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Development and Optimization of Starch-Based Bioplastics from Millet and Cassava Using Mixture Design Approach

Aminu D. MAHMUD[1*], Imran N. IBRAHIM[2]

[1]Department of Chemical Engineering, Ahmadu Bello University, Zaria, Nigeria
[2]Department of Process Engineering, Dangote Fertiliser Limited, Lagos State, Nigeria

[1*]admahmud@abu.edu.ng, [2]imranibrahim252@gmail.com

Abstract

The increasing environmental concerns associated with conventional petroleum-based plastics have driven the search for sustainable and biodegradable alternatives. This study investigates the extraction, characterization, and application of starches derived from millet (Pennisetum glaucum) and cassava (Manihot esculenta) for the production of bioplastic films. Starch was extracted using a wet extraction method and subsequently characterized for amylose and amylopectin content using iodine colorimetry. Bioplastic films were produced through solution casting with glycerol as a plasticizer, and the effects of varying starch blend ratios were evaluated using a simplex lattice mixture design. Results showed that cassava starch possessed a higher amylose content (~27.93%), while millet starch exhibited lower amylose (~18.17%) and higher amylopectin content. These compositional differences significantly influenced the mechanical and physical properties of the bioplastics. The tensile strength, strain, modulus of elasticity, and water absorption of the films were determined. Among the formulations, the 50:50 blend of millet and cassava starch demonstrated the best overall performance, exhibiting improved tensile strength and mechanical stability compared to single-starch films. However, all samples showed relatively high-water absorption, reflecting the hydrophilic nature of starch-based materials. Optimization using mixture design yielded a desirability value of 0.546, with predicted properties showing reasonable agreement with experimental results. The findings highlight the synergistic effect of starch blending in enhancing bioplastic performance and confirm the potential of locally sourced starches as viable raw materials for biodegradable plastic production. Nonetheless, further modifications are required to improve water resistance and broaden practical applications.

Keywords: *Bioplastics, Starch blending, Cassava starch, Millet starch, Amylose.*

1.0 Introduction

The widespread use of conventional petroleum-based plastics has generated significant environmental concerns due to their non-biodegradability, persistence in ecosystems, and contribution to global pollution. Accumulation of plastic waste in terrestrial and aquatic environments has intensified the need for sustainable and environmentally friendly alternatives [1], [2]. In response, biodegradable polymers derived from renewable resources have gained increasing attention as viable substitutes for synthetic plastics. Among these, starch-based bioplastics have emerged as one of the most promising candidates due to their abundance, low cost, renewability, and biodegradability.

Starch, a naturally occurring polysaccharide composed primarily of amylose and amylopectin, plays a critical role in determining the physicochemical and mechanical properties of bioplastic materials. The relative proportions of these two components significantly influence film strength, flexibility, and water sensitivity [3], [4]. High amylose content is generally associated with improved tensile strength and rigidity due to enhanced intermolecular hydrogen bonding, whereas amylopectin contributes to flexibility owing to its branched molecular structure. However, despite these advantages, starch-based bioplastics often suffer from inherent limitations such as poor mechanical strength, high brittleness, and high-water absorption, which restrict their practical applications, particularly in packaging [2], [5].

To overcome these limitations, recent studies have explored various modification strategies, including plasticization, chemical modification, and blending of starches from different botanical sources. Starch blending, in particular, has been identified as an effective approach for enhancing the performance of bioplastics by exploiting the complementary properties of different starch types [6], [7]. For instance, blending starches with varying amylose–amylopectin ratios can result in improved mechanical strength, flexibility, and overall structural integrity of the resulting films. Additionally, the use of plasticizers such as glycerol has been shown to improve film flexibility and reduce brittleness by increasing polymer chain mobility [8], [9].

Despite these advancements, most existing studies have focused predominantly on commonly used starch sources such as corn, potato, and cassava, with limited attention given to underutilized crops like millet, particularly in the context of bioplastic development. Furthermore, while several studies have demonstrated the benefits of starch blending, there remains a lack of systematic optimization of blend ratios using robust statistical tools such as mixture design approaches. Many studies rely on trial-and-error methods, which may not adequately capture

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the interaction effects between components or identify optimal formulations. In addition, there is insufficient empirical data comparing the synergistic effects of blending starches with contrasting compositional characteristics—such as high-amylose cassava starch and amylopectin-rich millet starch—on the mechanical and functional properties of bioplastics. Therefore, the key research gap lies in the limited application of mixture design techniques to optimize starch blending systems involving locally available but underutilized starch sources such as millet, as well as the insufficient understanding of how compositional differences influence the performance of resulting bioplastic films.

