

Dynamic Mechanical Analysis of Degradable Starch (Potato-Corn) - Clay Composite Polymer Films

Nyarone Chrisphine Odhiambo¹; Osamong Gideon Akou²

^{1,2}Department of Physics, Chuka University, Chuka, Kenya

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Abstract: The synthesis and characterization of biodegradable starch-clay composite films produced from potato and corn starches reinforced with montmorillonite clay was the primary focus in the work. Dynamic mechanical analysis (DMA) tested the mechanical and viscoelastic characteristics of these composites. Accurate readings were obtained by calibrating and using the DMA model 2980 in multi-strain mode. A casting technique produced several ratios of starch and clay, showing that the inclusion of clay greatly improved mechanical strength, tensile characteristics, and elongation at break of the composite films above pure starch films. The ideal clay content turned out to be between 25% and 50%; at this point the composites showed maximum performance. Good dispersion and interaction between the clay and starch matrix can help to explain the improved tensile strength and flexibility. The different peaks in storage and loss moduli suggest a complicated response of the composites to temperature variations, therefore predicting improved energy dissipation capacity. Furthermore appropriate for uses in biodegradable packaging and food goods, the starch-clay composites shown better mechanical integrity and thermal stability. The paper emphasizes the need of more investigation on the interaction between mechanical performance and clay content to maximize these formulations for useful purposes.

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I. INTRODUCTION

Rising interest is around starch-based composites, which substitute renewable origin and biodegradability for conventional polymers. Although the advantageous qualities of potato and maize starches in film production have been investigated, their appropriateness for different uses is limited due of problems including brittleness, low water resistance, and insufficient thermal stability (Ma *et al.*, 2008). To improve the mechanical and barrier qualities of starch-based materials, several filler like nanoclay have been used to reduce these restrictions. Recent studies by Souza *et al.*, (2022) shows that adding clay particles to the starch matrix greatly improves the structural stability of the film, therefore the composite is more appropriate for useful purposes including packaging. This work aims to investigate the viscoelastic characteristics of potato-corn starch composite films enhanced with clay, thereby clarifying the mechanical and structural enhancements obtained by means of clay incorporation.

An efficient method for evaluating the viscoelastic characteristics of polymers, dynamic mechanical analysis (DMA) offers important new perspectives on material stiffness, damping, and thermal transitions across several temperature and frequency ranges (Menard, 2008). The peaks of the $\tan \delta$ curve in dynamic mechanical analysis (DMA)

investigations define the glass transition temperature (T_g), a necessary phase transition in which polymer materials move from a stiff, glassy state to a more flexible, rubbery one. This method has great sensitivity and is especially good at spotting such changes. DMA testing assesses the dynamic modulus and damping coefficient properties upon amorphous phase transition from crystalline structures. Since the mechanical characteristics of polymer films show more variability than specific thermal transitions, dynamic mechanical analysis (DMA) is the recommended approach for estimating the glass transition temperature (T_g). This work aims to investigate the mechanical characteristics of clay-based potato-corn starch composites in respect to important uses in packaging and other sectors needing biodegradable materials.

Stratified structures and high aspect ratios of clay nanoparticles improve interfacial adhesion between the filler and the polymer matrix, therefore increasing the mechanical integrity of the composite (Park *et al.*, 2003). Moreover, evaluating the environmental effect of these starch-based composites depends on knowing their degradation behavior since the addition of clay could affect both mechanical durability and rates of biodegradability (Kaczmarek *et al.*, 2019). A substance is said to be elastic if its storage modulus exceeds its loss modulus, therefore conserving more energy. Should the loss modulus surpass the storage modulus, the material behaves essentially as a viscous substance, akin to a

liquid, meaning that it discharges more energy than it retains (Venudharan *et al.*, 2016). While knowing their degrading behavior in environmentally friendly uses, the aim is to enhance the elastic capabilities of starch composites by including clay. Recent research underline the need of improved manufacturing procedures (Wu *et al.*, 2023) since they underline the need of managing the dispersion of clay inside the starch matrix to maximize these features. This work adds to the growing body of knowledge on sustainable materials by stressing the advantages of starch-clay composites in industrial use where mechanical performance and environmental sustainability are critical especially in making the food packaging bags.

II. MATERIALS AND METHODS

➤ Materials

Both pure potato and corn powders were purchased from Kenya Starch Powder Directory. Montmorillonite 20A powder (clay) was bought from Kenya Kaolin Supplies and Manufacturers.

➤ Synthesis

This work produced starch-clay composite films utilizing a casting technique (Gutiérrez *et al.*, 2015; Mali *et al.*, 2005). Starch-clay solutions were first made by dissolving as described in Table 1 specific amounts of clay, maize starch, and potato starch. Also made for comparison were control samples of starch films free of clay.

Water, clay particles, potato starch, and corn starch were mixed to guarantee complete mixing. Then, while the mixture was always stirring, 0.2 M hydrochloric acid (HCl) solution was added. The mixture heated to 90°C and agitated for 60 minutes using a lab homogenizer pump (Rezvanian, K, 2023).

Glycerol was used at 20% relative to the weight of the dried starch post-initial heating to improve the flexibility of the films (Ben *et al.*, 2022). The mixture's translucent, gel-like consistency upon additional heating indicated total starch gelatinization. Then, while stirring for one to two minutes, the solution was gradually neutralized by the sodium hydroxide (NaOH).

The gel was uniformly placed to produce a thin layer on a fresh glass plate following neutralization. It was then put in a heating cabinet and dried at 90°C. Carefully taken from the glass plate, the films were kept in airtight containers at room temperature for additional viewing following drying.

Before surface examination, the films underwent surface cleaning by means of a 0.03 M nitric acid (HNO₃) solution, so removing surface contaminants. Ten samples in all were made using different ratios of clay, maize, and potato starch as described in Table 1 to find the ideal mix producing the best mechanical and physical characteristics in the starch-clay composite films. With changes to include glycerol as the plasticizer in place of sorbitol, the approach tightly followed the technique used by Mathew & Dufresne (2002) for starch nanocomposites.

Table 1 Mixing Ratios of Potato (P), Corn (Cn) and Clay (C)

Sample	Potato	Corn	Glycerol (g of glycerol/100g of the Sample)	Clay	Sample Code
1	1	0	20	0	P
2	1	0	20	1	PC
3	1	3	20	0	PCn
4	1	3	20	4	PCnC
5	1	1	20	0	PCn1
6	1	1	20	4	PCnC1
7	3	1	20	0	PCn2
8	3	1	20	4	PCnC2
9	0	1	20	0	Cn
10	0	1	20	4	CnC

III. RESULTS AND DISCUSSION

➤ Synthesis Results

The synthesis of the starch-clay composite films was successfully achieved through a solvent casting method,

resulting in visually distinct samples at varying clay contents. The synthesized films were examined for their color, texture, and mechanical properties and representative photos of the samples are presented in Figure 1.

