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Utilization of Rice Husk-derived Microsilica as a Reinforcing Agent in MOCAF Starch-based Bioplastics: Enhancing Mechanical Strength, Barrier Properties and Thermal Stability

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Abstract. *Starch-based bioplastics are increasingly recognized as eco-friendly substitutes for conventional synthetic plastics owing to their renewable origins and biodegradability. However, their poor mechanical properties and high affinity for water limit their practical applications. In this study, microsilica derived from rice husks was incorporated as a reinforcing agent to enhance the performance of bioplastics based on a modified cassava flour (MOCAF) starch matrix. This study aimed to determine the characteristics of bioplastics with a MOCAF starch matrix and microsilica added at various concentrations. Bioplastics were prepared with the solution casting method involving starch extraction, gelatinization, addition of microsilica at varying concentrations (0%, 1%, 2%, 3%, 4% and 5%), incorporation of a plasticizer (glycerol) and subsequent molding. The results demonstrated that microsilica addition significantly affected the physicochemical properties of the bioplastics. Tensile strength increased with microsilica content, reaching a maximum of 3.73 MPa at 5 wt%, followed by decreases at higher concentrations. Conversely, elongation at break decreased with increasing microsilica content, indicating reduced flexibility. Water absorption also decreased, indicating increased water resistance in the bioplastics. Fourier Transform Infrared (FTIR) analysis confirmed the interaction between microsilica and the starch matrix, while thermogravimetric analysis (TGA) revealed increased thermal stability after the addition of microsilica. Overall, the incorporation of microsilica effectively improved the mechanical, thermal and barrier properties of the MOCAF-based bioplastics, highlighting their potential for developing more durable and environmentally friendly materials.*

Keywords: *bioplastics; elongation; microsilica; MOCAF starch; tensile strength.*

Type of the Paper: Regular Article.

1. Introduction

The use of starch bioplastics as an alternative to non-degradable plastics has become widespread across various industrial sectors, reducing CO_2 emissions and the accumulation of conventional plastic waste in the environment [1]. Biodegradable plastics are typically derived from renewable natural resources, including plant- and animal-based materials such as starch,

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cellulose, protein, chitosan and hemicellulose, which are abundant and environmentally sustainable.

Among these materials, starch has been extensively investigated for bioplastic production due to its wide availability, low cost, biodegradability and eco-friendly characteristics. Various starches have been explored, including tapioca starch [2], taro tuber starch [3], yam starch [4], sweet potato starch [5,6], corn starch [7–9], casava starch [10], banana peel starch [11,12] and MOCAF starch [13]. These starches demonstrate significant potential as raw materials for the development of biodegradable bioplastics.

MOCAF, or modified cassava flour, is produced through the fermentation of cassava using lactic acid bacteria, resulting in improved physicochemical properties compared to native cassava flour. In addition to its application as a food substitute for wheat, rice and glutinous rice, MOCAF has also attracted attention as a promising biopolymer source for bioplastic production [14]. Previous studies have reported that bioplastics derived from MOCAF exhibited moderate mechanical performance. For instance, Rezekinta et al. [13] reported a tensile strength of 3.26 MPa, while Syamani et al. [15] obtained a tensile strength of 1.35 MPa without reinforcement and with a plasticizer content of 0.3% by weight of dry starch.

Despite these advantages, starch-based bioplastics generally suffer from inherent limitations, particularly high water sensitivity and relatively poor mechanical properties compared to conventional synthetic polymers [8,16,17]. To address these drawbacks, reinforcement strategies involving the incorporation of fillers, either organic or inorganic, have been widely applied. Among these, silica-based fillers, especially nanosilica, have been extensively studied and shown to significantly enhance the mechanical strength, thermal stability and barrier properties of biodegradable polymer systems [18–23]. The majority of existing studies have focused on nanosilica, which, while effective, is associated with high production cost and complex synthesis routes [24]. By contrast, microsilica, particularly that which is derived from rice husks (RH), an abundant agricultural byproduct, remains relatively underexplored, especially in MOCAF-based bioplastic systems. Rice husks generally contain 15–25 wt% silica [25], making them a cost-effective and sustainable precursor for microsilica synthesis. However, the application of silica at the microscale remains relatively underexplored, particularly in MOCAF-based bioplastic systems. This represents a critical research gap, especially considering that rice husk-derived silica, an abundant agricultural waste, has yet to be optimally utilized in biocomposite applications at the macro scale. Compared to nanosilica, microsilica offers several advantages, including lower production cost, simpler processing methods, reduced energy consumption, better feasibility for large-scale industrial applications and sustainable development potential. Unlike the extensively researched nanosilica reinforcement systems, the application of rice husk-derived microsilica in

MOCAF starch-based bioplastics remains extremely limited. Furthermore, studies evaluating the effectiveness of microsilica as a low-cost and scalable reinforcing agent are still scarce. Therefore, this study investigated the effect of 4 µm rice husk-derived microsilica on the mechanical, barrier and thermal properties of MOCAF starch-based bioplastics. The findings are expected to provide insights into the potential of microsilica as an effective and economically viable reinforcing agent for sustainable bioplastic development.