Addressing this gap is particularly important in developing countries like Nigeria, where both cassava and millet are widely available and hold significant potential as sustainable raw materials for biopolymer production. Leveraging such indigenous resources not only promotes environmental sustainability but also supports local agro-based industries and circular economy initiatives. The present study aims to develop and optimize starch-based bioplastics using blends of cassava (Manihot esculenta) and millet (Pennisetum glaucum) starches. A simplex lattice mixture design approach is employed to systematically evaluate the effects of varying blend ratios on the mechanical and physical properties of the resulting films. By integrating compositional analysis, mechanical characterization, and statistical optimization, this study seeks to provide deeper insights into the synergistic interactions between different starch sources and to identify optimal formulations for improved bioplastic performance.

2.0 Materials and Methods/Methodology

2.1 Materials

All materials used in this study were of analytical grade and utilized without further purification. Millet grains (*Pennisetum glaucum*) and dried cassava tubers (*Manihot esculenta*) were procured from a local market in Kaduna, Nigeria, ensuring their availability and relevance to indigenous biopolymer production systems. Distilled water was obtained from the Department of Chemical Engineering, Ahmadu Bello University. Chemical reagents employed in the analysis included sodium hydroxide (NaOH), ethanol (95%), acetic acid, glycerol, and iodine solution (0.2%), all of which are commonly used in starch characterization and biopolymer processing due to their effectiveness in gelatinization, plasticization, and colorimetric analysis. Glycerol was specifically selected as a plasticizer owing to its compatibility with starch matrices and its ability to enhance flexibility in biodegradable films. Standard laboratory equipment such as beakers, volumetric flasks, Erlenmeyer flasks, Petri dishes, glass rods, a heating mantle, oven, weighing balance, UV–Visible spectrophotometer, and tensile testing machine were used for processing and characterization. These instruments are widely adopted in starch-based biopolymer research for ensuring reproducibility and accuracy in physicochemical and mechanical analyses.

2.2 Methods

2.2.1 Starch Extraction

Starch was extracted from millet grains and cassava tubers using a conventional wet extraction method, which remains one of the most efficient and widely adopted techniques for isolating starch from plant sources due to its simplicity and high yield [10], [2]. Initially, the raw materials were thoroughly washed under running water to eliminate dirt, debris, and extraneous materials. The cleaned samples were subsequently subjected to mechanical size reduction using a blender to disrupt cellular structures and facilitate the release of starch granules. The resulting slurry was mixed with water and filtered through a sieving cloth to separate fibrous residues from the starch-containing liquid. The filtrate was then allowed to settle under gravity, enabling sedimentation of starch particles. The supernatant was carefully decanted, and the starch sediment was repeatedly washed with distilled water to remove soluble impurities such as proteins and sugars. The purified starch was air-dried at ambient conditions to reduce moisture content while preserving its native physicochemical properties. This approach aligns with established practices in starch processing, where minimal thermal degradation is desired to maintain functional characteristics [2].

2.2.2 Determination of Amylose and Amylopectin Content

The amylose content of the extracted starch samples was determined using the iodine colorimetric method, a widely accepted analytical technique based on the formation of a blue-colored complex between iodine and amylose helices [11], [3]. Approximately 100 mg of each starch sample was weighed into clean Erlenmeyer flasks, followed by the addition of sodium hydroxide (NaOH) and ethanol to facilitate starch dispersion and gelatinization. The mixtures were allowed to stand overnight to ensure complete solubilization of starch, resulting in a homogeneous viscous solution. The gelatinized solutions were diluted to a fixed volume using distilled water and further aliquots were prepared for analysis. Subsequently, acetic acid and iodine solution were added to each sample to initiate color development. The reaction mixtures were incubated in the dark for a specified period to ensure stabilization of the starch–iodine complex. Absorbance measurements were taken at a wavelength of 620 nm using a UV–Visible spectrophotometer. A calibration curve was constructed using standard amylose solutions,

