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Laboratory Study Of Sugarcane Bagasse Carboxymethyl Cellulose (CMC) Puruty on Filtration Loss in Drilling Mud

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Article Info	Abstract

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Drilling mud plays an important role in drilling operations, one of which is controlling filtration loss, which can affect formation stability. One common additive used to reduce filtration loss is Carboxymethyl Cellulose (CMC). This study aims to examine the effect of CMC purity synthesized from bagasse on the filtration loss value in drilling mud. Bagasse as an additive is processed through several chemical stages to produce CMC with a certain level of purity. CMC purity characteristics are carried out to determine purity and then compare performance with commercial CMC with concentrations of 2, 4, 6, and 8 grams. This test was carried out using the standard API filtrate test for 30 minutes. The results showed that increasing the concentration of CMC and commercial CMC reduced the filtrate loss value. CMC from bagasse with a purity of 99.64% showed excellent results and the results for filtration loss testing showed that it could be 14-10.8 ml, mud cake thickness of 1.37-1.54 mm, density of 8.53-8.59 ppg.

1. INTRODUCTION

In the oil and gas industry, the drilling process plays a crucial role in producing hydrocarbon resources. One key component of this process is drilling mud, which serves to cool and lubricate the drill bit, as well as maintain well stability. (Novrianti and Umar 2015). The use of thickening agents in the mud is crucial for controlling filtration loss, the amount of fluid lost into the rock formation during the drilling process. Filtration loss is the loss of fluid that seeps into the rock formation during the drilling process, which can cause various problems ranging from formation damage to increased operational costs. (Novrianti et al. 2019). CMC can be obtained from various sources, including bagasse, a waste product from the sugar processing industry. Using bagasse as a raw material for CMC not only helps reduce waste but also provides a more environmentally friendly and sustainable alternative. However, the quality of CMC produced from bagasse is greatly influenced by its purity. This purity is directly related to the content of contaminants and other interfering substances that can affect its performance in drilling

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mud. Furthermore, the quality of bagasse used as a raw material also varies. The levels of cellulose, lignin, and other contaminants in bagasse can affect the CMC extraction process. (Pane et al. 2023).

Carboxymethyl cellulose (CMC) is a water-soluble derivative of cellulose (hydrophilic colloid) which is effective in binding water so as to provide a uniform texture, increasing durability. CMC is filled with synthesis through two stages, namely alkalization and carboxymethylation. These two stages can take place in solid form and in the medium of water and organic solvents. Alkalization is carried out before carboxymethylation using NaOH. This process aims to activate the OH groups on the cellulose molecule. The carboxymethylation process is an esterification process. At this stage, the carboxylic acid group is bonded to the cellulose structure. Therefore, the carboxylic acid group in trichloroacetic acid can also be used.

After the synthesis process, the resulting CMC is further purified. The purpose of this study is to examine the effect of the purity level of CMC from bagasse on the filtration loss value in drilling mud and to compare it with commercial CMC. The quality of the resulting CMC can be seen from several parameters, namely: the value of the degree of substitution (DS), pH, viscosity, functional groups, and purity. The characteristics of CMC include: DS, pH, viscosity, water content, NaCl content, and purity. All CMC quality tests will be compared with SNI 06-3736-1995 and supported by analysis of the FT-IR spectra of cellulose and CMC.

2. METHODS

The sample used was bagasse. The materials used in this study were 17.5% NaOH, ethanol, methanol, distilled water, 3% H2O2, AgNO3, PP indicator, bentonite. The tools used in this study were a hot plate, scales, blender, sieve analyzer, spatula, LPLT, pH meter, oven, titration tool, mud mixer.

Procedure:

Sample Preparation

The process of making CMC (Carboxymethyl Cellulose) from sugarcane bagasse carried out in the laboratory is as follows:

The pulp of the mother that is taken is the stem, then washed with clean water. It is dried until it is dry for approximately 7 days under the sun to remove the very high-water content. The dried pulp of the mother is cut into small pieces to make it easier to blend and grind using a blender, then sieved with a 100-meish sieve.

Cellulose Isolation

Cellulose isolation is carried out in 2 stages, namely

Stage 1 Cellulose Extraction

20 grams of powdered sugarcane bagasse was placed in a beaker. Then, the delignification stage aimed to remove lignin and hemicellulose using 17.5% NaOH (Nuringtyas, 2010). 400 ml of 17.5% NaOH was added and heated for 2 hours at 80°C with constant stirring. Then, it was filtered and washed clean using distilled water. According to Robinson (1995), lignin removal with NaOH will be indicated by a blackish-red color in the solution.

