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The Mechanical and Morphological Properties of Nano Pineapple Crown Fiber (PCF) incorporated in Polyvinyl Alcohol, Carboxymethyl Cellulose (PVA/CMC) Bio-Nanocomposite for Food Packaging Applications

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Abstract
The essence of this study was to fabricate carboxymethyl cellulose polyvinyl alcohol (PVA/CMC) films and to improve their mechanical and morphological properties by using pineapple crown Nano fiber (PCF) as a reinforcement. The films were developed by incorporating PCF into the PVA/CMC (2:1) in 0wt.%, 3wt.%, 5 wt.%, and 7wt.% respectively, by the solution casting method in water as a solvent and 4.5 wt.% glycerol as a plasticizer. The particle size distribution of the treated pineapple crown fiber (PCF) powder was analyzed by TEM, revealing nanoscale, largely spherical particles ranging from 2.7 to 6.4 nm (average ≈4.5 nm). The dispersion of the Nano PCF contents in the film was analyzed by SEM, and it shows a more uniform dispersion of fibers within the polymer matrix and an enhanced interfacial contact. Thermal stability, degradation properties, and crystallinity were also studied. The biodegradability of cast films was also carried out by the soil burial method, and degradation increased as the number of days went by. 71% and 65% biodegradability was observed in 15 and 30 days for samples with 5wt.% and 3 wt.% PCF respectively. Tensile properties of blended film were tested, and it was found that the tensile strength 27.7MPa, % elongation at break 12.80%, and modulus of elasticity 2299MPa are higher in the blend with 3wt.% (PCF-B) as compared with the neat PVA/CMC film. This result tells the viability of this bio-nanocomposite material to be used in food packaging.
Keywords: CMC, PVA, Treated Pineapple Crown Fiber Bio-Nanocomposite, Packaging.

1. Introduction

Over the past few decades, plastics made from fossil fuels and disposables have contributed to environmental contamination. The depletion of fossil resources, environmental pollution, and global warming are the main drivers for the switch from fuel-based disposable polymers to biodegradable polymers (Amaregouda et al., 2022; Rodríguez et al., 2020). The use of nonrenewable and non-biodegradable petrochemical-based packaging in food preservation has put the environment at serious risk. Overcoming the issues brought on by non-degradable plastic garbage has been a difficult task. Finding an inexpensive, readily available, biodegradable, and nontoxic substance is necessary. The term "biodegradable" refers to materials that are broken down by naturally occurring microbes to produce an organic byproduct without releasing any hazardous gases (Gul et al., 2023; Sarwar et al., 2018). For packaging materials to be considered biodegradable, these biopolymers must be both renewable and biodegradable, and they must break down fully, collapse, and disintegrate into typical components without releasing any hazardous gases following disposal (Hussain et al., 2023; Suganthi et al., 2020). A new, environmentally friendly material should be sufficiently durable. It should preserve excellent food quality and prevent the growth of microorganisms that speed up food deterioration (Bajer & Burkowska-But, 2022).

Among various packaging materials, polymer-based materials are the best because they have a number of favorable characteristics, including ease of handling, light weight, transparency, and water impermeability. As edible, intelligent, and biocompatible materials that also meet the needs of the packaging industry, polymers of both natural and synthetic origin, such as polysaccharides (cellulose, starch, and chitosan), proteins (gelatin), polylactic acid (PLA), polyhydroxyalkanoates (PHAs), polyethylene (PE), etc., appear to be the most promising (Akhtar et al., 2022; Arefian et al., 2020; Ghosh & Katiyar, 2018).

The analysis of biodegradable polymers poly(vinyl alcohol) and carboxymethyl cellulose, which have been suggested for application in the food packaging sector, is the main subject of this study. One excellent polysaccharide-based biopolymer that is commonly accessible and well-used is carboxymethyl cellulose (CMC). It's being employed in pharmaceutical and biotechnology applications for its many beneficial attributes (Hussain et al., 2023). CMC is a naturally occurring polymer that comes from cellulose. CMC is essential in food packaging applications because of its biocompatibility, nontoxicity, and degradability (Dhandapani et al., 2022). Furthermore, CMC is widely employed in the food and pharmaceutical industries as a thickening and suspending agent due to its high hydrophilicity as a cellulose derivative. Scientists studying hydrogels for drug delivery applications have been particularly interested in CMC because of its good modifiability, swellability, and nontoxicity (Arefian et al., 2020).

