HYDROGEN
JURNAL KEPENDIDIKAN KIMIA
https://e-journal.undikma.ac.id/index.php/hydrogen/index
December 2024. 12(6)
e-ISSN: 2656-3061
p-ISSN: 2338-6487
pp. 1218-1229

Development and Characterization of Bioplastics from Straw and Rice Husk for: Effect of Addition of Glycerin, CMC, and TiO_2

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Article History
Received: 06-12-2024
Revised: 19-12-2024
Published: 31-12-2024

Keywords: bioplastic, rice straw, rice husk, glycerin, carboxymethyl cellulose (CMC), titanium dioxide (TiO_2)

Abstract
The development of environmentally friendly materials is crucial to mitigate the environmental impact of conventional plastics. This study focuses on developing bioplastics using rice straw and rice husks, combined with glycerine, carboxymethyl cellulose (CMC), and titanium dioxide (TiO_2). Glycerine acts as a plasticizer to enhance flexibility, CMC functions as a matrix binder, and TiO_2 serves as a reinforcing agent to improve mechanical strength. Bioplastics were produced using the casting method with specific proportions of these materials. Characterization tests included scanning electron microscopy (SEM) for morphology, biodegradability tests, water absorption analysis, and tensile strength measurements. Results revealed that the combination of rice straw and rice husks produced bioplastics with varied morphologies. TiO_2 enhanced mechanical strength and material homogeneity, though its distribution requires further optimization. Glycerine significantly increased flexibility, while CMC improved matrix cohesion. The novelty of this research lies in the integration of agro-industrial waste—rice straw and rice husks—with TiO_2, glycerine, and CMC, creating bioplastics that balance biodegradability, mechanical properties, and flexibility. This innovative approach demonstrates the potential of utilizing agricultural byproducts to produce sustainable alternatives to conventional plastics, offering customizable properties for diverse applications.

How to Cite: Hardi, G., Toruan, S., Dewi, B., Nurjanah, S., Nurohmat, N., Rifai, B., & Hidayat, A. (2024). Development and Characterization of Bioplastics from Straw and Rice Husk for: Effect of Addition of Glycerin, CMC, and TiO_2. Hydrogen: Jurnal Kependidikan Kimia, 12(6), 1217-1229. doi:https://doi.org/10.33394/hjkk.v12i6.13821

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INTRODUCTION

Bioplastic is a plastic material that can be naturally decomposed by microorganisms, making it an environmentally friendly alternative to traditional plastics. As awareness of plastic pollution grows, bioplastics are gaining traction in various industries, offering a sustainable solution that reduces the ecological footprint of packaging and products. it is environmentally friendly compared to conventional fossil fuel-based plastics. Bioplastics can be made from natural materials containing cellulose, starch, or protein as the main ingredients (Zhao, et al., 2023).

The demand for bioplastics is increasing along with attention to the negative impacts of conventional plastics, such as environmental pollution and marine ecosystems (Negrete & Guerrero, 2024). Straw and rice husks are abundant agricultural wastes that are usually burned or disposed of, which can pollute the environment. Both of these materials are rich in cellulose, which can be used to make bioplastics. The use of this waste will add economic value and reduce pollution (Goodman, 2020).

Glycerine is often used as a plasticizer in the manufacture of bioplastics to increase flexibility and reduce brittleness. Glycerine helps improve the mechanical properties of bioplastics by reducing stiffness and increasing elasticity. Different concentrations of glycerine can have a significant effect on the mechanical and physical properties of the resulting bioplastics (Benitez, et al., 2024).

CMC is a water-soluble cellulose-derived polymer, often used to increase the strength and stability of bioplastics. The addition of CMC can help bind molecules in bioplastics, providing higher resistance to moisture. Research on the use of CMC as an additive in straw and rice husk-based bioplastics provides an opportunity to optimize the mechanical and thermal properties of bioplastics (Han, et al., 2024). TiO_2 is an inorganic material commonly used as a filler in bioplastic composites to improve optical, mechanical, and UV resistance properties. The addition of TiO_2 to bioplastics can help increase tensile strength and resistance to environmental degradation. The effect of TiO_2 on straw and rice husk-based bioplastics still requires further research to understand its effect on properties such as transparency, strength, and durability (Cazan, Enesca, & Andronic, 2021).

