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Thermal and Biodegradation Behaviour of Acetylated Cassava Starch Bioplastics Films Reinforced with Kaolinite

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Abstract

This study investigates the thermal stability and biodegradability of acetylated cassava starch-based bioplastics reinforced with kaolinite and plasticized with ethylene glycol. Cassava starch was extracted and modified through acetylation, confirmed using Fourier Transform Infrared (FTIR) by the appearance of a carbonyl (C=O) peak at 1740 cm^{-1} in the FTIR spectrum. The acetylation process (degree of substitution 0.34) reduced the gelatinization temperature of starch from 65°C to 51°C, indicating enhanced processability. The films were prepared by varying the concentrations of ethylene glycol (2–5 g) and kaolinite filler (0 –1 g). Thermogravimetric Analysis (TGA) values with onset degradation temperature ranging from (180°-220°C) for 2 g ethylene glycol, (55°C-70°C) for 5 g ethylene glycol showed a consistent increase in thermal stability as kaolinite content increased for varying ethylene glycol content. Differential Thermal Analysis (DTA) also revealed that kaolinite enhanced the thermal stability of the films by an increase in the Tg from (60°C -70°C) for 2g ethylene glycol to (90°C-80°C) for 5g ethylene glycol content. Biodegradability was evaluated using soil burial tests, and the films were found to degrade significantly within four weeks, with the formulation of 5 g ethylene glycol and 0.5 g kaolinite showing the highest biodegradability with 70% degradability at week 3. These results indicate that the acetylated cassava starch-kaolinite composites offer a promising approach to sustainable, biodegradable packaging materials with improved thermal performance.

Keywords: Acetylated Cassava Starch, Kaolinite, Biodegradable Bioplastics, Thermal Stability.

1.0 Introduction

The accumulation of non-biodegradable plastics in the environment has led to significant concerns about pollution and waste management. This has driven global research into the development of bioplastics made from renewable resources, which offer an environmentally friendly alternative to conventional petroleum-based plastics. Among these, starch-based bioplastics are of particular interest due to their biodegradability, renewability, low cost, and availability as demonstrated by [1]. However, native starch is inherently hydrophilic, which results in films that are brittle, moisture-sensitive,

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and thermally unstable, limiting their practical applications as demonstrated by [2].

Chemical modification of starch, such as acetylation, has been widely studied to overcome these limitations. Acetylation involves replacing hydroxyl groups in starch with acetyl groups, making the material more hydrophobic, flexible, and thermally stable as demonstrated by [3]. This modification enhances the performance of starch-based bioplastics in applications where moisture resistance and thermal stability are essential. Acetylated starch films also exhibit improved flexibility, making them suitable for a broader range of applications, including food packaging as demonstrated by [4]

Incorporating fillers into starch-based films is another strategy used to improve their mechanical and thermal properties. Kaolinite, a naturally occurring clay mineral, is commonly used as a filler in bioplastics. Studies have shown that kaolinite improves the thermal stability and mechanical strength of starch-based bioplastics by acting as a reinforcement within the polymer matrix. The addition of kaolinite increases the onset degradation temperature and reduces the water absorption rate, which is a critical factor for the durability of bioplastics as reported by [5]. The interaction between kaolinite and starch also enhances the biodegradability of the films by creating a more compact structure, which facilitates microbial degradation during biodegradation as demonstrated by [6].

The choice of ethylene glycol as a plasticizer in this study is to improve the flexibility and processability of the acetylated cassava starch films. Ethylene glycol is a widely used plasticizer in starch-based bioplastics because it effectively reduces the brittleness of the material by lowering its glass transition temperature. This allows the bioplastic films to be more flexible and easier to process, which is essential for manufacturing films that are suitable for flexible packaging applications as demonstrated by [2]. Ethylene glycol also plays a role in enhancing the mechanical properties of the bioplastic by improving its elongation at break and tensile strength.

Cassava starch was chosen as the starting material due to its high yield and low cost. It is also readily available in many tropical countries, making it an attractive option for bioplastic production. Additionally, cassava starch is well-suited for acetylation, and its modification has been shown to produce bioplastics with desirable properties for various applications as demonstrated by [1]. By combining acetylated cassava starch with kaolinite and ethylene glycol, this study aims to develop a biodegradable bioplastic with improved thermal stability and mechanical properties, while maintaining the environmental advantages of starch-based materials.

This study focuses on evaluating the thermal stability and biodegradability of acetylated cassava starch-based bioplastics reinforced with kaolinite and plasticized with ethylene glycol.

Thermal stability was assessed through Thermogravimetric Analysis (TGA) and Differential Thermal Analysis (DTA), while biodegradability was evaluated using soil burial tests. By examining these two critical properties, the research aims to identify formulations that offer both environmental benefits and practical performance for sustainable packaging solutions.

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) and 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 methodology described by [7]. 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 was calculated using:

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

2.3 Preparation of Acetylated Starch

The native starch was modified via acetylation using methodology reported by [8] with slight modification. 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 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 Preparation of Biodegradable Plastic Film and Casting of films

The preparation was conducted following a refined modification of the method proposed by [9]. 10 g of the acetylated starch powder was weighed in a beaker, to which 100 mL of distilled water was added. The mixture was stirred at 350 rpm for 10 minutes on a magnetic stirrer. Kaolinite powder was then added at different weights: 0g (0% w/w), 0.5 g (5% w/w), and 1g (10% w/w), and stirred. Ethylene glycol was also added at different weights (2, 3, 4, 5 g) and stirred at 350 rpm for 15 min. The solution was heated to approximately 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^{O}C, and stored at room temperature.

2.5 Fourier Transform Infrared (Ftir) Spectroscopy

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 [10].