Stage 2 Bleaching

20 grams of residue was bleached using 200 ml of 3% H2O2 for 2 hours at 60°C. The bleaching process was repeated 3 times until the cellulose color became white.

CMC Making Procedure

Cellulose isolation is carried out in 2 stages, namely

Stage 1 Alkalization

Cellulose was weighed as much as 10 grams. Then it was put into a 300 ml Erlenmeyer flask placed on a hotplate stirrer at a speed of 100 rpm then added a solvent of 200 ml isopropanol 25 ml ethanol 25 ml aquadest and stirred for 10 minutes. Next, 20 ml of 15% NaOH solution was added drop by drop and the alkalization process lasted for 1 hour at a temperature of 24°C. The purpose of the alkalization process was to activate the hydroxyl groups in cellulose.

Stage 2 Carboxymethylation

After the alkalization process is complete, the carboxymethylation process is continued by adding 20 ml of sodium chloroacetate with varying concentrations (30%). The mixture is then heated at varying temperatures (55°C) for 3 hours, this process is carried out on a hotplate magnetic stirrer.

Stage 3 CMC Neutralization

After the carboxymethylation process is complete, the residue is filtered and proceeded to the neutralization process. The mixture is filtered and the residue is transferred to a beaker and its pH is measured. Then, 100 ml of 90% glacial acetic acid is added until the pH is neutral, namely 7. After the neutralization process is complete, the mixture is filtered and washed with distilled water. The resulting residue is then soaked in 100 ml of methanol solution for 24 hours to remove salts from the alkalization and carboxymethylation processes.

The solid obtained from the filtration was then dried in an oven at 70°C for 5 hours. After drying, it was ground using a blender and sieved with a 60-mesh sieve to obtain CMC powder.

Degree of Substitution Test

CMC of 0.5 g of sugarcane bagasse was put into an Erlenmeyer flask, then 100 ml of distilled water was added while stirring, then 25 ml of 0.05 N sodium hydroxide solution was heated for 15 minutes and titrated with 0.03 N hydrochloric acid and pp indicator.

pH Testing

Weigh 1 gram of CMC and add 100 ml of distilled water, heat on a hotplate until dissolved at a temperature of 70°C after measuring the pH.

Water Content Testing

Weigh 2 grams in a cup, then dry the cup and its contents for 3 hours at 105°C in the oven so that the weight remains constant.

Viscosity Testing

Weigh 2 grams of CMC, add 100 ml of distilled water, then heat for 2 minutes and transfer to a beaker and the viscosity is entered at a speed of 100 rpm for 2 minutes, then the scale analysis reading is repeated 3 times.

NaCl Level Testing

A total of 0.5 grams of dry CMC material was diluted with 1 ml of distilled water and heated at 70°C, after which the solution was titrated with K_2CrO_4 and 0.01 N $AgNO_3$.

CMC Purity Testing

The testing characteristics of the purity of CMC 100% minus the %NaCl value.

Drilling Mud Making Process

Prepare the mud mixer and cup mixer. Measure out 350 ml of distilled water and weigh 22.50 grams of bentonite, then add the sample (2, 4, 6, and 8 grams). Put the water into the vessel, then attach the vessel to the multimixer and add the bentonite and additives little by little after the mixer is running. Turn off the mixer once everything is mixed.

Filtration Loss Testing

Prepare the filter press tool and immediately install the filter paper as tightly as possible and place a measuring cup under the cylinder to collect the filtrate fluid. Pour the mud mixture into the cylinder to a limit of 1 inch below the surface of the cylinder, measure with a caliper, and immediately close it tightly. Then flow air with a pressure of 100 psi. Record the filtrate volume for 30 minutes using a stopwatch. Stop the air pressure to release the air pressure through the cylinder (bleed off) and pour the remaining mud back into the cylinder into the mixer cup. Determine the thickness of the mud cake using a caliper.

Density Testing

Calibrate the mud balance equipment as follows:

Cleaning the mud balance tool. Fill the cup with full water, then close it and clean the outside and dry it with a tissue. Set the rider on a scale of 8.33 ppg. Check the glass level, if it is not balanced adjust the calibration screw until it is balanced. Weigh 22.5 grams of bentonite and CMC bagasse at concentrations of 2, 4, 6, and 8 grams. Prepare the mixer cup and put it in a 350 ml water vessel then install the vessel into the multi mixer and add bentonite and CMC bagasse little by little while the multi mixer is running, after 20 minutes of stirring, take the vessel and fill the mud balance cup with the mud that has been made. Close the cup and clean the mud that sticks to the outside and inside of the tool. Place the mud balance in its position then adjust the rider until it is balanced. And read the density indicated by the scale. (API Specification 13A, 2015)

3. RESULT AND DISCUSSION

This study produced a CMC product that meets the requirements of SNI 06-3736-1995. The results obtained in this study can be seen in Table 1 which explains the degree of substitution, water content, pH, viscosity, NaCl content, and purity.