The widespread use of conventional plastics has led to significant environmental challenges, including persistent plastic waste accumulation and reliance on non-renewable resources. Additionally, large quantities of agricultural waste, such as rice straw and rice husks, remain underutilized, contributing to environmental pollution and waste management issues. These challenges underscore the urgent need for innovative solutions that address both plastic and agricultural waste problems. Despite advances in bioplastic development, achieving an optimal balance between biodegradability, mechanical strength, and flexibility remains a challenge. Furthermore, there is limited research on the use of rice straw and rice husks, combined with functional additives like glycerol, CMC, and TiO_2, in bioplastic formulations. This study addresses these gaps by developing and characterizing bioplastics derived from rice straw and rice husks, providing an eco-friendly alternative to conventional plastics while utilizing agricultural waste effectively.

METHOD

Materials & Equipment

Straw and rice husk utilized as raw materials were sourced from Indramayu agricultural products. The straw was washed, dried, pulverized, and sieved to a mesh size of 80. The chemicals utilized in the process include NaOH, Hydrogen Peroxide, NaCl, Glycerol, CMC, and TiO2. This study's instruments include a blender, 80 mesh sieve, hotplate, drying oven, magnetic stirrer, and a 25 cm x 15 cm mold.

Making Hemicellulose from Rice Straw and Rice Husk

Three steps are involved in turning straw and husk into bioplastic: preparation, delignification, and moulding (Wang, Asano, Kudo, & Hayashi, 2020). To help in delignification, the straw is ground in a blender until it reaches an 80 mesh size. An alkali delignification technique is used to process the smooth straw and husk. Using a 15% NaOH solution (30 grams of NaOH dissolved in 200 mL of distilled water), 25 grams of straw or husk are hydrolysed for 60 minutes at 100°C. The cellulose solid is dried in an oven at 50°C for three hours (Ashgar, et al., 2015). To manufacture bioplastic, soak the cellulose in 10% acetic acid (200 mL) and 20 grams of NaCl at 60°C for 60 minutes to smooth and clean the fibres (Mohammed, et al., 2022). The cellulose is then filtered, washed with clean distilled water, and dried again in the oven. The next stage is bleaching cellulose using a 7% H_2O_2 solution at 60°C for 60 minutes. After filtering, the cellulose is cleaned with pure distilled water and oven-dried once again. The following step involves bleaching cellulose for 60 minutes at 60°C with a 7% $H2O_2$ solution (Walawska, Olak-Kucharczyk, Kaczmarek, & H. Kudzin, 2024).

Making Bioplastic from Straw and Rice Husk

Table 1 shows four types of bioplastic samples made from a combination of main materials and additional materials. The main materials used are rice straw and rice husk, which are agricultural waste with high fibre content. This waste acts as a basic component to create an environmentally friendly bioplastic structure.

Table 1. Bioplastic Sample

No.	Sample Code	Main Ingredient	Additional Materials	Information
1	JP-CMC-GLY	Rice straw	CMC, Glycerol	Combination of rice straw with CMC and glycerol
2	SP-CMC-GLY	Rice Husk	CMC, Glycerol	Combination of rice husk with CMC and glycerol
3	JP-CMC-TiO2-GLY	Rice straw	CMC, TiO_2, Glycerol	Combination of rice straw with CMC, TiO_2, and glycerol
4	SP-CMC-TiO2-GLY	Rice Husk	CMC, TiO_2, Glycerol	Combination of rice husk with CMC, TiO_2, and glycerol

Each sample is equipped with additional materials such as CMC (Carboxymethyl Cellulose), glycerol, and TiO_2 (Titanium Dioxide). CMC is used as a binder to strengthen the polymer matrix, while glycerol functions as a plasticizer to increase the flexibility of the bioplastic product (Han, et al., Effects of carboxymethyl cellulose concentration on mechanical, viscoelastic properties, and thermal properties of starch/plant fiber foaming tableware materials with foam structure, 2024). The addition of TiO_2 to several samples aims to increase mechanical strength and resistance to UV light, making bioplastic more durable and stable (Amin, Chowdhury, & Kowser, 2019).