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and the amylose content of the samples was determined from this curve. The iodine-binding method remains highly reliable due to its sensitivity to amylose structure and its strong correlation with starch functional properties [12], [3]. Moisture content was determined concurrently using a moisture analyzer to improve accuracy in compositional analysis. The amylopectin content was subsequently calculated by difference, as the sum of amylose and amylopectin constitutes the total starch fraction

2.2.3 Production of Bioplastic Films

Bioplastic films were prepared using a starch blending approach based on a simplex lattice mixture design, which is commonly applied in optimizing multicomponent formulations due to its efficiency in evaluating interaction effects between components. Predetermined ratios of millet starch and cassava starch were blended and dispersed in distilled water. Glycerol was incorporated as a plasticizer to improve flexibility and reduce brittleness, while acetic acid was added to enhance gelatinization and improve intermolecular interactions within the polymer matrix. The mixture was heated at approximately 70°C with continuous stirring to induce starch gelatinization, a critical step that disrupts the crystalline structure of starch granules and enables film formation. The resulting homogeneous paste was cast into Petri dishes and allowed to dry under ambient conditions, followed by further drying in an oven to ensure complete removal of residual moisture. This method is consistent with widely reported procedures for starch-based bioplastic production, where controlled heating and plasticizer incorporation significantly influence film properties such as flexibility, transparency, and mechanical strength [2].

2.2.4 Tensile Strength Analysis

The mechanical properties of the produced bioplastic films were evaluated through tensile strength testing using a universal testing machine. Samples were cut into standardized shapes, and their dimensions were measured to determine cross-sectional area. Each specimen was subjected to uniaxial tensile loading until failure, and the stress–strain behavior was recorded. The tensile strength was defined as the maximum stress sustained by the material before fracture. Mechanical characterization is essential in evaluating the suitability of starch-based bioplastics for practical applications, particularly in packaging materials where strength and flexibility are critical performance parameters.

2.2.5 Water Absorption Capacity

Water absorption tests were conducted to assess the hydrophilic nature of the bioplastic films, which is a key limitation of starch-based materials. Pre-weighed samples were immersed in distilled water at room temperature for 24 hours. After immersion, the samples were removed, surface moisture was gently wiped off, and the samples were reweighed. Water absorption was calculated based on the percentage increase in weight relative to the initial dry weight. This method provides insight into the interaction between water molecules and the polymer matrix, which is largely influenced by the presence of hydroxyl groups in starch and the degree of plasticization. High water absorption is commonly reported in starch-based films and remains a major factor affecting their durability and application performance [2].

3.0 Results and Discussion

3.1 Amylose and Amylopectin Content

The results presented in Table 3.1 show clear compositional differences between millet and cassava starches. Millet starch recorded a mean amylose content of 9.09 mg/ml, corresponding to 18.17%, while cassava starch showed a higher mean value of 13.97 mg/ml, corresponding to 27.93%. Consequently, millet starch had a higher amylopectin content (81.83%) compared to cassava starch (72.07%). These findings indicate that cassava starch contains a greater proportion of linear polymer chains (amylose), whereas millet starch is richer in branched amylopectin structures. The amylose-to-amylopectin ratios were approximately 1:4.5 for millet and 1:2.5 for cassava, confirming the relatively higher amylose composition of cassava starch. The values obtained for millet starch are consistent with the findings of Tiwari et al. (2023), who reported amylose contents in the range of 18–22% for millet varieties, supporting the reliability of the analytical methods used. Similarly, the cassava starch results agree with reported amylose contents between 25–30%, depending on cultivar and processing conditions [14], [4]. From a structural standpoint, amylose contributes to film strength due to its linear configuration, which promotes intermolecular hydrogen bonding and chain alignment [3]. In contrast, amylopectin, being highly branched, enhances flexibility but reduces tensile strength due to steric hindrance and reduced chain packing efficiency [2]. Therefore, the higher amylose content observed in cassava starch suggests greater potential for mechanical strength, whereas millet starch is expected to impart flexibility to starch-based bioplastic films.

