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Effect of CMC Concentration from Oil Palm Empty Fruit Bunches and Tapioca Starch Ratio on Biodegradable Adhesive Properties

Elnis Seftiani Gaho[1], M. Prasanto Bimantio[2]✉

[1, 2] Agricultural Product Technology, Faculty of Agricultural Technology, Institut Pertanian STIPER Yogyakarta, Indonesia

✉ **Corresponding Author :** M. Prasanto Bimantio (e-mail: bimantiomp@instiperjogja.com)

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ABSTRACT

This study examines the use of empty oil palm bunch waste (EFB) as a raw material for Carboxymethyl Cellulose (CMC) for the development of biomass-based biodegradable adhesive. The novelty of this research lies in the application of EFB-derived CMC combined with tapioca starch as a biodegradable adhesive system that remains underexplored in bioadhesive development. The study used a randomized complete block design of two factors, namely the concentration of CMC (1%, 2%, and 3%) and the ratio of tapioca starch (6 g, 12 g, and 18 g). The parameters tested include viscosity, pH, adhesion, and drying time. The results showed that the concentration of CMC and tapioca starch had a significant effect on viscosity, adhesion, and sdrying time, but had no effect on pH. Increasing tapioca starch and CMC concentrations increased viscosity and adhesion through the formation of amylose–amylopectin gel network and hydrogen bond interactions with CMC, but slow down drying time due to increased water retention in the gel matrixThe best treatment was obtained in the C3P3 combination which produces the highest viscosity and adhesion values. This study confirms that EFB has the potential to be used as a raw material for CMC to produce biodegradable adhesive that is more environmentally friendly than synthetic adhesives and provides added value to palm oil industry waste.

INTRODUCTION

Palm oil is one of the strategic plantation commodities that contributes greatly to the Indonesian economy, especially as a producer of the country's foreign exchange through the export of its derivative products (Azahari, 2019). In the process of processing fresh fruit bunches (FFB) into crude palm oil (CPO), solid waste in the form of empty oil palm bunches (EFB) is produced in considerable quantities and until now its utilization is still not optimal (Erivianto et al., 2022). EFB production in Indonesia is estimated to reach around 1.9 million tons in dry conditions or the equivalent of ±4 million wet tons per year, so it has the potential to cause environmental problems if not managed properly (Siahaan & Darianto, 2020). EFB is classified as lignocellulose biomass because it is composed of the main components in the form of cellulose, hemicellulose, and lignin (Pranoto, 2016). High cellulose yields make EFB potentially used as a raw material in the development of value-added biomaterials, one of which is as a source of Carboxymethyl Cellulose (CMC) (Dewanti, 2018). In addition, the fiber content in EFB also has the potential to be used as a basic material for the development of biomass-based products that are more environmentally friendly (Kresnawaty et al., 2017). However, the use of EFB in the field is still relatively limited and is generally only used as organic fertilizer or allowed to accumulate in the factory area due to the limitations of processing technology and the high cost of material distribution (Abdillah & Eidil, 2020). These conditions can cause environmental pollution and disturb the balance of the ecosystem if not handled appropriately (Hizriani, 2023). In addition, the increasing amount of EFB waste is also a challenge in the plantation biomass management process (Praevia, 2025).

On the other hand, the need for adhesives or glues continues to increase as various industrial sectors such as the wood, paper, textile, packaging, and construction industries develop. Adhesives commonly used today are still mostly based on synthetic materials, such as polyvinyl acetate (PVA), polyurethane, urea formaldehyde (UF), phenol formaldehyde (PF), and melamine urea formaldehyde (MUF). Despite having good adhesion capabilities, the use of synthetic adhesives is known to have a negative impact on health and the environment because it produces emissions of volatile organic compounds (VOCs) and contains formaldehyde which is toxic and carcinogenic (Pranata et al., 2025). In addition, the continuous use of synthetic adhesives also has the potential to increase the risk of environmental pollution due to the content of chemicals that are difficult to degrade (Eskani et al., 2017).

Increasing attention to sustainability issues is driving the development of safer and more environmentally friendly biomass-based adhesives or bioadhesives (Zhang et al., 2024). One of the materials that has the potential to be used is Carboxymethyl Cellulose (CMC), which is a starch-based bioadhesive that has great potential as an environmentally friendly adhesive because it comes from renewable resources, has a lower environmental impact than petrochemical-based synthetic adhesives, and is able to form a stable and biodegradable adhesive system based on polysaccharides such as starch and cellulose (Arias et al., 2021). In addition, the development of biomass-based lignocellulosic adhesive and biodegradable glue continues to be carried out to produce adhesives with good adhesion characteristics and are safer for the environment (Zhang et al., 2024).