In this study, bioplastic was made with a ratio of 1:1:1. The solution was stirred and heated for 10-15 minutes at a temperature of 90°C until it thickened. This bioplastic solution is then moulded to a size of 25 cm x 15 cm with a thickness of 0.2 cm and dried at a temperature of 60°C until a bioplastic sheet is formed (Berghuis, et al., 2022).

Characterization and Test Parameters

Biodegradability

Biodegradability is a natural process that decomposes organic chemicals in the environment into simpler compounds, which can then be remineralized and circulated back through the cycle of elements such as carbon, nitrogen, and sulphur.

Bioplastics are made from basic materials such as starch and cellulose. Starch is a polymer that is abundant in plants, especially in straw and rice husks. From these plants, starch can be obtained in the form of powder or granules. Starch is often used as a raw material in film production due to fluctuations in the availability of conventional film resins. In biodegradable plastics, the decomposition process can take place quickly with the help of bacteria or naturally.

Biodegradability indicates the ability of bioplastics to decompose when exposed to microorganisms. Biodegradability tests were carried out using EM4. After being soaked in EM4 for 7 days, bioplastics will undergo a degradation process, and the amount of bioplastic that decomposes can be calculated using the following equation (Ngatin, Sihombing, Al-Ghifari, & Maulana, 2022):

$$\%Degraded = \frac{W0 - W1}{W1} x\ 100\%$$

Water Absorption Test

The water absorption test, or swelling percentage, was carried out based on the AOAC method (1983). The test process involves cutting plastic with a diameter of 60 mm, followed by weighing the sample. Samples measuring 60 mm x 60 mm were weighed to obtain the initial weight and then soaked in distilled water for 24 hours. After soaking, the sample was immediately weighed to obtain the final weight. The comparison of the initial and final weights is used to calculate the water absorption of bioplastics with the following formula:

$$Water\ Absorption\ Capacity\ (\%) = \frac{W1 - W0}{W0} x\ 100\%$$

Morphology and Tensile Test

A scanning electron microscope (SEM) test is performed to examine the surface morphology of bioplastic samples at a microscopic level. This test evaluates surface structure, such as the existence of pores, fissures, or uneven material distribution.

Tensile tests are used to evaluate the mechanical properties of bioplastics, namely the material's capacity to bear tensile forces before deforming or breaking. This test yields crucial results such as maximum tensile strength and elongation to break.

RESULTS AND DISCUSSION

The Process of Delignification

The first stage of delignification, which raises the amount of cellulose in straw, involves the use of alkali solvents. In order to make hemicellulose more accessible, this procedure seeks to open the lignocellulose structure (Bajpai, 2018). Figure 1 shows the changes in the state of the straw and husk following the NaOH delignification procedure.

(a) (b)

Figure 1. Straw (a) and rice husk (b) after treatment using 15% NaOH.

The hemicellulose structure can be broken down and dissolved by using the strong base NaOH. The alpha-cellulose content rises as the amount of hemicellulose dissolved increases with the amount of NaOH used (Prasetia, Deviana, Damayanti, Cahyadi, & Agus Gelge, 2018). Prior to alkali treatment, the straw had a coarse texture and vibrant color, as seen in Figure 1. Following treatment, the color darkened and the roughness smoothed out. This alteration results from the hydrolysis of lignocellulose during ethanol solution treatment, which creates fibers with a high cellulose content and gives the straw a darker appearance (Michele, et al., 2015).

(a) (b)

Figure 2. Rice Straw hemicellulose (a), Rice Husk hemicellulose (b)

Analysis of Hemicellulose

To compare the amounts of hemicellulose and lignin before and after the delignification process, the Chesson-Datta test was used. The Chesson-Datta test revealed that the hemicellulose content of rice husk was 10.09 grams and that of rice straw was 10.21 grams.

$$Percentage\ of\ Rice\ Straw\ Hemicellulose = \left(\frac{10,21}{25}\right) x\ 100\% = 41\%$$

$$Rice\ Straw\ Hemicellulose\ Percentage = \left(\frac{10,09}{25}\right) x\ 100\% = 40\%$$

The hemicellulose According to the statistics, the hemicellulose concentration of rice husk and straw ranges from 40% to 41%. The data's consistency suggests that the delignification process used is effective (Yang, Zhao, Liang, & Zhu, 2020).