2.6 Thermogravimetric/Differential Thermal Analysis (TGA/DTA)

TGA/DTA was conducted using the PerkinElmer TGA 4000 (Netherlands) to assess the thermal stability and decomposition behavior of the bioplastic film. This test provided insights into the materials resistance to heat and the efficiency of the composite's formulation and filler reinforcement.

2.7 Biodegradability Test

The biodegradable behavior of the bioplastic film samples was determined using soil burial decomposition test. Biodegradability test on the film samples were carried out using method proposed by [9] with slight modifications. Bioplastic films (3 inches by 3 inches) were weighed (W_1), buried in moist soil at a 3-inch depth for one week, and reweighed (W_2). The percent weight loss was calculated using:

$$(\%)Weight\ Loss = \frac{W1-W2}{W1} x100 \qquad (3)$$

3.0 Results and Discussion

3.1 FTIR

The various functional groups of the developed bioplastic films were analyzed using FTIR, as shown in Figure 1 and 2. The FTIR spectra observed the characteristic peaks of extracted and modified starch both were observed, confirming the modification process.

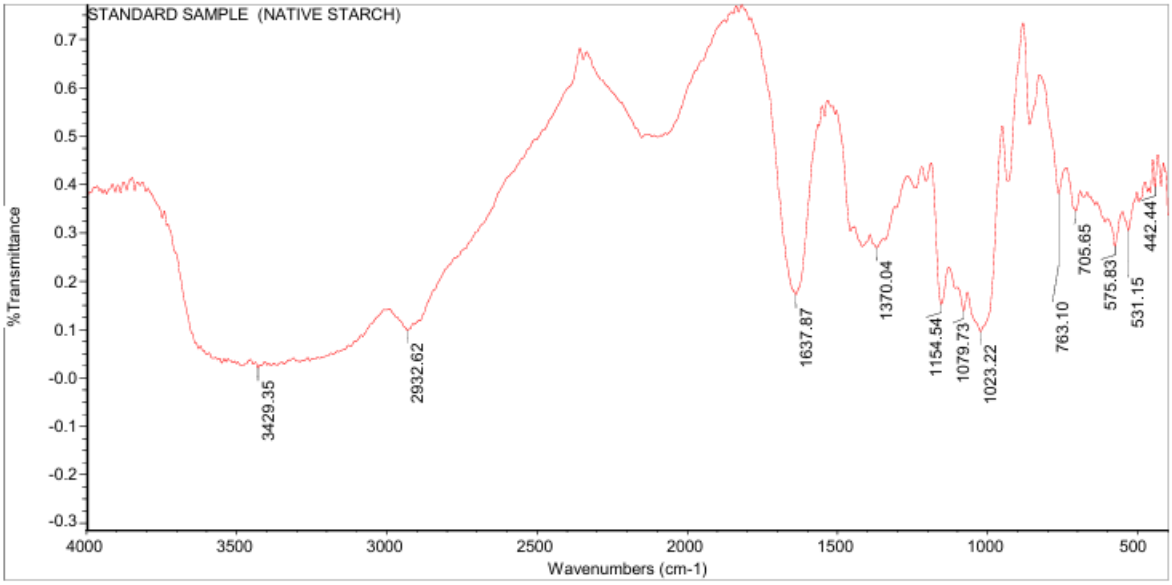


Figure 1: FTIR Spectrum for Native Starch

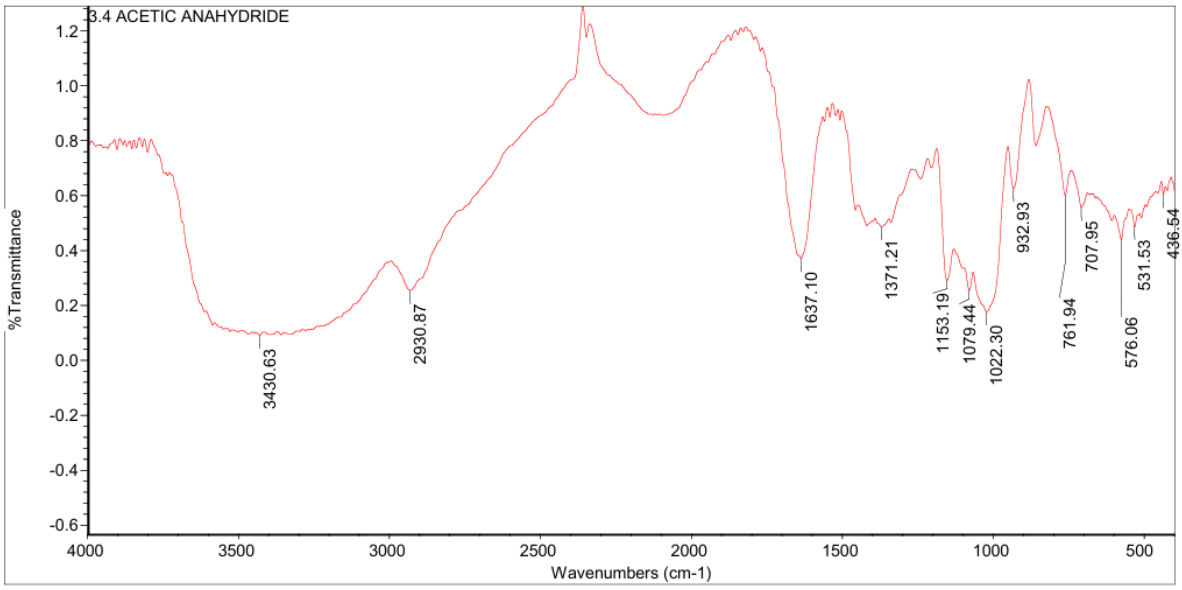


Figure 2: FTIR Spectrum for Acetylated Starch

From Figure 1, the native starch spectrum exhibited characteristic absorption bands typical of polysaccharides. A broad band around 3400 cm^{-1} corresponds to O–H stretching vibrations of hydroxyl groups involved in intra- and intermolecular hydrogen bonding [11]. The weak band near 2920 cm^{-1} arises from C–H stretching vibrations of methylene groups [12] while the strong absorption in the 1150–1000 cm^{-1} region represents C–O–C and C–O stretching of the glycosidic linkages [12]. A weak band around 1640 cm^{-1} is attributed to bound water molecules.