One of the materials that has the potential to be used as a bioadhesive is CMC, which is a cellulose derivative that has water-soluble properties, is able to form a film layer, and has a high enough viscosity that it is suitable for use as an adhesive material (Dimawarnita & Faramitha, 2019). In addition, CMC is also known to have the ability to be a binding and stabilizing agent in various polysaccharide-based biomaterial applications (Safitri et al., 2017). In this study, the use of CMC was combined with tapioca starch as a natural additive. Tapioca starch was chosen because it has adhesive properties, is easy to obtain, biodegradable, and is able to improve the viscosity and adhesion characteristics of natural adhesives due to its amylose and amylopectin content (Faridah & Thonthowi, 2020).

Research on the use of starch as an environmentally friendly adhesive material has been done because starch is known to be able to increase the viscosity and adhesion of biodegradable adhesives. Din et al. (2020) states that starch is a sustainable source of biomaterials that has great potential for the development of biomass-based adhesives. In addition, Watcharakitti et al. (2023) explained that modified starch has the ability to form a good polymer network so that it has the potential to be used as an environmentally friendly bioadhesive. Research on the synthesis of Carboxymethyl Cellulose (CMC) from EFB has also been conducted. Trisnawita & Putri (2024) shows that EFB-based CMC has good solubility and binding characteristics so that it has the potential to be used as a supporting material in adhesive systems.

However, previous research has generally focused on starch or CMC separately and has not investigated their combined application as the main component of biodegradable adhesive based on EFB. In addition, information regarding the interaction effect of EFB-derived CMC concentration and tapioca starch ratio on the physical properties of the adhesive, including viscosity, adhesion, pH, and drying time, remains limited. Therefore, this study aims to analyze the effect of variations in EFB-derived CMC concentration and tapioca starch ratio on the physical properties of the resulting biodegradable adhesive as a potential environmentally friendly adhesive based on renewable biomass.

RESEARCH METHODS

This research was carried out at the Laboratory of Agricultural Product Technology, the Central Laboratory of INSTIPER, and the Laboratory of the Faculty of Forestry. The main material used in this study is oil palm empty bunch fiber (EFB) as a source of cellulose. Chemicals used include nitric acid (HNO_3), sodium nitrite ($NaNO_2$), sodium hydroxide (NaOH), sodium sulfite (Na_2SO_3), sodium hypochlorite (NaOCl), hydrogen peroxide (H_2O_2), hydrochloric acid (HCl), sodium monochloroacetate, as well as additives such as tapioca starch, glycerin, sodium benzoate distilled water, and methanol. The tools used include beakers, erlenmeyers, measuring cups, pipettes, ovens, hot plates, magnetic stirrers, analytical scales, and pH meters. This study used a Randomized Complete Block Design (RCBD) with two treatment factors, namely the concentration of Carboxymethyl Cellulose (CMC) from EFB (1%, 2%, and 3%) and the ratio of tapioca starch (6 g, 12 g, and 18 g). The combination of the two factors resulted in nine treatments, each of which was repeated twice for a total of 18 trial units.

The synthesis of Carboxymethyl Cellulose (CMC) from empty oil palm bunches (EFB) begins with washing 250 g of EFB fiber thoroughly, then drying using an oven at 60°C for 24 hours. The dried fibers are ground and sifted using a 60 mesh sieve until a uniform particle size is obtained. A total of 75 g of EFB powder was then reacted using 1 L of HNO_3 3.5% added 10 mg $NaNO_2$ at 90°C for 2 hours. The filtered residue is digested using a mixture of 2% NaOH and Na_2SO_3 2% at 50°C for 1 hour, then a bleaching process is carried out using 1.75% NaOCl at a temperature of 70°C for 30 minutes. The residue was then re-reacted using 17.5% NaOH at a temperature of 80°C for 30 minutes and bleached using H_2O_2 10% at 60°C. The cellulose obtained is then dried using an oven at a temperature of 60°C for 9 hours. 20 g of cellulose is dispersed into 400 ml of distilled water until homogeneous, then 40 ml of 30% NaOH is added while stirring using a magnetic stirrer at 60°C for 1 hour. Next, 100 ml of monochloroacetic acid 25% is added slowly and the mixture is reheated at 50°C for 3 hours until coarse CMC is obtained. The mixture was then neutralized using 4 ml of glacial acetic acid, washed using distilled water, and soaked in 400 ml of methanol for 24 hours. CMC is then dried using an oven at 60°C for 3 hours and ground into a fine powder.