Table 1: Amylose and amylopectin content of cassava and millet starches

S/N	Sample	Amylose (mg/ml)	Mean Amylose (mg/ml)	Amylose (%)	Amylopectin (%)
1.	Millet	8.77, 9.40	9.09	18.17	81.83
2.	Cassava	14.44, 13.49	13.97	27.93	72.07

3.2 Tensile Strength of Bioplastics

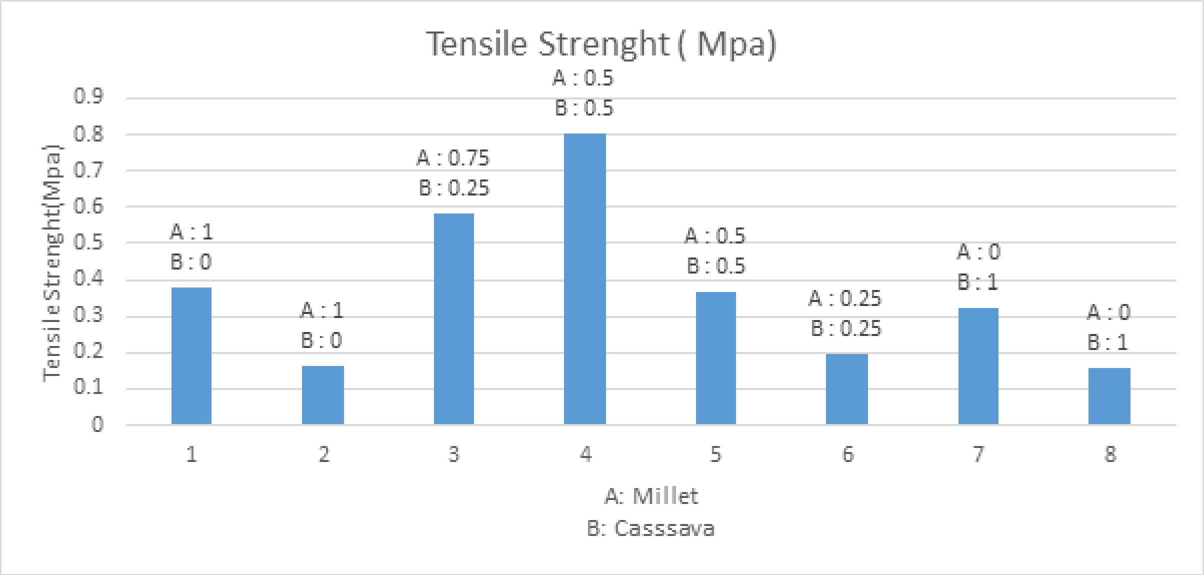

Figure 1: Tensile strength of bioplastic films at different millet (A) to cassava (B) starch mixing ratios. Values represent individual replicates where applicable.

From figure 1 above, samples with identical mixing ratios (e.g., 1 and 2; 4 and 5; 7 and 8) represent independent experimental replicates conducted to ensure reproducibility of the tensile strength measurements. The tensile strength results indicated that bioplastic films produced from a 50:50 blend of millet and cassava starch exhibited the highest tensile strength among all formulations, with values reaching approximately 0.802 MPa and 0.368 MPa. In comparison, films produced from single starch sources showed lower tensile strength. This result demonstrates a synergistic effect arising from starch blending, where the combination of the two polymer structures leads to improved mechanical performance. The enhanced tensile strength observed in the blended samples can be attributed to improved intermolecular interactions between amylose and amylopectin chains from different botanical origins, resulting in a more cohesive polymer network. Recent studies have consistently reported that starch blending improves mechanical properties due to enhanced compatibility and structural rearrangement within the polymer matrix [7], [6]. Specifically, cassava starch contributes rigidity due to its higher amylose content, while millet starch enhances flexibility, leading to an optimal balance between strength and ductility. Furthermore, the relatively low tensile strength values observed (compared to synthetic plastics) are characteristic of starch-based bioplastics and are largely attributed to their hydrophilic nature and weak intermolecular forces. However, such values remain within the acceptable range for biodegradable packaging applications [2], [1].