The acetylated starch spectrum in Figure 2 displayed a distinct absorption peak around 1740 cm^{-1}, which is attributed to the carbonyl (C=O) stretching vibration of ester groups, confirming acetylation. An additional peak at 1230–1260 cm^{-1} corresponds to C–O stretching of the acetyl ester linkage. The observed decrease and narrowing of the O–H stretching band intensity at around 3400 cm^{-1} reflect the replacement of hydroxyl groups by acetyl moieties, thereby reducing intermolecular hydrogen bonding. Slight intensity variations in the 1000–1150 cm^{-1} region also indicate modifications to the starch backbone [13].

Comparative analysis of the spectra clearly demonstrates that the acetylation chemical modifications were successfully achieved.

Table 2: Bioplastic Film Degradability

	Ethylene Glycol(g)	Kaolinite (g)	% degradability Week 1	% degradability Week 2	% degradability Week 3	% degradability Week 4
Acetylated Starch	2	0	22.5	43.0	63.0	Degraded
		0.5	17.0	32.0	52.0	Degraded
		1.0	10.0	20.1	40.1	Degraded
	3	0	25.5	49.0	69.0	Degraded
		0.5	18.6	35.2	55.2	Degraded
		1.0	12.0	22.0	52.0	Degraded
	4	0	31.0	58.0	78.0	Degraded
		0.5	22.0	42.0	62.0	Degraded
		1.0	17.0	32.0	52.0	Degraded
	5	0	34.0	61.0	81.0	Degraded
		0.5	26.5	50.0	70.0	Degraded
		1.0	23.5	45.0	65.0	Degraded

