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Effect of Sodium Chloroacetate towards the Synthesis of CMC (Carboxymethyl Cellulose) from Durian (*Durio zibethinus*) peel Cellulose

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Abstract— ***CMC (Carboxymethyl Cellulose) is a derivative of cellulose and are often used in the food industry. Food additives, CMC (carboxymethylcellulose) is used as a stabilizer, thickener and emulsifier. Due to a very wide utilization, easy to use, carboxymethyl cellulose into one of the substances of interest in the food industry. Based on these considerations, it takes an effort to produce a CMC from plant cellulose sources are widely available in Indonesia and less utilized optimally as durian peel. This study aims to determine the yield of cellulose from durian peel with NaOH solution variations in the isolation process and determine the yield of CMC produced with sodium chloroacetate variations in the synthesis process. Results cellulose insulation durian peel using NaOH with a ratio of durian peel and NaOH 10% (w / v) at 1:10, 1:15 and 1:20 is 27.18%; 64.72% and 30.84% respectively. CMC yield on varying the amount of sodium chloroacetate as many as 5, 6, 7, 8, 9 grams is 66.44%; 40.19%; 24.83%; 66.71%; 45.21% sequentially. The optimum conditions of the synthesis reaction CMC is variable with the amount of sodium monokloroasetat 7 grams that produces CMC with a degree of substitution of 1.632.***

Keywords— carboxymethyl cellulose, durian peel, sodium chloroacetate

I. INTRODUCTION

Carboxymethyl cellulose (carboxymethyl cellulose, CMC) is a derivative of cellulose dicarboxymethylation. CMC raw material can be derived from cellulose contained in plants (Awalludin et al, 2004). CMC is widely used as food additives as a stabilizer, thickener and emulsifier (Lestari et al, 2013). In addition, this material widely used in various industries such as: detergents, paints, ceramics, textiles and paper (Wijayani, 2005). Due to a very wide utilization, easy to use, carboxymethyl cellulose into one of the substances of interest in a variety of industries. Thus, it takes an effort to produce a CMC from plant cellulose sources are widely available in Indonesia and less utilized optimally as durian peel. Durian fruit produced in Indonesia and spread across several districts or cities. Based on data from the Indonesian Directorate General of Horticulture, in 2012 the production of durian fruit crops in Indonesia reached 888.127 tonnes.

This number is expected to continue to grow given the market demand for up to twenty years into the future is still promising. With the figure reaching nearly 900 thousand tons, durian also have the potential to produce waste in the form of peel which reached 60-75% of the total weight of the fruit (Untung, 2008). Based on research, durian peel contains a substance that is composed of cellulose as high as 50-60%, about 5% lignin and starch were lower by about 5%. Cellulose content in the peel durian high enough to have the potential to make durian peel as CMC (carboxymethylcellulose). Based on these considerations, the study was conducted in an attempt to produce a CMC of durian peel waste that can be a solution to the needs of CMC and reduction of environmental pollution. Manufacture of carboxymethyl cellulose alkalization which includes the step of reacting the cellulose with NaOH, followed by carboxymethylation reaction between the sodium salt alkaline cellulose with chloroasetate. The main factor to consider in the manufacture of CMC is alkalization and carboxymethylation because it determines the characteristics of the resulting CMC (Wijayani, 2005).

II. METHOD

The phase of the research study include sample preparation stage, the process of cellulose insulation, manufacture of CMC (including alkalization stage and carboxymethylation), and the neutralization process. Durian peel sample preparation starts from the peel cut into pieces, and then dried in the sun to dry. Durian peel dry milled and sieved to 60 mesh sieve. Flour dried durian peel back by using the oven for 1 hour at 60 ° C (Melisa et al, 2014). Durian peel powder obtained is then isolated the cellulose. Durian peel powder soaked with a solution of 10% sodium hydroxide with ratio durian peel porder to solvent 1:10 (w / v), 1:15 (w / v), and 1:20 (w / v), then stirred evenly until the entire powder durian peel perfect submerged. Soaking for 24 hours. After it is filtered using a filter cloth. The residue obtained is then soaked in a solution of hypochlorite (chlorine) 5% for 1.5 hours. Then the mixture is filtered and the resulting residue is washed with distilled water that has been boiled until the odor disappeared hypochlorite. The residue was then put into a petri dish and then dried in an oven with a temperature of 60 ° C to constant weight (Melisa et al, 2014). Extract yield can be calculated by the following equation (1) :