3.3 Strain of Bioplastics

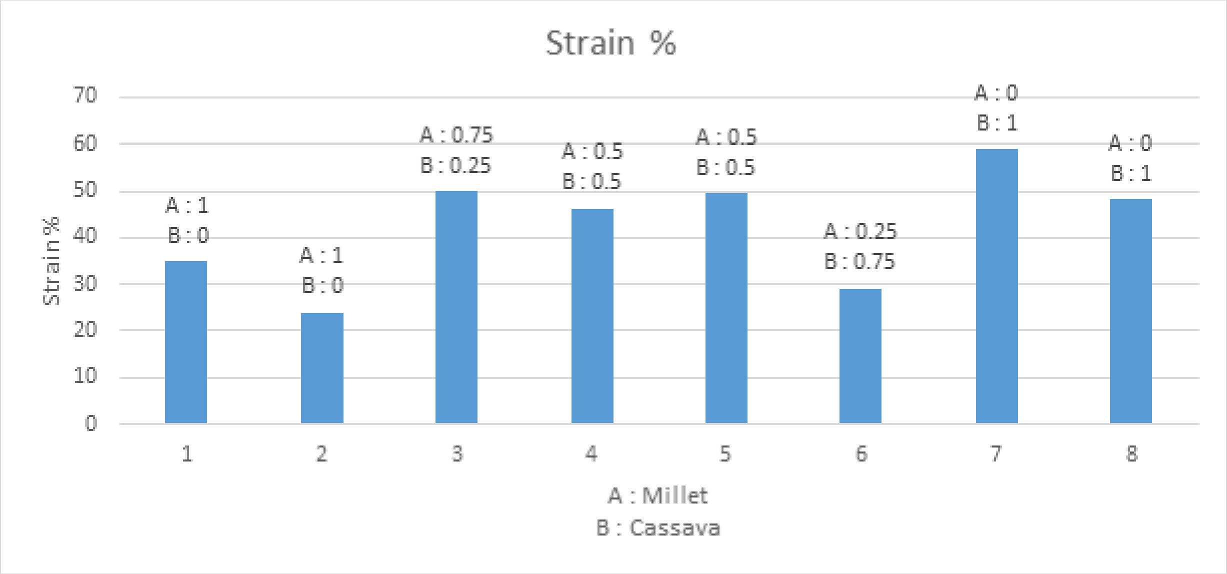

Figure 2: Percentage strain of bioplastics

The strain analysis showed that cassava-based films exhibited the highest elongation at break, with values of 59.12% and 48.4%, whereas millet-based films recorded lower strain values (24–35%). The blended samples (50:50) demonstrated intermediate but relatively high strain values (~46–49%), indicating good flexibility. This behavior can be directly linked to the molecular structure of starch components. Amylopectin-rich starches, such as millet, tend to enhance flexibility due to their branched architecture, which allows greater molecular mobility. However, in this case, cassava exhibited higher elongation, suggesting that other factors such as plasticizer interaction and gelatinization behavior also played significant roles. Recent literature highlights that glycerol, as a plasticizer, significantly increases elongation by reducing intermolecular forces and increasing free volume within the polymer matrix [8], [9]. Additionally, the gelatinization characteristics of cassava starch promote the formation of more uniform and continuous films, which enhances extensibility. The relatively high strain observed in the blended samples further supports the concept of polymer compatibility and network optimization, where the presence of both linear and branched chains allows for improved stress distribution during deformation [15].

