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RESEARCH ARTICLE

EFFECTS OF ACETYLATION, PLASTICIZATION AND KAOLINITE REINFORCEMENT ON MOISTURE AND MECHANICAL PROPERTIES OF CASSAVA STARCH-BASED BIOPLASTIC FILMS

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ABSTRACT

Effects of acetylation, plasticization (ethylene glycol) and filler(kaolinite) reinforcement on the moisture and water absorption characteristics of cassava starch films were investigated at various levels of plasticizer concentration (2, 3, 4, 5 g/100 g of starch), and kaolinite (0, 0.5g, 1 g/ 100 g of starch). The combined effects of the plasticizer and filler concentration on mechanical properties were also examined. Acetylation reduced the gelatinization temperature of native starch from 65 °C to 51 °C improving the processability and flexibility of the films. Moisture absorption of the films increased with higher ethylene glycol content, reaching 38.5% in films with 5 g of ethylene glycol and 0.5 g of kaolinite. Water absorption followed a similar trend, with the highest absorption of 38.3% observed in films with 5 g ethylene glycol. The tensile strength of the films was significantly improved by kaolinite reinforcement, with films containing 1 g of kaolinite exhibiting the highest tensile strength of 10.74 MPa. However, higher plasticizer content reduced tensile strength, indicating a trade-off between flexibility and strength. These results demonstrated the optimal combination of acetylation, plasticization, and kaolinite reinforcement to effectively balance flexibility, strength and resistance to moisture and water.

KEYWORDS

Acetylated Starch; Ethylene Glycol; Kaolinite; Water Absorption; Moisture Absorption; Tensile Strength.

1. INTRODUCTION

Plastic pollution, as a result of petroleum-based plastics, has become a major concern globally, which has led to a search for biodegradable plastics derived from renewable resources. Among the most prominent are starch-based plastics, which are advantageous because starch is readily available and cheaper, and it is biodegradable, especially in tropical countries where cassava cultivation is prominent. However, starch-based films are brittle and moisture-sensitive, which is a result of the high hydrogen bonding of starch molecules. As pointed out, starch-based films are not useful because of their brittleness, which is a result of hydrogen bonding (Krogars et al., 2002 and Liu et al., 2009).

Chemical modification of starch is a common method of improving starch-based films, and acetylation is a common method of modifying starch. As pointed out, acetylated starch has a reduced gelatinization temperature and improved flexibility (Lawal, 2011 and Shogren, 2003).

Although acetylated starch-based films are improved in processability and moisture sensitivity compared to native starch-based films, as pointed out, acetylated starch-based films are still poor in mechanical strength and stability, as pointed out (Bertuzzi et al., 2007; Lawal, 2011 and Shogren, 2003).

In order to improve flexibility and processability, plasticizers are typically integrated into starch-based films. As stated that chain mobility and flexibility are enhanced by the use of polyols, such as glycerol, sorbitol, and ethylene glycol (Talja et al., 2008). Nevertheless, the addition of plasticizers is said to increase the number of hydrophilic sites, as well as the free volume of the polymer network, which makes the films more vulnerable to water uptake, swelling, and the weakening of mechanical strength (Talja et al., 2008 and Liu et al., 2009). Thus, plasticizers improve flexibility, but worsen moisture sensitivity, which is an issue for packaging materials that need to withstand water or humidity.

Reinforcing fillers can help overcome weaknesses caused by plasticizers. Out of all fillers, kaolinite is the best at improving the mechanical strength, the thermal stability, and the water absorption of the starch-based films. Component interactions are probably responsible for the properties of the starch-kaolinite composite films. Because of its layered structure, kaolinite increases the tortuosity of diffusion pathways, hence, the composite's barrier properties are improved because the diffusion of water and small molecules is restricted. Additionally, as illustrated, clay reinforcement can also improve stress transfer in the polymer matrix, and consequently, the tensile strength and integrity of the films (Mbey and Thomas, 2012).