$$Extract\ yield\ (\%) = \frac{dry\ weight\ of\ extract\ durian\ peel}{weight\ of\ durian\ peel\ powder}\ x\ 100\% \qquad ...(1)$$

CMC synthesis done by 5 grams of durian peel extract (cellulose) is then added with 100 ml of distilled water in a 250 ml flat bottom flask. Then added 10 ml of 30% sodium hydroxide solution dropwise. Alkalization process is carried out for 1 hour at 25 ° C on a hotplate fitted shaker. After alkalization completed followed by carboxymethylation. In this research, various variations of the ammount of sodium chloroacetate (w / w) is added to the above mixture is 5, 6, 7, 8, and 9 grams. The mixture is then heated at a temperature of 60 ° C to 70 ° C for 2 hours, the process is carried out with oilbath Carboxymethylation. after which the mixture was filtered and the residue obtained in the form of CMC.

After the process carboxymethylation was completed, a neutralization residue obtained, soaked with 100 ml of methanol for 24 hours. Then the mixture was neutralized using a solution of glacial acetic acid. The mixture is then filtered again and the residue dried in an oven with a temperature of 60 ° C until its weight is constant. CMC obtained dried then crushed using a mortar and sieved to 60 mesh sieve. Carboxymethyl cellulose produced is determined (Melisa et al, 2014). Carboxymethyl cellulose yield is determined by the following formula (2) :

$$Crude\ yield\ of\ CMC\ (\%) = \frac{weight\ of\ product\ (CMC)}{weight\ of\ extract\ durian\ peel}\ x\ 100\% \qquad ...(2)$$

Having obtained the crude CMC then, next step is degree of subtitution determination with method performed by Melisa et al (2014).

III. Result and Discussion

Cellulose insulation is done to separate the cellulose and non-cellulose components such as lignin, hemicellulose, and pectin. On the particle size of the material used will be very influential in the extraction process, which will ultimately increase the amount of lignin and released hemicellulose. This study uses a durian peel with a particle size of 60 mesh, because the smaller the particle samples extracted the higher the yield of cellulose obtained. Durian peel powder extraction using durian peel powder with a water content of 4.8% and a basic compound, ie 10% NaOH solution with a ratio of durian peel and NaOH 10% (w / v) of 1:10, 1:15, and 1:20 with time soaking for 24 hours to determine the yield of cellulose to be obtained. 10% NaOH is used to dissolve the non-cellulosic materials contained in durian peel such as lignin, pectin, and hemicellulose. The immersion process is necessary because the durian peel lignocellulose compounds composed of hemicellulose, cellulose and lignin. These three components are held together tightly to each other as a result of the amorphous structure and β-1,4 glycoside bond in the cellulose and the lignin between cellulose chains (Melisa et al, 2014). The precipitate results delignification process, such as cellulose compounds. Lignin remaining cause brown color.

Bleaching process aims to eliminate the residual lignin in the pulp. In this process the color absorbing molecules (containing kromafor) will be oxidized, so that it becomes polar and soluble in water (Suyati 2008 in Melisa, 2014). Bleaching process will make the pulp into a brighter color or white. For that carried bleaching to remove lignin and bleaching compounds insulation products. Bleaching is done by immersion process using hypochlorite for 1.5 hours. By using chlorine bleaching process (hypochlorite) acts as an oxidant which can oxidize lignin structure. Hypochlorite is also a delignification agent that is widely used and can eliminate lignin and hemicellulose without significantly reducing the cellulose fiber. Hypochlorite is a reagent that is inexpensive and easy to obtain. hypochlorite ion negatively charged nucleophile is easy diadisikan at places of positive charge in lignin. These places are carbonyl and double bond structures in lignin. Hypochlorite ion is a strong oxidizing and will break C-C bonds in the structure proficiency level. Lignin remaining in the residue can be removed with the addition of hot water. The addition of hot water also serves to eliminate hypochlorite compounds and hemicellulose. Hemicellulose composed of short chains of glucose and branched, and hemicellulose more soluble in water.

