

DEVELOPMENT OF BIOPLASTICS BASED ON FOXTAIL MILLET (*Setaria italica*) SEED STARCH USING CaCO_3 AS A FILLER AND GLYCEROL AS A PLASTICIZER THROUGH MELT INTERCALATION METHOD

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Abstract

Starch-based bioplastics have attracted significant attention as a promising alternative to conventional plastics. One potential starch source for bioplastic production is foxtail millet (*Setaria italica*) seed starch. However, starch-based bioplastics exhibit low mechanical strength and high hydrophilicity, necessitating the addition of fillers and plasticizers to improve their physico-mechanical properties. This study developed the bioplastics using foxtail millet seed starch as the polymer matrix, with the addition of calcium carbonate (CaCO_3) as a filler and glycerol as a plasticizer. The objective was to evaluate the effect of CaCO_3 concentrations (2%, 4%, 6%, and 8%) on the morphological structure, elemental composition, functional groups, as well as physical and mechanical properties of the bioplastics. The bioplastics were fabricated using the melt intercalation method, which involve a solvent phase inversion principle. The results showed that the increasing of CaCO_3 concentration resulted in higher density and modulus of elasticity, but reduced tensile strength. The bioplastic with 2% CaCO_3 showed the most optimal physical and mechanical characteristics, achieving a tensile strength of 8.18 MPa. SEM-EDX revealed that the starch particles exhibit a semi-hexagonal shape with a particle size of approximately 10 μm . The addition of 2% CaCO_3 led to a denser bioplastic structure. Elemental analysis indicated the bioplastics were composed of oxygen (71.5%), carbon (15.6%), nitrogen (8.4%), calcium (2.6%), and phosphorus (1.9%). FTIR analysis showed an absorption at 1413.60 cm^{-1} which indicating the presence of CaCO_3 , and at 2927.95 cm^{-1} which corresponding to O–H stretching from glycerol.

Keywords: CaCO_3 , foxtail millet, melt intercalation, modulus of elasticity, SEM-EDX, tensile strength.

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1. Introduction

Plastic is widely used in various aspects of daily life due to its low cost, easy availability, durability, and versatility. However, the extensive use of plastic also poses significant environmental challenges, particularly related to its production and waste management. Plastic is primarily derived from limited fossil resources and is generally non-biodegradable, which means it can persist in the environment for a very long time and potentially pose risks to both human health and the ecosystem. To address these issues, bioplastics have emerged as a promising alternative by reducing dependence on fossil fuels and mitigating the associated environmental impacts. Bioplastics

are a class of plastics derived wholly or partially from renewable biomass and are capable of being decomposed by microorganisms in the environment [1].

Among the various types of bioplastics, starch-based bioplastics represent an innovative and sustainable solution. A wide range of starch sources has been explored for bioplastic production, including corn starch [2], mango seed starch [3], jackfruit seed starch [4], avocado seed starch [5], and cassava stem starch [6].

One underutilized yet promising starch source for bioplastic production is foxtail millet (*Setaria italica*). In Indonesia, foxtail millet is predominantly used as bird feed, despite its high nutritional value. The starch content of foxtail millet includes approximately 12.1% protein, 1.68% fat, 81.52% carbohydrates, and 7.8% dietary fiber [7], making it a viable candidate for bioplastic applications. Moreover, converting millet seeds into flour improves its storage stability, practicality, and blendability.

Nevertheless, starch-based bioplastics typically suffer from poor mechanical strength [8] and high hydrophilicity, leading to water solubility and limited water resistance [9]. These limitations can be addressed through the incorporation of filler materials, which can enhance performance without the need for specialized equipment or increased costs. Improvements are achieved through the formation of strong hydrogen bonds between the starch and the filler, with the filler expected to improve tensile strength and the viscosity of the starch matrix [10]. Calcium carbonate (CaCO_3) is one of the most commonly used fillers in starch-based bioplastics. For example, [11] demonstrated that the addition of up to 4% precipitated CaCO_3 increased tensile strength from 1.65 MPa to 3.38 MPa, while also reducing elongation at break by 6% to 10%. Conversely, [12] reported that the addition of CaCO_3 reduced the tensile strength of cassava starch-based bioplastics from 0.4% to 1%. Furthermore, [13] emphasized that improper incorporation of CaCO_3 may decrease intermolecular bonding, thus requiring the addition of a plasticizer such as glycerol to enhance flexibility and reduce brittleness. Due to its compatibility with amylose, glycerol is frequently used as a plasticizer in starch films, improving tensile properties by disrupting amylose packing and weakening intermolecular forces between starch molecules [10].