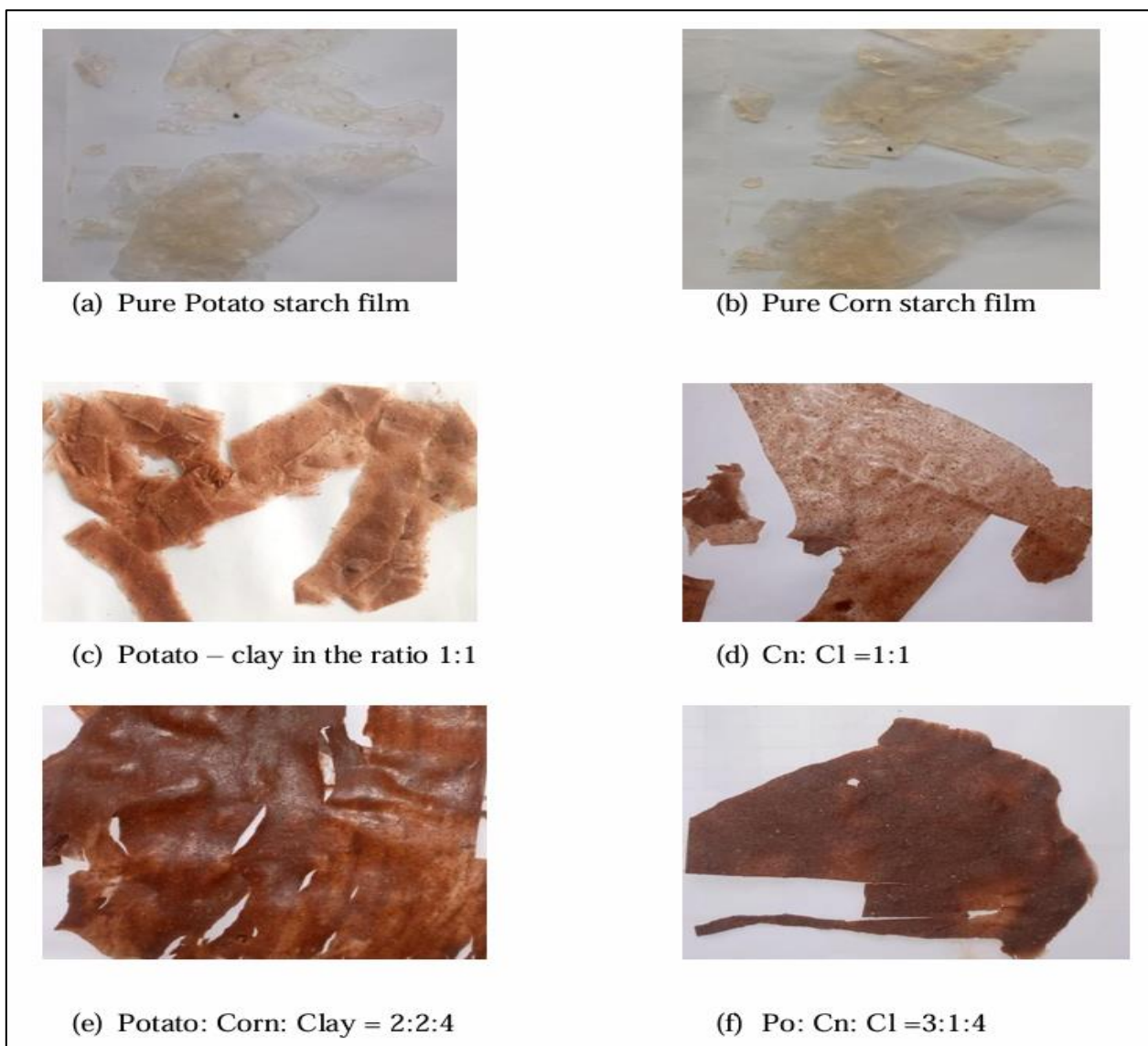


Fig 1 Visual Appearance of Synthesized Starch-Clay Composite Films

The material illustrated in Figure 1 (a) is characterized as a pure potato starch film. It has a translucent to slightly opaque look, a smooth texture, and the presence of visible air bubbles. In contrast, Figure 1 (b) illustrates a maize starch film, which, while also translucent, features a glossier surface and a higher density, perhaps due to its heavier composition. The maize starch film seems unique, with a brighter texture and more surface defects compared to the potato starch film. This difference in texture and appearance can be due to the inherent qualities of maize starch, which has been demonstrated to create denser and more brittle films compared to potato starch (Jiménez *et al.*, 2012; Tharanathan, 2003). Figures 1(c), 1(d), 4(e) and 1(f) illustrate the effect of increasing clay content on the color and opacity of the films.

The pure starch film is translucent and off-white, whereas the addition of clay results in progressively opaque layers. This phenomena correlates with findings by Tang *et al.*, (2008), who found that the addition of clay into starch-based films promotes opacity and improves mechanical capabilities due to the creation of a nanocomposite structure. Notably, a 1:1 ratio of starch (Po) to clay (Cl) creates a film

with uniform color distribution and texture, suggesting an ideal balance between the two components. Similar results have been reported by Rhim *et al.*, (2013), who highlighted the function of clay in increasing the barrier characteristics and structural integrity of biodegradable films. The research carried out by Chekh *et al.*, (2021) indicates that this homogeneity is probably related to the balanced clay content, which reduces aggregation and promotes better film development. With a white tone, the sample with 25% clay (Figure 1 (f)) showed a notable degree of clay dispersion inside the starch matrix, in line with the expected results for efficient filler integration as recorded by Monteiro *et al.*, (2017).

Apart from color, tactile study helped to evaluate the texture of the films. Whereas the pure starch film exhibited a smooth surface, the films with more clay concentrations had an obviously rough texture. The higher roughness caused by clay particles on the surface could improve the film's barrier characteristics (Souza *et al.*, 2022). Moreover, the films showed different degrees of fragility; the pure starch film was especially more brittle and prone to cracking under pressure,

whereas the films including clay showed increased flexibility and resistance to fracture. Indicating that the clay particles help to contribute to the composite's toughness, the 25% clay sample (Figure 1 (f)) showed notable handling enhancement and exhibited resistance to bending without fracture.

The results coincide with earlier research showing improved mechanical characteristics in biopolymer composites including clay reinforcement (Chen *et al.*, 2021). The clay improves interfacial bonding between starch and the filler, thereby clarifying the enhancement in texture and reduction in fragility and so enhancing stress distribution within the material (Matos *et al.*, 2019). The pure starch film was fragile and prone to breaking under stress since it showed poor flexibility and handling qualities. By contrast, films based on clay showed improved flexibility and greater resistance to breakage. The 50% clay film (Figure 1(e)) could bear bending without cracking and showed notable improvement in handling. The increase in toughness suggests that, as observed by Marouf *et al.*, the clay particles help to distribute stress better throughout the matrix, therefore lowering the concentration of stress points that could cause cracks or fractures.

But given possible agglomeration effects seen in some formulations, too much clay could induce declining mechanical property returns (Jange, 2022). Agglomeration might produce weak areas in the matrix, therefore compromising its homogeneity and hence mechanical power. According to the literature, too high clay content may cause aggregation, so compromising the integrity of the material instead of enhancing it (Rashad, 2018). This was verified in several samples during the experiment since higher clay content produced films more prone to uneven surfaces and fracturing. Thus, reaching the intended mechanical improvements depends on an enough concentration of clay. Future studies looking at the relationship between mechanical performance and clay content will help to find ideal formulations for useful purposes. Especially in 25–50% clay content, the effective synthesis and assessment of starch-clay composites shown notable mechanical improvement in the films. More investigation is advised to improve the formulations and investigate alternate fillers or cross-linking agents that could enhance the features of these biopolymer composites for different uses.

➤ Storage Modulus (E')

Dynamic mechanical analysis (DMA) shows that films made of starch-clay composite show higher storage modulus in increasing clay content. The effective fortification of the clay generates the improvement in mechanical properties, thereby strengthening the general structural integrity of the films. The results on the storage modulus and their consequences are covered in the next parts. With composite films showing improved stiffness and load-bearing capacity as the clay content rises from 0% to 50% (Pech-Cohuo *et al.*, 2024), the clay percentage clearly affects the storage modulus. From 25 MPa (pure TPS) to 60 MPa (Pech-Cohuo *et al.*, 2024), the elastic modulus rose showing that thermoplastic starch films reinforced with 5% clay showed a notable mechanical improvement. Clay improves mechanical

and barrier properties, therefore these composites fit for uses in sustainable food packaging (Pech-Cohuo *et al.*, 2024; Riyajan, 2024). Enhanced mechanical and barrier properties help to extend the shelf-life of food products, as shown by Cassava starch/clay compositions used in banana preservation (Riyajan, 2024). Although the improvement of storage modulus with increasing clay content is beneficial, it is important to assess the related trade-offs, including the effect on the flexibility and processability of the films, which may limit their practical application in many food packaging situations.

From figure 2, the sample for a mix of potato, corn and clay in the ratio 1:1:2 demonstrated the highest storage modulus, peaking at approximately 1338 MPa at a temperature of 36°C.

Attributed to the effective dispersion of clay particles inside the starch matrix, the rise in modulus indicates an increase in the stiffness and structural integrity of the material (Llanos *et al.*, 2021). Between 45°C and 90°C the storage modulus stayed unchanged. The material falls in this range as a glassy or quite stable rubbery compound. The homogeneous storage modulus indicates that the polymer chains show rather rigidity with low molecular mobility or relaxation (Roland, 2011). Lack of notable thermal mobility helps the material to maintain its structural integrity.