Biodegradability

Depending on their formulation and mechanical characteristics, the bioplastic samples displayed various physical changes during the five-day disintegration period. All samples were undamaged and intact on the first day, displaying their original colour and silky texture. On the second day, the liquid media displayed indications of material interaction with the surroundings, and certain samples started to exhibit colour and texture changes along with fissures in certain places.

(a) Sampel 1 – Sampel 3 – Sampel 4 – Sampel 2

(b) Sampel 1 – Sampel 3 – Sampel 4 – Sampel 2

(c) Sampel 1 – Sampel 3 – Sampel 4 – Sampel 2

(d) Sampel 1 – Sampel 3 – Sampel 4 – Sampel 2

(e) Sampel 1 – Sampel 3 – Sampel 4 – Sampel 2

Figure 3. Biodegradability Test Day 1 (a), Day 2 (b), Day 3 (c), Day 4 (d), Day 5 (e).

Depending on their formulation and mechanical characteristics, the bioplastic samples displayed various physical changes during the five-day disintegration period. All samples were undamaged and intact on the first day, displaying their original colour and silky texture. On the second day, the liquid media displayed indications of material interaction with the surroundings, and certain samples started to exhibit colour and texture changes along with fissures in certain places.

As the third day progressed, degeneration became more noticeable. Some samples softened significantly, their structure became more unstable, and certain components began to dissolve into the liquid media. On the fourth day, most of the samples lost their original structure, transforming into minute fragments or paste with varied degrees of disintegration. Samples 3 and 4 remained more stable than the others; however, samples 1 and 2 sustained more serious damage.

All the samples had nearly entirely broken down into paste or tiny particles by the fifth day, and the liquid medium seemed to be saturated with dissolved materials. Sample 4 showed comparatively superior resistance, whilst Sample 2 underwent the most degradation. According to these findings, bioplastics have the potential to be eco-friendly materials with further-optimized biodegradation capabilities. Additional tests can be performed to assess the residues created during the breakdown process to further the investigation. According to the results of the biodegradability test, commercial bioplastics took more than 60 days to fully decompose, whereas bioplastics made from rice and rice husks + CMC + glycerine were able to do so in 3 days. Bioplastics that also contained TiO_2 were also able to do so in 5 days (Ali, Isha, & Chang, 2023).

Water Absorption Test

Water absorption is one criterion used to evaluate the quality of bioplastics made from rice husks and straw. Because it impacts how long the bioplastic will remain in storage, the higher the water absorption, the worse the quality. Bioplastic materials used in this investigation were soaked at ambient temperature (28–30°C).

Table 1. Swelling Index Analysis Results

Sample	W0 (gr)	W1 (gr)	A(%)
SP + CMC + GLY	1,82	9,48	423%
JP + CMC + GLY	1,49	7,94	432%
SP + CMC + GLY + TiO_2	0,86	4,43	415%
JP + CMC + GLY + TiO_2	1,35	5,67	320%

The water absorption capacity of all samples was quite high, with increases ranging from 320% to 432%. In comparison to the samples without TiO_2, the SP + CMC + GLY + TiO_2 and JP + CMC + GLY + TiO_2 samples were able to decrease water absorption when TiO_2 was added. This bioplastic's high water absorption capacity means that it can absorb moisture more readily, which may have an impact on its practical uses and longevity, particularly when stored (Gerbie, Alemayehu, Belay, & Ewunetu, 2024).

SEM Results

The Scanning Electron Microscope (SEM) image of the CMC- and glycerol-based bioplastic above shows that the material's surface is smooth and wavy, and it appears to be rather uniform. The absence of substantial cracks or big pores suggests that the material's primary constituents—glycerol and CMC—are cohesive. This demonstrates that CMC serves as the primary matrix that gives the bioplastic its structure, while glycerol acts as a plasticizer that aids in enhancing the material's flexibility.

