cottonii* waste 15% had the highest tear resistance value, at 2496.37 mN. The result of microstructural observation using SEM was paper with *E. cottonii* waste of 15% seen to had less empty space (denser) than other paper. However, based on the grammage value and tensile resistance, paper with *E. cottonii* waste of 10% met the requirement of SNI 8218:2015 regarding the paper and cardboard condition for food packaging.

Recommendation

Further research regarding art paper from water hyacinth and *E. cottonii* waste is needed to know the bleach concentration and the adhesive that was proper for paper production. In addition, research is also needed on alternative paper materials other than water hyacinth that can be combined with *E. coottonii* waste, for example bagasse, rice straw or other sources of cellulose and hemicellulose. Therefore, art paper production was one of the waste treatment alternatives to reduces the usage of wood fiber as paper material. Examples of art paper usage were tissue boxes, picture frames, invitation card, and others with a high economic value.

AUTHOR'S CONTRIBUTIONS

AP conducted research and analysis of data. END was research idea owner, principle investigator, supervising, data interpretation, and wrote manuscript. RAK interpreted data and wrote manuscript. All authors agreed to this manuscript for publication.

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