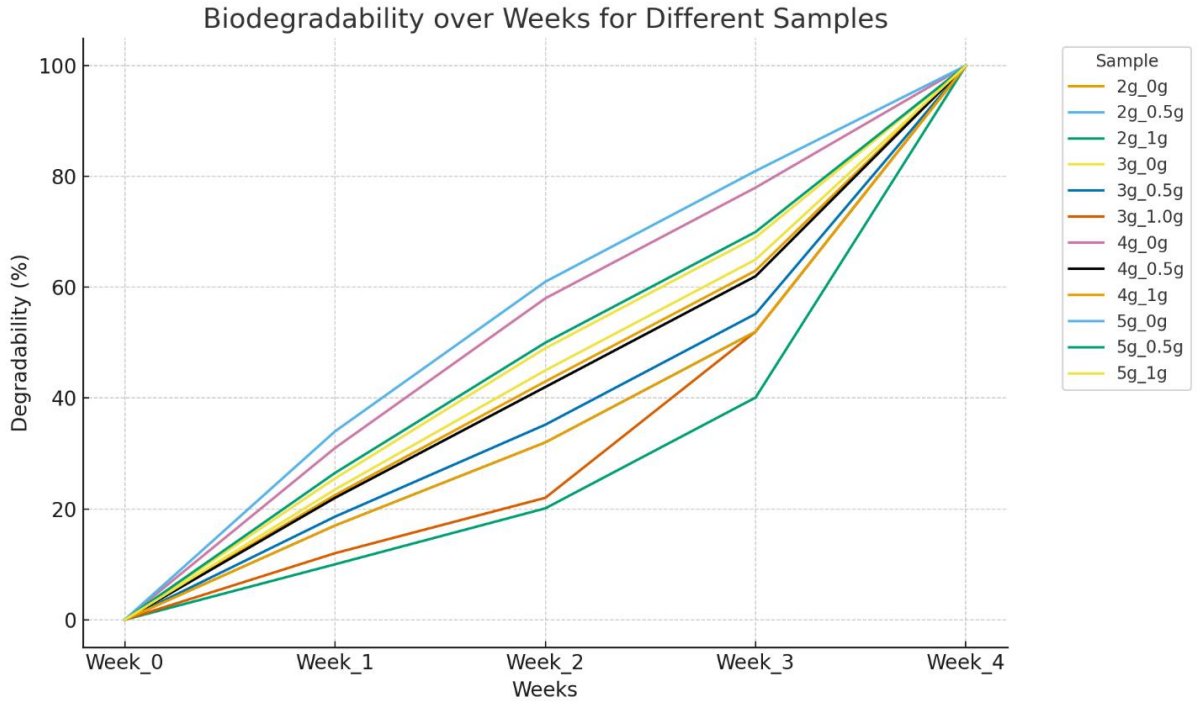


Figure 3: Effect of Acetylation on the Biodegrability of Starch Film

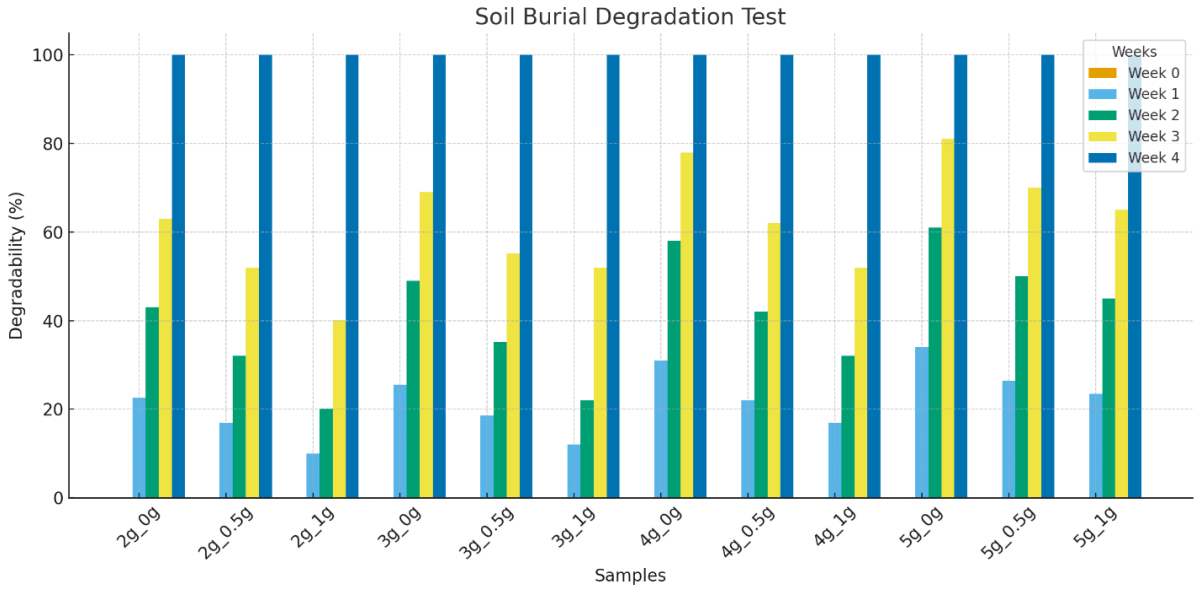


Figure 4: Effect of ethylene glycol and kaolinite on the biodegradability of the starch film at varying concentration.

The soil burial biodegradation test results, illustrated in the bar charts of Figures 4 and Table 2, show a clear increase in biodegradability for all samples over the four weeks. At the start of the test (Week 0), none of the samples had degraded (0 % biodegradability), as expected, since no exposure to the soil environment had yet occurred. From Week 1 onward, each sample began to decompose steadily, with the percentage of material broken down increasing week by week. By Week 4, most samples were almost completely degraded, with some formulations achieving full (100 %) biodegradability by the end of the testing period.

One key factor influencing the degradation rate was the amount of ethylene glycol used as a plasticizer in the films. Samples formulated with a higher ethylene glycol content (for example, 5 g) degraded more rapidly than those with a lower ethylene glycol content (such as 2 g) [14]. This trend suggests that increasing the plasticizer content improves biodegradability. A plausible explanation is that ethylene glycol, as a plasticizer, makes the polymer matrix more flexible, thereby facilitating easier penetration and breakdown by soil microorganisms. As a result, the films with 5 g of ethylene glycol showed very high biodegradability by Week 4. In contrast, the films containing only 2 g of ethylene glycol remained less degraded over the same timeframe [15].

Another factor examined was the effect of adding kaolinite filler on the biodegradation process. The presence of kaolinite influenced the early-stage degradation rates. In the initial weeks of burial, the films without any kaolinite filler (0 g) tended to degrade faster than those containing kaolinite (0.5 g or 1 g). This suggests that adding kaolinite

initially hindered degradation, possibly because the filler increased the rigidity of the bioplastic matrix, making it somewhat more difficult for microbes to penetrate the material [16]. However, this effect was only temporary. By Week 4, all samples, including those with kaolinite, had reached approximately 100% biodegradability. Thus, although kaolinite can slow down the initial degradation process, it does not prevent the bioplastic films from ultimately breaking down completely under soil burial conditions.

3.2 (TGA) and (DTA) of Bioplastic Films

The TGA/DTA analysis confirms that through chemical modifications and the incorporation of fillers and plasticizers, the thermal properties of cassava starch films can be tuned to optimize performance for specific applications, particularly in biodegradable plastic production

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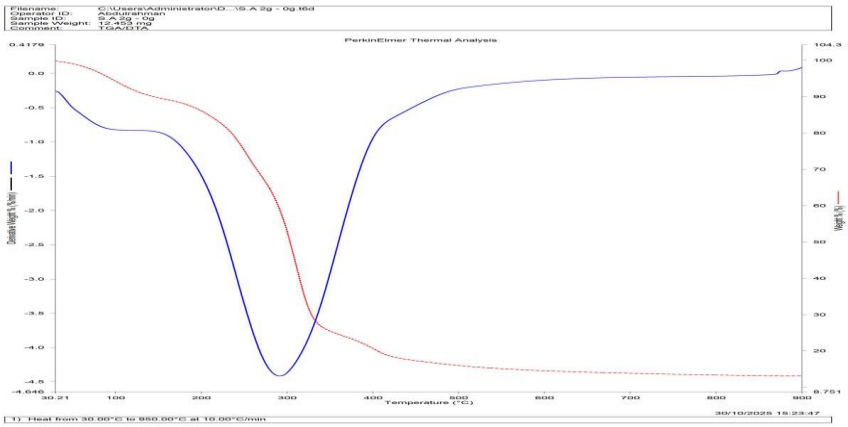


Figure 5: Effect of modification of the starch blend with 2 g ethylene glycol and 0 g kaolinite on the thermal stability.