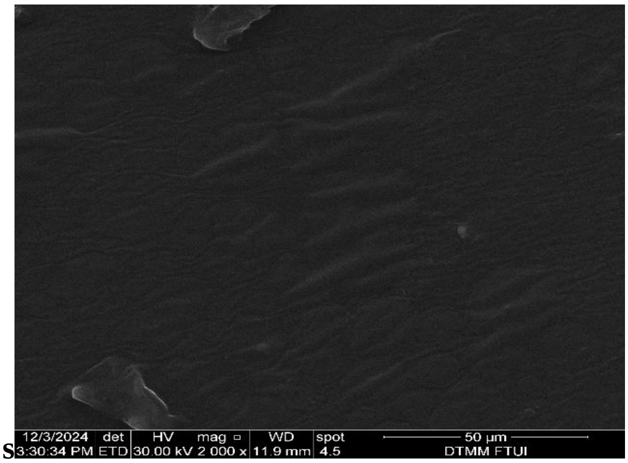
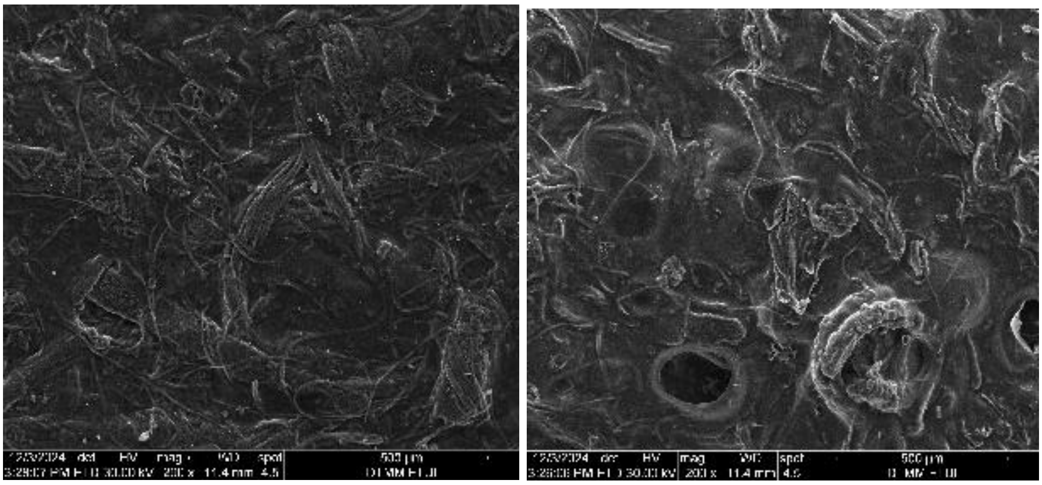


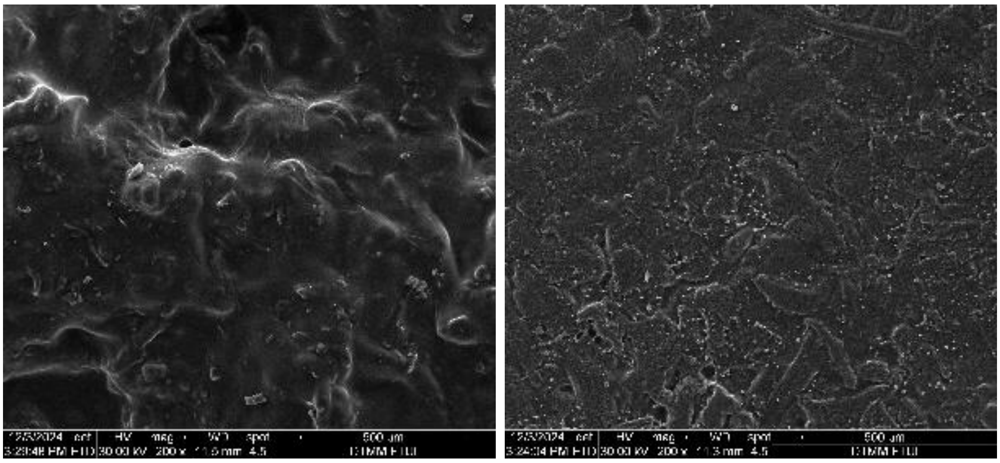
Figure 4. SEM results of CMC + Glycerol bioplastic



