Experimental and Gaussian Process Regression Validation of the Effects of Nano Clay (Montmorillonite) on Properties of Thermo Plastic Starch Hydroxyapatite

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Abstract: This study considers the development of composite from biodegradable bioplastic obtained from starch of wasted cassava peels using hybrid fillers which are hydroxyapatite from egg shell waste and Montmorillonite (MMT) nanoclay as reinforcing filler through solution casting. The experimental analysis was validated using the Gaussian Process Regression (GPR). The hybrid composite film was prepared by weighing thirty grams (30 g) of the starch inside a beaker, into which 8 ml of glycerol, 2 ml of olive oil, and 8 ml of vinegar were added. The control solution was prepared without hydroxyapatite and nanoclay and made up to 150 ml with distilled water. The solution was mixed thoroughly then the mixture was heated on a hot plate at 70˚C. The mixture was stirred continuously till it starts to gel, after which the mixture was spread on an aluminum tray and left for 2 to 3 days to dry. This procedure was repeated for the composite formulations by incorporating a fixed amount of 4.5 g of hydroxyapatite (corresponding to 15 wt.% relative to the dry starch weight) alongside varied amounts of montmorillonite nanoclay at 1 wt.% (0.3 g), 3 wt.% (0.9 g), and 5 wt.% (1.5 g) respectively, relative to the total dry weight of the starch matrix. The solution was thoroughly mixed, heated at 70 ˚C and stirred continuously till it started to gel, and thereafter dried for 3 days. The developed composite was evaluated via morphological, structural, thermal and mechanical analysis. From the scanning electron microscopy (SEM) test, the morphology of the composite surface showed homogeneity with no visible agglomerates, while the x-ray diffraction (XRD) results indicate that with increasing the volume fraction of the nanoclay in the polymer matrix, the crystallinity peaks of the biopolymer become wider to become amorphous. The 3% nanoclay offers the best balance between strength and flexibility before aggregation issues reduce performance in the 5% nanoclay However, the thermogravimetric analysis (TGA) shows that the thermoplastic starch with 5% nanoclay has the highest thermal stability at 790°C. The GPR model demonstrated strong predictive capability for the nanocomposite properties with R^2 values between 0.91-0.95, RMSE values ranging from 1.18-16.50 and MAE values between 0.75-13.30**.** Thus, this outcome indicates low prediction error and high agreement between predicted and experimental values. This study is significant as it enhances the application of bioplastics, encourages waste-to-wealth conversion, reduces waste generation, and promotes environmental sustainability.

Keywords: Bioplastic, Composite, Nanoclay, SEM, TGA, XRD.

1. INTRODUCTION

Plastics is a common material used widely for various applications ranging from domestic to industrial applications especially for packaging, and for the development of parts, and components for domestic, industrial, automotive and transport, medical and healthcare, construction, electronics and appliances, textile and clothing, agriculture, aerospace and defense amongst other [1-2]. Plastics boasts of some desirable features such as ease of use, availability low weight, and cost-effectiveness thus, making them suitable for packaging applications and product development [3]. However, plastics from petroleum resources may constitute threats to the environment due to their non-biodegradability [4]. Furthermore, improper disposal of fossil-based plastics may contribute to the destruction of the ecosystems [5]. The annual global plastic production has grown significantly from 1.5 million tons in 1950 to 390.7 million tons in 2021 [6]. This lends credence to the fact that the demand and use of plastics have increased considerably over the years with evolving society but the disposal and its non-biodegradability still pose significant threat to human lives, environment, and ecosystem [7]. The replacement of plastics with other alternatives also poses a technical challenge mainly because of its desirable properties that finds wide range of

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applications. Thus, the need to address the non-biodegradability of plastics through the development of bio-plastics that are environmentally friendly with its ease of disposal and degradability after its end of use. Recent researches have considered the development of biodegradable composite as possible alternative to the traditional plastics, because of its environmental compatibility [8-9]. Bioplastics can limit the use of fossil-based plastics thereby promoting environmental sustainability. Among all the biopolymers, starch stands out as a promising candidate because of its availability, biodegradability, biocompatibility, and ease of chemical modification [10]. It is not only biodegradable but can also enhance the degradability of other plastic blended, thereby making it safe and suitable for packaging foods such as snacks, vegetable, fruits or solid or dry products [11-12]. Despite its abundance in nature, some limitation of starch for packaging application include: brittleness, high water sensitivity and solubility, brittleness, poor thermal and mechanical properties, and poor gas barrier properties [13]. To overcome these limitations, some additives, such as a plasticizer can be added to the starch, to enhance its flexibility, thermal and mechanical properties as well as its processing ability and performance [14-15]. Recent advances have witnessed the development of thermoplastic starch (TPS) with improved thermal properties; however, the material remains susceptible to humidity. Thus, emerging solutions geared towards enhancing the properties of starch for bioplastic development include blending with other biopolymers, chemical modifications and also reinforcing with nanofillers.

Application of hydroxyapatite as nanofiller in food packaging materials could improve biopolymer properties because of its better properties such as biocompatibility, osteoconductivity, nontoxicity, and bioactivity [16]. This material has potential as a reinforce for polymers because of its high strength and stiffness. Moreover, hybrid fillers could be employed to obtain composites with improved properties. Thus, hybrid composites can be an alternative by integrating it into starch-based composites for improved properties compared to the use of a single filler. Nanoclay such as montmorillonite is cost-effective, abundant and has flexible surface chemistry hence, it can enhance the properties of the base starch [17-18]. Thus, it is feasible to enhance starch properties by filling its matrix with nanoclay [19-20]. Montmorillonite nanoclays has a high aspect ratio thus making it suitable for reinforcement in nanocomposite material. Furthermore, it is cost effective and highly stable [22]. Natural clays are cost-effective naturally occurring materials such as aluminum silicate comprising mainly of fine-grained minerals and various mineral clays [22]. Nanoclay finds increasing commercial usage having up to 70% of the total market value of nanoparticles [23-24]. The small size and shape nanoclays are considered a desirable property because its high surface area can enhance a large interface between biopolymer or matrix to be reinforced and the nanofiller thereby increasing the bonding. The larger the interface the better the properties of the bio-nanocomposites and vice versa [25]. These properties also make it suitable for food packaging by improving the ability of the bio-nanocomposites to withstand thermal and stresses that may result while processing, transporting or storing the food [26].