2. Materials and Methods

2.1. Materials

The main material used in this study was MOCAF produced by Rumah MOCAF Indonesia, where the cassava used was of the singkur and damar varieties. Additionally, the research utilized 4 µm microsilica from IR-42 rice husks, with a silica content of 95.759% [26]. Supporting materials included distilled water and glycerol, which served as a plasticizer.

2.2. Methods

2.2.1. MOCAF starch extraction

MOCAF starch extraction was carried out in several stages: mixing, filtering, centrifugation, drying, grinding and sieving. The mixing process involved adding water to MOCAF at a ratio of 1:10 (w/v) and blending for 10 seconds using a blender (Philips Type HR 2057). The MOCAF suspension was filtered using a 200-mesh sieve. The liquid that passed through the sieve was centrifuged (Corona 80-2) at 3,000 rpm for 5 minutes. The supernatant was then separated, leaving only the solid residue, which was then dried. This process was carried out using a dehydrator (Cosori) at 50°C for 5 hours. The dried solid was then ground and sieved using a 200-mesh sieve to obtain purified MOCAF starch.

2.2.2. Production of biocomposites with MOCAF starch matrix

Seven grams of MOCAF starch were dissolved in 70 mL of distilled water. The mixture was stirred at 400 rpm using a hot-plate magnetic stirrer (model SH-03) for 5 minutes. Then, microsilica was added according to the treatments with variations in concentration, ranging from 0 wt% to 5 wt%. It was stirred again for another 5 minutes. The mixture was heated until the temperature reached 85°C. The resulting gel was molded in a 15 × 15 cm glass mold, left at room temperature for 2 hours, and then dried in an oven (Universal Oven Memmert UN-55) for 36 hours (modified from Zuwanna et al. [27]). The resulting bioplastics were carefully removed and let sit at room temperature until it reached a constant weight. Then, measurements of tensile strength, elongation at break (Universal Testing Machine (UTM) Tensilon RTH-2410 and RTI-1310 brands), water absorption and thermal stability (thermogravimetric analysis; Perkin Elemer, Type

TGA 4000) were taken. Additionally, analysis of the functional groups of the bioplastics was carried out using Fourier Transform Infrared Spectroscopy (Shimadzu FTIR Tracer-100).

2.3. Observations

2.3.1. Tensile strength

The tensile strength of the biocomposite samples was tested using a Universal Testing Machine (UTM) (Model RTI-1310) at room temperature (23 ± 2°C), relative humidity (RH) of 50%, speed of 3 mm/minute and force of 10 kN. The samples were prepared according to the requirements and clamped at both ends of the panel [28]. The data derived from the test were subsequently calculated using Eq. (1).

$$\tau\ (\text{MPa}) = \frac{F_{\max}\ (\text{N})}{A\ (\text{mm}^2)} \quad (1)$$

2.3.2. Elongation at break

As with the tensile strength measurement, the elongation-at-break test was conducted using a Universal Testing Machine (UTM) (Model RTI-1310). The dried film sample was cut into strips with dimensions of 10 cm × 2.5 cm. Each end of the sample strips was secured in the grips of the testing machine, and the instrument was operated to stretch the sample strips until failure occurred [28]. The elongation percentage was determined using Eq. (2).

$$\%E = \frac{\Delta L}{L_0} \quad (2)$$

where

$\%E$ = Elongation (%)
ΔL = Increase in specimen length (mm)
L_0 = Initial specimen length (mm).

2.3.3. Water absorption

The sample, measuring 2 cm × 2 cm, was weighed using an analytical balance (W_0). It was immersed in a 250 mL beaker pre-filled with 30 mL of distilled water for 3 minutes. Then, it was removed from the water and allowed to dry using tissue paper [29]. Finally, it was reweighed (W_t). The water absorption value was calculated using Eq. (3).

$$\text{Water Absorption}\ (\%) = \frac{W_t - W_0}{W_0} \times 100\% \quad (3)$$

where

W_t = final weight (g)
W_0 = initial weight (g).