Bioplastics can be synthesized through various techniques, including exfoliation or adsorption, in-situ intercalative polymerization, prepolymer intercalation, and melt intercalation [14]. In this study, the melt intercalation method was selected. This technique involves solvent phase inversion, where the solvent is evaporated after casting the solution onto a glass plate. The method relies on thermodynamic principles, transitioning from a stable to an unstable state during the liquid-to-solid phase change [15]. Melt intercalation method also offers advantages over other polymer intercalation methods, due to its environmental friendliness as it avoids the use of organic solvents and aligns well with existing industrial processing methods [16].

Based on the above background, this study was carefully designed using the melt intercalation method to improve the properties of foxtail millet starch-based bioplastics. The research aims to evaluate the effect of CaCO_3 filler on the functional groups, morphology and elemental composition, physical, and mechanical properties of bioplastics derived from foxtail millet starch.

2. Materials and method

2. 1. Materials

Foxtail millet were obtained from the Barus Tani animal feed shop in Padang Bulan, Medan, Indonesia. CaCO_3 , glycerol, and aquadest were obtained from the CV. Rudang Jaya, Medan, Indonesia.

2. 2. Preparation of millet starch

The process of preparing millet starch involves several steps. First, the millet seeds are washed with water until clean. Then, 1 kg of millet seeds is soaked in 2 L of distilled water for 2 hours, then blended to form a porridge-like mixture. This mixture is then filtered to remove any solid particles, leaving behind a milky liquid. The remaining solid particles are mixed with 1 L of Aquadest, then blended again and filtered. The resulting suspension is left to settle for 24 hours, allowing the starch to precipitate. The precipitated starch is then dried using a 45°C

oven for 12 hours [17]. Once dried, the starch was ground and sieved to a 100-mesh size, ensuring a fine and uniform texture [5].

2. 3. Preparation of bioplastics

A total of 5 g of foxtail millet starch was added to 100 mL of aquadest in a beaker glass and heated on a hot plate while stirring to a temperature of 70°C [18] until gelatin was formed for 10 minutes. Then, glycerol was added as much as 40% (w/v) [17] of the starch weight as a plasticizer. CaCO₃ was added with varying concentrations (0%, 2%, 4%, 6%, 8%) of the starch weight for each run. The mixture was stirred and cooled to a temperature of 50°C. The mixture was poured into an acrylic container measuring 250 mm × 250 mm × 2 mm [11]. After that, the film was dried in the oven at 50°C [17] for 4 hours. Film was cooled to a room temperature and stored in a desiccator for 48 hours.

2. 4. Fourier transform infra-red (FTIR)

The analysis was conducted at the Integrated Research Laboratory at Diponegoro University, Semarang, Central Java. Equipment specifications used: Perkin-Elmer UATR Spectrum Two. The samples were cut into small pieces and ground with KBr until smooth to form a thin layer. Then, they were placed in a sample container for testing. The cluster spectrum was recorded on the monitor screen at 4000–400 cm⁻¹ wavelength.

2. 5. Scanning electron microscope-energy dispersive X-Ray (SEM-EDX)

The analysis was carried out at the Teaching Factory Manufacturing of Electronics (TFME) Laboratory at Batam State Polytechnic. Morphological analysis of the upper cross-section of bioplastics was carried out using the SEM-EDX phenom/proxy model. The sample is cut to 10 × 10 mm to be attached to the holder (sample container for testing). The sample was inserted into the SEM-EDX equipment chamber to set the position and record the image with a magnification of 5000 times.

