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Hydrogel derived from hybrid water hyacinth carboxymethyl cellulose and banana peels pectin hydrogel via citric acid crosslinking

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Abstract

Water hyacinth (WH) is a major pest in aquifer and likewise of banana peel waste that pollutes the environment. WH and banana peels have potential to produce carboxymethyl cellulose (CMC) as well as pectin. Both CMC and pectin are applicable for fabrication of hydrogels that focus on the natural ingredients for their use as food packaging materials. The application of CMC and pectin as a hydrogel material are splendid for increasing its mechanical, biodegradable and environment-friendly properties. This study determined the effect of citric acid as a cross-linking agent on the swelling properties of CMC-pectin based hydrogels and investigated their functional groups. Preparation of hybrid CMC-pectin hydrogels was started by extracting WH cellulose. Through bleaching and dehemicellulose process. Cellulose was modified into CMC in two steps, alkalization and carboxymethylation. At the alkalization stage, the cellulose was mixed with NaOH 10% solution. For carboxymethylation, natrium chloroacetate (Na-CA) was added and stirred at 55°C for 3.5 hrs. Fabrication of hydrogel was mixed with 5% ratio 70:30 (w/w.%) of CMC:pectin. Citric acid (CA) was added as the cross-linking agent with concentrations of 5%, 10%, and 15% on heat treatment. The result of hybrid bio-hydrogels (HBH) was a sheet form film of semi-transparent, and brownish in color. HBH CMC/pectin with the addition of a cross-linking agent in the form of citric acid (5%) gave the highest swelling ability (6.64 wt., within 1 hr). Also, the presence of carboxyl bonds with hydroxyl groups was observed via Fourier transformed infrared spectrometry (FTIR) analysis.

1. Introduction

As it is known that water hyacinth (WH) grows quickly in water areas. The growth speed can reach 400 – 700 tons of biomass per hectare per day (Rakhmania *et al.*, 2017). As a result of very fast growth, WH can cover the surface of the water. WH is considered a weed plant and becomes an environmental problem because it interferes with ecosystem life in the waters. Moreover, South Kalimantan is mostly peat land and swamps, so water hyacinth is easy to grow. When cleaning the water hyacinth area, it will also be wasted. Whereas water hyacinth has a cellulose content of up to 60% (Ajithram *et al.*, 2021). Likewise with banana peel waste. In South Kalimantan, there are many cadgers and food-entrepreneurs of banana-based foods. These business activities produce banana peels which are no longer used in the production process so that they become waste.

Literally, the waste can produce other products. Water hyacinth extracted cellulose and the highest cellulose content in the stem (Rakhmania *et al.*, 2017). From cellulose synthesized into carboxymethyl cellulose (CMC). CMC is a cellulose derivative that has a long and linear chain, is soluble in water, absorbs water, and anionic (Mondal *et al.*, 2015). Generally, CMC is employed as an emulsifier, food preservative, thickener and suspending agent (Ezati *et al.*, 2022).

So far, banana peel waste has only been

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implemented as plant fertilizer or animal feed, even thrown away. So, it is better to use it as other products such as pectin. Pectin is a natural polymer found in most plants (Haas *et al.*, 2021), including bananas. Pectin is also a hydrophilic substance with a negative charge and is gel-forming (Zhang, Wang, Yu *et al.*, 2021). Pectin has been widely used in food, pharmaceuticals, as thin film (Elma, Ghani, Rahma *et al.*, 2022; Elma, Nata, Maulida *et al.*, 2022; Elma, Rahma, Kusumawati *et al.*, 2022; Rahma, Elma, Aliah *et al.*, 2022; Rahma, Elma, Rampun *et al.*, 2022) and cosmetic industries (Guo *et al.*, 2018). Currently, still doing a lot of imports.

The waste product in the form of CMC and pectin is investigated to produce hydrogel. It is found that CMC as a hydrogel is very good for absorbing water molecules. However, it has the disadvantage of rapid biodegradability (Raafat *et al.*, 2012). The strength of CMC-based hydrogels is affected by their shelf-life. Longer shelf life can weaken the structure and bond and reduce the water resistance attached to the hydrogel (Dong *et al.*, 2021). Therefore, the addition of pectin as a composite material is expected to improve the shortcomings of CMC-based hydrogels. Based on the advantages of pectin which can improve crosslink (Martău *et al.*, 2019). The combination of two natural polymers as a hydrogel material can be called a hybrid bio-hydrogel (HBH).

The application of a CMC-pectin mixture as a hydrogel is more effective in the formation of cross-links with the addition of a cross-linking agent. The cross-linking agent used is a carboxylic acid compound. One of them is citric acid. Citric acid (CA) is often used because it is cheap and non-toxic (Ghorpade, 2020). Cross-linking gives the hydrogel an ability to absorb water on a large scale (Rahmani *et al.*, 2022). This research aimed to fabricate a hydrogel made from a mixture of water hyacinth CMC and banana peel pectin with citric acid as cross-linking agent. Analyzing the addition of citric acid variations on the swelling ability and functional groups in the hydrogel.