The resistance of traditional plastic-based packaging materials to biodegradation contributes to the increasing environmental wastes and ecosystem challenges. Global production of plastic increased to 350 million tons in 2017, while 40% of the total plastic demand was tailored to packaging applications [27]. Due to lack of capacity for recycling in some areas coupled with improper disposal, some plastic materials constitute waste at their end of use either in landfills, water-bodies or lands thereby impacting the environment negatively [28]. Sequel to this, researchers continue to seek for a safe and environmentally friendly, bioplastic as packaging materials which my serve as a possible replacement to the traditional petrochemical-based plastics either in the pure or blended form. Starch-based polymers due to its biodegradability and non–toxic nature appeal as a replacement to some synthetic polymers in order to mitigate the problem caused by the traditional plastic wastes to the environment.

This study is aimed at producing and characterising biodegradable composite from wasted cassava peels using hybrid fillers which are hydroxyapatite from egg shell waste and montmorillonite nanoclay as reinforcing filler through solution casting and predicting the material's properties using the Gaussian Process Regression (GPR). The specific objectives include the preparation of starch from wasted cassava peel and thermoplastic starch without filler as well as preparation of thermoplastic starch with hydroxyapatite filler and the production of environmentally friendly starch composite reinforced with hydroxyapatite and varied amount of nanoclay filler (hybrid filler). This is followed by the characterisation of the thermoplastic starch and composite reinforced with fillers using Fourier Transform Infrared (FTIR), SEM-EDX, TGA and XRD to determine their mechanical properties using the Universal Testing Machine. Thus, the mechanical properties thermal stability, structural properties and morphological aspects of the composites were studied. Lastly, the prediction of the material's properties using the GPR.

The thermoplastic starch (TPS) reinforced with hydroxyapatite (HAp) and montmorillonite (MMT) nanoclay is a biodegradable material that has potentials for packaging and biomedical applications. Nevertheless, its structure is a function of multiple interacting factors such as the percent composition ratios of the TPS, HAp, and MMT, morphology such as particle size, structural properties such as crystallinity, and interlayer spacing as well as processing conditions such as temperature.

The traditional method of characterization of this material via physical experimentation may be time-consuming and expensive while conventional regression methods might not adequately capture the nonlinear relationships between input and output variables. Hence, the need for a predictive modelling. The GPR technique can handle small datasets and capture nonlinear behaviour between the input and output variables thereby making it useful in material design.

This study provides a data-driven optimization of TPS/HAp/MMT nanocomposite that enables the prediction of mechanical and thermal properties without exhaustive experimentation. The implementation of the GPR model as demonstrated in this study supports decision-making in the quest for the development of sustainable and biodegradable composites.

This study is also significant in that it is need-driven as it contributes to environment sustainability and the protection of ecosystem through the production of degradable bio-plastics. Technically and empirically, it presents a scientifically validated approach for the production of bio-plastics. The findings of this study may assist polymer developers in harnessing the potentials of natural materials such as starch for the development of bio-plastics tailored towards specific domestic or industrial applications.

This study is novel in that the investigation of the effect of nano clay (montmorillonite) on properties of thermo plastic starch hydroxyl apatite has not been sufficient highlighted in the existing studies.

Specifically, the evaluation of the bioactive properties of binary composites such as starch/MMT or starch/HAp have been independently conducted, while limited studies have reported on the optimization of their combined ternary interactions. In standard binary systems, there is usually a trade-off between high MMT loading which may trigger brittle matrices and high HAp configurations which may cause severe particle agglomeration and poor interfacial adhesion. This study addresses this gap by developing a ternary TPS/HAp/MMT hybrid composite. The utilization of high-aspect-ratio planar sheets of MMT to significantly alter the spatial nucleation and dispersion of spherical HAp nanoparticles within the amorphous TPS matrix represents a core novelty of this study. This study contributes methodologically to literature by establishing the procedural steps for the establishment of co-reinforcement and concurrently optimizing the mechanical tensile strength, water barrier resistance, and bioactivity with outcomes which exceed the performance of previously reported binary starch composites.

2. LITERATURE REVIEW

Starch is a major carbohydrate reserve in plants and is extensively used in various industrial and research applications due to its renewable, biodegradable, and inexpensive nature [29]. It comprises of two major glucose polymers: amylose, which is mostly linear with α-1,4 glycosidic linkages, and amylopectin, a highly branched molecule with α-1,4 and α-1,6 linkages [30]. Some of the outcome of existing studies on thermoplastic starch (TPS) and MMT systems [14, 31-38] include:

- Tensile strength and modulus increase with MMT content (up to an optimal point), while elongation decreases.
- XRD patterns show that starch addition can expand the MMT layer spacing due to intercalation, or even make the peak disappear upon full exfoliation.
- SEM micrographs generally show clay agglomerates at higher loadings, but uniformly dispersed layers at low loadings.
- FTIR spectra of TPS/MMT often reveal stronger OH stretching bands or new peaks, suggesting hydrogen bonding between starch and clay surfaces.
- Thermogravimetric analysis (TGA) indicates that the onset of starch decomposition shifts to higher temperature (10-20 degrees Celsius) with MMT, as the clay layers insulate the polymer and catalyze the char.
- Barrier properties; water vapor transmission rate (WVTR) can drop by 20-50% with a few % clay. All these outcomes support the utility of MMT.

For TPS specifically, investigations of HA and MMT together are sparse, but analogous systems (*e.g*. PCL/HA/Clay scaffolds) suggest benefits. A study on Hydroxyapatite-montmorillonite (HA-MMT) nanocomposites showed that HA nanoparticles can deposit on clay surfaces, creating a hybrid network that

improves strength and osteoconductivity. In polycaprolactone (PCL), a HA/Clay double filler improved both modulus and bioactivity compared to single fillers. Drawing from these, we anticipate the following for TPS/HA/MMT; MMT will be preferentially intercalated by the starch matrix, while HA particles may situate on starch or near clay. Good dispersion of both is crucial. Challenges include preventing HA-HA or MMT aggregation and ensuring interfacial adhesion. If successful, such ternary nanocomposites could achieve better mechanical and moisture properties than TPS/HA or TPS MMT alone. This will thus extend prior work by systematically adding clay to a TPS/HA system, filling a gap in the literature.