When used to make polymer nanocomposites, poly(vinyl alcohol) (PVA), a commonly used polymer, has remarkable chemical and physical properties as well as strong tensile and compression strengths(Haghighi et al., 2021; Islamipour et al., 2022; Youssef et al., 2019). PVA is a synthetic polymer that is hydrophilic, semicrystalline, and has a planar zigzag structure. It is resistant to deterioration in the majority of physiological conditions and has both chemical and thermal stability. Additionally, because of its high polarity, high biocompatibility, and nontoxicity, it is soluble in water. As a result, processing the mixture is simple. Additionally, PVA is very capable of creating biodegradable films (Oun et al., 2022; Thungphotrakul & Prapainainar, 2024).

The world's most popular fruit is the pineapple, although its crown is thrown away as waste. Nonetheless, the bioactive compounds and chemicals found in these pineapple crowns (PaCn) have a wide range of potential uses in the pharmaceutical, cosmetic, food and beverage, biofuels, bioplastics, paper and pulp products, and other industries. It has been demonstrated that the waste from PaCn conversions reduces waste generation, and the products produced by the conversion would help the waste-to-value idea (Arumugam et al., 2025). Cellulose, hemicellulose, lignin, and pectins are among the materials that often make up the pineapple crown. This residue's high cellulose content, roughly 80%, makes it very appealing for extraction and use in a variety of applications. Previous studies such as Fareez et al. (2018) and Prado and Spinacé (2019)indicate that the pineapple crown is interesting for cellulose extraction.

Lignin bound to cellulose significantly hinders the effective utilization of cellulose. In nature, cellulose is not found in a pure form but is closely associated with hemicellulose and lignin within lignocellulosic structures (Asrofi et al., 2018). Lignin is highly resistant to degradation due to its strong interactions with cellulose and hemicellulose. It comprises more than 30% of plant biomass and provides structural rigidity by reinforcing cellulose microfibrils and binding to hemicellulose (Asrofi et al., 2018). Consequently, lignin removal is essential and is typically achieved through delignification processes (Manurung et al., 2018), which may be carried out using physical, chemical, physicochemical, or biological methods (Kunusa et al., 2018).

Natural fiber-reinforced polymer composites have garnered significant interest as eco-friendly substitutes for petroleum-derived materials because of their biodegradability, lightweight nature, renewability, and advantageous mechanical characteristics. Among the different lignocellulosic fibers studied, fibers derived from pineapple have appeared as potential reinforcements due to their high cellulose levels and remarkable specific strength. Earlier research has mainly concentrated on pineapple leaf fiber (PALF) Glória et al. (2017); Gnanasekaran et al. (2021); Rahman et al. (2021); Sethupathi et al. (2024) as reinforcement in thermoplastic and thermosetting matrices, while relatively less emphasis has been placed on pineapple crown fiber, an agricultural byproduct frequently discarded post-fruit harvest. Pineapple crown fiber is an overlooked biomass resource that holds considerable promise for enhanced applications. While numerous studies have indicated the extraction of cellulose or nanocellulose from pineapple leaves Almendárez-Camarillo et al. (2022); Chawalitsakunchai et al. (2021); Cherian et al. (2010); Nguyen et al. (2021), there is a scarcity of research on integrating treated pineapple crown nanofibers into biodegradable polymer film systems. Additionally, the majority of reported studies have concentrated on single-polymer matrices or traditional composite uses, with minimal exploration of pineapple crown nanofibers in films made from polysaccharide–synthetic polymer blends for sustainable packaging purposes. The current research fills this void by employing chemically modified pineapple crown nanofibers as a reinforcement in a poly(vinyl alcohol) (PVA)/carboxymethyl cellulose (CMC) composite. The uniqueness of this study resides in (i) the utilization of pineapple crown waste as a source of nanoscale cellulose reinforcement, (ii) the addition of treated pineapple crown nanofibers into a PVA/CMC composite system, and (iii) the thorough assessment of the resulting films via mechanical, structural, thermal, and morphological analysis. In contrast to earlier research that mainly targeted pineapple leaf fibers or micro-scale reinforcements, this study examines the influence of nanoscale pineapple crown fibers on the crystallinity, interfacial interactions, and performance of biodegradable PVA/CMC films. The results offer fresh perspectives on creating high-efficiency, eco-friendly packaging materials using agricultural waste resources.

2.0 Materials and methods

2.1 Materials

Pineapple crown wastes were obtained from a local fruit vendor along Regina road, Okpuno Awka, Anambra State. Nigeria. Carboxymethyl Cellulose (CMC, Central Drug House (P) LTD. India), Polyvinyl Alcohol (PVA, MonoChem, India), Glycerol ($C_3H_8O_3$, Merk, Darmstadt, Germany), were used for film formation. The distilled water used in this study was purchased from a reliable chemical vendor at the main market, Upper Iweka, Onitsha. Anambra State.