2.3.4. Fourier Transform Infrared (FTIR) Spectroscopy

FTIR spectroscopy was conducted to analyze the chemical functional groups of the bioplastics. This test is capable of detecting molecular interactions with electromagnetic radiation within the wavelength range of 0.75–1,000 µm or at wavenumbers of 13,000–10 cm^{-1}. In this test,

the sample was cut into strips measuring 1 cm × 1 cm. Testing was conducted at a wavenumber range of 600–4,000 cm^{-1} at room temperature.

2.3.5. Thermogravimetric analysis (TGA)

Thermogravimetric analysis (TGA) aimed to determine the thermal resistance of the film. It was conducted using a PerkinElmer TGA 4000 instrument at temperatures ranging from 30°C to 900°C, with a heating rate of 20°C per minute. The samples used weighed between 10,150 mg and 11,637 mg.

3. Results and Discussion

3.1. Mechanical Properties

The mechanical properties of the bioplastics as a function of microsilica content are presented in Table 1. The tensile strength (TS) increased significantly with increasing microsilica concentration from 0 wt% to 4 wt%, rising from 1.69 MPa to a maximum of 3.73 MPa, before decreasing to 3.26 MPa at 5 wt%.

Table 1. Mechanical properties of bioplastics with microsilica reinforcement and MOCAF starch matrix.

Content of microsilica (%)	Tensile strength (MPa)	Elongation (%GL)	Water absorption (%)
A (Microsilica 0%)	1.69 ± 0.55	10.36 ± 0.37	27.5 ± 0.76
B (Microsilica 1%)	2.33 ± 0.42	9.02 ± 0.29	26.9 ± 0.47
C (Microsilica 2%)	2.89 ± 0.10	8.72 ± 0.58	26.5 ± 0.26
D (Microsilica 3%)	3.51 ± 0.15	7.85 ± 0.22	24.9 ± 0.34
E (Microsilica 4%)	3.73 ± 0.25	5.83 ± 0.19	23.4 ± 0.28
F (Microsilica 5%)	3.26 ± 0.34	5.12 ± 0.35	22.9 ± 0.23

This trend indicates that microsilica acts as an effective reinforcing agent within the MOCAF starch matrix. The improvement in tensile strength at low-to-moderate filler loadings can be attributed to enhanced interfacial interactions between the hydroxyl groups (–OH) of starch and the silanol groups (Si–OH) of silica, which promote stress transfer efficiency across the matrix–filler interface. The increase in tensile strength is closely related to the intermolecular interaction between silica and MOCAF starch in bioplastics [30]. As a result, the polymer network becomes more rigid and resistant to deformation. However, at higher microsilica content (5 wt%), the tensile strength decreased, which is likely due to particle agglomeration. Xie et al. [31] stated that an increase in microsilica content can lead to a decrease in tensile strength due to agglomeration and insufficient interfacial interaction. Excessive filler loading can lead to poor dispersion and the formation of stress concentration points, thereby weakening the composite structure. In addition, larger particle size and no uniform distribution of microsilica may reduce effective interfacial bonding, consistent with previous findings that highlight the detrimental effects of filler agglomeration on mechanical performance [32].

Simultaneously, elongation at break decreased progressively from 10.36% to 5.12% with increasing microsilica content, indicating a transition from a ductile to a more brittle behavior. This reduction in flexibility is commonly observed in reinforced biocomposites and reflects the restriction of polymer chain mobility due to filler incorporation.

3.2. Water absorption

Water absorption results (Table 1) showed a decreasing trend with increasing microsilica content from 27.5% (0 wt%) to 22.9% (5 wt%). The reduction in water uptake can be attributed to both physical and chemical effects. Physically, microsilica particles occupy void spaces within the polymer matrix, thereby reducing free volume and limiting water diffusion pathways. Chemically, interactions between the silica and hydroxyl groups of starch reduce the availability of hydrophilic sites for water binding.

This dual mechanism leads to improved water resistance, which is critical for the practical applications of starch-based bioplastics. Furthermore, the improved barrier properties suggest a denser and more compact microstructure, consistent with the observed increase in tensile strength. Similar trends have been reported in silica-reinforced starch systems, where filler dispersion and interfacial bonding play key roles in reducing hydrophilicity. Microsilica functions as a filler that fills the pores or gaps between polymer chains, thereby inhibiting water diffusion into the bioplastics. This finding is supported by Ismail and Zaaba [30], who stated that adding 2 wt% silica reduces water absorption by up to 35%. Furthermore, the impact of silica powder addition on water absorption hinges on particle size, dispersion and interfacial interaction [33].