3. MATERIALS AND METHODS

3.1. Starch Isolation

Cassava peels gotten from a garri processing industry in Ado Ekiti, Nigeria were thoroughly washed with water, the brown part of the peel was removed with knife, and the peels were blended for 3 min to ensure homogeneity.

The resulting slurry was put into a muslin cloth and placed in a distilled water. The cloth and the contents were repeatedly squeezed to expel the starch into the water. Thereafter, the starch was allowed to settle for 12 hrs and the supernatant was decanted. This process was repeated until the supernatant was clear. Wet starch was extracted and air-dried (Figure **1a**).

3.2. Extraction of Hydroxyhapatite

Eggshells are a rich in $CaCO_3$ which can be converted into hydroxyapatite ($Ca_{10}(PO_4)_6(OH)_2$); a biocompatible material for the production of bio-plastic. First the eggshells (Figure **1b**) are collected from the restaurants of Afe Babalola University, Ado Ekiti, Nigeria and the impurities and inner membranes are removed by soaking it in warm water for 30 min. They are further washed in distilled water to remove organic residues and dried at 100°C in a hot air oven for 3 hrs. The dried eggshell was grinded with a mechanical grinder to a fine powder and sieved to obtain a uniform particle size of <100 µm (Figure **1c**).

The fine powder was thereafter heated at 900℃ for 2 hrs, to decompose according to equation 1 and allowed to cool at room temperature.

$$CaCO_3 \rightarrow CaO + CO_2 \quad (1)$$

The calcined CaO is dissolved in distilled water to form calcium hydroxide according to equation 2.

$$CaO + H_2O \rightarrow Ca(OH)_2 \quad (2)$$

Next phosphoric acid was added drop wise to the calcium hydroxide solution and stirred continuously for 1 hr at room temperature to hydroxyapatite according to equation 3 while maintaining the pH between 8-10 using NH_4OH solution.

$$10Ca(OH)_2 + 6H_3PO_4 \rightarrow Ca10(PO_4)6(OH)2 + 18H_2O \quad (3)$$

The resulting product was filtered, washed using distilled water and dried in an oven at 120 ℃ and calcined at 900℃ for 12 hrs [39].

Figure 1a: Dried starch.

Figure 1b: Collected egg shells.

Figure 1c: Dried, fine and sieved eggshell powder.

3.3. Preparation of Bio-Plastic

The hybrid composite film was prepared by weighing thirty grams (30 g) of the starch inside a beaker, into which 8 ml of glycerol, 2 ml of olive oil, and 8 ml of vinegar were added. The control solution was prepared without hydroxyapatite and nanoclay and made up to 150 ml with distilled water. The solution was mixed thoroughly then the mixture was heated on a hot plate at 70˚C. The mixture was stirred continuously till it starts to gel, after which the mixture was spread on an aluminum tray and left for 2 to 3 days to dry.

This procedure was repeated for the composite formulations by incorporating a fixed amount of 4.5 g of hydroxyapatite (corresponding to 15 wt.% relative to the dry starch weight) alongside varied amounts of

montmorillonite nanoclay at 1 wt.% (0.3 g), 3 wt.% (0.9 g), and 5 wt.% (1.5 g) respectively, relative to the total dry weight of the starch matrix. [40].

All filler concentrations for hydroxyapatite (HAp) and montmorillonite (MMT) nanoclay are expressed as weight percentages (wt.%) calculated relative to the total dry weight of the starch matrix. For a standard baseline batch utilizing exactly 30 g of dry starch (with a fixed 30 wt.% glycerol plasticizer ratio), a 15 wt.% HAp loading corresponds precisely to the addition of 4.5 g of HAp nanoparticles.

3.4. Scanning Electron Microscopy (SEM)

The SEM (Hitachi SU8030 FE-SEM Tokyo, Japan) was employed to observe the shape and size of the native and modified starch granules at an accelerating potential of 5.0 kV. All samples were sputter-coated with thin layer of gold-palladium (Au/Pd) before the examination to prevent charging and enable effective signal detection. This is due to the fact that the coating enables a conducting surface that allows electron to flow away from the sample to prevent the development of static charges that can distort the image and enhance the resolution of the images.

3.5. X-Ray Diffraction (XRD)

The XRD test was conducted using a copper anode x-ray tube (Cu-Kά1 radiation) with the aid of a X-ray diffractometer (Rigaku D-max Tokyo, Japan). The starch powder was tightly packed in the sample holder while ensuring the surface is smooth and flat to prevent displacement during rotation. It was exposed to the X-ray beam at 40 kV and 20 mA while the scan range of the diffraction angle (2θ) varies between 5° to 50° at step size count of 2 s and scan speed of 2°/min for a better resolution.

3.6. Thermal Analysis

The thermogravimetric analyzer (TGA), Shimadzu TG-DTA model TG-60 (Shimadzu, Kyoto, Japan) was employed for the thermal analysis conducted at a flow rate of 120 ml/min. in an open alumina crucible having 5 mg of starch at a heating rate of 12°C/min, with the initial and final temperature being 30°C and 800^{o}C respectively.

3.7. Tensile Test

The Tensile Strength (TS), elongation and the Young Modulus (YM) test of the bioplastics were conducted under standard procedure according to the guidelines of ASTM D882-91 using the universal testing machine. The cut samples of the bioplastic produced having a rectangular shape of 50.8 mm by 152.5 mm were loaded onto the machine the elongation and TS.

3.8. Gaussian Process Regression

This section details on the Gaussian Process Regression (GPR) modelling to predict the structure property relationships in TPS/HA/MMT nanocomposites using experimental characterization data obtained from SEM, XRD, and TGA analyses.

The GPR is a non-parametric, Bayesian supervised learning technique used for regression, especially with a small dataset [41-43]. The GPR was employed to model the complex non-linear relationship among the input variables such as percent composition, morphology, and structural features of TPS/HAp/MMT nanocomposites and the output variables such as the YM, TS etc. Table **1** presents the dataset employed.