2.2 Preparation and Delignification of Pineapple Crown Fiber

According to the method used by de CM Miranda et al. (2019) Minmunin et al. (2018), with slight modifications, pineapple crown leaves were first thinned, washed thoroughly with clean water, and dried in a laboratory oven at 65 °C for 48 h. After drying, the leaves were ground with a milling machine to reduce the particle size, after which the material was passed through a mesh sieve (75µm) to obtain a uniform fiber size suitable for subsequent chemical treatment.

The sieved biomass (PCF) was subjected to an alkaline delignification process using a 10% w/v sodium hydroxide (NaOH) solution, following the procedure described by Mittal and Chaudhary (2018). To prepare the NaOH solution, 100 g of NaOH pellets was measured using a digital weighing balance and dissolved in 1000 mL of distilled water. A total of 15 g of the biomass was then placed into a beaker and mixed with 250 mL of the prepared NaOH solution. The mixture was stirred thoroughly until a homogeneous suspension was obtained.

The prepared mixture was placed in a water bath maintained at 60 °C for 2 h with continuous stirring, in line with the method reported by (Mikulski & Kłosowski, 2022). Following alkaline treatment, the mixture was filtered using a fabric sieve and rinsed repeatedly with distilled water to ensure complete removal of solubilized lignin. The remaining solid residue (delignified fiber) was finally dried in an oven at 50 °C and stored for further analysis.

2.3 Preparation of Nano PCF

Nano-PCF was produced from waste pineapple crown through a mechanochemical milling approach, as described by (Gbadeyan, Adali, Bright, & Sithole, 2021). A dry milling technique was applied to obtain nano-sized particles. Initially, 30 g of pre-ground PCL powder was weighed and subjected to dry milling using a planetary ball mill (Retsch PM 100, Germany) to achieve finer particle refinement (Gbadeyan, Adali, Bright, Sithole, et al., 2021). The milling system consisted of a 500 mL stainless-steel jar (inner diameter: 100 mm) and 50 stainless-steel balls, each 10 mm in diameter. Milling was carried out at 450 rpm for 30 minutes in a clockwise rotation. Following the milling process, the resulting powder was sieved using a mechanical sieving shaker (Retsch AS 200 Basic, Germany) to obtain particles with sizes in the range of 2.7 - 6.4nm.

2.4 Preparations of PVA/CMC Composite Films added with Nano Pineapple Crown Filler

The films were fabricated using a solution-casting technique adapted from Mohammadi et al. (2019) Amaregouda et al. (2022), with minor modifications. Briefly, 150 mL of distilled water was used to dissolve 10g of PVA and 5g of CMC separately. The PVA solution was heated in a 90 °C water bath, while the CMC solution was maintained at 50°C; both were continuously stirred for approximately 30 minutes to ensure complete solubilization.

After dissolution, the two polymer solutions were combined, mixed thoroughly, and returned to the water bath for an additional 10 minutes. Glycerol was incorporated as a plasticizer, with its quantity for each formulation calculated as 30wt.% of the corresponding PVA/CMC mass. The glycerol was added using a 10-mL syringe, followed by 30 minutes of mixing.

0, 3wt.%, 5wt.%, and 7 wt.% of Nano-PCF were weighed using an electronic balance, dispersed in 10 mL of distilled water, and subsequently added to the PVA/CMC/glycerol mixture. The resulting solution was divided into 40-mL portions and cast into plastic Petri dishes. This procedure was repeated four times to obtain four film replicates.

The cast films were dried at ambient conditions for approximately 96 hours (4 days) and then transferred to a desiccator to minimize exposure to environmental moisture.

2.5 Transmission Electron Microscopy (TEM) Analysis of the Pineapple Crown Powder

The ball-milled pineapple crown powder was deposited on a copper grid and was investigated using JEM-ARM 200FG transmission electron microscope at 80kV. The morphological image obtained was analysed for particle size of the PCF sample using Image J 1.52 visualization software by measuring and averaging the size of over 30 - 50 nanoparticles.

2.6 Morphological and Chemical Structural Analysis of Nanocomposite Films

The dispersion of the nano-pineapple crown fillers within the polymer matrix was examined by scanning electron microscope on a JOEL-JSM-7600F at 5kV. The film samples were dried overnight under vacuum at 40 ^{O}C and then prepared by cryogenically fracturing to expose their cross-section for examination. To improve contrast in the SEM, the samples were coated with a 5 nm layer of platinum before analysis.

2.7 Thermogravimetric Analysis

The TGA was conducted using UW_MSE TA Instruments TGA Q50 thermogravimetric analyser to investigate the thermal degradation process of the films. The samples were heated from 30 °C to 900 °C at a heating rate of 10 °C/min and under a nitrogen atmosphere.