2. Materials and methods

2.1 Chemicals and materials

Water hyacinth (WH) taken from Gambut river, South Kalimantan-Indonesia (3°22'50.3"S 114° 38'41.4"E). The extraction process for carboxymethyl cellulose (CMC) require several chemicals such as toluene (99%, UPT BPPTK LIPI), 96% ethanol, sodium chlorite ($NaClO_2$) (80%, Sigma-Aldrich), NaOH, CH_3COOH, demineralize water, isobutanol (1.00984.0250, Merck), isopropyl alcohol, and natrium chloroacetate (Na-CA) (607-158-00-5, Merck). Hybrid bio-hydrogel (HBH) fabrication required CMC extracted from WH, banana peels pectin bought from Chemical Retail Shop Surabaya-Indonesia, and citric acid (1.00244.1000, Merck) as cross-linking agent.

2.2 Preparation of CMC derived from water hyacinth

The WH is the highest cellulose content compared to other parts. WH was cut into smaller pieces with a size of 10 cm. It is then dried under the sun and then heated up in the oven at 100°C for 2 hrs. The dried WH was ground to be powder with a size of 30/40 mesh. WH powder was then extracted by Soxhlet at 115°C for 3 hrs employing toluene and 96% ethanol as solvent with ratio 1:1. Afterwards, it was bleached with 3% NaClO for 2 hrs at 80°C. Thereafter, it is rinsed with demineralized water until the powder color becomes whiter. Dehemicellulose process was conducted by soaking the powder in 17.5% NaOH solution for 3 hrs. It was washed with demineralized water and CH_3COOH until reaching pH 4. Then it was dried in the oven up to 60°C for 5 hrs.

The results obtained in the form of cellulose was synthesized into CMC by the alkalization process. Cellulose was mixed with isobutanol-isopropyl alcohol 1:4 and stirred for 10 mins. It is followed by adding 10% NaOH solution and stirred for an hour. Then the carboxymethylation process started by adding Na-CA and stirred for 3.5 hrs at a temperature of 55°C. At the end, drops of CH_3COOH solution were added to reach pH 7. It was then washed with 96% ethanol at the several times. The CMC powder obtained was dried for 5 hrs at 60°C.

2.3 Fabrication of hybrid CMC-pectin hydrogels

Hybrid CMC-pectin hydrogel fabrication was carried out by mixing the CMC-pectin 5 wt.% in every 200 mL of demineralized water. The composition of CMC:pectin ratio was 70:30 wt.%. The mixtures were stirred for 30 mins until it became homogeneous. It was then added with the various citric acid concentrations (5, 10 and 15 wt.%) for 1 hr mixing at 100°C. The hybrid CMC-pectin hydrogels solution was cast on a silicone mold in order to obtain sheet film hydrogels. Hybrid CMC-pectin hydrogels were dried at 55°C for 5 hrs.

2.4 Measurement of swelling ability

Swelling ability (SA) is one of the main parameters in hydrogel testing by calculating water absorption. SA is inversely proportional to water retention ability (Lv *et al.*, 2019). The measurement of SA test was carried out by cutting the HBH sample with a size of 2 × 2 cm, it was then weighed. Then, it was soaked into demineralized water for an hour at room temperature (27±2°C). After an hour, the remaining liquid on the surface of the sample was dried with a tissue. Samples were reweighed. The swelling ability was calculated by the equation:

$$\text{Swelling ability (SA)} = (W_t - W_o)/W_o \quad (1)$$

Where W_o is initial weight of hydrogel in dry condition (in g), and W_t is the weight of the hydrogel after soaking for a certain time (in gram), in this case for 1 hr.

2.5 Characterization of hybrid CMC-pectin hydrogels using FTIR analysis

Functionalization of hybrid CMC-pectin hydrogels were analyzed based on the standard ASTM-E1252 method using Fourier transform infrared (FTIR). The application of FTIR proves the presence of cross-linking materials in HBH by providing information on functional groups in the network. The spectrum used was 400-4000 cm^{-1}.

3. Results and discussion

3.1 Extraction of CMC from water hyacinth

CMC is modified from cellulose and processed by esterification of hydroxyl groups with chloroacetic acid (CA) compounds. The CMC synthesis process consists of alkalization and carboxymethylation processes. In the alkalization process, a mixture of isobutanol and isopropyl alcohol solvent were added to water hyacinth cellulose. Then the addition of NaOH may affect the process of alkalization of cellulose on the –OH bonds. On the other hand, the addition of excess NaOH may reduce the performance. NaOH solution is difficult to substitute for –OH and becomes more soluble. It is due to –OH is not optimally substituted, the cellulose structure becomes crystallized, and it will be more difficult to carry out the carboxymethylation process.