(a) **(b)**

Figure 5. SEM Results of Bioplastics (a) Rice Straw + CMC + Glycerol and (b) Rice Straw + CMC + TiO_2 + Glycerol

The surface structure of the bioplastic formed from rice straw, CMC, and glycerol is not uniform, as shown by the results of Scanning Electron Microscopy (SEM) in figure 5(a). In the polymer matrix, the fibers of rice straw are irregularly scattered and stand out. This suggests that the filler material, rice straw, has not been evenly distributed throughout the CMC and glycerol mixture. Furthermore, a lack of contact and adherence between the filler and the polymer matrix is indicated by the existence of gaps or fissures between the fibers and the matrix (Ubaidah, Puad, Nor, Hamzah, & Azmi, 2024). Low compatibility between rice straw and matrix materials is often indicated by rough and uneven surfaces. This may have an impact on bioplastics' mechanical qualities, including their elasticity and tensile strength. It could be necessary to employ a compatibilizer to strengthen the link between the filler and the matrix in order to solve this issue. To produce bioplastics with superior mechanical and physical properties, the mixing procedure must also be enhanced to ensure a more even distribution of components (Qin, Soykeabkaew, Xiuyuan, & Peijs, 2024).



(a) (b)

Figure 5. SEM results of bioplastics (a) Rice Husk + CMC + Glycerol and (b) Rice Husk + CMC + TiO2 + Glycerol.

The surface of the bioplastic appears to be highly complicated, with rice straw fibers still visible in multiple places, according to Scanning Electron Microscopy (SEM) data on bioplastics synthesized from rice straw, CMC, TiO_2, and glycerol. This suggests that the fibers have not been completely distributed throughout the matrix. Large pores on the surface are a sign of voids, which may have an impact on the material's mechanical strength. This is probably the result of a less-than-ideal mixing procedure or variations in the compatibility of the CMC matrix enhanced with glycerol and TiO_2 and rice straw. This material combination demonstrates that even though rice straw, CMC, TiO_2, and glycerol interact, the production process for bioplastics still needs to be optimized in order to create a more uniform morphology, decrease holes, and improve material compatibility.

The surface morphology of bioplastics derived from rice husk, CMC, and glycerol is smoother than that of bioplastics derived from rice straw, according to SEM data. Even while there are still some spots with tiny particles and voids visible, the surface structure is generally more uniform. Better compatibility between rice husk as a filler and the CMC matrix and glycerol as a plasticizer is indicated by the more equal distribution of rice husk in the polymer matrix. However, the appearance of small pores at some spots may suggest that the material mixing is not properly optimal, affecting the bioplastic's mechanical qualities. This smooth surface may also indicate improved interaction between the rice husk filler and the matrix, resulting in greater adherence than rice straw bioplastic.

The SEM image of bioplastics created from rice husks, CMC, TiO_2, and glycerol above shows a more complicated surface than prior samples. The material's surface exhibits a more equal particle distribution, with small particles dispersed randomly throughout. These particles, most likely TiO_2, serve as a filler or reinforcing ingredient to enhance the mechanical properties of bioplastics. Rougher patches suggest uneven distribution of TiO_2 particles in the CMC-glycerol matrix, potentially affecting the material's strength and consistency. TiO_2 can improve UV resistance and mechanical qualities in bioplastics, but further research is needed to fully understand its impact on material strength and durability.