From Table **1**, the input variables (X) are MMT, d-spacing, onset temperature and, particle size while the responses are crystallinity, decomposing temperature, YM, and TS etc. First, the data set was gathered by extracting numerical features from the SEM, XRD, and TGA characterization. The extracted data was cleaned by removing the missing values and ensure consistency of measurement units. This was followed by the feature scaling to normalize input variables using StandardScaler. The GPR model was selected with Matern or RBF kernel. This was followed by cross-validation which entails the apply Leave-One-Out Cross-Validation (LOOCV) due to small nature of the dataset. The model was trained using the model using training data and the predicted values were obtained. The model was evaluated by

Table 1: The Dataset Employed

MMT (%)	d-spacing (nm)	Onset temp. (°C)	Particle size (nm)	Crystallinity (%)	Decomposing temperature (^{o}C)	YM (MPa)	TS (MPa)
0	1.2	300	450	22	500	1.38	0.20
1	1.6	305	300	22.5	580	0.62	0.14
3	1.8	320	180	23.15	750	40.36	2.48
5	1.5	325	260	24.15	790	66.94	3.28

considering the R^2, Root Mean Square Error (RMSE), and Mean Absolute Error (MAE).

4. RESULTS AND DISCUSSION

4.1. Tensile Test of the Bio-composites

Table **2** shows that the TS of the bioplastic ranges from 0.20-3.28 MPa, with the bioplastic reinforced with 5% nanoclay having the highest value and bioplastic without nanoclay having the least TS. This is the stress the material can withstand when stretched before deformation. The addition of nanoclay increased the TS of the biopolymer exceeding the Moderate Class Plastic mechanical properties of 1-10 MPa.

As shown in Table **2**, the sample without nanoclay is weak and soft due to its low structural integrity while the sample with 1% nanoclay has a slight improvement in the TS and YM due to the presence of nanoclay particles that reinforces the matrix. Sample with 3% and 5% nanoclay showed improved and significant improvement respectively in the TS and YM due to good dispersion of nanoclay which creates a strong interfacial bonding. Nanoclay acts as a reinforcing filler, which restricts the mobility of polymer chains and enhancing load-bearing capability. However, the elongation in the 5% nanoclay reduces significant due to possible agglomeration. Elongation provides an insight into the film's flexibility and elasticity via the measurement of the maximum length it can reach before breaking. It is a measure of a material's ductility and toughness as well as the amount of deformation it can withstand without breaking. The elongation decreases with increase in the percentage of nanoclay as shown in Figure **2**. The lower the elongation, the lower the ductility and toughness of the material, and vice versa. The elongation of a plastic is a function of its application. The elongation of the bioplastic ranges between 1.72-8.03 samples with the bioplastic with 5% nanoclay having least elongation at break and nanoplastic with 1% nanoclay having highest value. Thus, the results of the elongation presented in Table **2** show that the bioplastic with 5% of nanoclay possesses the lowest ductility and toughness than others under the same loader conditions.

YM otherwise called the modulus of elasticity of a material, is the ratio of the stress developed in the material to its corresponding strain. This property is useful in estimating the tensile or compressive stiffness of a material. The higher the YM value, the stronger and stiffer the bioplastic would be. The YM increases with increase in the percentage of nanoclay. The results of the biopolymers range between 0.62 and 66.94 MPa with the biopolymer with 2% nanoclay having the least value (0.62 MPa) and the biopolymer with 5% nanoclay having the highest YM value (66.94 MPa). This implies that the bioplastic with 5% nanoclay will be stiffer than others and will be able to withstand deformation and changes in length when under tension or compression without yielding to failure. The increase in the elastic modulus of the bioplastic with nanoclay may be ascribed to the existence of hydrogen bonds between the molecules of the starch and the glycerol. The presence of nanoclay restricts deformation, thus increasing the stiffness and rigidity of the bioplastic.

Thus, the TS and YM of the developed hybrid composite films were found to increase with increase in the nanoclay content up to 3 wt.%. This may be attributed to enhanced mechanical reinforcement and strong interfacial bonding between the high-aspect-ratio MMT sheets and the starch matrix. Conversely, the elongation at break decreases due to the fact that the clay layers restrict polymer chain mobility, thereby rendering the material more rigid. This finding is directly validated by SEM observations, which show a highly uniform, cohesive cross-sectional morphology at 3 wt.% MMT, signaling a homogeneous filler dispersion and excellent stress transfer. Therefore, the 3 wt.% loading offers the optimal balance between tensile strength and matrix flexibility. Beyond this threshold, at 5 wt.% MMT, a decline in the mechanical performance was observed. This decline aligns closely with SEM micrographs, which show signs of filler aggregation. These localized nanoclay clusters act as internal structural flaws and stress-concentration zones, prematurely inducing brittle failure and suppressing the overall ductility of the biopolymer composite.

4.2. Surface Morphology of the Produced Biopolymers

The essence of the morphology analysis is to observe the surface structure, dispersion of nanoclay, and fracture behavior of the bioplastics. Figure **2**(**a-d**) presents the surface morphology of the biopolymers. The thermoplastic without nanoclay (Figure **2a**) showed a smooth and relatively homogeneous surface, however, there were visible microvoids and starch granule due to poor interfacial bonding. The biopolymers with nanoclay showed (Figure **2b-2d**) a less smooth, uniform and homogeneous surface compared to Figure **2a**. There is evidence of slight surface roughening which increases as the percentage of nanoclay in the bioplastic increases. The surface of the samples shown in Figure **2b-2d** showed better adhesion between the base starch and the nanoclay with the disappearance of the microvoids due to the presence of nanoclay which fills the voids as the

percentage of nanoclay in the bioplastic increases. There was no evidence of phase separation which implies good structural integrity as the nanoclay was well incorporated to thermoplastic matrix without visible agglomerates observed as the percentage of the nanoclay in the bioplastic increases. Adewumi *et al*. [17] reported similar result on Africa yam bean starch blended with nanoclay. In line with the findings of this study, Adeodu *et al*. [44] indicated that proper dispersion of nano-platelets can enhance the mechanical, thermal and electrical properties of polymer nanocomposites. Samples with 3% and 5% nanoclay noticeably appear rougher, although with a more compact surface indicating a well-dispersed nanoclay platelets with fewer voids and cracks due to effective matrix–filler interaction. The homogeneous surface of thermoplastic starch/ hydroxyl apatite nanoclay films were an indicator of good mechanical properties such as high resistance and elongation at break. The homogeneous film surface of the thermoplastic starch reinforced with HA and nanoclay could make an improved biodegradable bio-blend for food packaging.