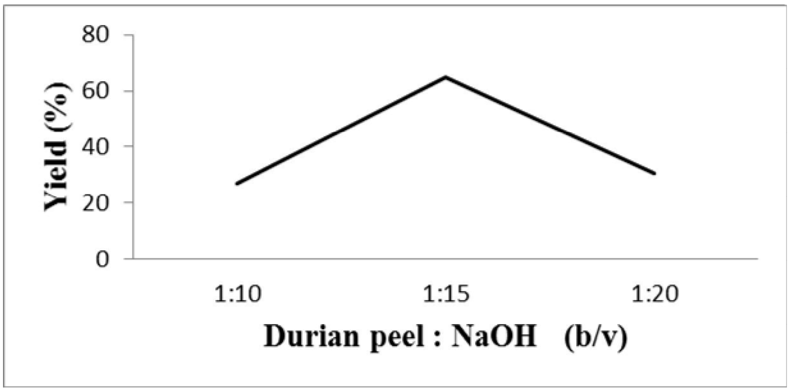

Fig. 1 Effect of durian peel Ratio: NaOH to yield of cellulose

Durian peel powder extraction results in Figure 1 shows that the yield of durian peel extract at a ratio of durian peel and a solution of NaOH 10% (w / v), was 27.18% 1:10, 1:15 is 64.72%, and 1 : 20 was 30.84%. On these results showed that the use of 15% NaOH solution to get the most yield of cellulose. Research conducted by Bidin (2010) with the same treatment and the different materials that rice straw with a yield of 36.335% and the research conducted by Melisa (2014) is made from sweet corn cob with a yield of 36.165%. Therefore the extraction of cellulose used for the manufacture of CMC uses the ratio of durian peel and a solution of NaOH 10% (w / v) 1:15 with a water content of 4.8% and a yield of 64.72% cellulose.

The main factors to be considered in the synthesis of CMC is the process of alkalization and carboxymethylation, because it will determine the characteristics of the resulting CMC (Setiawan et al, 1990 in Arum et al, 2005). Optimization of the synthesis reaction CMC, is determined by the amount of sodium chloroacetic reagents used in the process carboxymethylation. Arum et al, (2005), states that the amount of reagents used will affect the substitution of anhydroglucose units. The amount of influence in the process chloroacetic carboxymethylation, it will largely determine the quality of the resulting carboxymethyl cellulose. That is because the increasing substitution reactions that occur in the process carboxymethylation, then the better the quality of the CMC.

TABLE I
EFFECT OF THE AMOUNT OF SODIUM CHLOROACETATE TO THE CMC YIELD

THE AMOUNT OF SODIUM CHLOROACETATE (G)	THE RESULTS OF CMC SYNTHESIS (G)	YIELD (%)
5	3,32	66,44
6	2.01	40,19
7	1,24	24,83
8	3,34	66,71
9	2,26	45,21

The optimum conditions carboxymethyl cellulose synthesis reaction, namely when the addition ratio of 8: 5 grams of sodium chloroacetic, carboxymethyl cellulose yield obtained at 66.712% as shown Table 1. Previous research conducted by Melisa (2014) from samples of sweet corn cobs optimum conditions of the synthesis reaction carboxymethyl cellulose during the addition ratio of 7: 5 grams of sodium monokloroasetat, carboxymethyl cellulose produced produce yield of 55.79%.

The degree of substitution is an important parameter in determining the quality of a carboxymethyl cellulose. Arum (2005), stated that in terms of quality, the greater the degree of substitution rates, the better the quality CMC because the greater solubility in water. Based on applicable standards, the degree of substitution is in the price range between 0.7 to 1.2 and the pH of a 1% solution between 6.0 to 8.0.