Out of the numerous studies conducted in the field, few studies have looked into the combined effect of acetylation, plasticization, and kaolinite reinforcement in relation to the moisture, water, and alkaline/acid chemical uptake, and the mechanical properties of starch films, while, even fewer have cassava starch as their focus. Since cassava is an abundant and often overlooked material in many developing countries, getting to know the optimization of its modified films will likely result in great advancements in the area of sustainable packaging.

The goal of this research is to determine how different proportions of plasticizer (ethylene glycol) and filler (kaolinite) will affect the moisture absorption, water uptake, acid resistance, alkaline stability, and tensile strength of acetylated cassava starch film. The end result will produce a

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formulation that has good balance between flexible and strong films, while also maintaining their ability to resist degradation within the environment, thus allowing for their use as biodegradable packaging material.

2. Materials and Methods

2.1 Sample Collection/Sample Area

Cassava tuber were purchased from a farm in Ekosodin community Benin City, Edo State. It is located along the longitude 5° 451 and 6° 151 east and latitude 5° 151 and 6° 451 north of the central province of Edo state. Additional materials such as HCl (Reagent 36%- WW), Sodium hydroxide pellets (MOLYCHEM-98% Purity), Acetic Anhydride (APC- 98% Purity), Ethylene glycol (SRL-99% Purity), Kaolinite were sourced from Pyrex-IG Scientific company Benin City, Edo State Nigeria.

2.2 Preparation of Native Starch

Extraction of cassava starch was carried out using method described by (Ezeoha and Ezenwanne 2013). The cassava tubers were mechanically grated after being manually peeled and cleaned with distilled water three times as much water as the shredded cassava was added to the mixture. A coarse sieve and a filter cloth were used to sieve and filter the mixture, respectively. The filtrate was allowed to settle for six hours, then mixed with an equal amount of water and left to settle for a day. At the end of 24 hours, the wet starch was decanted, manually dewatered, and then oven-dried at 105 °C for 4 hours to reduce its moisture content.

$$(\%)\ Starch\ Yield = \frac{weight\ of\ extracted\ starch\ (g)}{weight\ of\ cassava\ tubers} x100 \quad (1)$$

2.3 Preparation of Acetylated Starch

The native starch was chemically modified by acetylation using method reported with slight modification by (Henry 2007). 20 g of starch was dispersed in 100 cm^3 of distilled water and then stirred constantly for 30 minutes. The slurry was adjusted to pH 8.0 with 3% NaOH, and 1.2 g of acetic anhydride was added to the slurry. After the addition of acetic anhydride, the reaction was allowed to proceed for an additional five minutes. The pH of the slurry was adjusted to 4.5 with 0.5 M HCl, and then it was filtered through Whatman No. 1 filter paper. The residue obtained was washed four times with distilled water to remove acids that may be present in the product, and finally air-dried at room temperature. %Yield were calculated using:

%Yield was calculated using:

$$(\%)\ Yield = \frac{weight\ of\ acetylated\ starch\ (g)}{weight\ of\ starch\ (g)}\ x\ 100 \quad (2)$$

Table 1: Experimental Design For Biodegradable Plastic Film Formulation

	Ethylene glycol (Plasticizer) (g)	Kaolinite (Filler) (g)
Acetylated Starch	2	0 0.5 1.0
	3	0 0.5 1.0
	4	0 0.5 1.0
	5	0 0.5 1.0

2.4 Biodegradable Plastic Film Casting

The casting of the biodegradable film was done following the method proposed with slight modification by (Nwaka et al., 2025). Ten gram of the starch acetate powder was weighed in a beaker, then 100 mL of distilled water was added to it. The mixture was stirred at 350 rpm for 10 mins on a magnetic stirrer. Kaolinite powder was then added at different concentration: 0g (0% w/w), 0.5 g (5% w/w), and 1g (10% w/w), and stirred. Ethylene glycol was also added at different concentration (2, 3, 4, 5 g) and stirred at 350 rpm for 15 min. The solution was heated to about 80°C with continuous stirring to form a gel. The slurry was then poured onto a mold, dried in a hot air oven at 50 ^{0}C, and stored at room temperature.