Chloroacetic acid is added to achieve the carboxymethylation process. The carboxymethyl group is substituted for the cellulose molecule on the side of the hydroxyl group. As reported by Kanikireddy *et al.* (2020) that the carboxymethyl group ($-CH_2-COOH$) is attached to the hydroxyl group on the glucopyranose chain of cellulose. So, the process produces the equation:

$$ClCH_2COOH + NaOH \Rightarrow ClCH_2COONa + H_2O \quad (2)$$

$$[C_6H_7O_2(OH)_2ONa]_n + n\ ClCH_2COONa \Rightarrow \quad (3)$$

3.2 Fabrication of hybrid CMC-pectin hydrogels with citric acid crosslinking

HBH was fabricated by CMC extracted from WH and added with banana peel pectin with a ratio of 70:30 (%). Improved stability of natural polymers, such as CMC, other natural polymers can be added to form stronger cross-links (Zhang, Xu, Tao *et al.*, 2021). Banana peel pectin addition is intended to add a carboxyl group. It can bond with the cross-linking agent, citric acid (CA). The results of HBH film fabrication can be seen in Figure 1.

In Figure 1, it is shown that the color of HBH film is semi-transparent and slightly brownish. The pectin addition causes the color of the film to become brownish. Then, the color slightly changes when the temperature starts to rise during hydrogel fabrication, non-enzymatic browning reaction, and Maillard reaction (Gouveia *et al.*, 2019). Judging from color of the sample, the higher the concentration of citric acid used, the brighter the HBH film. Sample A looks more brownish, the brown color in sample B looks faded, and the color of sample C is brighter and more transparent. CA has a good ability to reduce enzymatic browning reactions (Macedo *et al.*, 2019). The addition of citric acid combined with heating is done to reduce the enzyme activity (Anyasi *et al.*, 2015).

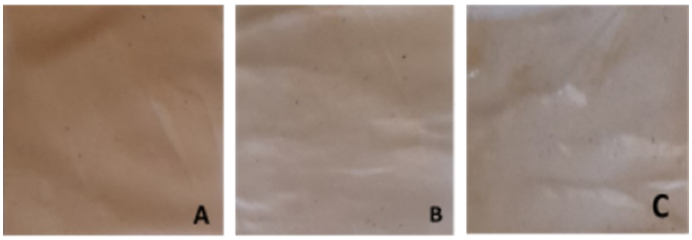

Figure 1. HBH film of CMC from WH and banana peels pectin with citric acid as cross-linking agent: (A) 5%, (B) 10%, (C) 15%.

3.3 Swelling properties

Measurement of swelling ability is needed in food packaging testing. It can also determine the ability of the hydrogel to absorb water. Pectin addition to CMC-based hydrogel can increase water absorption. Figure 2 shows that the swelling ability of the hydrogel using a citric acid concentration of 5% has the greatest swelling ability (6,647 wt.) within 1 hr.

As shown in Figure 3, the HBH film with a higher concentration of citric acid had a stiffer texture after the swelling test. The color is still brownish in the center of the 15% CA sample indicates the presence of cross-links. Number of crosslinks that are intertwined is higher and inversely proportional to the swelling ability as in Figure 2 occurs because the higher concentration of CA, the crosslinks that are intertwined with CMC and pectin are more and dense (Hashem *et al.*, 2013).

At a concentration of 5%, CA produces cross-links that are not too tight. There will be more space to accommodate the absorbed water. In contrast to the

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concentration of citric acid at 10% and 15%, it showed a decrease in the swelling ability. As reported by Kowalski *et al.* (2019), the swelling ability decreases as the concentration of the cross-linking agent increases. Drastic decrease in swelling ability of the 5% CA to 10% CA samples was due to the dissociation of the hydrophilic CMC carboxylate groups. Therefore, water absorption occurs drastically fills the pores (Tang *et al.*, 2014) on the 5% CA sample. The effect of high hydrogen bond interactions between natural polymers CMC/pectin and citric acid can lead to reduced expansion and loosening of polymer chains (Mali *et al.*, 2018).