2. 6. Density

Density analysis was conducted at the analytical chemistry laboratory, Faculty of Engineering, Universitas Sumatera Utara. The procedure for determining bioplastic density is as follows [19]. The sample's mass, m (g), is weighed with a digital balance. Then, the 10 mL measuring cup is filled with water to 5 mL, and the bioplastic sample is placed in the measuring cup filled with water. After 15 minutes, the new water volume (v) was recorded to calculate the actual film volume by the difference between the final and initial volumes. So, it is possible to obtain the density (ρ) with the equation (1)

$$\text{Density (g/mL)} = \frac{m \text{ (g)}}{v \text{ (mL)}}. \quad (1)$$

2. 7. Tensile strength

Bioplastics measuring 250 mm × 250 mm × 20 mm, made into dumbbell-shaped specimens, were tested for tensile strength and elongation at break according to ASTM D 638 using the ZP Recorder Imada 50N Strength test equipment. After the samples were installed, the initial limits were recorded. Then, a tensile force was applied to the sample until it breaks. So, let's obtain data such as the final limit and maximum force magnitude.

3. Results and Discussion

3. 1. Fourier transform infra-red (FTIR) of millet starch, bioplastic without filler, and bioplastic with 2% of CaCO₃ filler

Fourier transform infra-red (FTIR) spectra of millet starch, bioplastic without filler, and bioplastic with 2% of CaCO₃ filler is shown in **Fig. 1**. **Fig. 1** (Sample C): millet seed starch shows an absorption peak with a wave number of 3272.95 cm⁻¹, indicating the O–H group. This absorption came

from amylose and amylopectin compounds in starch [20]. A wave number of 2927 cm^{-1} indicates the presence of an alkane C–H group, a wave number of 1645.82 cm^{-1} indicates a C=O group in the middle of an amino acid [21], a wave number of 1077.41 cm^{-1} indicates a C–O ether group [22]. A wave number of 930.15 cm^{-1} indicates a C=H group, and a wavenumber of 658.83 cm^{-1} indicates an aromatic C–H group. The wave number 1337 cm^{-1} indicates the presence of the C–N amine group, which is thought to originate from proteins contained in starch. Muthamilarasan [20] reported the total protein content of barley seed starch, namely $12.3/100\text{ g}$, so the presence of the C–N amine group is thought to originate from the protein contained in starch [23]. The wave number 1077.41 cm^{-1} indicates the presence of the C–O ester group. Sharma and Niranjana [24] reported that the crude fat content of barley starch was $4.3/100\text{ g}$, so the presence of the C–O ester group is thought to originate from the fat contained in the starch.