2.5 Physicochemical Properties

The physicochemical properties of the starch acetate were analyzed using standard methods. Parameters like moisture content, pH, solvent solubility, gelatinization temperature and degree of substitution were analyzed.

2.5.1 Moisture Content and pH

The moisture content and pH were analyzed using the AOAC method. A crucible was dried in an oven at 105 °C, then cooled in a desiccator and weighed as (W_0). 2 g of the starch sample was added to the crucible, and the weight was recorded as (W_1). The crucible was then placed in the oven at 105 °C for 6 hours. After drying, it was transferred to a desiccator to cool and subsequently weighed as (W_2). The crucible was returned to the oven for another 6 hours to ensure complete drying. After the second drying period, the crucible was weighed again to confirm weight consistency. The pH was measured by dissolving 2 g of starch in 50 mL of distilled water and using a pH meter.

%Moisture content was calculated using:

$$(\%)\ Moisture = \frac{W1-W2}{W1-W0}\ x\ 100 \quad (3)$$

2.5.2 Solvent Solubility Test

0.3 g of bioplastic film was cut into pieces and placed in a test tube containing 3 mL of ethanol. The solubility was observed at 30 °C after one hour as demonstrated by (Nwaka et al., 2025).

2.5.3 Gelatinization Temperature

1 g of dried starch sample was weighed into a beaker filled with 10 mL of distilled water; the pH of the starch was recorded using a calibrated FP20 Mettler Toledo pH meter before it was subjected to heat using a magnetic stirrer with hot plate. The mixture was continuously stirred while the temperature was monitored. The gelatinization temperature was recorded using a thermometer.

2.5.4 Degree Substitution Of Acetylated Starch (Ds)

The acetyl group (AG expressed as percentage on dry basis) and the degree of substitution (DS) of cassava starch were determined as demonstrated by (Mark and Mehltretter, 1972). 5 g of starch sample was weighed, transferred to a 250 mL conical flask and dispersed in 50 mL distilled water. Few drops of phenolphthalein indicator were added and titrated with 0.1N sodium hydroxide to permanent pink colour. Then 25.0 mL of

0.45N NaOH was added to it and shaken vigorously for half an hour. The stopper and neck of flask was flushed with little distilled water and then the excess alkali was titrated with 0.2N HCl to disappearance of pink colour. A total of 25.0 mL of 0.45N NaOH was titrated as blank. Acetyl group and degree of substitution were calculated as follows:

$$(\%)\ AD = \frac{(Blank-sample)mLx\ M(HCl)\ x\ 0.43\ x100}{weight\ of\ sample} \quad (4)$$

$$DS = \frac{162\ X\ \%Acetyl}{4300-(42\ x\ \%Acetyl)} \quad (5)$$

2.6 Absorption Properties

The absorption properties of biodegradable plastic films are critical in determining their interaction with moisture, and other environmental substances. These films can adsorb water and other polar molecules onto their surface or within their polymer matrix, which affects their mechanical performance and degradation behavior

2.7 Moisture Absorption Resistance

Moisture absorption resistance analysis was carried out as reported with slight modifications by (Nwaka et al., 2025). Moisture absorption resistance was determined by drying the bioplastic films in a desiccator until a constant weight (W1) was achieved, then placing them in a normal atmosphere for 24 hours and weighing them again (W2). The percentage moisture absorption was calculated using:

$$(\%)\ Moisture\ Absorption = \frac{W2-W1}{W2} x100 \quad (6)$$

2.8 Water Adsorption Resistance

Water absorption resistance analysis was carried out as reported by Nwaka *et al*. (2025) with slight modifications. Various samples of the bioplastic films were evaluated by soaking them in water at room temperature for one hour, drying them with cotton pieces, and weighing them. The percentage of water absorption was calculated using:

$$(\%)Water\ absorption = \frac{wet\ \ weight-dry\ weight}{dry\ weight} x100 \quad (7)$$