Table 1. Results of CMC Characteristics of Sugarcane Bagasse

Parameter	CMC Sugarcane Bagasse	CMC (SNI 06-3736-1995)	
		Quality I	Quality II
Degree of Substitution	0,95	0,7-1,2	0,4-1,0
pH	6,64	6-8	6-10
Viscosity	17 ,3 cP	>26 cP	< 26 cP
Purity	99,64%	99,50%	65,00%

The degree of substitution test is an important parameter for determining the quality of CMC. The result obtained was 0.95, which meets quality standard I.

The purpose of this study was to determine the number of substituted hydroxyl groups and determine the solubility of CMC in bagasse. The higher the degree of substitution, the better the quality of carboxymethyl cellulose, as it is more soluble in water (Rofifah and Kelly 2020). A higher degree of substitution contributes to better solubility of CMC, thus forming a stable and effective gel structure in drilling mud, thereby reducing filtration loss.

Moisture content testing was performed to determine the quality of bagasse. In this study, samples and cups were weighed and then dried in an oven for 3 hours at 105°C. After calculations, the moisture content test result was 9%. This 9% moisture content indicates a low moisture content, which will increase stability.

The pH test obtained a value of 6.64 and met quality standard I, indicating that the bagasse CMC has good stability in drilling mud. pH is measured on a scale of 0 to 14, with 7 being neutral. A value below 7 will cause acidity, while a value above 7 will cause alkalinity. Therefore, the bagasse CMC pH value of 6.64 is below 7, which is acidic.

This viscosity test yielded a result of 17.3 Cp. Although this is a low value, it is still acceptable for use in drilling mud. A low viscosity can reduce the CMC's ability to form an effective filter cake layer, resulting in higher filtration loss compared to CMC with a high viscosity.

In this study, the NaCl value was found to be 0.36%. The purpose of this NaCl test is related to purity. The NaCl content greatly influences the purity value because the lower the NaCl, the higher the purity.

The purity test yielded a value of 99.64%, categorizing it as high as expected. This high purity indicates that the purification process was effective in removing impurities and lignin from the bagasse.

Table 2. Filtration Loss Testing

Mud Sample	CMC Sugarcane Bagasse (ml)	CMC Industry (ml)	API Standard (ml)
LS + 2 gr	14	8,7	Maximal 15
LS + 4 gr	12,5	7,8	
LS + 6 gr	11,6	6,2	
LS + 8 gr	10,8	5,6	

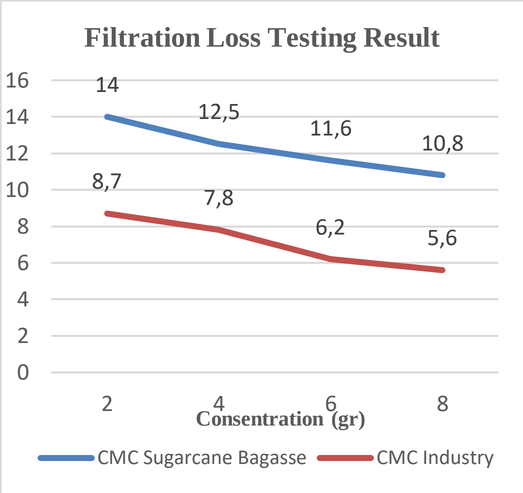

Fig. 1. Filtration Loss Testing

Based on Table 1, filtration loss testing shows that bagasse CMC is able to reduce fluid loss in drilling mud, although it lags behind compared to industrial CMC. At a concentration of 2 grams, the filtration loss of bagasse CMC is 14 ml, while industrial CMC is more effective with a value of 8.7 ml. Increasing the concentration of bagasse CMC from 4 to 8 grams shows a decrease from 12.5 ml to 10.8 ml, but remains higher than industrial CMC from 7.8 to 5.6 ml. Filtration loss that is too large has a negative effect on the formation and mud because it can cause a reduction in permeability to oil and the mud will lose a lot of fluid (Fitrianti 2012). However, all results still meet API standards, indicating that bagasse CMC is an additive to drilling mud.