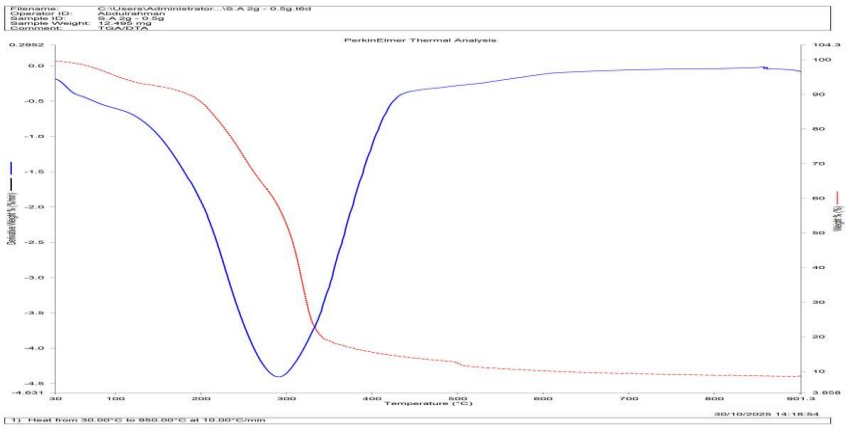


Figure 6: Effect of modification of the starch blend with 2 g ethylene glycol and 0.5 g kaolinite on the thermal stability.

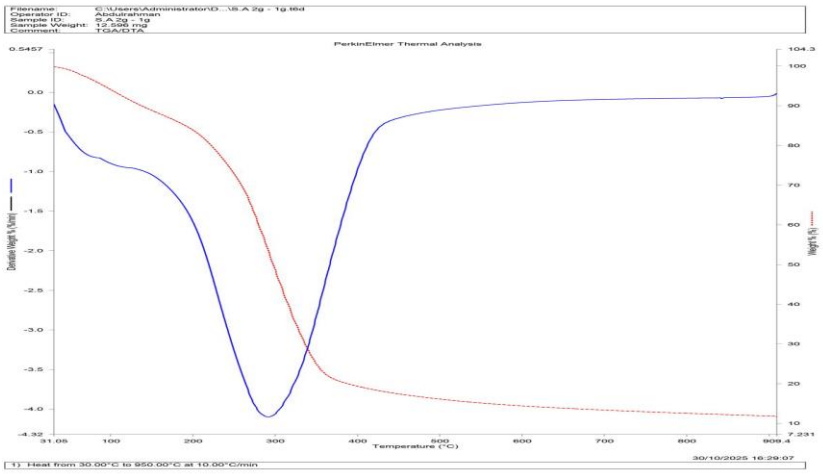


Figure 7: Effect of modification of starch blend with 2 g of ethylene glycol and 1 g of kaolinite on the thermal stability

From Figure 5, formulations containing 2 g of ethylene glycol without kaolinite show degradation beginning around 180°C and peaking at approximately 240°C. Although the film is relatively flexible, it exhibits poor thermal stability, largely due to ethylene glycol weakening the starch acetate network and promoting early thermal breakdown. From Figure 6, when 0.5 g of kaolinite is introduced, the onset temperature of degradation shifts upward to about 200°C, indicating that the filler improves film rigidity while reducing moisture uptake [17]. The stabilizing effect becomes even more pronounced when the kaolinite concentration is increased to 1

g, at which point degradation begins at approximately 220°C, as shown in Figure 7. At this stage, the film decomposes more slowly, and the enhanced structural reinforcement provided by kaolinite becomes evident [18].

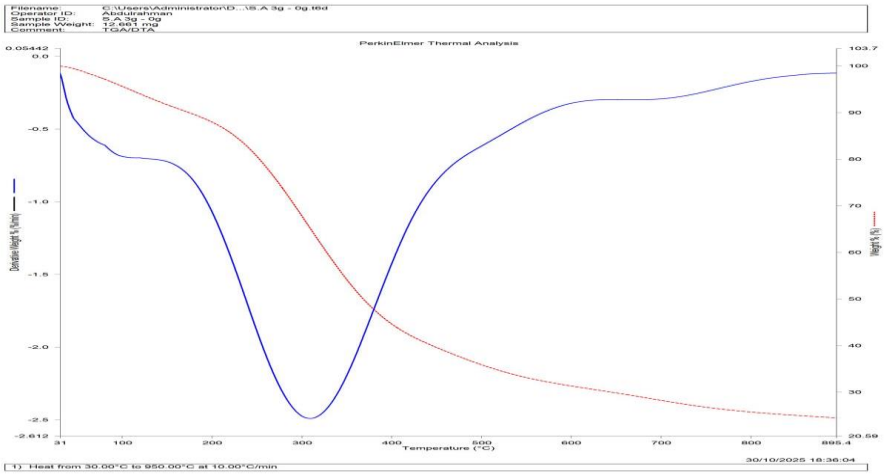


Figure 8: Effect of the modification of starch blend with 3 g ethylene glycol and 0 g kaolinite on thermal stability.

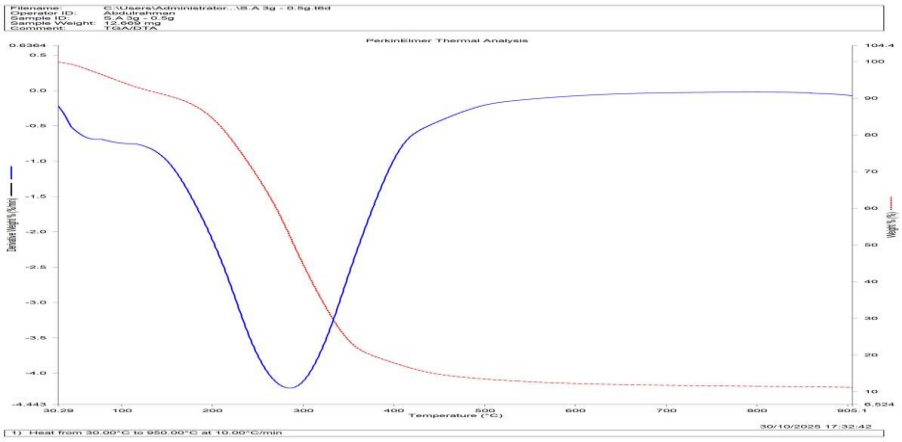


Figure 9: Effect of modification of starch blend with 3 g ethylene glycol and 0.5 g kaolinite on the thermal stability.