2.9 Acid Absorption Resistance

Acid absorption resistance analysis was carried out as reported with slight modifications by (Nwaka et al., 2025). Various samples of the bioplastic films were soaked in 1M hydrochloric acid solution, and their weights were recorded after one hour. The percentage of acid absorption was calculated using:

$$(\%)Acid\ absorption = \frac{wet\ \ weight-dry\ weight}{dry\ weight} x100 \quad (8)$$

2.10 Base Absorption Resistance

Base absorption Resistance analysis was carried out as reported with slight modifications by (Nwaka et al., 2025). Bioplastic samples were tested by soaking the films in 1M sodium hydroxide solution and weighing them at regular intervals. The percentage of base absorption was calculated using:

$$(\%)Base\ absorption = \frac{wet\ \ weight-dry\ weight}{dry\ weight} x100 \quad (9)$$

2.11 Characterization

Fourier Transform Infrared (Ftir) Spectroscopy And Thickness Of The Film

The FTIR spectra of native starch and acetylated starch were acquired on a Perkin Elmer FTIR spectrophotometer (Perkin Elmer, Inc., MA, USA) using a potassium bromide (KBr) disc prepared from powdered samples mixed with dry KBr. The spectra were recorded (16 scans) in the transparent mode from 4000 to 400 cm-1, as demonstrated by (Bernardino-Nicanor *et al*., 2017) The thickness of the bioplastic film was determined using the micrometer screw gauge. Each sample was recorded at five different points. The mean value was recorded as the thickness of the bioplastic.

2.12 Mechanical Property

Tensile Strength Of Bioplastic Film

Tensile strength was determined using the Tensile Strength Test Machine TM 2101-T7**,** following ASTM D638 with a maximum force of 10kN**.**

3. Results and Discussion

Table 2: Percentage Yield, Moisture And Ph Of The Extracted Native Starch

Yield (%)	Moisture (%)	pH	Gelatinization temp(°C)
62.3	12.3	6.0	65

Cassava starch extraction processes typically have a moderate extraction efficiency. The starch yield of 62.3% for this process is considered to be moderate. The moisture content of the starch has a large effect on the overall performance of bioplastics and their mechanical properties and biodegradability (Sajjad et al., 2020). The moisture content found in this process is 12.3%, which means there is only a small amount of moisture in cassava starch extract, preventing microbial growth and keeping the bioplastic stable. The pH of cassava starch extract has a value of 6.0, which means it is slightly acid almost to neutral and can be used for food-related bioplastics because a neutral pH prevents unwanted chemical reactions or degradation. The gelatinization temperature (GT) of cassava starch has been found to be consistent at 65 °C by both and the current research (Sajjad et al., 2020). This temperature is important in the processing of starch-based materials, as it indicates the temperature at which starch will experience irreversible changes and be thickened.

Table 3: Degree Of Substitution (Dos), Percentage Yield And Gelatinization Temperature Of Acetylated Starch

Sample	Yield (%)	DOS	Gelatinization temp(°C)
Acetylated starch	61	0.34	51

The yield of native starch after acetylation modification reached 61% which represents a minor decrease from native starch yield because the acetylation procedure resulted in some material loss. The results of this study match the previous findings from which demonstrated that acetylation process decreases yield (Gani et al., 2019). The process involves exchanging hydroxyl groups for acetyl groups which leads to partial material breakdown. The Degree of Substitution (DOS) value of 0.34 shows moderate acetylation level which matches the results from a different research study that proved acetylation can be used to alter starch physical characteristics for different uses (Gani et al., 2019). The acetylated starch maintained its native starch characteristics because the DOS value decreased. The acetylated starch reached a gelatinization temperature of 51°C which represents a substantial decrease from the native starch temperature due to acetylation's effect on starch thermal characteristics. Acetylation adds acetyl groups to starch molecules which decreases the starch chain intermolecular connections that results in lower gelatinization temperature according to (Zhang et al., 2015). The reduction in gelatinization temperature provides advantage for starch-based bioplastic production because it allows for processing at lower temperatures which decreases energy needs while improving material processing abilities.