3.4 Modulus of Elasticity

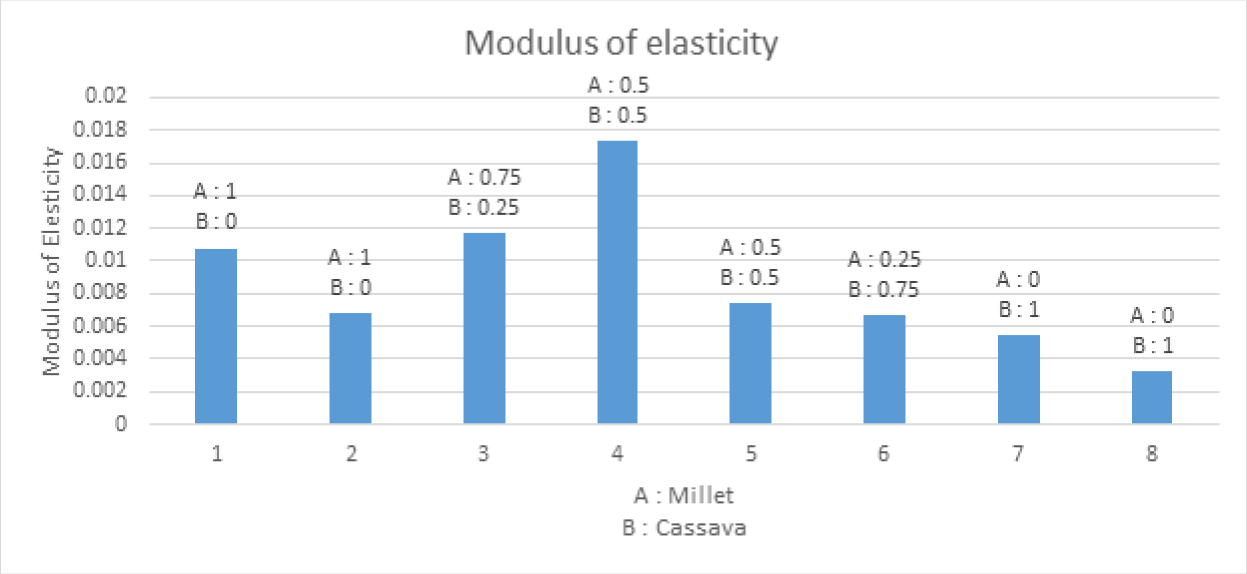

Figure 3: Modulus of elasticity of bioplastics

The modulus of elasticity results followed a trend similar to tensile strength, with the 50:50 starch blend exhibiting the highest modulus value (~0.017 MPa). This indicates that the blended films possess greater stiffness compared to films derived from single starch sources. The modulus of elasticity reflects the resistance of a material to elastic deformation, and its increase in blended samples suggests improved structural integrity of the polymer network. This can be attributed to stronger intermolecular bonding and better packing efficiency resulting from

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the interaction between different starch components. Studies have shown that the modulus of starch-based films is highly dependent on amylose content, degree of gelatinization, and plasticizer concentration [3], [6]. The improved modulus observed in the blended system indicates an optimal balance between rigidity (from amylose) and flexibility (from amylopectin), which is desirable for functional bioplastic materials.

3.5 Water Absorption

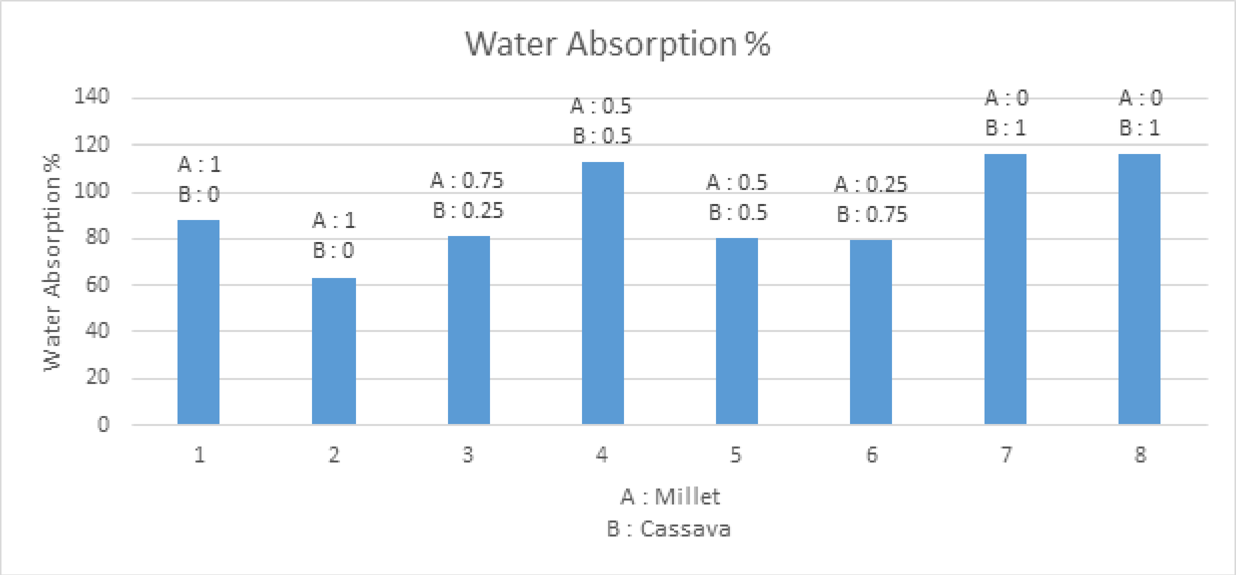

Figure 4: Water absorption values of bioplastics

Water absorption analysis revealed that cassava-based films exhibited the highest water uptake (up to 116.1%), while millet-based films showed significantly lower absorption (~63.38%). The blended samples displayed intermediate values, though still relatively high. This behavior is primarily attributed to the hydrophilic nature of starch, which contains abundant hydroxyl (-OH) groups capable of forming hydrogen bonds with water molecules. The higher water absorption observed in cassava starch films can be linked to its greater amylose content, which increases the availability of hydrophilic sites for water interaction. Recent studies confirm that starch-based bioplastics generally exhibit high water sensitivity, which limits their application in moisture-rich environments [2], [5]. Additionally, plasticizers such as glycerol further increase water uptake due to their hygroscopic nature. The reduced water absorption observed in millet-based films may be attributed to differences in granule structure and molecular arrangement, which can influence water diffusion pathways. Blending the starches partially mitigates excessive water absorption, although not sufficiently to match synthetic polymers.