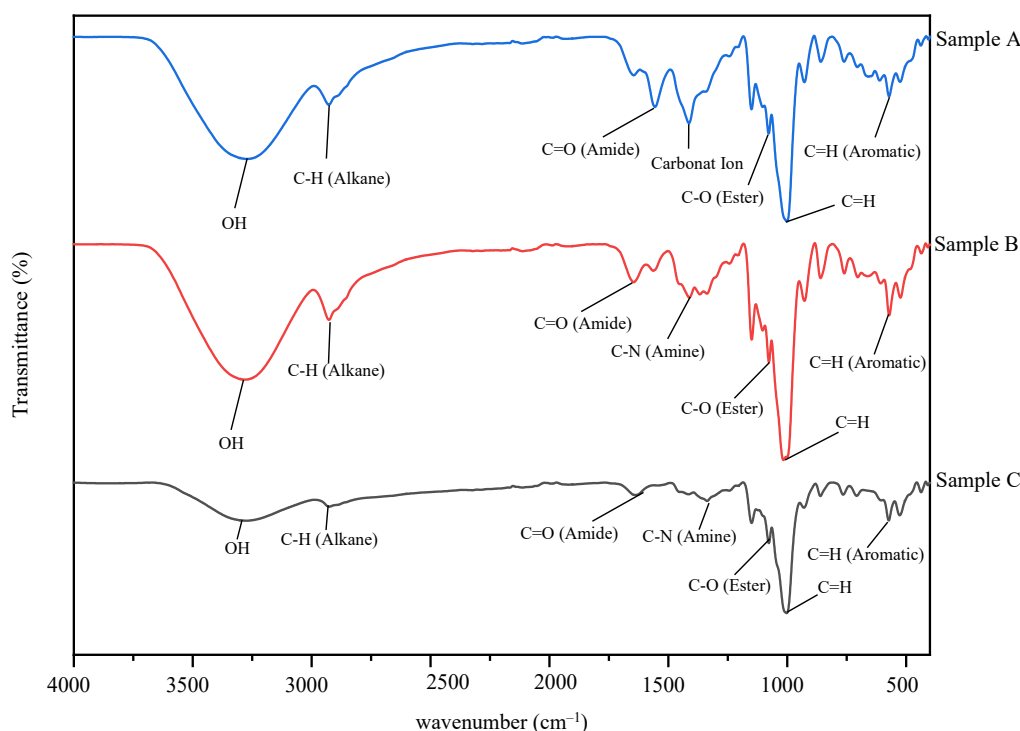


Fig. 1. Fourier transform-infrared spectra of bioplastic with 2% of CaCO_3 filler (sample A), bioplastic without filler (sample B), and millet starch (sample C)

Fig. 1 (Sample B): bioplastic without CaCO_3 filler shows a wave number of 3274.95 cm^{-1} , showing that the O–H group has slightly shifted. The wave number of 2927.22 cm^{-1} indicates the C–H alkane group. The wave number of 1337.70 cm^{-1} indicates the C–N amine group, and the wave number of 1077 cm^{-1} indicates the C–O group, indicating the presence of protein and fat in millet starch [23].

Fig. 1 (Sample A): bioplastic filled with 2% of CaCO_3 shows a wavenumber of 1413.60 cm^{-1} , indicating the presence of CaCO_3 filler [25]. The wavenumber of 3272.95 cm^{-1} indicates the OH group that has experienced a slight shift. The wavenumber of 2927.95 cm^{-1} indicates the C–H group of the alkane. The wavenumber of 1556.10 cm^{-1} indicates the C=O amide group. The wavenumber of 1078.10 cm^{-1} indicates the C–N amine group. The last, the wavenumber of 859.14 cm^{-1} indicates the aromatic C–H group.

3. 2. Scanning electron microscope energy dispersive X-ray (SEM-EDX) of millet starch

Analysis of millet starch using SEM-EDX aims to observe the morphology of the starch and determine the elemental composition of the millet starch. SEM-EDX results of millet starch are shown in **Fig. 2**.

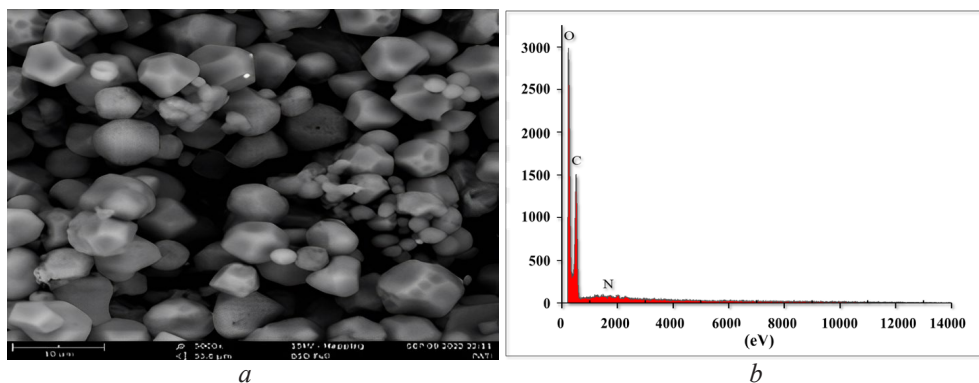


Fig. 2. SEM-EDX analysis results of millet seed starch with a magnification of 5000 times; *a* – morphology; *b* – diffractogram

Fig. 2 shows that the particle size of millet starch is 10 μm , and the granules are semi-hexagonal. This is in line with the research by [17, 18], which reported the size and shape of millet starch to be around 6.8–11.8 μm , with starch shapes such as pores and hollow polygonals. The size and shape of this starch depend on the plant source. Based on the size of the starch, millet seed starch is included in the small-size starch [26]. SEM-EDX analysis also revealed the elemental composition of millet starch, including 75.9% of oxygen, 14.9% of carbon, and 9.2% of nitrogen. The presence of oxygen, carbon, and nitrogen are thought to originate from the components of the hydroxyl ($-\text{OH}$) and carbonyl ($\text{C}=\text{O}$) bonds from the carbohydrate group, as well as the amine ($\text{N}-\text{H}$) from the protein group. These results are supported by **Fig. 1** (Samples A, B, and C), which are the FTIR spectra of these functional groups [20, 23, 27].