Table 3. Mud Cake Testing

Mud Sample (gr)	CMC Sugarcane Bagasse (mm)	CMC Industry (mm)	Standar API
LS + 2 gr	1,37	0,26	Maximal 2
LS + 4 gr	1,41	0,34	
LS + 6 gr	1.48	0,47	
LS + 8 gr	1,54	0,58	

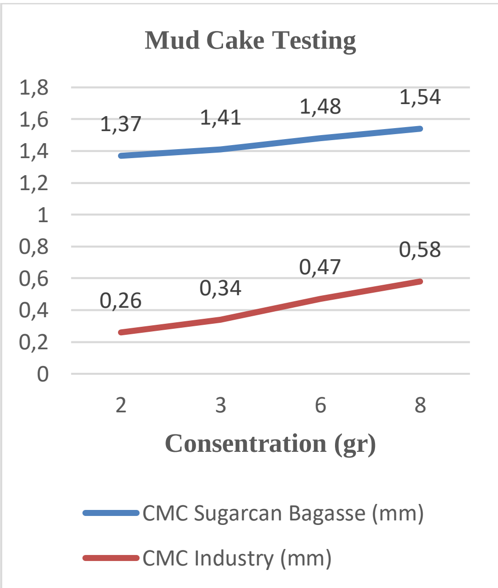

Fig. 2. Mud Cake Testing

Based on the test results in table 2, it can be concluded that the CMC of bagasse increased when the addition of bagasse CMC to the drilling mud sample resulted in an increase in drilling mud particles of 1.37 - 1.54 mm. While the results of the mud cake of industrial CMC mud were 0.26 - 0.58 mm. From these results, it shows that the CMC of sugarcane amps is higher than the industrial CMC because bagasse is made from natural materials that have fibers so that the results of sugarcane amps are higher than the industrial CMC. However, the results of the mud cake test for CMC of sugarcane amps and industrial CMC, both results still meet the standard of 2 mm. The importance of good mud cake thickness is because if the results are thick, it can cause the drilling pipe to be pinched (Angriani, Purnamawati, and Husbani 2023).

Table 4. Density Testing

Mud Sample (mm)	CMC Sugarcane Bagasse (ppg)	CMC Industry (ppg)	API STANDARD Drilling Mud Density (ppg)
LS + 2 gr	8.53	8.62	8,5 – 9,6
LS + 4 gr	8.55	8.65	
LS + 6 gr	8.57	8.68	
LS + 8 gr	8.59	8.70	

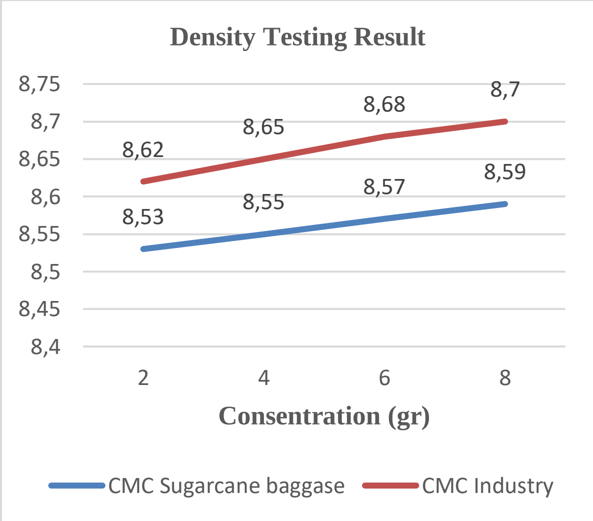

Fig.3. Density Testing

Table 4 shows that the mud density increases with the addition of CMC concentration, which initially increased from 8.53 ppg in standard mud to 8.59 ppg with an additive mass of 2-8 grams. This means that bagasse CMC shows good performance as an alternative to industrial CMC in drilling formulations because it meets API standards. (Amin 2014)

4. CONCLUSION

The purity of CMC was obtained at 99.64%, which is quite good. In the tests carried out on mud with the addition of CMC Bagasse additives with sample concentrations of 2, 4, 6, and 8 grams, it affected the filtration loss in the drilling mud. In the CMC Bagasse sample, the filtration loss was 14-10.8 ml and in the sample with the addition of industrial CMC additives, it was 8.7-5.6 ml. Meanwhile, the thickness of the mud cake in the CMC Bagasse additive was 1.37-1.54 mm and for the thickness of the mud cake in the sample with the Industrial CMC mixture, it was 0.26-0.58 mm. For the density test of CMC Bagasse, it was 0.53-0.59 ppg, while for the density of Industrial CMC, it was 8.62-8.70 ppg. From the tests carried out between standard mud with CMC Bagasse and mud with Industrial CMC, it was directly proportional, based on the filtration loss test, it was able to overcome the filtration loss in drilling mud that met the API 13A standard.

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