2.8 X-ray Diffraction (XRD)

The films were examined using Rigaku D/Max- IIIC Xray diffraction at 40 kV X-ray radiation. Scattered radiation was detected in the angular range (2 θ) between 5°/min and 50°/min at room temperature with a CuKa radiation set at 40Kv and 20mA, and was plotted as a curve used to determine the crystallinity percentage of the film in accordance with the method described in the literature.

The percentage crystallinity can be calculated using the equation (Jain et al., 2019).

$$\% \text{ Crystallinity} = \frac{I_{002} - I_{am}}{I_{oo2}} \times 100 \quad (1)$$

Where I_{002} is the peak intensity of the crystallographic plane, and I_{am} is the intensity scattered by the amorphous part of the sample.

2.9 Differential Scanning Calorimetry (DSC).

The melting and crystallization properties of the matrix polymer and the composites were examined utilizing a TA Equipped with an Instruments 2920 Modulated Differential Scanning Calorimeter (DSC). Using a cooling connection under a nitrogen atmosphere, the data were gathered at a continuous heating temperature between 15 °C and 200 °C at a heating rate of 5 °C/min. A sample weight of approximately 10 milligrams was used. The samples were sealed in aluminum pans, and the sealed samples were placed on an empty reference aluminum pan and the furnace's heating surface. The heat movement and energy shifts in and out of the samples in the sealed aluminum pans were noted using an empty aluminum pan as a comparison. The melting temperature was taken from the apex of the heating curve.

2.10 Mechanical test

Tensile testing was performed on the film samples using a Model Zugfestigkeit 440gu philips universal mechanical testing machine in line with ASTM D882 guidelines. The films were cut into rectangular strips measuring 100 mm by 20 mm. Before each test, the thickness of every specimen was determined with a digital caliper (Traceable® Digital Calipers 6 in, Fischer Scientific).

2.11 Soil Burial Degradation

The produced films were assessed for soil burial degradation using the technique described according to (Patil et al., 2021). 20. After drying, the film sample (1.415 cm × 1.415 cm) was weighed. Following weighing, the dried films were buried in black dirt at a depth of 10 cm. For fifteen days, ten milliliters of water were given daily. The samples were carefully taken out of the soil after 18 days, dried, and weighed again to determine the weight loss.

3.0 Result and Discussion

3.1 Mechanical Properties

Table 3.1: Results of tensile, % elongation, and modulus of elasticity of nano composite film

Sample ID	Tensile Strength (MPa)	Elongation (%)	Modulus of Elasticity (MPa)
A-P.C.F	20.6± 4.8	10.75± 0.14	2070± 350
B- P.C.F	27.7± 3.5	12.80± 0.13	2299± 300
C- P.C.F	25.2± 3.3	10.60± 0.10	2055± 220
D- P.C.F	24.3± 3.0	10.40± 0.12	2242± 310

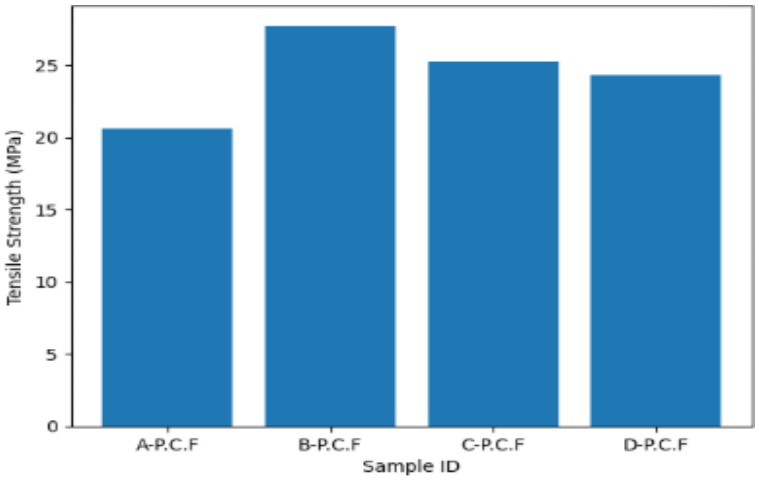


Figure 3.1: Tensile Strength of P.C.F Composite Samples (A–D)

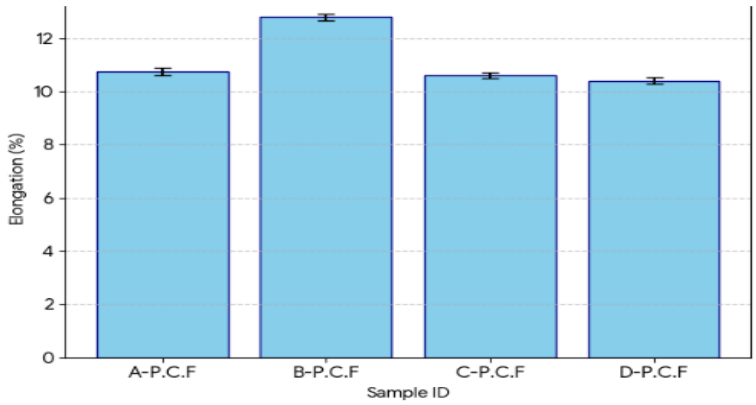


Figure 3.2: % Elongation of P.C.F Composite Samples (A–D)