3.3. Thermogravimetric analysis (TGA)

The thermal stability of the bioplastics was evaluated using TGA, as shown in Fig. 1. The degradation profile revealed that the incorporation of 4 wt% microsilica significantly altered the thermal behavior of the material. The bioplastic without microsilica exhibited as rapid a mass loss, starting from approximately 280°C, whereas the sample containing 4 wt% microsilica showed delayed degradation, with the main decomposition occurring at a higher temperature between 290°C and 310°C. These shift indicates enhanced thermal stability due to the presence of microsilica.

Significant peaks in mass loss were observed for both types of samples in the temperature range of 300–380°C. This range is associated with the breakdown of the main polymer structure in starch during the second stage of the bioplastic's thermal degradation process. This aligns with the statement of Syafri et al. [34] that mass loss in the second stage is associated with the formation of ethers and unsaturated structures during the thermal condensation of hydroxyl groups from starch chains. Notably, the microsilica-reinforced samples (4 wt%) exhibited a slower degradation

rate in this region, indicating that microsilica acts as a thermal barrier, reducing heat transfer and slowing the release of volatile degradation products.

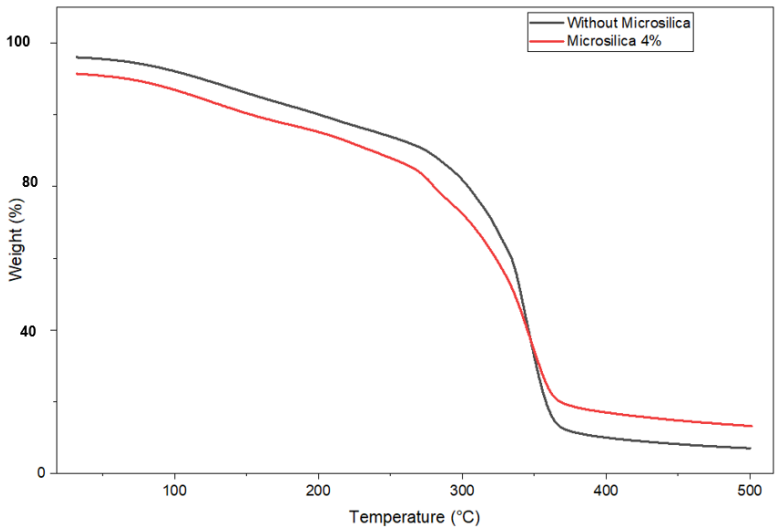

Fig. 1. TGA of MOCAF starch-based bioplastics without microsilica and with 4% microsilica.

At higher temperatures ranging from 500°C to 600°C, the residual mass of the microsilica-containing bioplastic (2.0–2.5%) was higher than that of the control sample (± 1.05%), confirming the presence of a thermally stable inorganic residue. These results further support the role of microsilica in enhancing thermal resistance and char formation. This finding aligns with that of Chen et al. [35], showing that adding nanosilica to starch biocomposites enhances heat resistance by forming interfacial bonds and thermal barriers. The improved thermal stability is consistent with previous studies on silica-reinforced biocomposites, where silica contributes to structural integrity at high temperatures. Similar behavior was reported by Warsiki et al. [20] for nanosilica-reinforced cassava peel starch-based bioplastics. The enhanced thermal stability imparted by microsilica makes these composites more suitable for applications involving moderate thermal processing or storage conditions.

3.4. Fourier Transform Infrared (FTIR) Spectroscopy

FTIR analysis was conducted to investigate the interaction between the MOCAF starch matrix and microsilica within a wavenumber range of 4,000–500 cm^{-1} (Fig. 2). The absorption bands at 2920–2930 cm^{-1} correspond to the C–H stretching vibrations of aliphatic groups. A decrease in peak intensity in the microsilica-reinforced bioplastics indicates restricted molecular mobility, suggesting stronger intermolecular interactions within the composite [36].

The bands observed at 1640–1650 cm^{-1} correspond to the O–H stretching vibrations associated with water bonding in the starch matrix. Syafri et al. [34] stated that the peak at 1627 cm^{-1} represents bonding water molecules and is attributed to the scissoring of O–H. The shift and reduction in intensity of this peak in the presence of microsilica indicate a decrease in free water molecules, which is consistent with the reduced water absorption observed experimentally. This

suggests that microsilica interacts with the hydroxyl groups in starch, leading to a more compact and less hydrophilic structure.