Table 2: Mechanical Properties of the Thermoplastic starch (TPS)

Samples	TS (MPa)	Elongation (%)	YM (MPa)
TPS without hydroxyapatite	0.20±0.03	8.03±0.44	1.38±0.05
TPS with 1% nanoclay	0.14±0.02	7.63±0.21	0.62±0.02
TPS with 3% nanoclay	2.48±0.14	5.05±0.52	40.36±0.04
TPS with 5% nanoclay	3.28±0.31	1.72±0.22	66.94±0.23

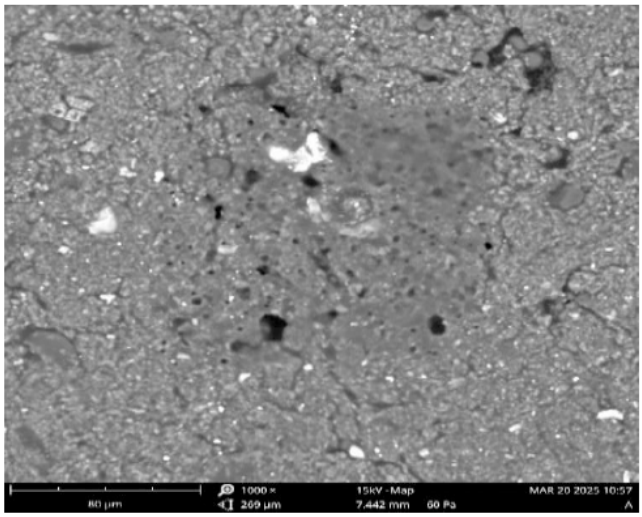

Figure 2a: SEM of Bioplastic without filler nanoclay.

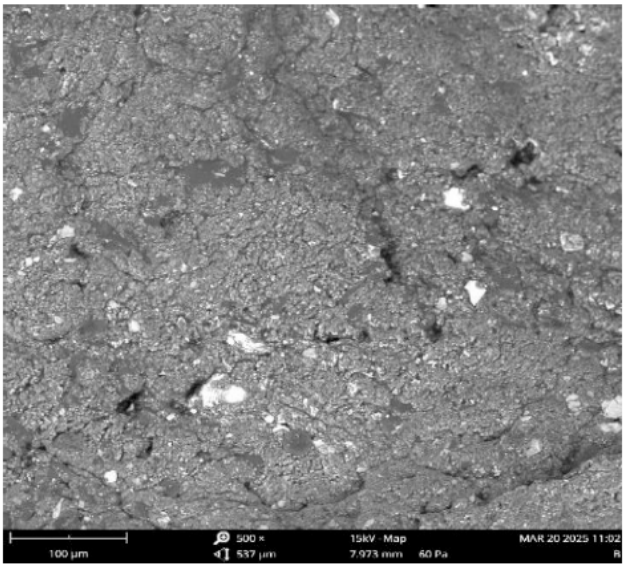

Figure 2b: SEM of Bioplastic with 1% nanoclay.

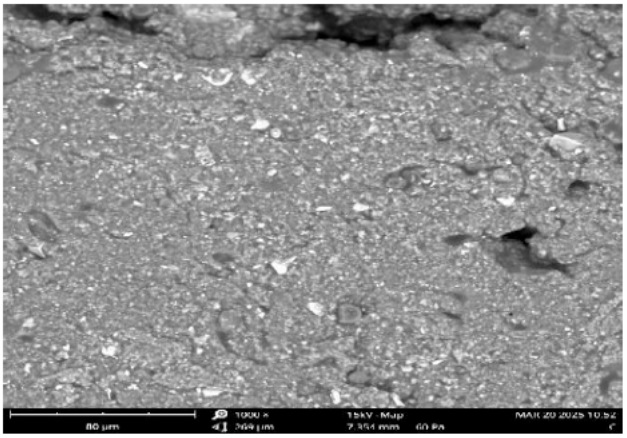

Figure 2c: SEM of Bioplastic with 3% nanoclay.

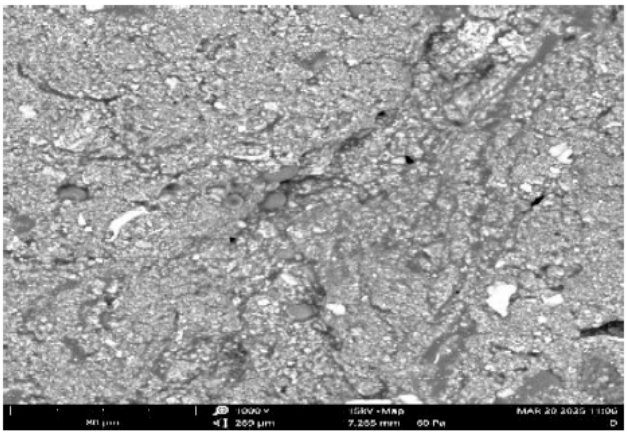

Figure 2d: SEM with 5% nanoclay.

4.3. The XRD- diffraction Pattern of the Produced Biopolymers

The essence of the XRD analysis is to study the crystalline structure, intercalation and exfoliation of nanoclay as well as the phase changes. Figure **3**(**a-d**) presents the XRD- diffraction patterns of the

biopolymers. The bioplastic without nanoclay (Figure **3a**) showed a broad amorphous structure peak at 2θ of 15.5° and minor crystalline peaks at 2θ of 22.17°-22.17° allocated to the Vh-type crystals. The Vh-type crystallinity is enabled by heat treatment, triggering the replacement of the OH groups in the molecules of the starch with hydrogen bonds developed between starch and the plasticizer during processing [45-46]. Biopolymer with 1% nanoclay (Figure **3b**) showed sharp peak at 2θ of 16° and 22.5°, 3% showed peak at 23.15 and 5% showed peak at 16.5° and 24.15 of 2θ. However, it was observed that with increasing the volume fraction of the nanoclay in the polymer matrix (Figure **3b-3d**), the crystallinity peaks of the biopolymer become wider to become amorphous, which may be due to reduction in the formation of glycerol amylose complex as a result of restricted movement of amylose molecules as similarly observed by Adewumi *et al*. [2]. The microstructural changes within the hybrid films were indicated by their crystalline profiles. For instance, as the nanoclay content increases up to 3 wt.%, the composite films show a localized increase in structural order, correlating with the higher tensile strength and stiffness observed in the mechanical testing. The appearance of a well-defined reflection peak for the 3 wt.% nanoclay sample, indicates a structured, arrangement where the long starch molecular chains have slipped into the tiny spaces inside the clay layers, thereby pushing them apart. Conversely, the 5 wt.% nanoclay sample shows alteration of peak intensity and notable peak broadening signaling a reduction in long-range structural order and smaller crystallite domain sizes within the host matrix. The reduction in peak sharpness at higher loading may be as a result of severe disruption of the starch matrix's structural arrangement induced by localized nanoclay cluster formation and uneven filler distribution. The clay aggregates may act as stress concentrators under mechanical loading, thereby inducing significant disruption on the starch matrix.