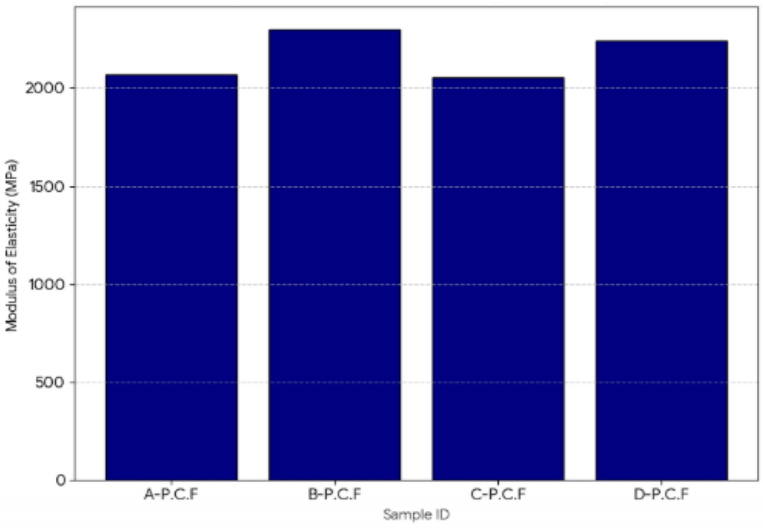


Figure 3.3: Modulus of Elasticity of P.C.F Composite Samples (A–D)

The observed and calculated results of tensile strength, Young's modulus, and elongation at break of blend films are tabulated in Table 3.1. The interfacial bonding between the PCF and polymer matrix has been found to affect the properties of the polymer blend hybrid bio-composite. It was observed that sample B with 3wt.% of PCF composites showed a higher and optimum value of tensile strength 27.7 MPa, tensile modulus 2299 MPa, and percentage elongation at break of 12.80%.

Pineapple crown fiber contains high levels of structural cellulose (56%-83%), characterized by stiff crystalline domains (Prado & Spinacé, 2019). When scaled down to the nanoscale, the significant specific surface area of these particles enhances extensive hydrogen bonding with the hydroxyl (OH) groups of PVA and the carboxyl (-COO-) groups of the CMC matrix. At this particular loading threshold, the nanoparticles are evenly distributed, facilitating effective stress transfer from the pliable PVA/CMC matrix to the stiff filler.

The enhancement in tensile strength can be linked to the elevated cellulose content and rigidity of nanomaterials derived from pineapple, which promote stress transfer from the polymer matrix to the reinforcing particles during tensile loading. Comparable enhancements have been noted for PVA films strengthened with cellulose nanofibers derived from pineapple biomass, with tensile strength rising by about 28% because of strong filler–matrix interactions and enhanced crystallinity (Forti et al., 2021; Mahardika et al., 2024; Noor Mohamed et al., 2017).

The neat biofilm has less tensile strength, and it is increased to 27.7 MPa in blends of PVA with CMC and PCF. This increase in strength might be a result of better cross-linking between CMC and PCF (Wongkom & Jimtaisong, 2017). Raising the filler concentration above the ideal level results in a slight decrease in tensile strength to 25.2 MPa (C-P.C.F) and 24.3 MPa (D-P.C.F). This pattern is a frequent occurrence in biocomposites reinforced with nanocellulose (PNC-type films). Due to its elevated surface energy, surpassing the critical loading threshold causes self-aggregation of particles through strong intermolecular hydrogen bonding. These agglomerates function as localized stress holders instead of supportive entities, triggering early micro-cracking during mechanical strain. Comparable patterns have been observed in PVA composites reinforced with cellulose nanofibers, where mechanical characteristics are enhanced until reaching an optimal filler concentration, after which they fall due to particle clustering and diminished dispersion quality (Madivoli et al., 2022; Mahardika et al., 2024). From the above result, it is evident that the composite made with the inclusion of 3wt.% PCF will perform well. The range of tensile strength obtained in this study falls within the range obtained by Johny et al. (2023); Kumar et al. (2022) in their studies.