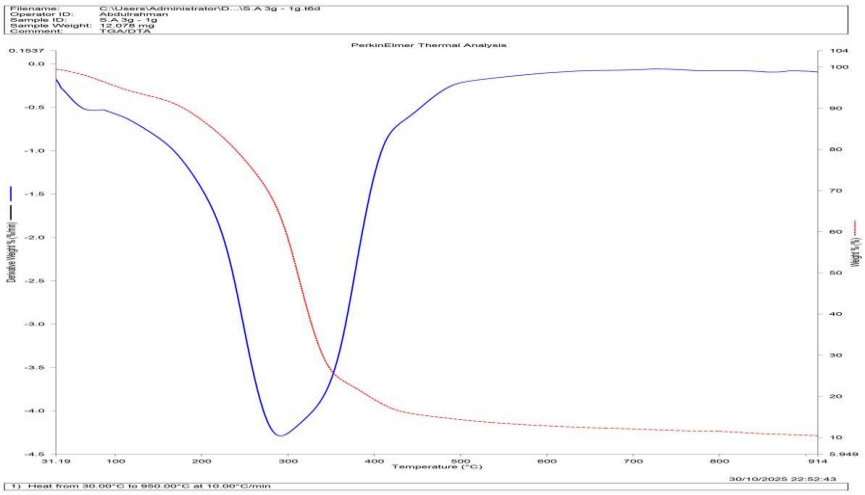


Figure 10: Effect of modification of starch blend with 3 g ethylene glycol and 1g kaolinite.

As the ethylene glycol content increases in Figures 8, 9, and 10, the trend reverses. The sample containing 3 g of ethylene glycol without kaolinite shows a significant decline in thermal stability, with degradation beginning at only 150°C. This drop is attributed to higher moisture absorption and the excessive softening effect of the plasticizer, which disrupts the starch acetate matrix [19]. However, when 1 g of kaolinite is added to this formulation, thermal stability is restored, and the onset of degradation temperature once again reaches about 220°C. This confirms that kaolinite can counteract the destabilizing effects of high plasticizer content [20].

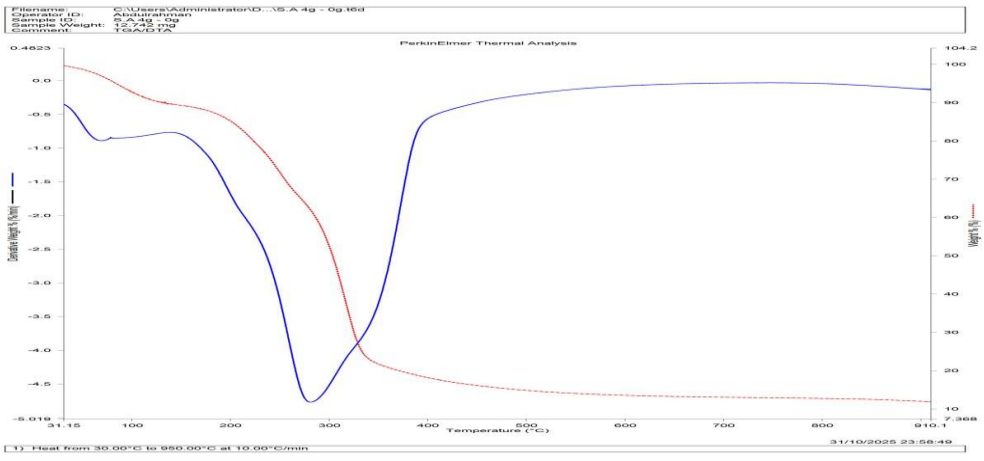


Figure 11: Effect of modification of starch blend with 4 g ethylene glycol and 0 g kaolinite on the thermal stability.

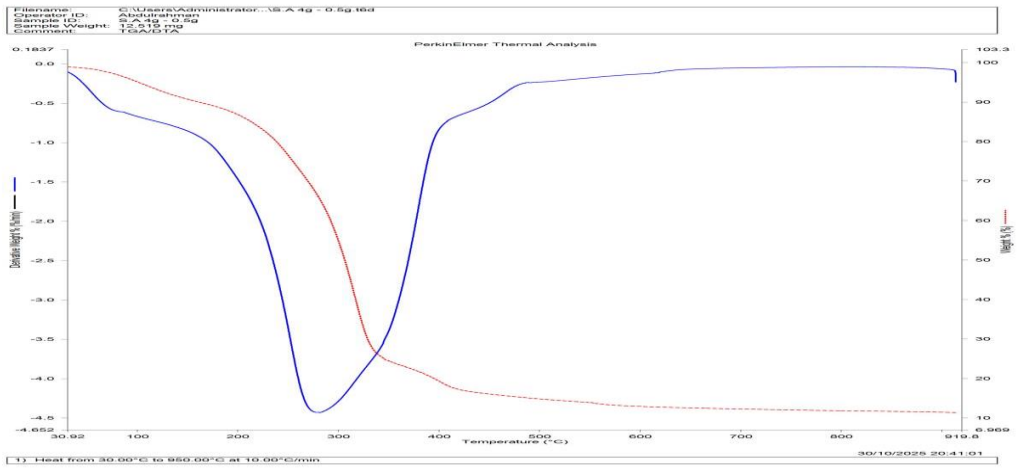


Figure 12: Effect of modification of starch blend with 4 g ethylene glycol and 0.5 g kaolinite on the thermal stability.