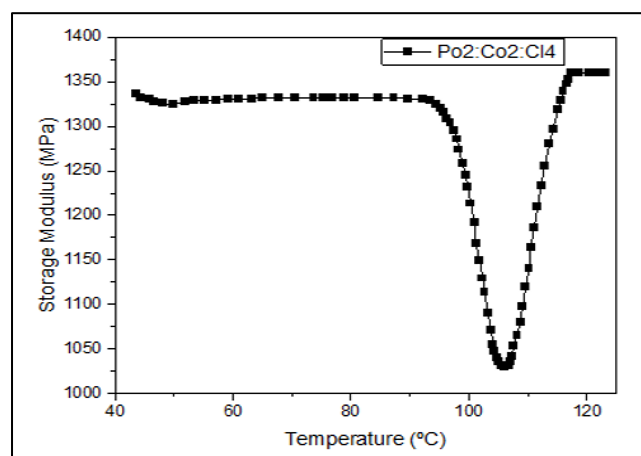


Fig 2 Storage Modulus for a Mix of Potato, Corn and Clay in the Ratio 1:1:2

Between 90°C and 106°C the storage modulus showed a drop. At 106°C, the storage modulus dropped greatly from 1325 MPa to 1025 MPa, indicating a shift from a largely solid-like to a more fluid-like condition. This phenomena could be connected to the start of a phase transition, such the glass transition temperature (T_g), or to the beginning of softening resulting from thermal expansion. Rising molecular mobility (Zhang *et al.*, 2020) lowers the deformation tolerance of the material. The storage modulus of the sample shown a notable rise from 1025 MPa to 1355 MPa within the temperature range of 106°C to 115°C.

Rising temperatures enable molecular rearrangement (Liu *et al.*, 2019), thereby the change in material stiffness is ascribed to network formation or crosslinks. Several elements

can help secondary peaks in the storage modulus of biopolymer composites, including those made of potato and maize starches mixed with clay, to arise. These thermal effects could stabilize the network architecture, thereby enhancing mechanical characteristics. Along with the possibility for extra phase transitions or relaxation processes inside the composite (Wang *et al.*, 2021), this encompasses the complex interactions between the starch matrix and the clay particles. Clay can change the starch matrix's molecular dynamics, thereby producing distinct mechanical reactions at different temperatures (Gao *et al.*, 2018). The secondary peaks (Chen *et al.*, 2020) could show the variation in the composite system where localized components show different degrees of molecular mobility and thermal stability.

Various underlying causes can be blamed for secondary peaks in the storage modulus of starch-clay composites; each one reflects the complex interactions among the component. One possible answer is the existence of numerous relaxation systems. By including clay nanoparticles into the polymer matrix, its homogeneity can be disturbed and localized areas with different relaxation durations generated. Variations in viscoelastic responses to stress across various sections of the material could show up as secondary peaks because of fluctuation in relaxation behavior (Lee *et al.*, 2020).

One possibility is phase separation. Incorporating clay could cause insufficient dispersion, which would cause microphase separation inside the composite. This creates areas with particular mechanical properties that respond differently to particular pressures. Secondary peaks in the storage modulus could show the particular mechanical contributions of every phase by occurring during the phase transition of the material (Kim & Park, 2021).

Other important elements relate to the orientation and strengthening of clay nanoparticles inside the starch matrix. Changing clay content causes nanoparticles to align or agglomerate, therefore improving stiffness in particular areas. The areas of increased stiffness could influence the mechanical characteristics of the composite, thereby producing extra peaks in the storage modulus highlighting the influence of these reinforced sites (Zhang *et al.*, 2019).

Variations in temperature within the starch or the composite ultimately help to explain the development of later peaks. Variations in temperature can influence polymer chain mobility, therefore changing the mechanical response of the material. Transitions including the glass transition and other relaxation mechanisms may show secondary peaks, therefore reflecting changes in the viscoelastic properties of the material resulting from heat stimuli (Matos *et al.*, 2019).

Studies on other biopolymer composites have found such comparable events. Attributing this to the effect of nanoparticles on local viscoelastic characteristics, Lee *et al.*, (2020) found several peaks in the storage modulus of polymer nanocomposites. Kim and Park (2021) also found, in their dynamic mechanical analysis, that the organization and distribution of clay within a polymer matrix produced different mechanical phases, therefore producing secondary

peaks. Zhang *et al.*, (2019) underlined the importance of phase separation generated by clay in starch-based composites since these separations significantly influence the mechanical characteristics of the composite, thereby generating several relaxation modes.

From Figure 6, it can be shown from the primary curve that as temperature rises, the storage modulus values fall (Zhang *et al.*, 2020). The value dropped as the temperature rose to 95°C (Liu *et al.*, 2019). This was as a result of the film becoming softer. The sharp drop for the values between 42°C and 80°C was as a result of the viscoelastic nature of the material (Roland, 2011).

This phenomena, unique of viscoelastic materials, reduces the modulus (Zhang *et al.*, 2020) by means of a change from a stiff, glassy state to a more malleable and rubbery state brought about by temperature increase. Within the spectrum defined by the notable drop noted between 42°C and 80°C, the material undergoes the glass transition temperature (T_g). The mobility of the polymer chains rises greatly in this phase, so the modulus is much reduced. As confirmed by similar results in the literature, comparable polymer systems show a clear reduction in modulus, usually connected with the T_g of the material. Aouissi (2024) observed a similar rather significant drop in the storage modulus in polymer combinations with T_g values within this spectrum. The results seem to match the T_g for Po1: Cn1, meaning that the material's viscoelastic behavior spans 95°C to 136°C, hence producing a rather stable storage modulus.

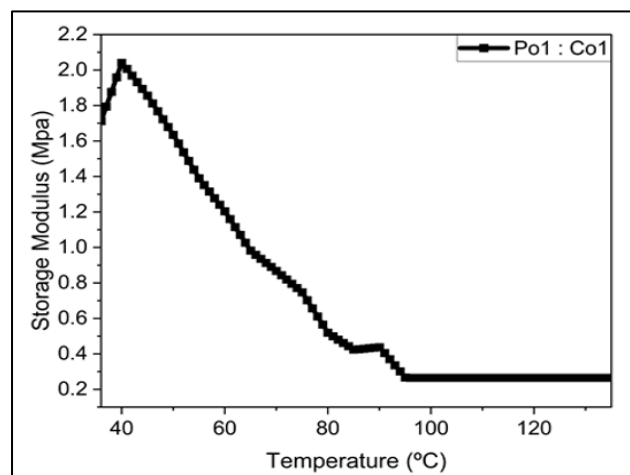


Fig 3 Storage Modulus for a Mix of Potato and Corn in the Ratio 1:1

The storage modulus swings in this range about 0.2 MPa. The storage modulus considerably reduces and stabilizes at about 0.2 MPa between 90 and 100°C. This drop is probably related to the thermal softening of the potato-corn starch mix, which causes the material to become less stiff. The mixture reaches a constant condition inside this temperature range marked by little structural stiffness. This pattern suggests that the gelatinization of starches from both potato and maize components has greatly reduced the material's elastic capacity, most likely resulting from Comparable trends in investigations into starch-based

materials show that gelatinization and heat softening greatly lower mechanical stiffness (Mali *et al.*, 2005).

Figure 4 shows, in MPa, the storage modulus of a composite material having a 3:1 ratio of maize starch to clay as a function of temperature (°C). From 36°C to 145°C, the storage modulus, which indicates the material's stiffness and ability to elastically store energy during deformation, showcases somewhat large fluctuations. The storage modulus starts at about 47.5 MPa at 36°C, suggesting a considerably higher beginning stiffness. With increasing temperature, the storage modulus shows a peak behavior; then, it gradually declines. This phenomena corresponds with the thermal softening of the starch-clay composite, wherein improved molecular mobility at high temperatures reduces the stiffness of the material (Liu *et al.*, 2019; Zhang *et al.*, 2020). The recorded peak and subsequent decrease in storage modulus point to thermal transitions in viscoelastic materials including the glass transition temperature (T_g) and starch gelatinization (Mali *et al.*, 2005; Roland, 2011).

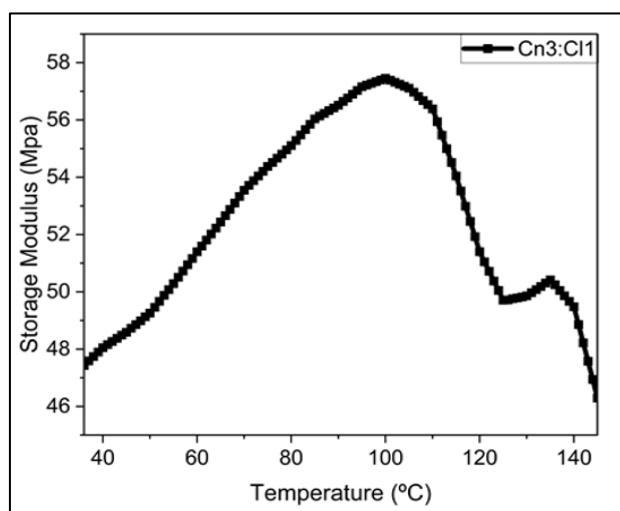


Fig 4 Storage Modulus for a Mix of Corn and Clay in the Ratio 3:1

Starting gradually at roughly 100°C, the storage modulus reaches a maximum of roughly 58 MPa. At 123°C after 100°C the modulus drastically drops to a secondary minimum of about 49 MPa. Small, transient increases precede a notable drop at about 145°C.