Elongation at break in neat PVA/CMC is 10.75%, which is increased in blend films in the range 10.40–12.80% due to the consequences of both cross-linking and plasticizing effects. The Young's modulus of blend films is decreased by up to 10%. This sharp decrease in modulus may be attributed to the plasticizing effect of glycerol (Kalambettu et al., 2015). Generally, incorporating a rigid filler limits the movement of polymer chains, resulting in increased tensile strength but significantly reduced ductility. Notably, Sample B-P.C.F differs from this trade-off, exhibiting concurrent rises in both tensile strength (27.7 MPa) and elongation (12.80 ± 0.13%). This behavior indicates that at this particular concentration, the filler interacts synergistically with the matrix. The small particle size can change the semi-crystalline structure of the PVA blend, enabling nearby polymer chains to slide along the filler interfaces prior to breaking. (Esquivel-Alfaro et al., 2025)

In a nutshell, it may be inferred that tensile strength is mainly directed by the cross-linking effect, Young's modulus by plasticizing effect, and % elongation by both cross-linking and plasticizing effects in the present study. The result of mechanical studies shows that the ternary blends have high tensile strength and high elongation at break, which are better than those of binary PVA/CMC and PVA/PCF blends Kalambettu et al. (2015) and might be used in food packaging and other applications in which high mechanical properties are required.

3.2 Transmission Electron Microscopy

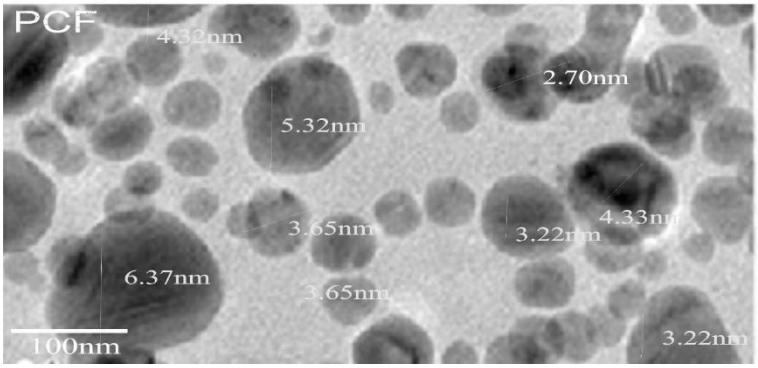


Figure 3.4: Transmission Electron Microscopy Image of PCF Powder

TEM analysis of treated pineapple crown fiber (PCF) powder exposed nanoscale, largely spherical particles with sizes ranging from 2.7 to 6.4 nm (average ≈4.5 nm). The treatment greatly reduced the fibers into uniform nanoparticles by eliminating amorphous regions, resulting in cellulose-rich structures with greater crystallinity and surface reactivity. Alwani et al. (2014) suggested that nanoscale morphology and dispersion reveal the potential of treated PCF as a bio-based nanofiller for reinforcing polymer composites and upholding mechanical, thermal, and barrier properties in sustainable material applications.