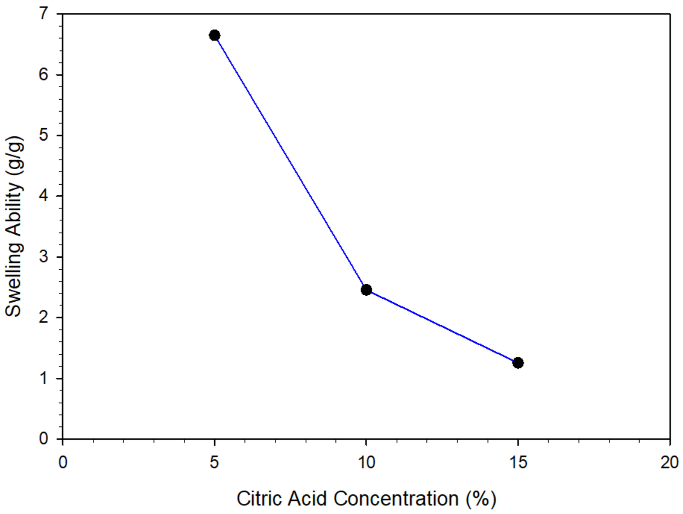

Figure 2. Effect of citric acid concentration on swelling ability of hybrid CMC-pectin hydrogels.

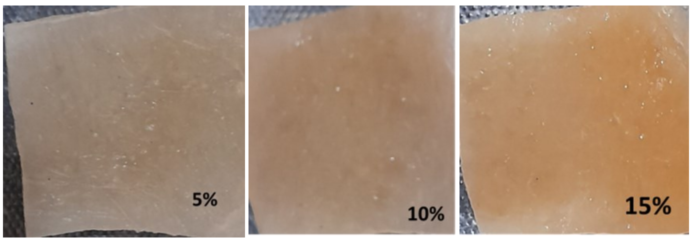

Figure 3. HBH film after swelling test.

3.4 FTIR results

Examination of HBH samples using FTIR to determine the functional groups. Analysis was carried out on HBH samples with CA concentrations of 5%, 10% and 15% w/w. The results of the HBH FTIR analysis are shown in Figure 4.

Figure 4 shows the presence of a hydroxyl group (-OH) that occurs in the range 3255 – 3403 cm^{-1}, and the peak is 3351 cm^{-1}. In this case, it means that hydrogen bonding occurs which gives HBH its hydrophilic properties. There is a small peak only in the 5% CA sample at wavelengths 1081 cm^{-1} and 1278 cm^{-1} indicating stretching of the C-O ester group that binds to the CMC. Associated with the presence of the 1416 cm^{-1} peak there is a carboxymethyl group which indicates that the –OH group in water hyacinth cellulose has been substituted into CMC. CMC has the ability to absorb water very well.

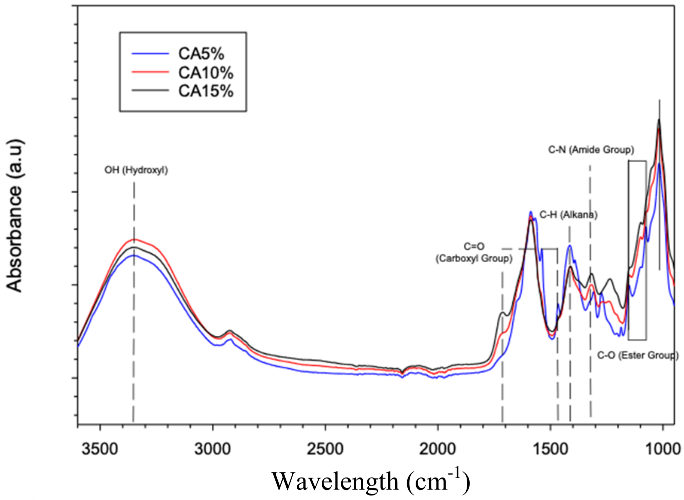

Figure 4. FTIR spectra of hybrid CMC-pectin hydrogel by addiction varied of citric acid concentration as cross-linking agent.

At a wavelength of 1471 – 1716 cm^{-1}, it provides information that cross-linking occurs due to the esterification reaction between the hydroxyl group (-OH) and the carboxylic group (–COOH). The peak was most visible in the 15% CA sample, indicating the density of cross-linking that occurred. Several peaks between the range 1233 – 1319 cm^{-1} indicate an amide group (C–N). Indicates the occurrence of cross-linking also with pectin. Citric acid will form a cyclic anhydride which reacts with reactive OH groups from adjacent polymer chains to form ester bonds (Ghorpade *et al.*, 2018). Furthermore, other unreacted carboxylic groups allow attachment to other polymer groups to increase hydrogen bonds to become stronger (Demitri *et al.*, 2008).

4. Conclusion

HBH film based on water hyacinth CMC with the addition of banana peel pectin at a concentration ratio of 70:30 (%) has been successfully fabricated. From the variations in the concentration of citric acid as a cross-linking agent combined with the peaks in the FTIR analysis, confirmed the presence of a carboxyl group bonding with a hydroxyl group. The highest swelling performance was in HBH immersion for 1 hr in the 5% CA sample of 6.647 wt. The ability of HBH to absorb water and swell can be applied to plastic food packaging to reduce excess water content.

Conflict of interest

The authors declare no conflict of interest.

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