3.6 Effect of Starch Blending on Overall Properties

The results demonstrate that starch blending significantly enhances the performance of bioplastics compared to single-source starch films. The 50:50 blend consistently showed improved tensile strength, strain, and modulus of elasticity, confirming the presence of synergistic interactions. This observation aligns with recent findings that blending different starch sources improves film homogeneity, mechanical strength, and functional properties due to complementary molecular characteristics [16], [15]. Similar studies on corn–potato and cassava–rice starch blends have also reported enhanced mechanical and barrier properties.

3.7 Model Prediction and Optimization

Model prediction and optimization were carried out using *Design-Expert software* (Stat-Ease Inc., USA) based on a mixture design in which millet starch (A) and cassava starch (B) served as the independent variables, constrained such that A + B = 1. The responses evaluated were tensile strength (Y_1), which was targeted for maximization, and water absorption (Y_2), which was targeted for minimization. A quadratic mixture model was fitted to the experimental data to describe the relationship between the component proportions and the responses.

Numerical optimization was performed using the desirability function approach, where tensile strength was maximized and water absorption minimized, with equal importance assigned to both responses. The overall desirability function was defined as the geometric mean of individual desirabilities. The optimum formulation was obtained at a millet-to-cassava ratio of approximately 0.52:0.48, with a predicted tensile strength of 0.53 MPa and water absorption of 90.7%. Experimental validation yielded values of 0.38 MPa and 85%, respectively, indicating reasonable agreement despite some deviations, which may be attributed to experimental variability and model limitations. The desirability value of 0.546 suggests moderate optimization efficiency, indicating a balance between competing response goals but also highlighting the need for further improvement. Similar findings have been reported in the literature, where mixture design models effectively predict formulation behavior but may exhibit discrepancies due to complex interactions in multicomponent systems [17], [18], while desirability-based optimization provides a practical approach for balancing multiple objectives [19] and guiding further formulation enhancement strategies such as reinforcement or crosslinking [15].

4.0 Conclusion

This study successfully demonstrated the extraction, characterization, and application of starches derived from millet (Pennisetum glaucum) and cassava (Manihot esculenta) in the production of biodegradable bioplastic films. The results established that both starch sources possess suitable physicochemical properties for biopolymer development, with notable differences in their amylose and amylopectin compositions significantly influencing material performance. The compositional analysis revealed that cassava starch contained a higher amylose content compared to millet starch, while millet exhibited a higher proportion of amylopectin. These differences played a critical role in determining the mechanical and functional properties of the resulting bioplastics. Specifically, higher amylose content contributed to improved tensile strength and stiffness, whereas higher amylopectin content enhanced flexibility and elongation. Among the various formulations investigated, the blended starch system—particularly the 50:50 ratio of millet to cassava—consistently exhibited superior overall performance. This formulation achieved a balanced combination of tensile strength, strain, and modulus of elasticity, indicating a synergistic interaction between the two starch types. The findings confirm that starch blending is an effective strategy for optimizing the structural and mechanical properties of biodegradable films. Water absorption analysis highlighted a major limitation of starch-based bioplastics, with all samples exhibiting relatively high moisture uptake due to the hydrophilic nature of starch molecules. However, variations between samples indicated that starch source and composition influence water sensitivity, suggesting opportunities for further material improvement. The application of mixture design and optimization techniques provided valuable insights into the relationship between formulation variables and material properties. Although the predictive models showed reasonable agreement with experimental results, some deviations were observed, reflecting the inherent complexity of multicomponent biopolymer systems. This study confirms that locally sourced starches from millet and cassava can be effectively utilized in the development of biodegradable plastics. The results further demonstrate that blending different starch sources significantly enhances material performance, making such systems promising candidates for sustainable packaging applications. However, improvements in water resistance and mechanical strength are necessary to expand their practical applicability.

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