3.3 Scanning Electron Microscopy

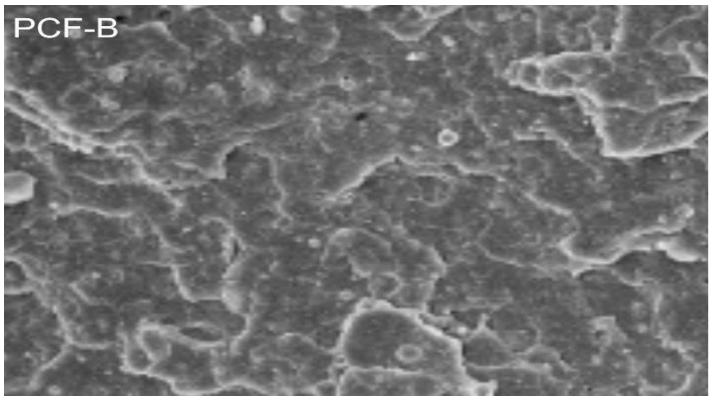


Figure 3.5: Scanning Electron Microscopy of PVA, CMC, PCF Bio-Nanocomposite

In contrast to what is usually observed in composites with untreated fibers Johny et al. (2023), the image in Fig. 3 .5 shows a comparatively compact, uniform, and consistent surface structure devoid of significant large cracks, gaps, or pronounced phase separation, in contrast to pure PVA/CMC films that usually show smooth, glassy fracture surfaces denoting brittle failure (Niedźwiecka et al., 2025). The inclusion of PCF results in a ductile or pseudo-ductile fracture feature. The coarse surface indicates that the nanofibers successfully hinder crack development within the matrix. There is no indication of significant agglomeration of the nano-treated pineapple fibers. The even distribution of the reinforcing phase is essential for load transfer. Even dispersion at the nano-scale verifies efficient surface treatment of the PCF, enabling it to interact smoothly with the PVA-CMC mixture through hydrogen bonding (Esquivel-Alfaro et al., 2025). The lack of clear, deep voids or gaps surrounding the pulled-out fibers is a crucial characteristic that suggests that the hydrophilic matrix and the nanocellulose have good interfacial adherence (Nizardo et al., 2024). Nevertheless, the presence of fiber pull-out features indicates that, despite the strong interfacial adhesion between the matrix and the reinforcement, failure occurred predominantly through fiber debonding and slippage rather than fiber fracture. This failure mechanism is commonly observed in thermoplastic composites reinforced with cellulosic fibers, where the interfacial bond strength, although significant, may not be sufficient to prevent fiber extraction under applied stress completely. (Kabir et al., 2012; Pickering et al., 2016). The uniformity of the background phase (PVA/CMC) is significant. The absence of phase separation between PVA and CMC indicates the creation of a miscible blend, enabled by intermolecular hydrogen bonds between the hydroxyl groups of PVA and the carboxyl groups of CMC (Hamza & Habeeb, 2024; Kabir et al., 2012; Niedźwiecka et al., 2025).

The SEM analysis verifies that nano-treated Pineapple Crown Fiber acts as a strong reinforcement for PVA/CMC matrices. The rough fracture surface, consistent fiber distribution, and strong interfacial bonding account for the enhanced mechanical properties noted in the literature for these sustainable composites. The lack of notable voids indicates that ideal processing conditions were met, producing a dense, uniform material appropriate for packaging use.

3.4 Thermogravimetric Analysis

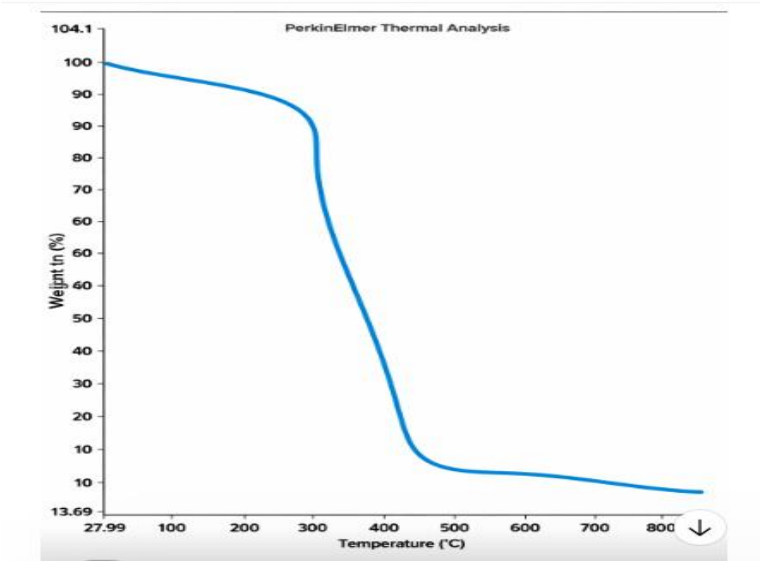


Figure 3.6: TGA of PVA, CMC, PCF Bio-Nanocomposite

The TGA graph of CMC/PVA reinforced with Pineapple crown as filler showed a multi-step thermal degradation trend Fig 3.6. The initial weight loss was observed at less than 200 °C due to moisture evaporation and loosely bound water in the film (Kapila & Kirtania, 2025; Li et al., 2018). Furthermore, a major weight loss event occurred between 320 °C and 470 °C, indicating a more thermally stable char formation due to the presence of the filler (Krishnasamy et al., 2021). The Lignin, cellulose, and silica components of the pineapple crown are responsible for the stability of the film structure upon heating (Hossain et al., 2024), which signals the main decomposition of the polymer matrix (CMC and PVA) Adnan et al. (2025), thereby losing their structural integrity, and the decomposition curve is consistent with biopolymer blends (Pereira et al., 2021).

Ultimately, the incorporation of treated PCF as a nano filler significantly enhances the thermal stability and char yield of the CMC/PVA matrix (Jayaprakash et al., 2023). The high residual mass of 27.9% suggests improved flame retardancy and a potential for high-temperature application (Al-Muntaser et al., 2023; Lee et al., 2020).