4.3. The Thermographic Analysis Results of the Produced Biopolymers

The essence of the TGA is to study the thermal stability of the bioplastic samples under the application of heat. Figure **4**(**a-d**) shows the thermographic analysis results of biopolymers. The TGA curve of thermoplastics starch shows that degradation take place in three steps. The biopolymer film without nanoclay (Figure **4a**) was seen to decompose initially between 100 °C- 120 °C. Furthermore, the weight loss experienced in the first stage is attributed to dehydration or evaporation of free water [47], while the weight loss in the second stage observed between 450 and 500 °C can be linked to the evaporation of bound

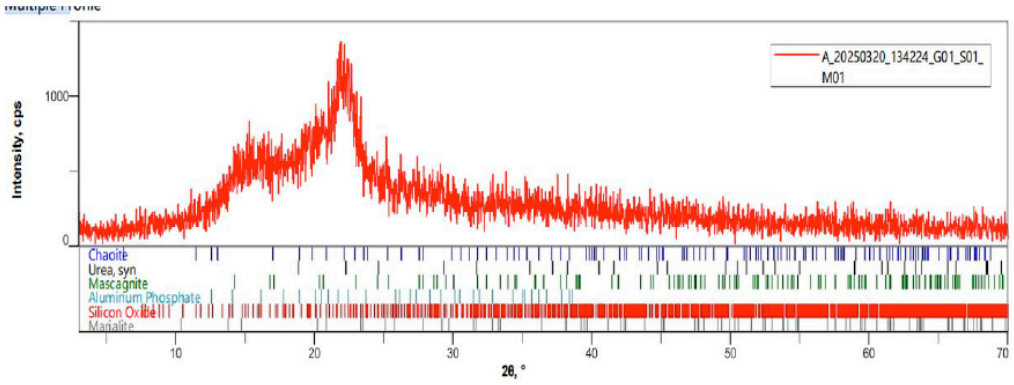

Figure 3a: XRD Spectrum of Bioplastic without nanoclay.

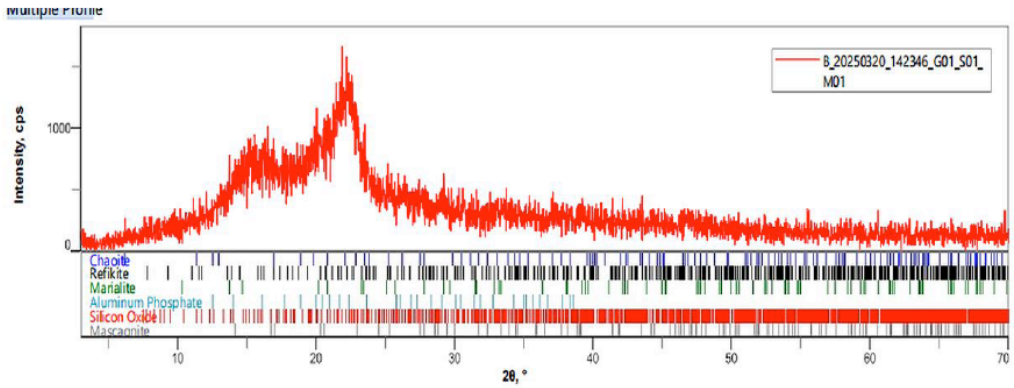

Figure 3b: XRD Spectrum of Bioplastic composite with 1% nanoclay.

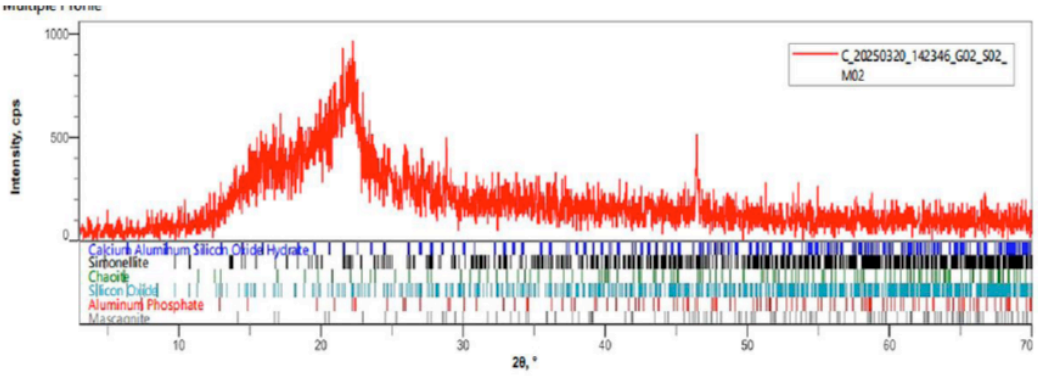

Figure 3c: XRD Spectrum of Bioplastic composite with 3% nanoclay.