Table 4: Absorption And Solubility Test On Biodegradable Plastics Films

	Ethylene glycol(g)	Kaolinite (g)	%Water absorption resistance	%Moisture absorption resistance	%HCl absorption resistance	%NaOH absorption resistance	Ethanol absorption resistance

Table 4 (cont): Absorption And Solubility Test On Biodegradable Plastics Films							
	2	0	20.0	9.0	20.0	Soluble	Insoluble
		0.5	33.3	7.5	26.6	Soluble	Insoluble
		1.0	25.0	7.0	23.8	Soluble	Insoluble
Acetylated starch	3	0	26.5	20.1	28.4	Soluble	Insoluble
		0.5	28.5	10.3	29.5	Soluble	Insoluble
		1.0	24.3	10.0	24.1	Soluble	Insoluble
	4	0	31.3	28.0	35.5	Soluble	Insoluble
		0.5	28.9	17.0	36.1	Soluble	insoluble
		1.0	28.3	12.0	29.4	Soluble	Insoluble
	5	0	38.3	25.0	43.0	Soluble	Insoluble
		0.5	38.5	21.4	37.2	Soluble	Insoluble
		1.0	32.1	14.0	33.3	Soluble	Insoluble

The moisture absorption of the films from Table 4 depended on both the ethylene glycol and kaolinite effect. The moisture absorption increased with higher ethylene glycol content because of its hygroscopic properties and the resulting increase in free volume within the starch matrix. The addition of kaolinite to the plasticizer system decreased moisture absorption because the filler created a more complex pathway for water vapor to travel while it strengthened the polymer matrix. The films with higher ethylene glycol content absorbed more moisture, and the films with 5 g of ethylene glycol and 0.5 g of kaolinite showed the highest moisture absorption at 38.5%. The data shows that ethylene glycol makes the films more hydrophilic because it creates more amorphous spaces in the starch matrix which helps the films maintain moisture. The films absorb more moisture, which shows they cannot be used in applications that need materials with low moisture sensitivity. Water absorption patterns followed a similar trend to moisture absorption, with the films absorbing up to 38.3% water when 5 g of ethylene glycol was used. The films with lower plasticizer content showed an increase in water absorption when compared to the other films. The films show strong water absorption, which makes them vulnerable to water damage, thus restricting their ability to function in areas with high humidity for extended periods. The film samples were analyze to measure their acidic and alkaline solution absorption capabilities, which showed that the films dissolved in 1M NaOH but remained a insoluble in ethanol. The acid uptake results showed a mid-level absorption across all samples, with the highest absorption occurring in the 5 g ethylene glycol and 0.5 g kaolinite combination, which reached 43.0%. The results show that the films will experience greater alkaline degradation, which will reduce their operational capacity in areas that face basic environmental conditions.