The purpose of the scanning electron microscope energy dispersive X-ray (EDX) on bioplastics is to see the morphology and elemental composition of the bioplastics. **Fig. 3** shows the SEM-EDX results of bioplastics with and without of 2% of CaCO_3 filler.

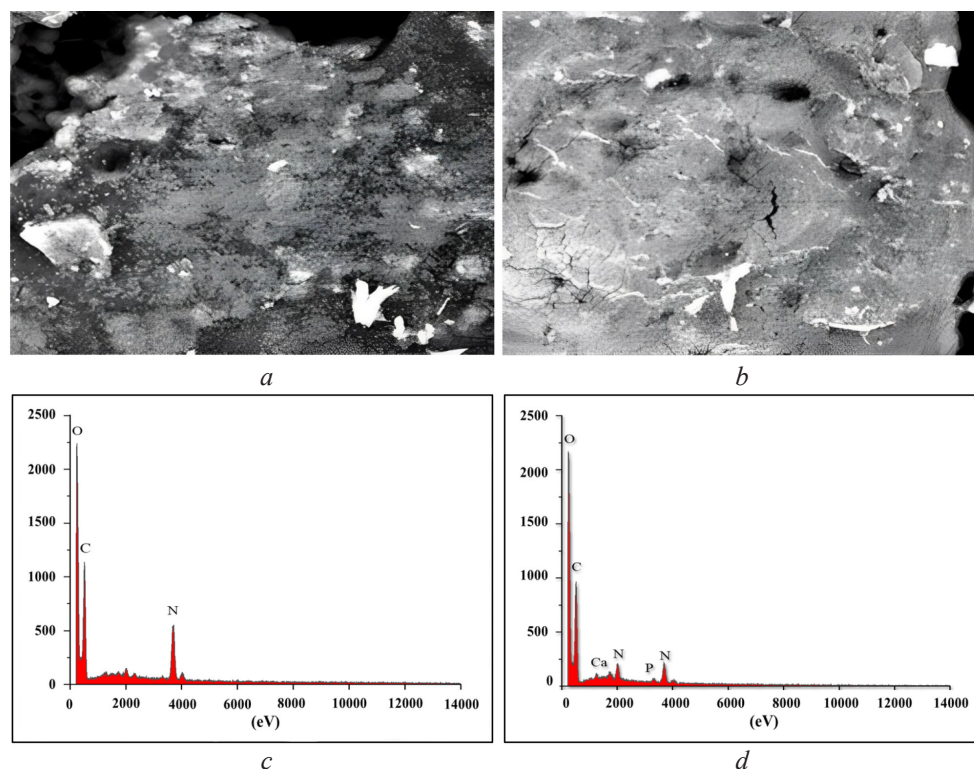


Fig. 3. SEM-EDX analysis results of bioplastics: *a–c* – without filler; *b–d* – with 2% of CaCO_3 filler

Fig. 3, a shows that uneven white dots mark the bioplastic without CaCO_3 filler and show starch clumping due to the non-uniform size of the starch molecules. **Fig. 3, b** shows that the addition of 2% of CaCO_3 filler is indicated by the presence of white dots on the surface of the bioplastic, which becomes denser and has no distance between particles. It is suspected that the CaCO_3 filler fills the space between starch particles.

Fig. 3, c shows that the elemental composition of millet starch bioplastic without CaCO_3 filler is dominated by elements: 75.7% oxygen, 15.7% carbon, and 8.6% nitrogen. **Fig. 3, d** shows the elemental composition of millet starch bioplastic filler 2% CaCO_3 : 71.5% oxygen, 15.5% carbon, 8.4% nitrogen. The presence of 2.6% calcium Ca elements is thought to be from the CaCO_3 filler, causing the percentage of O elements in bioplastics to decrease to 71.5%.

3. 3. Effect of CaCO_3 addition on the density of bioplastics

Density analysis aims to determine the physical properties of bioplastics, particularly the density/porosity of bioplastics. The effect of CaCO_3 addition on the density of millet starch-based bioplastics is presented in **Fig. 4**.