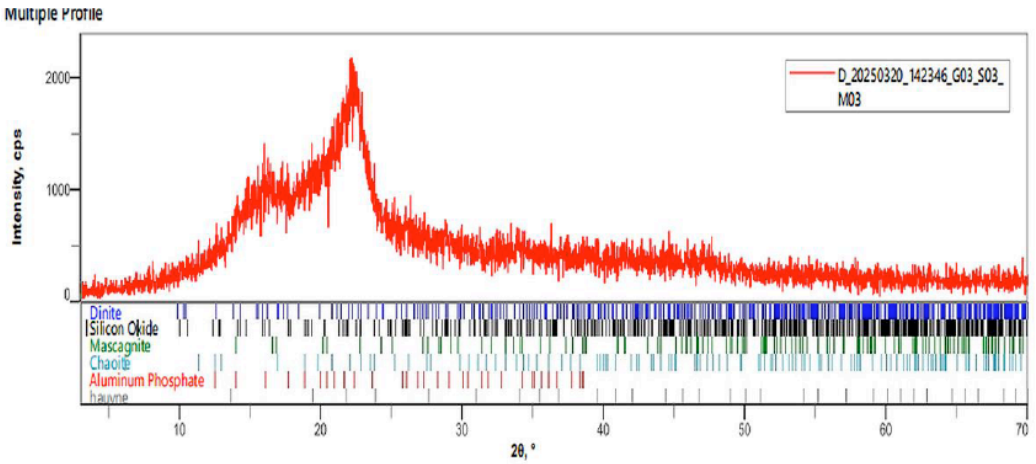

Figure 3d: XRD Spectrum of Bioplastic composite with 5% nanoclay.

water [48]. At 580 °C, weight loss is triggered by depolymerisation and decomposition of the functional group chain in the polymeric films [49]. The biopolymer film with 1% nanoclay (Figure **4b**) was observed to initially decompose between 100 °C- 120 °C. There was a slight increase in thermal stability in the bioplastic with 1% nanoclay compared to the bioplastic without nanoclay and the thermal stability increases with increase in the percent of nanoclay in the bioplastic. The thermal stability is attributable to the presence of nanoclay in the bioplastic which acts as a heat barrier. The weight loss in the second stage was seen between 500-550 °C while at the third stage, further weight loss was noticed at 720 °C. Eventually the structural matrix collapsed at 750 °C thus, indicating high capacity for thermal resistance which makes the bioplastic suitable for high temperature application within the range observed and reported in this study. The biopolymer film with 3% nanoclay (Figure **4c**) was observed to decompose at the initial stage between 100 °C- 120 °C. The weight loss in the second stage was observed between 500-550 ^{o}C. Further weight loss in the third stage was observed at 750 °C. At 750°C the structural matrix was collapsed. Initially, the biopolymer film decomposed with 5% biopolymer (Figure **4d**) between 100 °C- 120 °C. At the second stage, the weight loss was seen between 570-590 °C while further weight loss was experienced in the third stage at 790 °C. At 790°C, there was a complete collapse of the structural matrix. This result indicates that nanoclay improved the thermal stability of the biopolymers. The biopolymer comprising of 5% nanoclay has the highest thermal stability while the one without nanoclay has the least thermal stability. Biopolymer with 5% nanoclay gave better thermal stability in comparison to the other biopolymers. The increase in the thermal stability of the biopolymers reinforced with nanoclay could be ascribed to a strong intermolecular hydrogen bond between the starch and nanoclay [50]. The results obtained showed a slight trade off as the bioplastic with 3% nanoclay offers the best balance between strength and flexibility while the sample with 5% showed improved morphology, crystallinity and thermal stability. Bioplastic sample without nanoclay showed poor mechanical, structural and thermal properties while the one with 1% nanoclay showed minimal reinforcement. However excessive nanoclay reinforcement may lead to agglomeration, thereby defeating the purpose of enhancing the properties of the bioplastic.

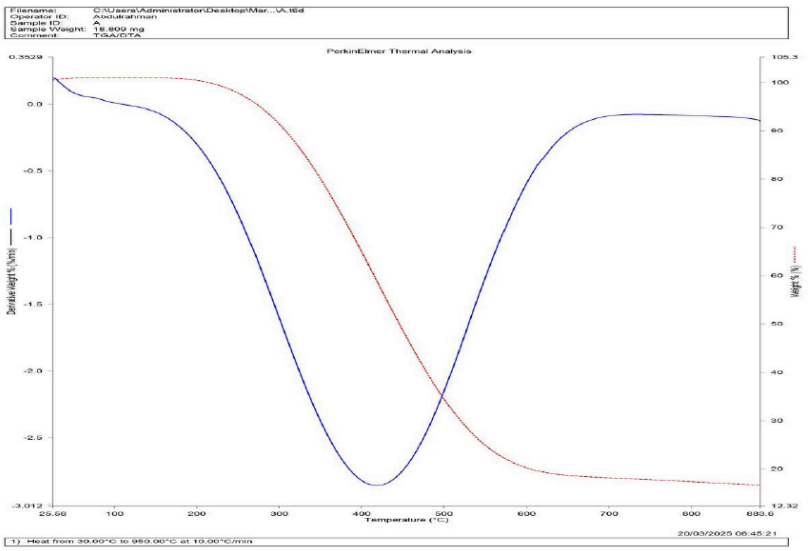

Figure 4a: TGA of bioplastic composite without nanoclay.

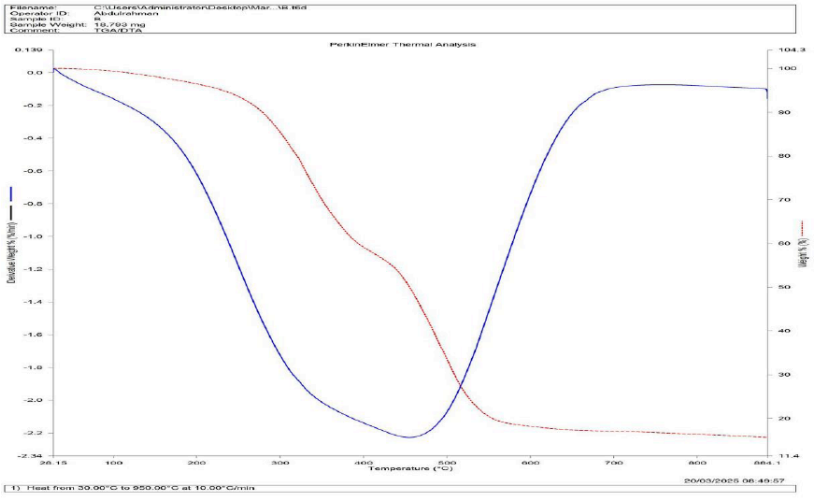

Figure 4b: TGA of bioplastic composite with 1% nanoclay.