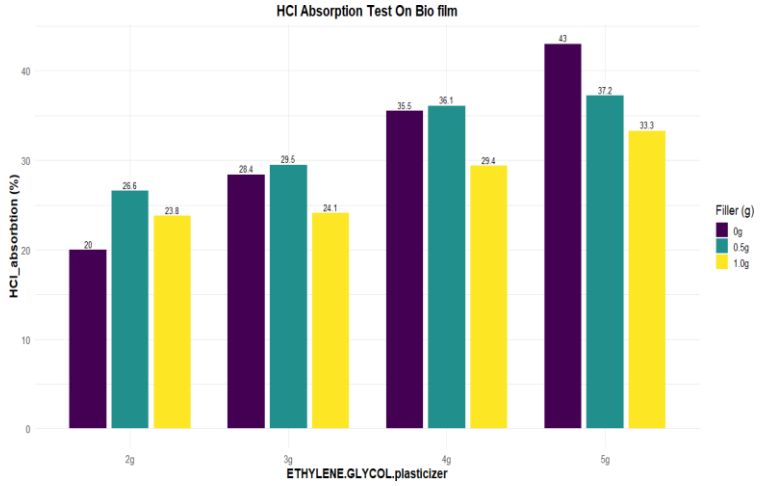

Figure 1: HCl absorption test on Bioplastic Film

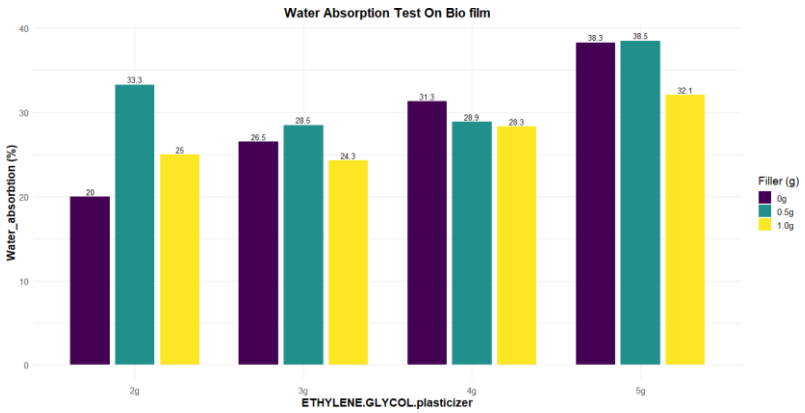

Figure 2: Water absorption test on Bioplastic Film

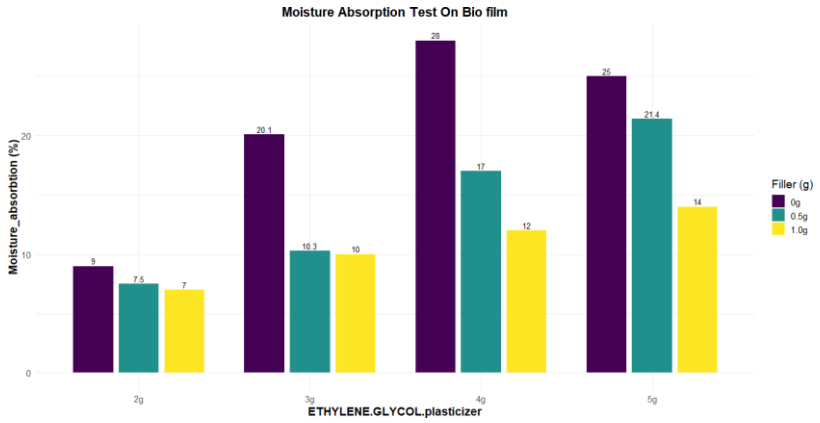

Figure 3: Moisture absorption test on Bioplastic Film

3.1 FTIR Spectroscopy

FTIR (Fourier Transform Infrared) was use to confirm functional groups in both native starch and acetyl-modified starch. The FTIR spectra of native starch and acetylated (modified) starch can be found in Figures 4 and 5, respectively. The unique structural differences shown for each sample indicate that the starch molecules underwent a successful chemical modification process.