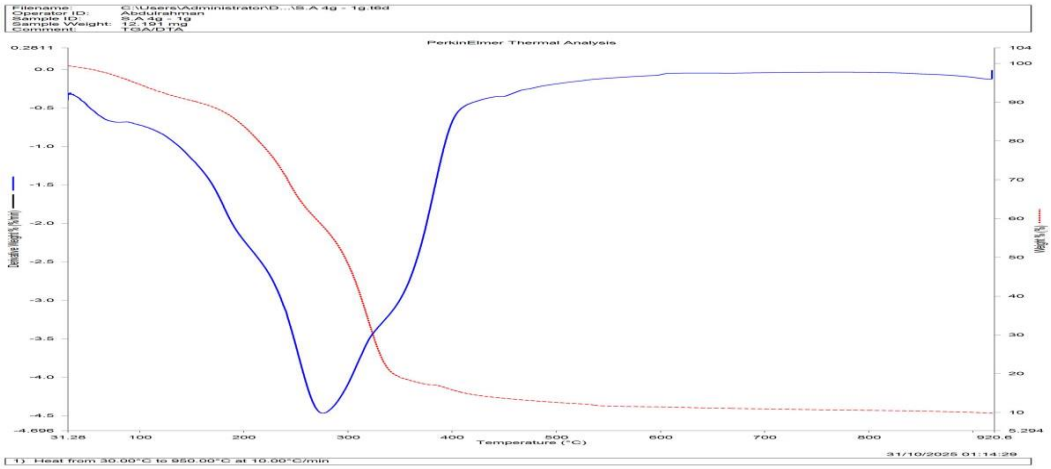


Figure 13: Effect of modification of starch blend with with 4 g ethylene glycol and 1 g kaolinite on the thermal stability.

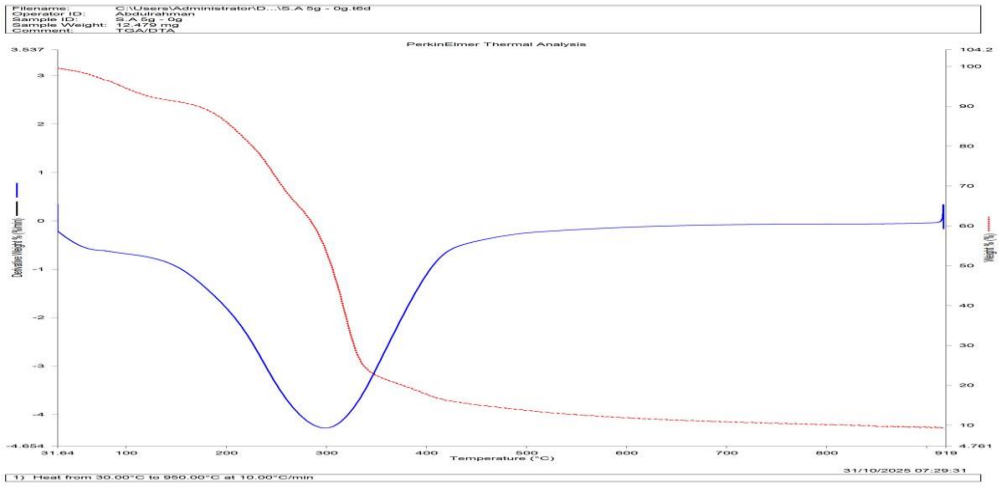


Figure 14: Effect of modification of starch blend with 5 g ethylene glycol and 0 g kaolinite on the thermal stability.

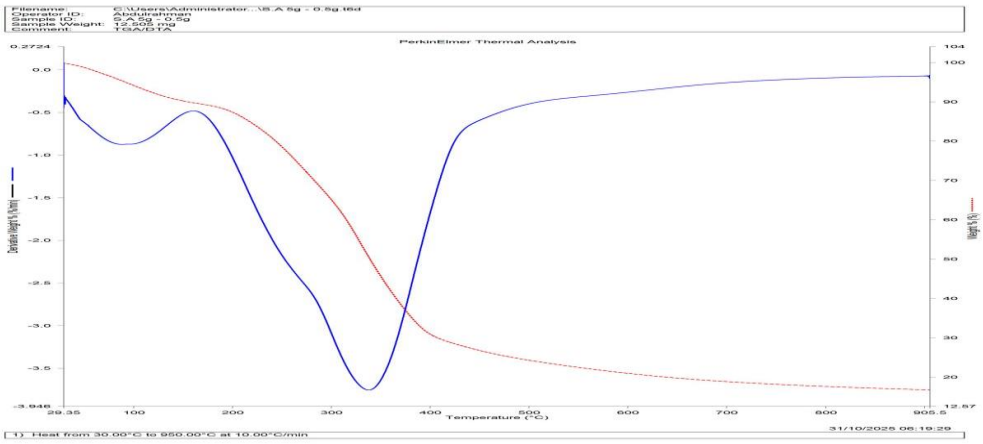


Figure 15: Effect of modification of starch blend with 5g ethylene glycol and 0.5 g kaolinite on the thermal stability.

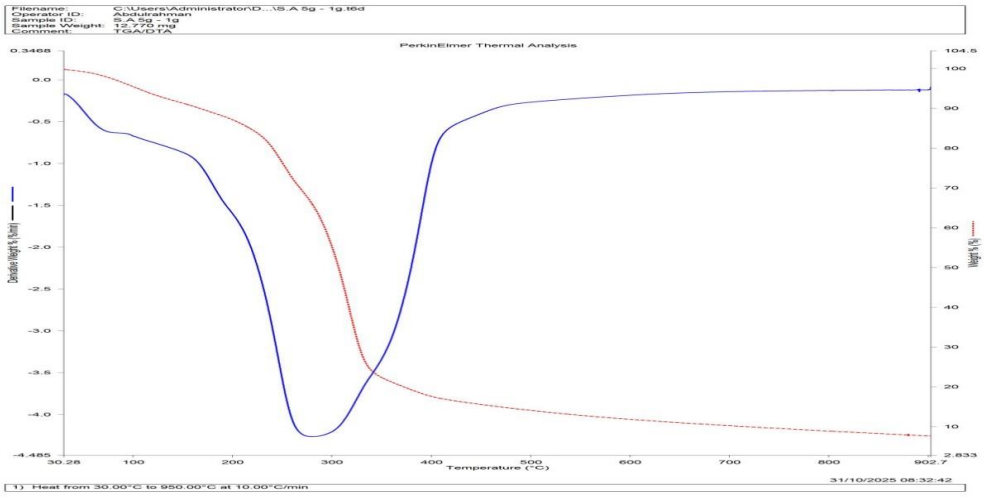


Figure 16: Effect of modification of starch blend with 5 g ethylene glycol and 1 g kaolinite on the thermal stability