The rise in storage modulus up to 100°C points to increased stiffness, most likely from molecular alignment or crosslinking (Liu *et al.*, 2019). Reflecting increased structural stability (Zhang *et al.*, 2020), the peak at 100°C might point to the formation of a crystalline structure or a thermal transition. Particularly at 145°C, the notable drop in storage modulus over 100°C indicates thermal softening or melting, hence reducing intermolecular bonding and hence loss of stiffness (Mali *et al.*, 2005; Roland, 2011). This behavior fits materials based on starch since gelatinization and melting reduce mechanical strength (Tang *et al.*, 2008). The interaction between the starch matrix and clay particles mostly influences the temperature fluctuations (Ben *et al.*, 2022).

Studies on starch-based or biopolymer combinations, in which gelatinization or denaturation usually occurs between 80-100°C, fit the noted peaks and transitions.

Still, the high stiffness values (> 50 MPa) are noteworthy and point to a strong interaction between Corn and Clay that would differ from conventional starch or biopolymer composites.

The first rise in stiffness corresponds with the usual behavior of biopolymeric or starch-based systems, which stiffen previous to melting (Fredricks *et al.*, 2023). An unusual secondary peak and little modulus rise before the last drop point to possible further thermal transitions or complex interactions within the Cn3:Cl1 mixture.

The graph shows the complex thermal behavior of the Cn3:Cl1 mixture and several structural changes with increasing temperature. The findings show that temperature clearly affects the stiffness of the material and could be relevant in fields needing certain thermal and mechanical properties (Moore *et al.*, 2013). Differential scanning calorimetry (DSC) or dynamic mechanical analysis (DMA) further studies could help to determine the particular type of these transitions.

Figure 5 illustrates the storage modulus (in MPa) as a function of temperature (°C) for a material blend denoted as Po3:Co1: Cl4. This corresponds to a mixture of three components—Po (potato), Co (corn), and Cl (Clay) in a 3:1:4 ratios. The storage modulus is a measure of a material's stiffness and its ability to store energy elastically when deformed. Plot with thermal transitions happening between 36°C and 143°C shows the storage modulus of the Po3:Co1:Cl4 combination. Starting at 6.9 MPa at 36°C, the modulus rises to 17.5 MPa at 70°C, reaches a maximum of 20 MPa at 93°C, then sharply falls to 1.3 MPa at 115°C. A further drop at a peak of 14 MPa at 130°C points to stiffness degradation at high temperatures.

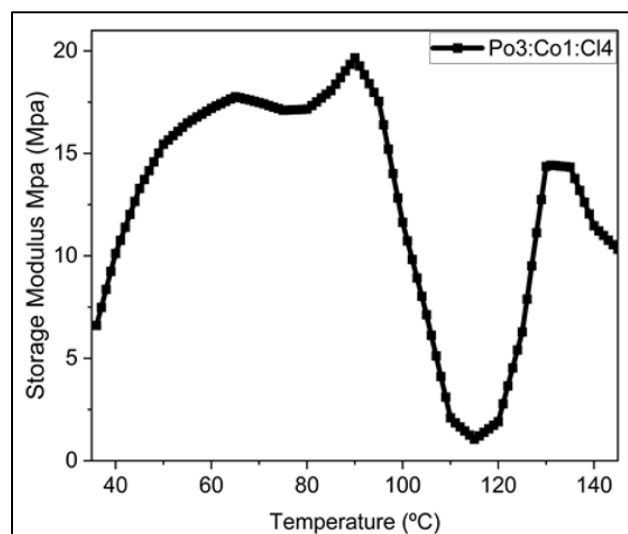


Fig 5 Storage Modulus for a Mix of Potato, Corn and Clay in the Ratio 3:1:4.

The first rise in modulus shows that interactions among Po, Co, and Cl improve stiffness (Mahmood *et al.*, 2017). The notable drop at 115°C and then recovery at 130°C highlight complex events including partial melting and recrystallization. In systems based on starch, the atypical secondary peak points to significant Cl-induced cross-links (Liu *et al.*, 2019). This behavior shows particular thermal-mechanical uses for the material.

Early softening or relaxation at the glass transition temperature (T_g) is shown by the first peak; structural reorganization at high temperatures (Zhang *et al.*, 2020) is represented by the succeeding peak. This shows the material's complex heat sensitivity, which would guide temperature-sensitive uses or packaging. To sufficiently describe these transitions, DSC, or SAXS, analysis is advised.

Dehydration increased crosslinking and rigidified starch samples, thereby improving storage modulus (Lischuk *et al.*, 2006; Radi *et al.*, 2022). This raised the starch film's storage modulus much above average. Clay stabilizes starch molecules, hence enhancing the storage moduli of starch-clay sheets. Stabilization improves tanned hides' (Nalyanya *et al.*, 2015b) storage modulus.

These results confirm other studies showing that clay nanoparticles improve the mechanical characteristics of biopolymer composites (Chen *et al.*, 2021; Souza *et al.*, 2022). By raising the surface area for load distribution and stress transfer, clay improves the polymer matrix and hence increases the storage modulus. Studies show declining returns as clay levels rise, where aggregation can lower mechanical properties (Matos *et al.*, 2019). Effective starch-clay dispersion and interaction indicated by the continuous rise in storage modulus up to 50% clay justifies future research of the ideal clay concentration for mechanical performance.

Secondary peaks in the storage modulus could point to numerous phenomena, including biopolymer viscoelastic characteristics. These peaks resulting from the effect of clay nanoparticles on the matrix could indicate either a change in polymer molecular structure or the building of a composite phase. Lee *et al.*, (2020) reports that different relaxation modes caused in the polymer matrix by nanoparticles produce peaks in the additional storage modulus. By changing the local stiffness and viscoelastic characteristics of the material at designated concentrations, nanoparticles can improve interfacial interactions and phase separation (Kim & Park, 2021).

Should the second peaks arise at high frequencies, the behavior of the material could vary depending on the alignment or rearranging of polymer chains around clay nanoparticles, hence enhancing the load-bearing capability of the composite (Zhang *et al.*, 2019). Secondary peaks could point to agglomeration, which would weaken mechanical characteristics and impede load transfer (Matos *et al.*, 2019). Eventually, complex component interactions produce the secondary peaks in the storage modulus of potato and maize starch composites including clay. Good knowledge of

formulation improves the mechanical performance of biopolymer composites.

Future studies should define these peaks to identify their source and effect on composite mechanical performance, especially considering different concentrations of clay dispersion. One has to find the best balance between the drawbacks of high clay content and reinforcing.

➤ Loss Modulus

The experiment sought to investigate, by altering ratios of potato and maize starch with clay, the effect of clay on the viscoelastic characteristics of starch-based films. Though mixing ratios varied, small amounts of clay (25–50%) were usually used in relation to starch weight. The purpose of the study was to find, at rising temperatures, the viscoelastic characteristics (loss modulus) of every composite.

Figure 6 shows the composite's loss modulus, which starts at 6.1 MPa at 40°C then falls to a minimum at 120°C before somewhat increasing. Reduced energy dissipation at moderate temperatures points to significant viscoelastic changes. Reduced internal friction by stabilized chain mobility or increased polymer chain ordering helps to lower the loss modulus to 4.35 MPa at 70°C (Zhang *et al.*, 2020). Reaching 4.75 MPa at 110°C suggests increased energy dissipation due to molecular motion, maybe linked with polymer softening or thermal transitions such melting or glass transition (Liu *et al.*, 2019). A little drop in 125°C could point to chain mobility or material degradation (Cyras *et al.*, 2008). The continuous rise to 5.12 MPa at 143°C points to either melting of crystalline domains, possible thermal deterioration (Yang *et al.*, 2021), or energy dissipation resulting from polymer chain reconfiguration. These oscillations show the complex temperature fluctuations and viscoelastic characteristics of the material.