The manufacture of biodegradable adhesive is done by heating 40 ml of distilled water to a temperature of 60°C, then CMC according to the treatment (1%, 2%, and 3%) is added while stirring using a magnetic stirrer to form a homogeneous solution. The tapioca starch as treated (6 g, 12 g, and 18 g) is mixed with 20 ml of distilled water and heated to 75°C to form a transparent thick paste due to the gelatinization process. The starch paste is then mixed into the CMC solution and stirred at 70°C for 10 minutes until homogeneous. Next, 2 ml of glycerol and 0.1 g of sodium benzoate were added which had been dissolved in 5 ml of distilled water. The mixture is transferred to a measuring cup and distilled water was added to a final volume of 100 ml, then cooled at room temperature and stored in a sealed bottle until testing is carried out (Susilawati & Rahmaniar, 2018).

RESULTS AND DISCUSSION

Glue Viscosity Test

Viscosity testing was carried out to determine the level of viscosity of biodegradable adhesive resulting from various combinations of CMC EFB concentration and tapioca starch ratio. Viscosity is an important parameter that affects the ease of application, spreading, and stability of the adhesive system.

Table 1. Results *of Two Way ANOVA* Biodegradable adhesive Viscosity Test

Sources of Diversity	SS	df	MS	F	Sig
C	990.824	2	495.412	462.445	.001
P	6157.414	2	3078.707	2873.837	.001
C x P	2013.431	4	503.358	469.862	.001
Total	30517.568	18			

Based on Table 1, ANOVA results showed that the concentration of CMC (C), the ratio of tapioca starch (P), and the interaction of the two (C × P) had a significant effect on the viscosity of biodegradable adhesive ($p < 0.05$). The tapioca starch factor has the most dominant influence compared to other factors, indicating that starch is the main component controlling the rheological properties of the adhesive system. The increase in viscosity is caused by the gelatinization process of tapioca starch during heating. In this condition, starch granules undergo swelling due to water absorption, followed by disruption of crystalline structure and amylose release which then forms a three-dimensional polymer network. The network increases the viscosity resistance so that the viscosity increases. Amylupectin plays a role in maintaining system stability through its viscoelastic properties, while CMC EFB functions to strengthen the gel matrix through the interaction of hydrogen bonds with the starch hydroxyl group, thereby increasing the homogeneity of the adhesive system. Haryanti et al. (2014) stated that the increase in viscosity in starch-based systems is related to the formation of gel structures due to the gelatinization process. Din et al. (2020) It also explains that starch plays an important role in improving rheological properties in biomass-based bioadhesives. Compared to the study, the results of this study show a tendency of more stable and higher viscosity, which is thought to be influenced by the contribution of CMC EFB in strengthening gel tissues and improving the cohesiveness of polymer structures.

Table 2. Duncan Viscosity Test Results

C	P			
	P1	**P2**	**P3**	**Average P**
C1	46516,00	26333,00	60842,50	44563,83
C2	7320,00	34136,00	53797,50	31751,17
C3	2630,65	11745,50	66611,50	26995,88
Average C	18822,22	24071,50	60417,17	

Based on the Duncan test, the combination of C3P3 treatments yielded the highest viscosity values compared to other treatments. This indicates a synergy between tapioca starch and CMC EFB in forming a tighter gel network that significantly increases the viscosity of the adhesive system.

Glue pH Test

pH testing aims to determine the acidity or alkaline level of the biodegradable adhesive produced. The pH value is important to ensure the safety of use as well as the stability of the product.

Table 3. Results *of Two Way ANOVA* Biodegradable adhesive pH Test

Sources of Diversity	SS	df	MS	F	Sig
C	.000	2	.000	.	.
P	.000	2	.000	.	.
C x P	.000	4	.000	.	.
Total	648.00	18			

Based on test results *Two Way ANOVA* in Table 3, it is shown that the concentration factor (C), treatment (P), and the interaction of the two (C × P) do not affect the pH of biodegradable adhesive. This is shown by the absence of data variation, where all treatments have the same pH value of 6. This condition causes the value of F and significance to not be calculated. This constant pH value indicates that the formulation and manufacturing process of biodegradable adhesive are relatively stable, so changes in the concentration of CMC and the ratio of tapioca starch do not affect the acidity level of the product. Thus, further testing was not carried out because there was no real difference between the treatments.

Glue Adhesion Test

Adhesion testing is performed to evaluate the glue's ability to bond two paper surfaces. In this study, the score method was used to facilitate qualitative assessment.