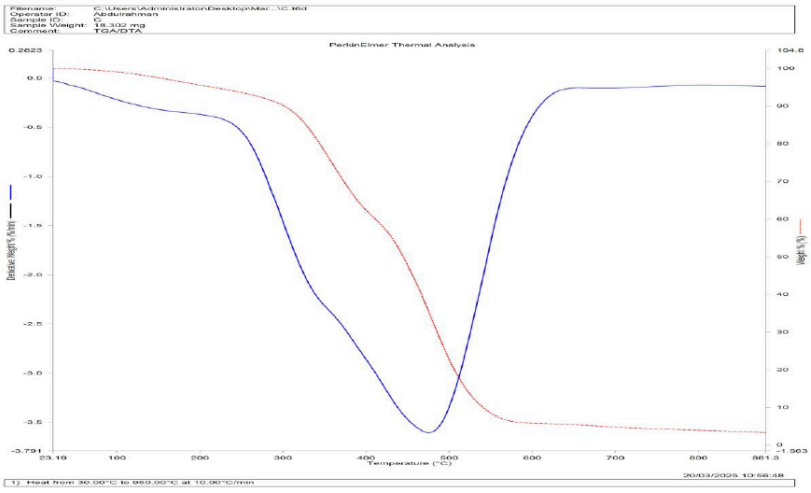

Figure 4c: TGA of bioplastic composite with 3% nanoclay.

4.4. GPR Validation

Table **3** presents the evaluation metrics for the predicted property using the GPR model.

The GPR model was employed as a preliminary validation tool to evaluate the mathematical trends of the nanocomposite properties. The model produced R^2 values ranging from 0.91 to 0.95, RMSE values from

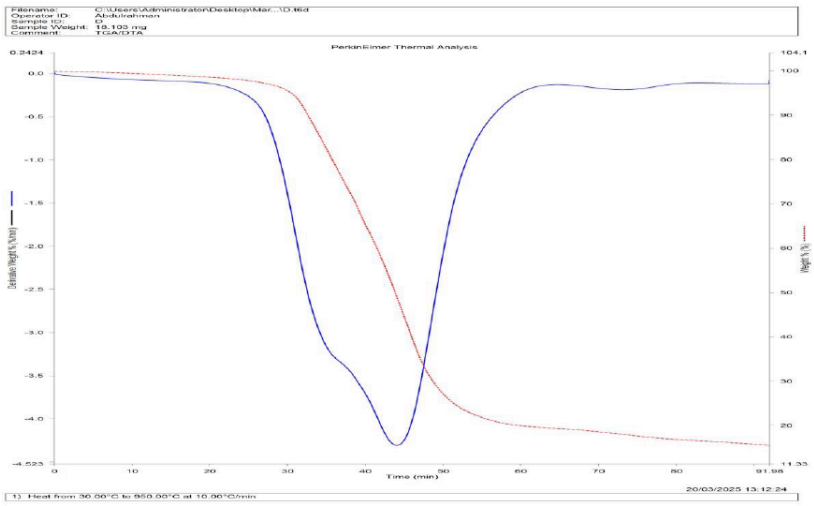

Figure 4d: TGA of bioplastic composite with 5% nanoclay.

1.18 to 16.50, and MAE values from 0.75 to 13.30. Although these evaluation metrics indicate low interpolation errors within this specific experimental set-up, however, it might be a definitive proof of strong predictive capability because the analysis is heavily constrained to a very small dataset containing only four experimental formulations (n = 4). Statistical metrics such as R^2 are highly sensitive and sometimes prone to overestimating model capability. Thus, this GPR model serves as an initial exploratory interpolation validating the internal consistency across the tested formulation intervals. A significantly larger experimental dataset would be required to establish the actual generalizability and robustness of the model as a forecasting framework.

Table 3: The Evaluation Metrics for the Predicted Property

Predicted Property	R^2	RMSE	MAE
Crystallinity (%)	0.93	2.20	1.25
Decomposing Temperature (°C)	0.95	4.90	3.30
YM (MPa)	0.92	16.50	13.30
TS (MPa)	0.91	1.18	0.75

5. CONCLUSION AND RECOMMENDATIONS

The morphology of the composite surface showed homogeneity with no visible agglomerates, while the x-ray diffraction (XRD) results showed that with increasing the volume fraction of the nanoclay in the polymer matrix, the crystallinity peaks of the biopolymer become wider to become amorphous. The result shows that bioplastic with 5% nanoclay has the highest TS and YM values but the lowest elongation at break indicating the lowest ductility and toughness. Hence, 3% nanoclay offers the best balance between strength and flexibility before aggregation issues reduce performance in the 5% nanoclay. However, the morphology and XRD patterns indicates that the samples with 5% nanoclay in the bioplastic had the best morphology and crystallinity. Furthermore, the thermogravimetric analysis (TGA) shows that the thermoplastic starch with 5% nanoclay has the highest thermal stability at 790°C.

The GPR model validated the internal consistency across the tested formulation intervals with R^2 values from 0.91-0.95, RMSE values from 1.18-16.50 and MAE values ranging from 0.75-13.30. Thus, this outcome indicates low prediction error.

This study provides a feasible and viable pathway for enhancing the application boundaries of starch-based bioplastics, thereby advancing environmental sustainability through circular economic principles. By utilizing organic waste such as starch extracted from cassava peels and bio-calcium derived from eggshell waste, this study contributes a methodological procedural for waste-to-wealth conversion. The developed ternary biopolymer films showed improved tensile strength, higher elastic modulus values, and enhanced thermal stability metrics up to a 3 wt.% MMT nanoclay loading. Conversely, the elongation at break systematically decreased, a trend directly attributed to the restricted mobility of the starch polymer segments within the tightly packed nanoclay network. Hence, findings indicate that nano-biocomposites show promising potential as short-term alternatives to conventional petroleum-based plastics in specialized food packaging applications. Future work should focus on the optimization of processing configurations within these specific packaging boundaries. Furthermore, this study is to a very small dataset containing only four experimental formulations (n = 4), a significantly larger

experimental dataset would be required to establish the actual generalizability and robustness of the GPR model as a forecasting framework.

DECLARATION

Availability of Data and Materials

The data that support the findings of this study will be made available upon a reasonable request.

Competing Interests

The authors declare no conflict of interest.

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Authors Contributions

The conceptualization, experimentation, results analysis, writing original draft, visualization, data curation, format analysis and editing were the collective work of the authors and all authors consent to its publication are the collective works of the authors

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