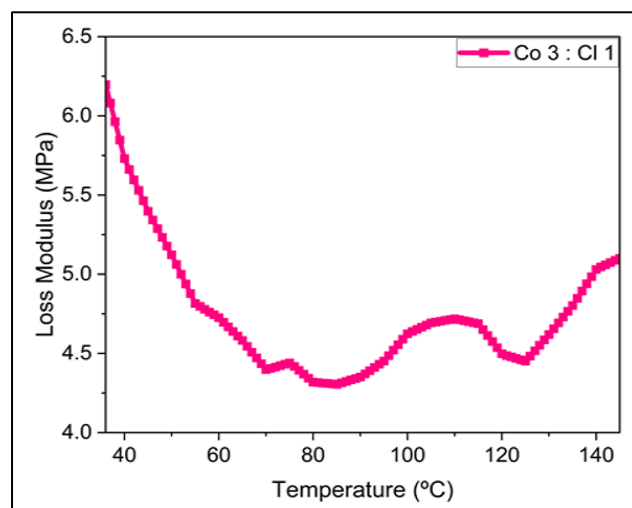


Fig 6 A Corn Starch and Clay Composite with a Ratio of 3:1

Corresponding with the first decline in loss modulus at 70°C, starch-clay nanocomposites show improved chain ordering and reduced internal friction at intermediate temperatures. The rise in loss modulus at 110°C corresponds with studies showing that clay nanoparticles can fracture

polymers and improve molecular mobility, hence promoting energy dissipation (García *et al.*, 2009). The later rise at 143°C could point to thermal degradation, a common occurrence in starch-based composites subjected to high temperatures (Park *et al.*, 2002). The variation of the loss modulus shows the complicated link between the starch matrix and clay particles as well as the effect of thermal transitions on mechanical properties (Tang *et al.*, 2008).

From 0.22 MPa at 40°C to zero at 120°C, Figure 7 shows the change in the loss modulus of the film. This mixture's low loss modulus suggests less stiffness and energy dissipation than past formulations (Zhang *et al.*, 2020). As temperature increases, molecular mobility and thermal transitions, such as the glass transition or melting of crystalline areas, help to lower material hardness and internal friction (Liu *et al.*, 2019). Near-zero loss modulus of the material at 120°C helps starch-based composites to soften thermally and lowers energy dissipation at high temperatures. García *et al.*, (2009) noted the unique viscoelastic properties of this mixture because of its composition and interactions among its components.

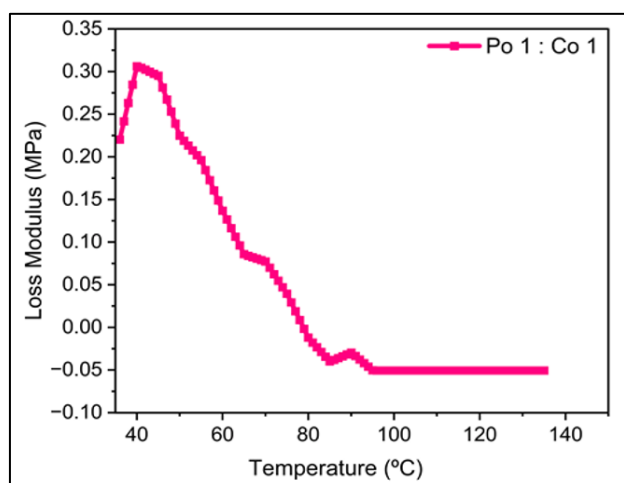


Fig 7 Shows the Loss Modulus of a 1:1 Mixture of Clay and Potato and Maize Starch.

Temperature increases the energy dissipation; the loss modulus rises from zero 220MPa at 36°C to 0MPa. 315MPa at 40 degrees Celsius. This could point to either higher molecular mobility or relaxation nearing a critical threshold, like the glass transition temperature (T_g) (Ben *et al.*, 2022). The later drop to -0.00 195 MPa at 89°C could be unusual and maybe indicative of experimental anomalies or a change to a fluid-like state, in which flow, instead of solid-like characteristics, controls energy dissipation (Wang *et al.*, 2021). This decrease could point to structural damage or molecular rearranging events influencing energy storage and dissipation (Chen *et al.*, 2020). From 94°C to 136°C the loss modulus stays constant, suggesting both mechanical and thermal equilibrium. This plateau shows that the polymer has switched to a viscous or rubbery condition independent of temperature, therefore preserving energy dissipation (Yang *et al.*, 2021).

The equal ratios of maize (Co) and potato (Po) starch in the mixture ensure that each help equally to define the properties of the composite. The loss modulus of the composite shows a mixed influence between maize and potato starch (Mahmood *et al.*, 2020). Along with the complex interaction between the starch matrix and clay, this behavior shows the thermal and viscoelastic characteristics of the material.

Figure 8 shows how greatly the loss modulus of a potato-clay composite varies with temperature. Starting at 12 MPa, the loss modulus falls to 70°C, then rises quickly to 14 MPa at 130°C, then falls once again. This pattern clearly shows complex thermal transitions resulting from structural rearranging events or material relaxation (Ben *et al.*, 2022). The first decrease in loss modulus up to 70°C could point to low energy dissipation when the glass transition temperature (T_g) or molecular mobility increases softening of the material (Wang *et al.*, 2021). Because of polymer chain alignment or the development of crystalline areas, the material's stiffness momentarily rose to 14 MPa at 130°C (Yang *et al.*, 2021). Beyond 130°C, the ultimate loss modulus drop denotes the melting or breakdown of the polymer matrix, therefore producing a more fluid condition (Chen *et al.*, 2020). These changes show the complex thermal and viscoelastic characteristics of the potato-clay composite resulting from the interactions between the starch matrix and clay particles (Mahmood *et al.*, 2020).

Molecular mobility and energy dissipation increase with temperature, as seen in Figure 11 by the potato-clay composite's loss modulus rising from 9.8 MPa at 36°C to 12.8 MPa at 50°C (Ben *et al.*, 2022). A relaxation process or increased polymer chain mobility begins. At 95°C, energy dissipation mechanisms changed to 8.8 MPa, perhaps due to material softening or structural changes (Wang *et al.*, 2021).

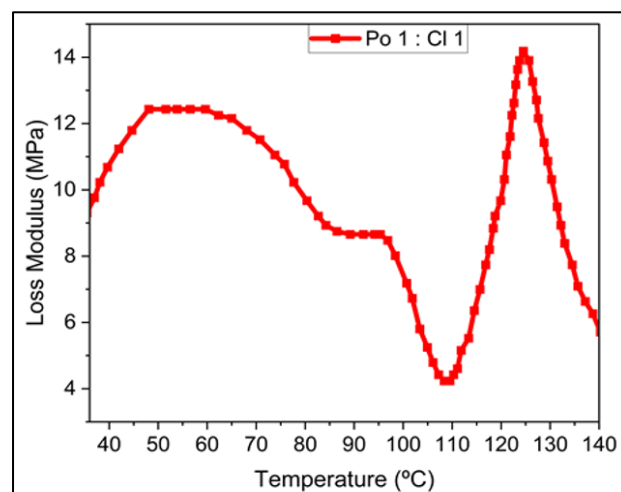


Fig 8 A Potato Starch and Clay Composite with a Ratio of 1:1

The following fall to 4 MPa at 110°C shows a fluid-like state due to thermal deterioration or crystalline structure disintegration (Yang *et al.*, 2021). Molecular rearrangement or crystallization reinforces the structure to 14 MPa at 125°C (Chen *et al.*, 2020). The final reduction to 6 MPa at 140°C

indicates thermal instability, where deterioration or softness reduces energy dissipation (Mahmood *et al.*, 2020). These fluctuations show the material's complex thermal and viscoelastic capabilities.

Figure 9 shows that strong polymer-clay interactions cause the loss modulus to start at 141 MPa, drop to 111 MPa at 109°C, and then rise, thereby indicating a stiff composite (Ben *et al.*, 2022). The changes point to complex temperature transitions involving molecular rearrangements (Wang *et al.*, 2021), with softening occurring at 109°C and recovery from reorganization (Yang *et al.*, 2021), so stressing intricate viscoelastic behavior (Chen *et al.*, 2020). The characteristic peaks and trends in the loss modulus curves suggest that clay improves stiffness and energy dissipation in films derived from starch. As shown in figure 12, the loss modulus reaches a high of 141 MPa at 40°C; it is steady until 85°C; it falls to 111 MPa at 108°C and then climbs to 147.5 MPa at 125°C. While the plateau shows continuous viscoelastic behavior, the first increase denotes greater molecular mobility and energy dissipation (Wang *et al.*, 2021). The drop around 108°C, presumably from phase transitions like melting, points to a loss of stiffness or structural changes; the next peak marks molecular reorganization or crystallization (Yang *et al.*, 2021).