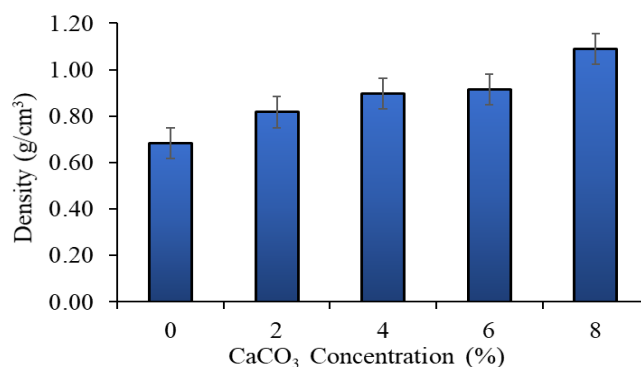


Fig. 4. Effect of CaCO_3 addition on the density of bioplastics

The density of bioplastics increases with the increasing of CaCO_3 concentration. The highest density, up to 1.089 g/cm^3 , was achieved at a CaCO_3 concentration of 8%. The increasing density is due to the CaCO_3 filler having a higher density than starch [28]. Furthermore, this increasing density is indicated by the presence of white dots on the surface of the bioplastic, which becomes denser because the CaCO_3 filler is thought to fill the space between the starch particles, as shown by the SEM-EDX results in **Fig. 3, b**.

3. 4. Effect of CaCO_3 addition on the tensile strength of bioplastics

Tensile strength analysis is a measurement of the strength and resistance of a material to stress applied in the tensile direction. **Fig. 5** depicted the tensile properties of bioplastics at various CaCO_3 concentration, showing a decrease in tensile strength with increasing CaCO_3 content.

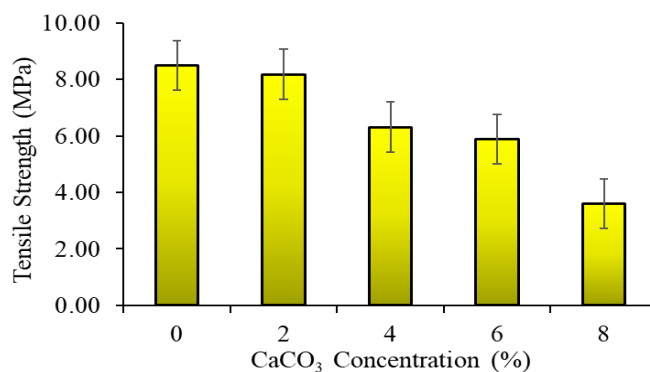


Fig. 5. Effect of CaCO_3 addition on the tensile strength of bioplastics

The highest tensile strength (8.18 MPa) is achieved by using 2% CaCO_3 . In essence, the presence of fillers such as CaCO_3 serves to address one of the main drawbacks of bioplastics, namely their low tensile strength. The addition of an appropriate amount of CaCO_3 can enhance the tensile strength of bioplastics. This is due to the role of CaCO_3 as a filler within the bioplastic matrix, which increases the material's density and strength. However, excessive addition of CaCO_3 may reduce the tensile strength of the bioplastic. This occurs because CaCO_3 can occupy the spaces between polymer molecules, thereby reducing the number of intermolecular bonds that can form, ultimately decreasing tensile strength. This finding is supported by research by Nuriyah [12], more CaCO_3 loading, weaker the tensile strength of the bioplastic.

3. 5. Effect of CaCO_3 addition on the modulus of elasticity of bioplastics

Analysis of the modulus of elasticity in bioplastics aims to determine the ability of the bioplastic to restore its shape after a tensile or compressive force is applied. **Fig. 6** shows that the addition of CaCO_3 filler up to 4% increases the modulus of elasticity from 0.52 MPa to 0.94 MPa. The increase in modulus of elasticity is related to the interaction between starch and CaCO_3 filler. Adding CaCO_3 up to 8% decreases the modulus of elasticity of bioplastic; this is because CaCO_3 is hygroscopic and disrupts the interaction between the starch polymers that form bioplastic. The elastic modulus value is influenced by tensile strength; the more significant the tensile strength value, the greater the elastic modulus [11].