The influence of kaolinite becomes more obvious in formulations containing 4 g and 5 g of ethylene glycol. For instance, the combination of 4 g of ethylene glycol with 1 g of kaolinite yields a moderate improvement in stability, with degradation commencing at 75 °C and reaching maximum decomposition at approximately 260^{o} C with 52% of the sample degraded which is higher than that of 2 g of ethylene glycol with 1 g of kaolinite with 40.1% degradation. Despite the high plasticizer content, the kaolinite reduces moisture absorption and enhances rigidity. A similar pattern is observed in the 5 g ethylene glycol 1 g kaolinite film, where the film remains flexible but demonstrates better heat resistance than its kaolinite-free counterpart. Moisture absorption drops significantly in this case as well, confirming that the filler effectively reduces the bioplastic's hygroscopic [20].

Overall, the TGA results highlight two key observations. First, kaolinite consistently enhances the thermal stability of the starch acetate films by increasing the onset degradation temperature and reducing moisture absorption. Its reinforcing effect becomes more visible as the filler content increases. Second, although ethylene glycol enhances flexibility, it also reduces the thermal resistance of the films, particularly at higher concentrations. This delicate interaction between the plasticizer and filler determines the material's overall thermal performance.

The DTA results further support these findings by showing how the films behave during heating. In the 2 g ethylene glycol film with no kaolinite, the endothermic peak at about 60°C reflects the softening of the plasticizer, while the early exothermic peak near 240 °C confirms poor stability. When kaolinite is added such as in the 3

g ethylene glycol + 1 g kaolinite sample both the softening and decomposition temperatures shift to higher values. The endothermic transition occurs near 70°C, and the exothermic degradation peak appears at around 240°C, demonstrating that the filler delays the breakdown of the material and increases its glass transition temperature. The 5 g ethylene glycol 1 g kaolinite sample reinforces this trend, exhibiting an endothermic peak at approximately 80°C and an exothermic peak at around 270°C. These shifts indicate that, despite the high plasticizer content, kaolinite strengthens the heat resistance of the films while still preserving flexibility [20].

Taken together, the combined TGA and DTA results clearly indicate that kaolinite is a highly effective reinforcing agent for enhancing the thermal stability of acetylated cassava starch bioplastics. While ethylene glycol plays a crucial role in providing flexibility, its presence also makes the films more vulnerable to heat, especially at higher levels. The optimal balance is observed when kaolinite is used in conjunction with a moderate plasticizer content, such as the formulation with 2 g of ethylene glycol and 1 g of kaolinite. This combination achieves a high onset degradation temperature of about 220°C and shows a gradual decomposition pattern, indicating stronger structural resilience. The reduced moisture absorption in kaolinite-containing samples also supports more controlled biodegradation, further enhancing their sustainability. Ultimately, the findings suggest that the synergy between kaolinite and ethylene glycol can be fine-tuned to produce biodegradable starch-based films that are both thermally stable and mechanically suitable for packaging applications (The scope of this study did not determine the final % weight remaining at the end of the heating program nor was it compared with standard reference values).

Table 4: Thermal Properties of Bioplastic Film

Ethylene glycol (g)	Kaolinite (g)	Onset Temperature (°C)	Max Degradation Temperature (°C)	Tg (°C)	Moisture Absorption (%)	Thermal Stability	Observation
2	0	180	240	60	High	Low	Flexible, low thermal stability, quick decomposition
	0.5	200	250	65	Moderate	Moderate	Kaolinite improves stability and rigidity
	1	220	250	70	Low	High	Enhanced rigidity, good heat resistance

3	0	150	220	50	High	Low	High moisture absorption, low thermal stability
	0.5	200	230	55	Moderate	Moderate	Improved stability with kaolinite, better heat resistance
	1	220	240	60	Low	High	Enhanced thermal stability and rigidity
4	0	55	200	70	High	Very Low	High moisture absorption, low thermal resistance
	0.5	70	250	75	Moderate	Moderate	Kaolinite improves rigidity and thermal stability
	1	75	260	80	Low	High	Excellent rigidity, improved thermal stability
5	0	55	170	90	Very High	Very Low	Highly flexible but thermally unstable
	0.5	65	200	85	High	Low	Improved rigidity but low thermal stability
	1	70	210	80	Moderate	High	Highly flexible but more heat-resistant due to kaolinite

Conclusion

The study demonstrates that acetylated cassava starch bioplastics, reinforced with kaolinite and plasticized with ethylene glycol, achieve improved thermal stability and biodegradability. The incorporation of kaolinite enhances thermal stability by acting as a heat barrier, while varying ethylene glycol content influences flexibility and biodegradation rates. The most stable formulation, with 1 g kaolinite and 2–3 g ethylene glycol, showed increased thermal tolerance without compromising biodegradability. The bioplastics fully biodegraded within four weeks under soil burial, with kaolinite formulations initially slowing degradation, but ultimately not affecting complete biodegradation. These findings highlight the potential for developing sustainable, biodegradable packaging materials that balance thermal durability and eco-friendly disposal with a % degradability of 52%.

Acknowledgement

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