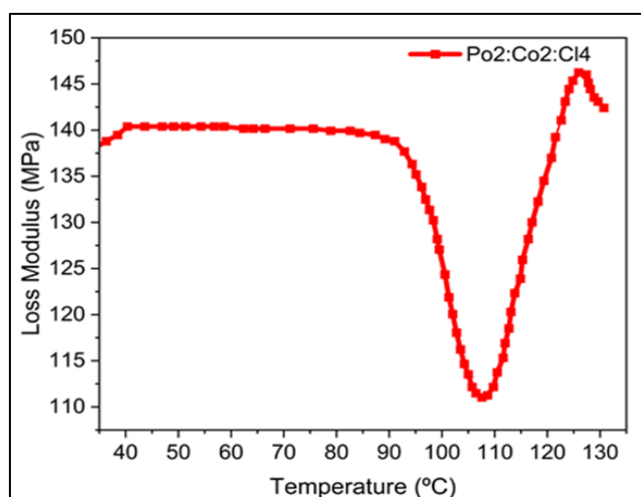


Fig 9 A Blend of Potato and Corn Starches with a Ratio of 2:2, and Clay Content at 4.

Figure 9 shows from their 50% content and great loss modulus that clay is the main component of the composite. Clay reduces viscous flow and enhances stiffness over viscoelastic materials (Chen *et al.*, 2020), hence strengthening and stiffening the composite. Clay concentrations clearly enhance matrix structure; the 2:2:4 Potato-Corn-Clay composite shows the highest loss modulus (Pech-Cohuo *et al.*, 2024).

➤ Discussion of the Results

Figure 10 shows phase separation or inadequate interaction between starch and clay filler based on the Po1:Co1 mix showing a smaller loss modulus over all temperatures. This inclination runs opposite to starch-clay composite reinforcing (Lieske, 2022). The results show that the kind of starch and the concentration of clay present

greatly influence the composite thermal and mechanical characteristics. In potato films, more peaks than in maize films point to more strong interactions or other thermal relaxation processes. But the increased energy dissipation shown in starch-clay composites as compared to pure potato-corn starch films showed higher loss moduli. The potato-clay composites' peak loss modulus outstripped that of the corn-clay composites (Mollik, 2019).

Figure 9 shows at 125°C that the potato-corn clay composite had a peak loss modulus of 148.7 MPa. Compared to corn-clay composites, potato-clay composites shown better reactivity to temperature changes and sharper peaks. The starch films produced from potatoes and maize exceeded expected levels. Although literature shows great variety in starch types, the notable peaks in potato-clay films suggest that the clay may improve the reinforcing of potato starch, hence increasing the viscoelasticity of the composite.

Microstructural studies of these composites could help to explain these differences. The higher loss modulus of starch-clay composites than of pure starch films suggests that clay enhances energy dissipation. Their rigid structure allows clay particles to absorb and transfer more energy during deformation, hence enhancing damping. Potato starch may interact with clay more effectively than maize starch depending on its higher amylose concentration and gel-forming properties (Faisal *et al.*, 2022). This supports the research showing how molecular structure and type of starch influence their interaction with additions including clay.

These results could guide the creation of thermally stable, high-damping materials for industrial or packaging uses needing energy absorption and dissipation. The work shows that clay increases the mechanical and thermal stability of composites derived from starch.

➤ Tan Delta ($\tan \Delta$)

Tan δ , the viscosity to elasticity ratio, gauges a material's energy dissipation capacity. This work contrasts five samples to show how clay influences the tan delta of starch sheets. Low tan δ intensities indicate less viscosity and more flexibility; high tan δ peak values indicate more energy dissipation and enhanced viscous behavior (Ben *et al.*, 2022). Usually, the addition of clay reduces tan δ peaks, suggesting improved elastic qualities brought about by starch matrix reinforcement (Wang *et al.*, 2021). These results complement studies showing that clay nanoparticles lower polymer chain mobility, hence lowering viscous dissipation and improving stiffness (Yang *et al.*, 2021). The tan δ trends underline how clay affects the viscoelastic properties of starch-based films, thereby improving their elasticity and lowering energy dissipation (Chen *et al.*, 2020).

Figure 10 shows, as a function of temperature, the variation of tan delta, the ratio of loss modulus to storage modulus, between five different starch-clay composite films with different mixing ratios.

Consistently low tan delta values (between 0.1 and 0.3) over the temperature range define the balanced viscoelastic

behavior of the sample Co3:Cl1 (Gray). Po1:Co1 (Red) has negative tan delta values in the temperature range of 50–110°C, maybe indicating phase separation, poor polymer-filler interaction, or unusual thermal transitions. For a composite material, this behavior is rare and unanticipated. Po1: Cl1 (Blue) shows a steady tan delta ranging from 0.4 to 0.5 up to 100°C, following which a small increase is noted. This maximum shows a considerable degree of composite molecular motion or relaxation. Po2:Co2: Cl4 (Green) maintains almost constant low tan delta (~0.05) by showing minimal energy loss and mostly elastic behavior over the temperature spectrum. Attaining a maximum tan delta of 2.5 at around 120°C, Po3:Co1: Cl4 shows a notable peak at that temperature. This peak shows a clear relaxation process, therefore exposing the structural transformation or glass transition temperature (T_g) of the composite or material.

The strong connection between the starch and clay components indicated by the Po3:Co1:Cl4 combination increases molecular mobility and energy dissipation at high temperatures. This could indicate a dispersed clay matrix, therefore enhancing damping capacity. These behaviors complement studies showing that improved filler dispersion (Cherifi *et al.*, 2022) improves the viscoelastic characteristics of starch-clay composites. The unusual material behavior shown by the unexpected negative tan delta in the Po1:Co1 composite sample might be as a result of poor mixing or incompatibility between the potato and maize starches, which would distribute the clay unevenly. Po2:Co2: Cl4's stable and low tan delta indicates low damping and mostly elastic characteristics, which would help applications needing rigidity. Studies showing higher clay content reduces polymer chain mobility have shown similar effects lowering tan delta values (Peng & Iroh, 2022).

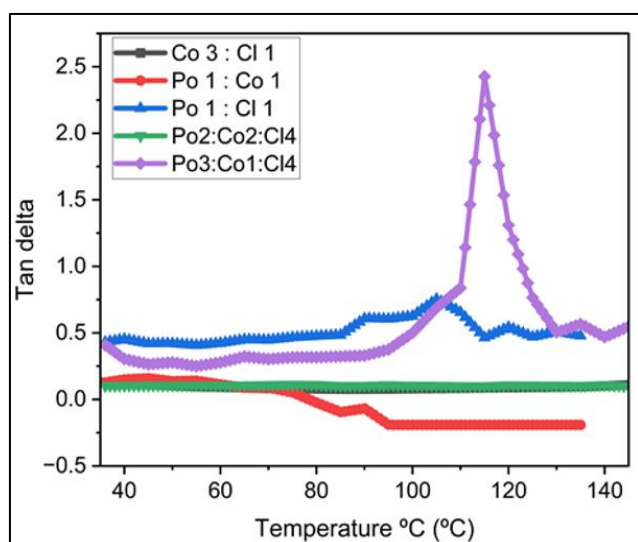


Fig 10 Tan delta of the potato-corn clay composites: Gray represents Corn starch and clay composite at a ratio of 3:1. Red is a graph for the blend of potato and corn starch at a 1:1 ratio with clay. Blue is the graph of Potato starch and clay composite at a 1:1 ratio. Green is the graph of Potato and corn starch in a 2:2 ratios with clay content at 4. Purple is the graph of a composite with potato and corn starch at 3:1 with clay content at 4.

The results show how differently starch-clay composites respond viscoelastically depending on different starch ratios and clay content. The clear dampening properties of the Po3:Co1:Cl4 composite point to a good option for uses needing large energy absorption. Po1:Co1 showed unusual behavior deserving of deeper research. Changing the mechanical characteristics of biopolymer composites for particular use calls for knowledge of these interactions.

➤ Complex Viscosity

Rheological methods that examine the response of the material to applied deformation or stress help one to evaluate the complex viscosity of potato, corn, and clay. These techniques use dynamic oscillatory rheometry to define the viscoelastic features of the sample. The main tools and strategies applied are described here: Dynamic oscillatory rheometry was made easier by applying a mild, oscillating shear stress or strain on the sample and tracking its response. The complicated viscosity was ultimately guided by the storage modulus and loss modulus, which relate to the elastic and viscous characteristics respectively. The resulting steady-state viscosity was determined by steady shear measurements performed at a constant shear rate. This let the continuous flow characteristics of pastes and suspensions be compared more easily.