Tensile Test Results

The mechanical properties of bioplastics manufactured from straw and rice husks are affected by the quantity and kind of components used, such as rice straw, rice husks, CMC, TiO_2, and glycerol. The level of affinity between components is an essential aspect in determining bioplastics' mechanical characteristics. Affinity refers to the ability of molecules or atoms in a material to bind with one another. The greater the affinity between components, the more molecular bonds are created, resulting in superior mechanical properties (Negrete-Bolagay & Guerrero, 2024). Bioplastics' mechanical qualities include tensile strength and % elongation. Tensile strength determines the maximum stress that bioplastics can sustain before failing, whereas percentage elongation tells how far the bioplastic film can extend before and after being pulled. Thus, the compatibility of rice straw and rice husk materials with polymer matrices such as CMC and glycerol is critical for producing bioplastics with good mechanical performance (Nuriyah, Saroja, Ghufron, Razanata, & Rosid, 2018).

Tensile strength testing was carried out at the Balai Besar Kulit, Karet, dan Plastik Yogyakarta Laboratory utilizing test equipment that met ASTM D-638-02 standards. The test results include measurements of tensile strength, which is a material's ability to endure maximum stress before breaking, and % elongation, which describes the level of material elongation prior to damage. This data is used to assess the mechanical qualities of the bioplastics evaluated.

Table 2. Bioplastic Mechanical Test Results

Test/Name of Sample	**JP + CMC + GLY**	**SP + CMC + GLY**	**JP + CMC + GLY + TiO2**	**SP + CMC + GLY + TiO2**
Tensile Strength (N/mm2)	3	1,32	1,02	1,64
Elongation at Break %	18,07	30,70	27,70	43,48

The tensile test results and the percentage of elongation at break for a number of bioplastic samples are displayed in Table 2. The JP + CMC + GLY sample had an elongation at break of 18.07% and a tensile strength of 3 N/mm². This demonstrates that this material is strong but less elastic because it has the highest tensile strength of any sample tested and the lowest elongation at break. The SP + CMC + GLY sample had an elongation at break of 30.70% and a tensile strength of 1.32 N/mm². This material is more elastic but less strong than the last sample since it has a higher elongation but a lower tensile strength.

The JP + CMC + GLY + TiO_2 sample had an elongation at break of 27.70% and a tensile strength of 1.02 N/mm². This demonstrates that while TiO2 has superior elongation, it lowers tensile strength when compared to the JP + CMC + GLY sample. Lastly, the SP + CMC + GLY + TiO_2 sample exhibits an elongation at break of 43.48% and a tensile strength of 1.64 N/mm². In comparison to the SP + CMC + GLY sample, this sample exhibits an improvement in both tensile strength and elongation, suggesting that the addition of TiO_2 improves both characteristics and makes the material more elastic and robust.

CONCLUSION

Glycerine, CMC, and TiO_2 were successfully utilized as additives to create bioplastics from rice husk and straw. The bioplastics exhibited good biodegradability, with certain formulations degrading entirely within three to five days. TiO_2 significantly enhanced mechanical strength, while CMC and glycerin improved the material's flexibility. The novelty of this study lies in the use of rice husk and straw as agro-industrial waste materials, combined with TiO_2, glycerin, and CMC, to develop bioplastics with tunable biodegradability and mechanical properties, providing an innovative approach to sustainable materials development.

Despite these promising results, various technical challenges were observed, including uneven fiber distribution and suboptimal compatibility between the filler (straw/rice husk) and the polymer matrix. These limitations highlight the need for a compatibilizer and a more efficient mixing technique to improve the homogeneity and mechanical properties of the bioplastics.

For future research, it is recommended to explore alternative compatibilizers and advanced mixing methods to enhance the material's uniformity and performance. Further investigation into the scalability of the production process and the environmental impact of large-scale bioplastic manufacturing is also necessary. Additionally, testing the bioplastics under varying environmental conditions (e.g., humidity, temperature) and evaluating their performance in specific applications will provide valuable insights for practical implementation.

ACKNOWLEDGEMENTS

The authors would like to express their deepest appreciation to Politeknik Negeri Indramayu (Polindra) for their assistance and financing supplied through the Polindra Penelitian, Pengabdian kepada Masyarakat, dan Pengembangan Unit (P3M), which allowed this research to be completed effectively. Without Polindra's aid, particularly through P3M, this research could not have been carried out as effectively. We also want to thank everyone who offered ideas, time, and technical assistance throughout the research process. Hopefully, the findings of this research will be valuable in the advancement of existing science and practice.

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