Table 4. Results *of Two Way ANOVA* Biodegradable adhesive Adhesion Test

Sources of Diversity	SS	df	MS	F	Sig
C	4.000	2	2.000	9.000	.007
P	12.333	2	6.167	27.750	.001
C x P	15.667	4	3.917	17.625	.001
Total	162.000	18			

Based on test results *Two-Way ANOVA* in Table 4, shows that the concentration of CMC (C), the ratio of tapioca starch (P), and the interaction of the two (C × P) have a significant effect on adhesion ($p < 0.05$). The tapioca starch factor exerts the greatest influence on the adhesion value, indicating that starch is the main component of forming adhesive strength. Increased adhesion occurs due to tapioca starch's ability to form a continuous and flexible polymer film on the surface of the substrate. The film is composed of a network of amylose and amylopectin, where amylose contributes to an increase in cohesive strength, while amylopectin provides elastic properties that increase contact between surfaces. CMC EFB acts as a structural reinforcer through the interaction of hydrogen bonds with starch thereby improving the stability of the adhesive film matrix. Watcharakitti et al. (2023) states that modified starch is able to form stable polymer tissues and contributes to improved bioadhesive adhesion properties. Arias et al. (2021) It also emphasizes that polysaccharide-based bioadhesives have good adhesion properties and are more environmentally friendly than synthetic adhesives. When compared to the research of Susilawati & Rahmaniar (2018) which used starch as a single filler, the results of this study showed an increase in adhesion stability. This indicates that the addition of CMC EFB provides a strengthening of the film structure so as to improve the overall adhesion performance.

Table 5. Duncan's Spacing Test Results Adhesive Test

C	P			
	P1	**P2**	**P3**	**Average P**
C1	1	3	3	2,3
C2	1	1,5	4,5	2,3
C3	2,5	5	2,5	3,3
Average C	1,5	3,2	3,3	

Based on the results of the Duncan test in Table 5, there were differences in adhesion values among the treatment combinations. The highest adhesion value was obtained in the C3P3 treatment combination, indicating that the increase in tapioca starch concentration together with CMC from EFB produced a synergistic effect in improving adhesion strength. This indicates that the C3P3 formulation was the optimal treatment for producing the highest adhesion strength compared to the other treatments. The increase in adhesion is related to the formation of a stronger, more flexible, and more stable polymer film network on tshe substrate surface.

Glue Dry Time Test

Dry-time testing is carried out to determine the speed of the glue in achieving perfect adhesion conditions. The assessment was carried out using the scoring method.

Table 6. Results of Two Way ANOVA Biodegradable adhesive Dry Time Test

Sources of Diversity	SS	df	MS	F	Sig
C	5.444	2	2.722	9.800	.006
P	11.111	2	5.556	20.000	.001
C x P	8.556	4	2.139	7.700	.006
Total	161.000	18			

Based on test results *Two-Way ANOVA* in Table 6, shows that the concentration of CMC (C), the ratio of tapioca starch (P), and the interaction of the two (C × P) have a significant effect on the drying time ($p < 0.05$). This shows that the composition of the material plays an important role in controlling the drying rate of the adhesive system. The difference in drying time is due to the ability of starch and CMC to bind water through the formation of gel structures. The increased concentration of both components causes more water to be trapped in the gel matrix, so the evaporation process takes place more slowly. In addition, the formation of a film layer on the surface of the glue serves as a barrier to the diffusion of water into the environment. Zhang et al. (2024) states that biomass-based bioadhesives form a layer of polymer films that play a role in controlling the rate of water evaporation during the drying process. Compared to a single starch-based system, the results of this study showed a longer drying time, which indicates that CMC EFB increases the water retention capacity in the adhesive system.

Table 7. Duncan Combined Distance Test Results Dry Time Test

C	P			
	P1	**P2**	**P3**	**Average P**
C1	4	2	2,5	2,8
C2	2,5	1	2,5	2
C3	5	3,5	1,5	3,3
Average C	3,8	2,2	2,2	

The results of the Duncan test showed that there was a difference in drying time for each combination of treatments. The most optimal drying time (most suitable for the drying characteristics of biodegradable adhesive) was obtained in the C1P2 treatment with the fastest drying time, while the longest drying time was found in the treatment with higher starch content, especially C3P3. This suggests that increased concentrations of tapioca starch tend to slow down the drying process due to the increased ability of the system to bind water and form a denser gel structure. Thus, starch and CMC formulations have an important role in regulating the balance between the adhesive strength and drying speed of biodegradable adhesive.

CONCLUSION

Based on the results of this study, variations in the concentration of CMC from EFB and the ratio of tapioca starch significantly affected the physical characteristics of biodegradable adhesive, particularly viscosity, adhesion, and drying time. The C3P3 treatment produced the highest viscosity and adhesion values, indicating that the combination of 3% CMC and 18 g tapioca starch was the optimal formulation for improving adhesive performance. Meanwhile, the pH value remained stable in all treatments and was not affected by variations in formulation. Overall, the combination of EFB-derived CMC and tapioca starch has strong potential to be developed as an environmentally friendly biomass-based adhesive alternative to synthetic adhesives.

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