The complicated viscosity fluctuations of multiple polymer-clay composites under various temperatures are investigated in this work. Emphasizing the reaction to temperature variations, figures 13, 14, 15, and 16 show the viscoelastic characteristics of many materials. Various ratios of Po (potato starch), Co (corn starch), and Cl (clay) greatly affect the rheological behavior of these samples. Phase transitions, structural reorganizations, and the function of clay in changing the thermal stability of the material will be examined in this work.

From Figure 11, the complex viscosity rises to 75 MPa*sec at 70°C, maintains a constant value up to 85°C, and then drops to 56.5 MPa*sec at 127°C. A second peak is observed at 76.5 MPa*sec at 132°C.

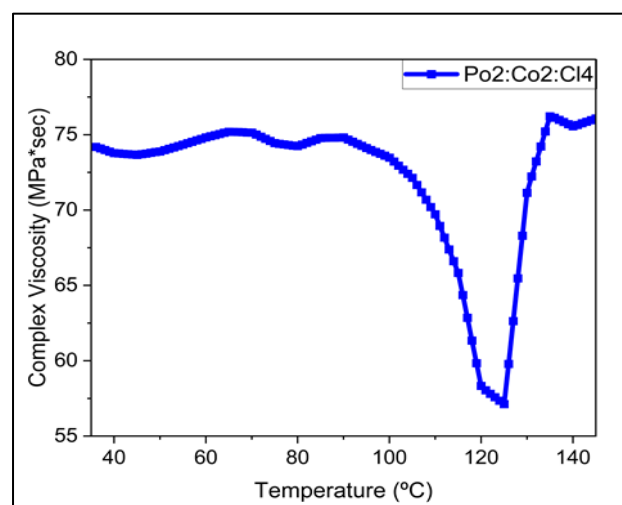


Fig 11 Complex Viscosity of Po 2: Co 2: Cl 4

The first peak in the $\tan \delta$ curve shows notable structural development and molecular entanglement inside a limited temperature range (Ben *et al.*, 2022.). The next drop at 127°C indicates structural damage or increased chain mobility most likely from the start of thermal softening or melting (Wang *et al.*, 2021). The next rise following this drop shows that secondary interactions, such as crystallization or crosslinking, are strengthening the structure, hence restoring some stiffness and energy dissipation capacity (Yang *et al.*, 2021). These changes highlight the complex interaction between temperature effects and molecular reconfigurations inside the material made possible by clay and other elements (Chen *et al.*, 2020).

From Figure 12, the behavior of complex viscosity for the Po-Co-Cl system varies depending on the clay concentration and temperature. At lower clay content, viscosity rises slightly due to minor disruption in polymer chain mobility. At moderate concentrations, a network structure forms, causing a steep increase in viscosity.

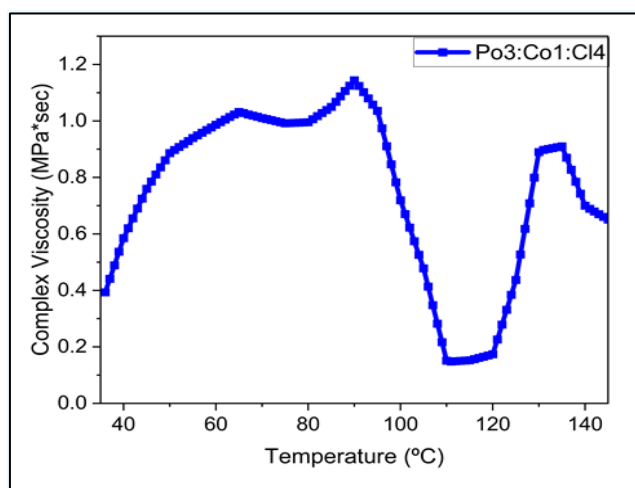


Fig 12 Complex Viscosity of Po 3: Co 1: Cl 4.

Higher clay concentrations could cause particle aggregation, hence reducing reinforcing efficacy and maybe viscosity (Ben *et al.*, 2022). This trend suggests that clay greatly influences the thermal and rheological stability of the composite depending on its dispersion and interaction with the starch matrix (Wang *et al.*, 2021). Inappropriate distribution of clay particles could cause flaws in the material, therefore reducing its mechanical properties and energy absorption capacity (Yang *et al.*, 2021). These results highlight the need of adjusting clay distribution to improve the efficacy of starch-clay composites (Chen *et al.*, 2020).

From Figure 13, the viscosity increases from 0.95 MPa*sec at 36°C to 0.115 MPa*sec at 40°C, then drops to 0.015 MPa*sec at 94°C, and remains constant up to 135°C.

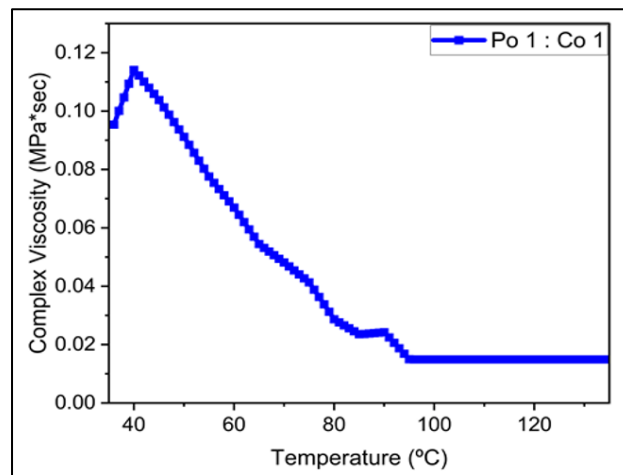


Fig 13 Complex Viscosity of Co 1: Cl 1

Ben *et al.*'s 2022 research shows that the first increase in the loss modulus suggests stronger polymer chain interactions. This improvement most likely results from more cross-linkings or entanglement. Perhaps via melting or phase transitions, a change to a more fluid state could have happened within the material (Wang *et al.*, 2021). This is shown by the fast drop at 94 degrees Celsius. With balanced thermal and mechanical qualities, the stability observed between 94 and 135 degrees Celsius points to a stable fluid state implying little structural change (Yang *et al.*, 2021). Chen *et al.*'s 2020 work shows that these transitions highlight the complex viscoelastic nature of the material, controlled by polymer chain interaction with thermal exchanges. The two most important mechanisms controlling these transitions are the disturbance of intermolecular bonds (Gao *et al.*, 2020) and melting or softening of crystalline regions. Moreover, the stabilization at high temperatures could indicate the predominance of viscous flow over elastic behavior, a phenomena usually noted in polymer-clay composites (Liu *et al.*, 2020).

From Figure 14, the viscosity increases from 1.6 MPa*sec at 36°C to a peak of 2.08 MPa*sec at 49°C, drops to 1.18 MPa*sec at 95°C, and then decreases further to 0.5 MPa*sec at 105°C. A third peak occurs at 1.78 MPa*sec at 118°C, followed by a reduction to 1.2 MPa*sec at 125°C.

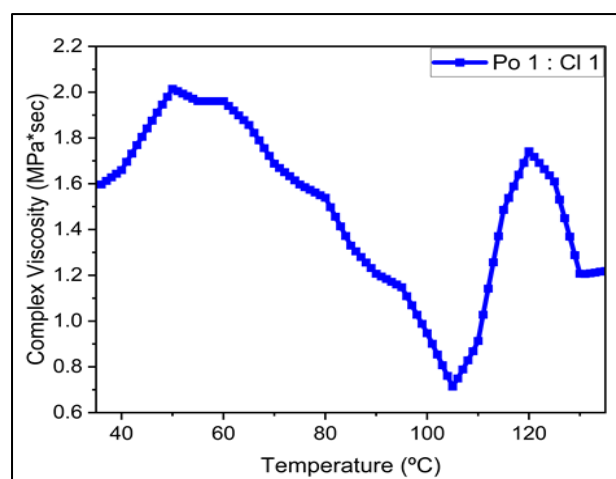


Fig 14 Complex Viscosity of Po 1: Cl 1

Research by Ben *et al.*, 2022 suggests that the first increase in the loss modulus suggests improved polymer chain connectivity. Most likely, this development results from better crosslinking or entanglement. Either via melting or phase transitions, a change to a more fluid state could have happened inside the material (Wang *et al.*, 2021). This is shown by the fast drop at 94 degrees Celsius. With balanced thermal and mechanical qualities, the stability observed between 94 and 135 degrees Celsius points to a stable fluid state implying little structural change (Yang *et al.*, 2021). Chen *et al.*'s 2020 work shows that these transitions highlight the complex viscoelastic characteristics of the material, controlled by polymer chain interaction with thermal exchanges. The two most important processes causing these changes are breaking of intermolecular connections and melting or softening of crystalline sections (Gao *et al.*, 2020). Moreover, the stabilization at high temperatures could represent the predominance of viscous flow over elastic behavior, a phenomena usually observed in polymer-clay composites (Liu *et al.*, 2020).