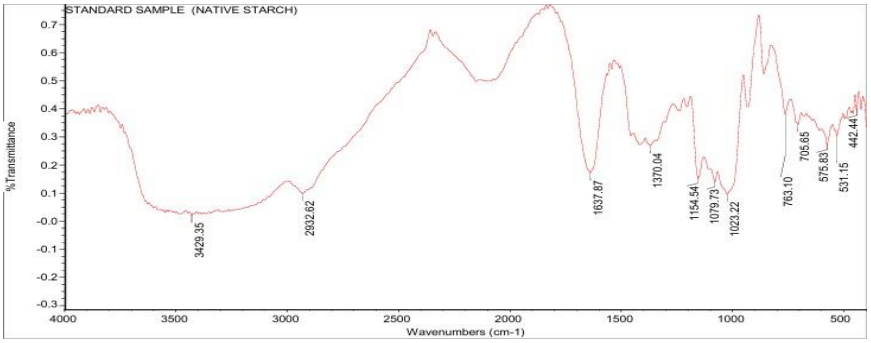

Figure 4: FTIR Spectra of native starch

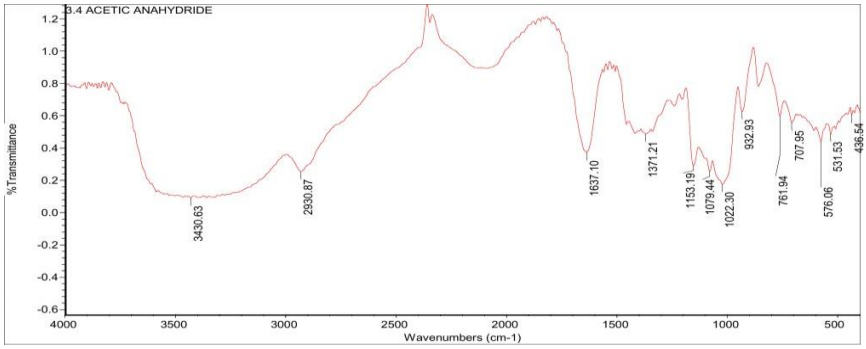

Figure 5: FTIR Spectra for acetylated starch

The original native starch spectrum (Figure 1) showed the same kinds of absorption bands that are generally associated with polysaccharides. The broad band at around 3400 cm-1 is consistent with the O–H stretching vibrations from the hydroxyl groups involved in intra and intermolecular hydrogen bonding. The weak band that is approximately 2920 cm-1 is due to the stretching C–H vibrations from the methylene groups. The strongest absorption within the range of 1150 to 1000 cm-1 is attributed to the stretching vibrations of the C–O–C and C–O from the glycosidic linkage. A weak band that is located around 1640 cm-1 is because of the bound water molecules.

The acetylated starch spectrum (Figure 2) had a peak that was very distinct at approximately 1740 cm-1; the peak is the carbonyl C=O stretching vibration of the ester group, and confirms the acetylation. A peak was also evident in the range of 1230 to 1260 cm-1; this peak results from the C–O stretching of the acetyl ester linkage. The O–H stretching band at approximately 3400 cm-1 exhibited a decrease and narrower width in comparison to the original spectrum, indicating replacement of the hydroxyl groups with the acetyl moieties and a corresponding decrease in the amount of intermolecular hydrogen bonding. Minor changes in the intensity of the bands in the range of 1000 to 1150 cm-1 are also an indication of some modification to the starch backbone. The spectra comparison made it obvious that the starches' acetylation chemical modifications were made successfully.

Table 5: Bioplastic Formulation; Colour And Thickness

Ethylene glycol (g)	Kaolinite (g)	Colour	Thickness (mm)
2	0	White	0.413
	0.5	Milky	0.512
	1.0	Milky	0.518
3	0	White	0.412

Table 5 (cont): Bioplastic Formulation; Colour And Thickness			
	0.5	Milky	0.520
Acetylated starch	1.0	Milky	0.525
4	0	White	0.420
	0.5	Milky	0.521
	1.0	Milky	0.522
5	0	White	0.421
	0.5	Milky	0.530
	1.0	Milky	0.531

As shown in Table 5, increasing ethylene glycol concentrations resulted in changing the color of each of the films from white to milky. The discoloration of the film may be due to the effect that the plasticizers have on film transparency. The addition of ethylene glycol to the formulation has a small, but statistically significant, positive effect on the thickness of each of the films produced; thus, it can be stated that plasticizers affect the density and structure of the film. The thickness of each of the films produced by initial formulation yielded a fairly similar thickness (i.e., 0.413 – 0.531 mm), suggesting that differences in formulation would not produce significant changes in the overall thickness of any of the films.