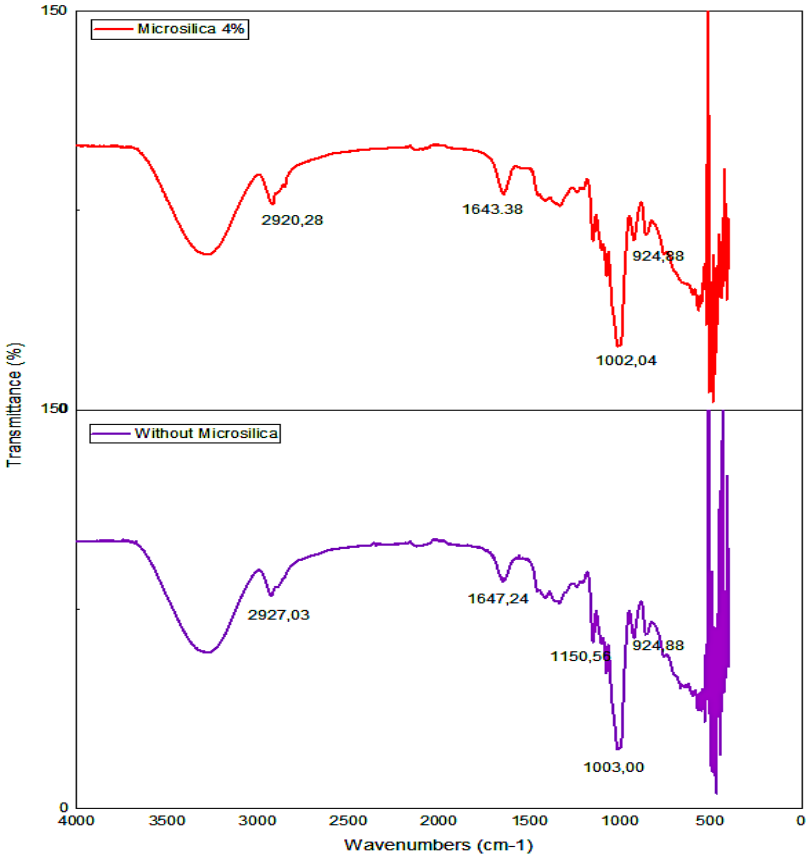

Fig. 2. FTIR spectroscopy results of the bioplastic with MOCAF starch matrix.

The C–O stretching vibrations of the C–O–C bands in the anhydroglucose ring were identified in the FTIR spectrum within the range between 924.88 cm^{-1} and 1003.00 cm^{-1}. The spectrum range of 900–1030 cm^{-1} is characteristic of anhydroglucose stretching [37]. Within this range the characteristic C–O–C stretching of the anhydroglucose unit overlaps with Si–O–Si and Si–O–C vibrations. The slight shift from 1003 cm^{-1} to 1002.04 cm^{-1} upon microsilica addition confirms the formation of interfacial interactions between the starch matrix and silica particles, likely through hydrogen bond formation or Si–O–C covalent bonds.

Overall, the FTIR results provided strong evidence of physical and possible hydrogen bonding interactions between microsilica and starch, which contribute to the improved mechanical, thermal and barrier properties of the bioplastics.

4. Conclusions

This study demonstrated that the incorporation of rice husk-derived microsilica at varying concentrations (0–5 wt%) significantly influenced the performance of MOCAF starch-based bioplastics. The tensile strength increased with increasing microsilica content, reaching an optimum value at 4 wt% (3.73 MPa), followed by a decline in elongation at break with increasing

microsilica content, indicating reduced flexibility as a consequence of restricted polymer chain mobility. In addition, water absorption decreased with microsilica incorporation, reflecting improved water resistance and a denser composite structure. FTIR analysis confirmed the presence of interactions between microsilica and the starch matrix, which contribute to the reduction in hydrophilicity. Thermogravimetric analysis (TGA) further revealed enhanced thermal stability, as evidenced by delayed degradation and higher residual mass in microsilica-reinforced samples. These findings confirm that rice husk-derived microsilica is an effective and economically viable reinforcing agent for MOCAF-based bioplastics. Future studies should investigate the biodegradability of this composition, conduct scanning electron microscopy (SEM) to characterize filler dispersion morphology and explore surface modification of microsilica to further improve compatibility with the starch matrix.

Abbreviations

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Data Availability Statement

Data will be shared upon request by the readers.

CRediT Authorship Contribution Statement

Ruri Wijayanti: investigation, writing –original draft, data curation, methodology, conceptualization, formal analysis, resources. **Emriadi**: review & editing, conceptualization supervision, validation, data curation, formal analysis. **Anwar Kasim**: writing - review & editing validation, conceptualization, supervision, data curation, formal analysis. **Nalwida Rozen**: investigation, formal analysis, writing – review & editing.

Declaration of Competing Interest

The authors declare that there are no conflicts of interest.

Declaration of Use of AI in the Writing Process

Nothing to disclose.

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