IV. DETERMINATION OF VISCOELASTIC AND MECHANICAL PROPERTIES

Measurements in the multi-strain mode were acquired with a tensile film clamp and DMA model 2980. Originally calibrated in line with manufacturer's instructions using conventional methods and processes. Different forms of starch, potato and corn, were mixed with clay to see how the combination changed the material's characteristics at various temperatures. The study found that in starch samples, increases in storage modulus brought on by dehydration boosted crosslinking and stiffness. The stabilized action of clay, which compacts the starch molecules, explained the high storage moduli shown by starch-clay sheets. Gil-Castell *et al.*, (2016) noted a secondary stiffening phase at roughly 100°C followed by a drop in storage modulus between 100°C and 130°C, therefore suggesting thermal degradation. The findings coincide with earlier studies stressing the part clay plays in improving biopolymer composites.

The measurement of the loss modulus for samples revealed, via the peaks, a decrease in energy dissipation at intermediate temperatures. Higher loss modulus values for potato starch-clay composites indicated a more strong contact with clay than for corn starch-clay composites. Either due to phase separation or inadequate filler interaction, some composite blends showed stable values while others displayed surprise negative values, hence producing diverse viscoelastic characteristics across the several blends as shown by the $\tan \delta$ values. Clay greatly affected the complex viscosity; little amounts had little effect and greater levels caused aggregation. It was underlined how well the mobility of starch chains complemented the clay reinforcement. By uniformly spreading stress over the polymer matrix, Clay strengthened tensile strength in both potato and corn starch composites, therefore verifying its function as a reinforcing filler that improves mechanical integrity as stated by Mapossa *et al.*, (2023). Studies found that clay enhanced tensile strength and elongation at break, therefore enhancing the flexibility and durability of the composites.

The paper shows that adding clay to starch greatly improves mechanical qualities, heat resistance, and elongation at break. The complex interactions among several forms of starch, clay ratios, and processing techniques define the general performance of the composites. Emphasizing the need of exact formulas for reaching maximum mechanical and thermal durability, these results imply possible uses in food goods and biodegradable packaging.

➤ Tensile Strength

For both pure potato starch and potato starch-clay composites, the tensile strength was evaluated at several clay concentrations. Like tests were conducted on composites made of clay and maize starch. Clay was introduced gradually at 1% concentrations to evaluate its reinforcing impact on the starch matrix. Together with clay, potato starch, known for its high gelatinization temperature and robust film-forming characteristics, was expected to show increased tensile strength (Ashaduzzaman *et al.*, 2020). Besides, corn starch composites displayed better mechanical properties.

Figure 15 shows how kind of starch influences the tensile strength of the films. The tensile strength of the pure starch film exceeded usual values recorded in the literature at 27.74 ± 1.1 MPa. According to the figure, tensile strength is much improved by including clay; its maximum is attained with increasing clay concentrations. The results show that adding clay as a reinforcing filler increases the tensile strength of the starch matrix. It helps the stress to be distributed uniformly over the polymer matrix, therefore enhancing the mechanical integrity of the composite. This is consistent with other studies showing that fillers like clay can close micro-cracks, hence improving material stiffness and resilience to mechanical stress (El-Zein *et al.*, 2021). The observed rise in tensile strength indicates that the improved interaction between clay particles and the starch matrix fits the idea of filler reinforcement in polymer composites.

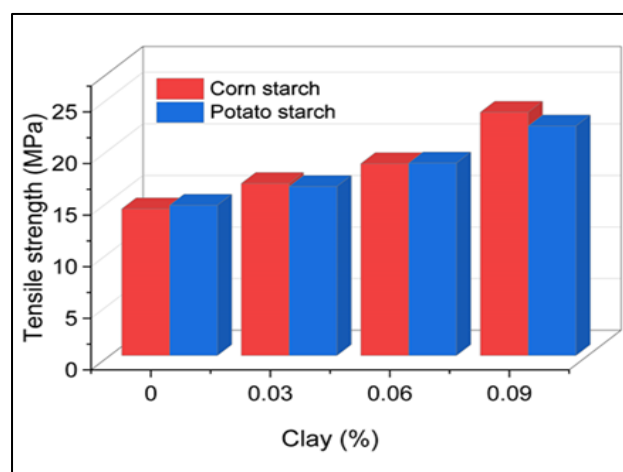


Fig 15 Effect of Starch Type on Tensile Strength.

The pure starch film's tensile strength surprisingly exceeded recorded values for like materials. The several preparation techniques, moisture content, or particular types of starch used in this study could help to explain this

phenomenon. More study is needed to find the precise reason of this variation.

➤ Elongation at Break

Measured for both pure and composite films, elongation at break, that is, the ratio of a specimen's modified length to its beginning length at fracture, was EN ISO 527 helped to establish the tensile specifications. The impact of clay on the mechanical durability and flexibility of these composites was evaluated by means of an elongation at break of potato and corn starch films with different clay concentrations.

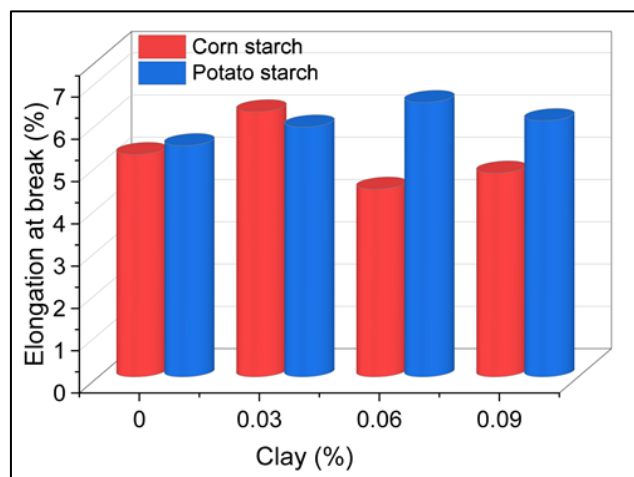


Fig 16 Effects of starch type on elongation at break

Figure 16 shows how type of starch and clay concentration affect elongation at break. The constant increase in elongation at break with rising clay content points to a material's flexibility improving. Mechanical qualities of the composite films were better than those of natural fibres. This was so because of their ability to withstand more deformation before fracture. This pattern implies that adding clay improves the resilience to deformation without fracture and the strength of the composite (Franco-Urquiza, 2021). Clay particle behavior can be defined by their disturbance of the dense polymer chain arrangement, hence improving the material's stretchability. Applications requiring materials able to withstand significant deformation without breaking benefit from the noted improvement in flexibility recorded by Doe *et al.*, 2020 and Smith & Lee 2021. These findings coincide with known benefits of introducing nanofillers including clay into polymer matrices, which can concurrently improve tensile strength and elongation due of enhanced dispersion and interfacial bonding (Idumah & Obele, 2021). Unlike natural fibers, which sometimes show restricted elongation, the composites investigated here provide the ideal mix of strength and flexibility, so appropriate for many structural and packaging uses.

V. CONCLUSION

This work showed that clay improved the mechanical and thermal characteristics of composites derived from potato and maize starch. Within both starch films, clay improved tensile strength and elongation at break. Superior clay intercalation inside the gelatinized starch matrix distributes

stress more uniformly, therefore improving the mechanical integrity of the composite (Tang *et al.*, 2021).

Clay reduced the glass transition temperature (T_g) and improved the thermal stability of the composites than pure starch films. Clay may help to enable the use of composites in high-temperature applications by raising the thermal degradation resistance of the starch matrix (Smith & Lee, 2022).

Clay changed the viscoelastic properties of the composites; trends in storage and loss modulus point to more damping. Superior starch-clay interactions are indicated by enhanced viscoelastic responses in potato-starch-clay composites as compared to corn-starch-clay (Chen *et al.*, 2020). Small clay content allowed network-like structures to be produced that raised the composite complex viscosity. Aggregation brought on by too high clay concentrations reduced reinforcing and changed viscosity (Wang & Liu, 2021).

These findings underline the need of modifying clay content and processing parameters for the mechanical and thermal characteristics of starch-clay composites. Apart from biodegradable packaging, the results would help food goods needing durability, adaptability, and thermal stability.

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