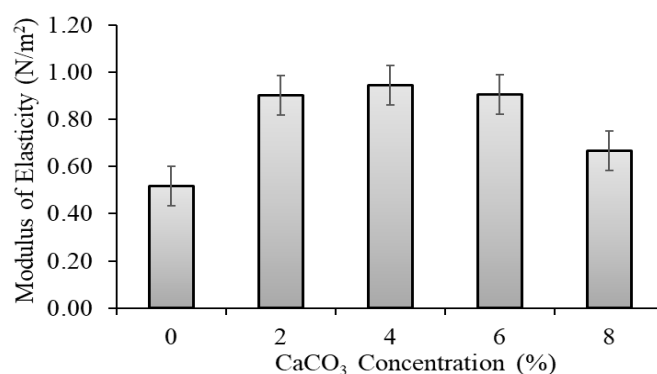


Fig. 6. Effect of CaCO_3 addition on the modulus of elasticity of bioplastics

3. 5. Limitations of the study and future directions

This study successfully demonstrates the fabrication of bioplastic films derived from foxtail millet-seed starch using glycerol as a plasticizer and CaCO_3 as a filler through the melt intercalation method. While the findings highlight the potential of foxtail millet-seed starch as an underutilized raw material for bioplastic production, several limitations should be acknowledged. First, the current work is limited to the fabrication of thin-film bioplastics and does not extend to the production of fully developed bioplastic products with practical form factors. Consequently, the applicability of the resulting bioplastic under real industrial or consumer conditions has not yet been assessed. Second, the characterization in this study focused primarily on the physicochemical properties of bioplastics, without evaluating other essential aspects such as biodegradability, antimicrobial activity, and their performance in food packaging applications. These untested properties may influence the suitability of the bioplastics for specific use and therefore represent important directions for future inquiry.

The experimental procedure and analytical methods described in this study were conducted under controlled laboratory conditions and can be reproduced by other researchers. However, it is important to note that variations in starch source, processing conditions, and environmental humidity may affect the reproducibility and consistency of the resulting bioplastic properties. Therefore, researchers intending to replicate or apply these findings in practical settings should ensure standardized preparation and controlled environmental parameters during synthesis and testing.

Future research should be directed toward expanding the functional assessment of fox-tail millet-seed starch-based bioplastics. Specifically, studies evaluating their biodegradation behavior under different environmental conditions, antimicrobial performance against foodborne pathogens, and suitability as active packaging materials are recommended. In addition, scaling up the melt intercalation process to pilot or industrial levels would provide valuable insights into the material's feasibility for commercial production and its potential contribution to sustainable bioplastic development.

4. Conclusions

This research showed that the addition of CaCO_3 filler into millet starch-based bioplastics had a significant effect on physical and mechanical properties of bioplastics. Bioplastic with 2% of CaCO_3 filler has the highest tensile strength, up to 8.18 MPa. SEM-EDX results show no distance between starch particles. Bioplastic was composed of several elements, including 71.5% of oxygen, 15.6% of carbon, 8.4% of nitrogen, 2.6% of calcium, and 1.9% of phosphorus. The results of FTIR analysis showed the absorption at 1413.60 cm^{-1} and 2927.95 cm^{-1} on the bioplastic, indicating the presence of CaCO_3 groups as fillers and O–H glycerol groups as plasticizers.

Conflict of interest

The authors declare that there is no conflict of interest in relation to this paper and the published research results, including the financial aspects of conducting the research, obtaining and using its results, as well as any non-financial personal relationships.

Financing

There is no financial support in this research.

Data availability

Data will be made available on reasonable request.

Use of artificial intelligence

During the preparation of this paper, the authors used ChatGPT solely to improve the language and readability of the text. After using this tool/service, the authors reviewed and edited the content as necessary and took full responsibility for the content of the published article. No artificial intelligence assistance was used in the acquisition or analysis the data presented in this paper.

Authors' Contributions

Farizal Hamdi: Data curation, Writing – original draft.

Muhammad Ferdiansyah Hasugian: Data curation, Visualization.

Afiifah Radhiyatullah: Project administration.

Muhammad Hendra S. Ginting: Supervision, Methodology, Validation.

Viqry Pramananda: Writing – review & editing.

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