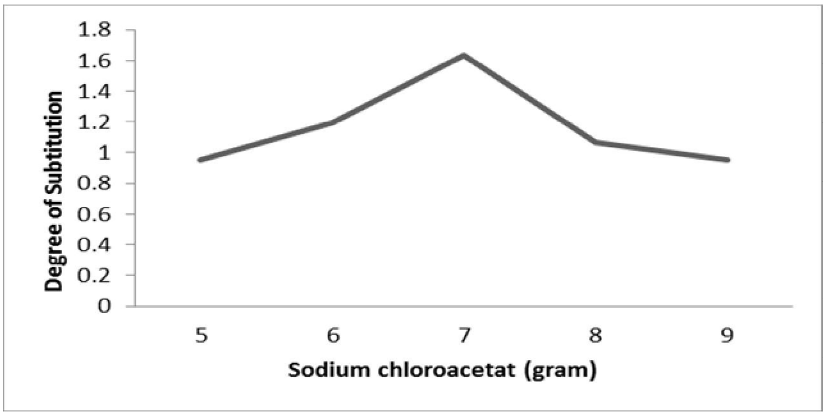

Fig. 2 Effect of Sodium Chloroacetate to the Degree of Substitution of Carboxymethyl Cellulose of Durian Peel

In Figure 2 it can be seen that the optimum conditions carboxymethyl cellulose synthesis reaction by varying the amount of sodium monokloroasetat at a ratio of 7: 5 grams with a degree of substitution of carboxymethyl cellulose 1,632. The result is better when compared to the results of research conducted by Melisa et al (2014), namely carboxymethyl cellulose synthesis reaction by varying the amount of sodium monokloroasetat at a ratio of 7: 5 grams with a degree of substitution of carboxymethyl cellulose 1.403. In the previous study is 0.821 which uses material from sago waste on condition addition of 6 grams of sodium monokloroasetat ratio (Pushpamalar et al, 2005), Arum et al, (2005) in the results using cellulose from water hyacinth carboxymethyl cellulose produced by 0.85 with the amount of sodium monokloroasetat much as 1 gram. Bidin (2010) reported a degree of substitution of carboxymethyl cellulose from straw at a ratio of 1.477 at monokloroasetat sodium cellulose 6: 5 grams.

The addition of 5 to 7 grams of sodium monokloroasetat the carboxymethylation process, showing the value of the degree of substitution of carboxymethyl cellulose higher. The rise in the amount of salt dissolved monokloroasetat will simplify and accelerate the diffusion of salt monokloroasetat into the reaction center is a hydroxy group (Kentjana, 1998 in Arum et al, 2005). The addition of 8 to 9 grams of sodium monokloroasetat value of the degree of substitution obtained decreases. This is because more and more byproducts are formed serperti NaCl, thus lowering the degree of substitution of a carboxymethyl cellulose. Wijayani et al, (2005), stated that the purity of CMC research decreased when $ClCH_2COONa$ increasingly rising, as a result of the increasing number of formed NaCl (sodium chloride) and $HOCH_2COONa$ (sodium glycolate) resulting decline in the degree of substitution. On the addition of sodium monokloroasetat more than 6.0 grams, the formation of glycolic higher and lower reaction efficiency (Pushpamalar et al, 2006).

IV. CONCLUSIONS

Durian peel waste can be used as raw material in the manufacture of CMC because they contain a high cellulose. The amount of 10% NaOH solution which produces cellulose greatest yield is as much as 15% (w / v) is 64.72%. The amount of sodium chloroacetate used during the synthesis process CMC CMC influence the amount of yield. The amount of sodium chloroacetate that gives the highest yield is 8 grams which generate as much as 66.71% CMC. The optimum conditions of the synthesis reaction CMC is variable with the amount of sodium monokloroasetat 7 grams that produces CMC with a degree of substitution of 1.632.

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