Table 6: Tensile Strength Of Bioplastic Film		
Ethylene Glycol(g)	Kaolinite(g)	Tensile Strength (MPa)
2	0	3.05
	0.5	6.62
	1	10.74
3	0	2.00
	0.5	4.40
	1	10.05
4	0	0.79
	0.5	1.79
	1	2.36
5	0	0.91
	0.5	1.38
	1	1.67

The tensile strength increased with kaolinite additions, indicated by the highest tensile strength film measured at 10.74 MPa, created by the combination of 2 g ethylene glycol and 1 g kaolinite. This illustrates that high filler loading with low plasticizer content produces the greatest tensile strengths.

Kaolinite's re-enforcing impact increases the rigidity of the starch films. Too much plasticizer content (>5 g ethylene glycol) reduces tensile strength. Therefore, even though plasticizers give flexibility to the starch films, they reduce mechanical integrity of the films at higher concentrations. The optimal tensile strength was achieved with a balanced combination of 2 g ethylene glycol and 1g kaolinite.

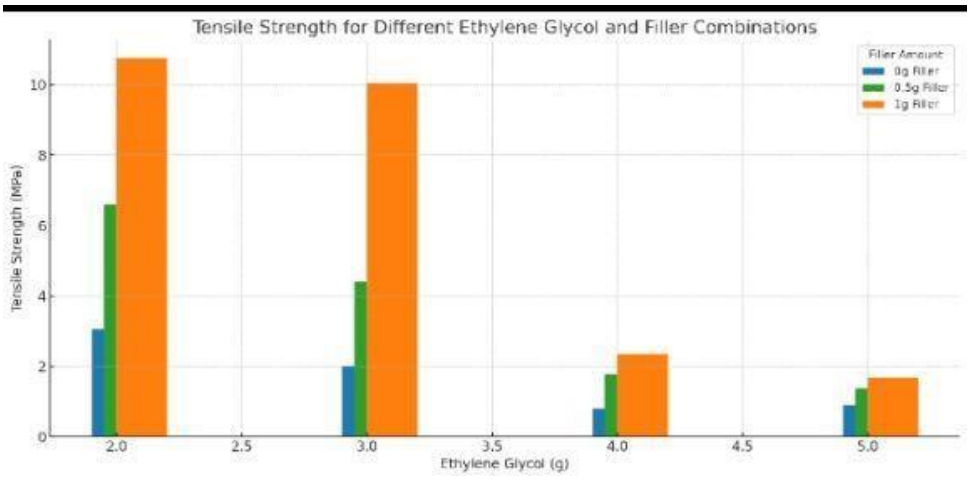

Figure 6: Bar Chart of tensile strength of bioplastic film at different concentration of ethylene glycol and kaolinite

4. Conclusion

This study shows that cassava starch can be modified in such a way that it can be used to make films that are easy to process and have a good balance between strength, flexibility and water resistance. Cassava starch was modified by acetylating, plasticizing with ethylene glycol, and reinforcing with kaolinite. The acetylation of the native starches reduced their gelatinization temperatures, while improving the flexibility of the films; however, the yield of the acetylated starch was also slightly lower. Plasticizing with ethylene glycol increased the moisture absorption of the films, which indicates that they are hydrophilic and therefore more likely to degrade when exposed to water. When kaolinite was added in the formulation, the tensile strength and water resistance of the films improved; the films containing 1g of kaolinite had the best mechanical properties.

Kaolinite reinforcement improved tensile strength significantly, while ethylene glycol plasticization improved flexibility, but decreased mechanical strength. The best tensile strength was achieved when combining 2 g of ethylene glycol with 1g of kaolinite, which would be suitable for use in applications where mechanical integrity is critical; however, a higher level of ethylene glycol would be recommended for use in applications where flexibility is more important than strength.

The results also demonstrate the versatility of cassava starch, a widely available and sustainable resource, in the production of functional, eco-friendly bioplastics. This study contributes to the advancement of starch-based films that can reduce plastic pollution, offering a promising alternative to conventional plastics derived from petroleum.

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