3.5 Differential Scanning Calorimetry (DSC).

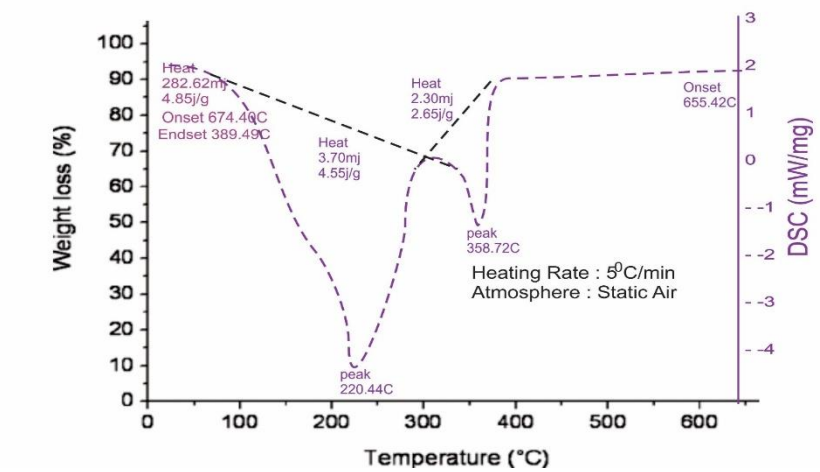


Figure 3.7: DSC of PVA, CMC, PCF Bio Nanocomposite

The DSC graph of CMC/PVA loaded with pineapple crown as filler PCF-B reveals multiple distinct peaks at lower and intermediate temperatures between 220 °C and 358 °C. This shows several thermal events representing phase transitions, which represent endothermic events due to water loss and polymer decomposition. The Tg of the material showing phase change was observed at the onset temperature range of 674.40 °C – 655.420 °C. The increased number of transitions in Fig. 3 .7 implies greater material complexity, more tunable thermal behavior, better dispersion of the filler, improved interfacial adhesion, and higher char residue. The energies associated with

these transitions are shown by the area under the curve (2.65 J/g at 358.72 ºC), quantifying the energy absorbed during these changes. The implication of more peaks at lower temperatures is enhanced material complexities with new and more controllable transitions, meaning better processability and application tenability. With respect to the physical property enhancement, the extra thermal events imply a stronger interface, improved mechanical strength, greater flexibility, and better energy absorption due to improved filler-polymer interaction. For the thermal stability, the extra peak shows that the filler improves resistance to heat-induced degradation and benefits packaging. Pineapple crown as filler has been shown to possess the ability to add transitions, as observed. which means the film properties can be tailored for specialized uses and justify their use in food packaging.

3.6 X-ray Diffraction (XRD).

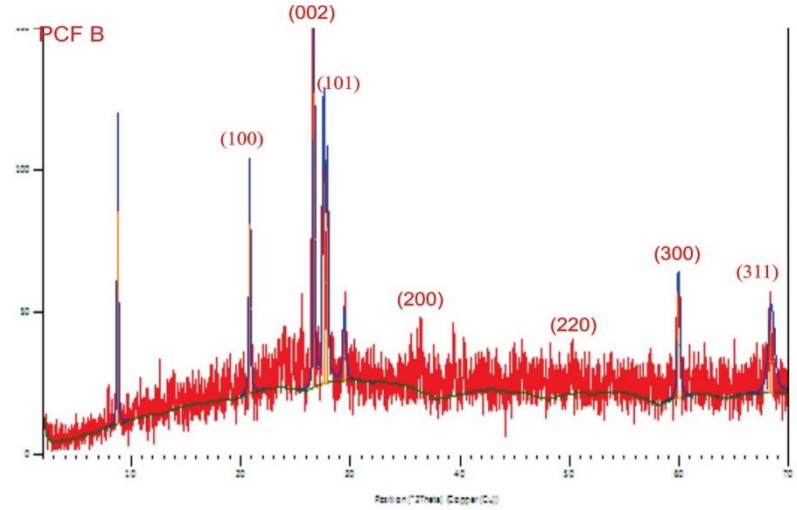


Figure 3.8: XRD of PVA, CMC, PCF Bio Nanocomposite

The PVA/CMC/Treated Pineapple Crown Fiber (PCF-B) composite exhibits a distinctive semi-crystalline X-ray diffraction (XRD) pattern, which reflects the cooperative integration of the polymer matrix and the nano-reinforcement. The diffractogram exhibits distinct sharp reflections superimposed on a broad amorphous halo. The Cellulose I polymorph is indicated by the major crystalline peaks seen at 2̣ values that correspond to the (100), (002), and (101) planes. The presence of highly organized cellulose crystallites formed from the treated pineapple crown fibers is specifically confirmed by the high-intensity peak at the (002) plane (≈ 26.50). The narrow Full Width at Half Maximum (FWHM) of the (002) reflection and the refinement of the diffraction peaks indicate that non-cellulosic components like lignin and hemicellulose were effectively eliminated by the PCF-B's chemical treatment. By increasing the fibers' Crystallinity Index (CI), this purification procedure enables them to function as efficient nucleating agents in the PVA/CMC matrix. A considerable level of long-range structural order is shown by the existence of higher-order reflections (200), (220), (300), and (311) at higher 2̣ angles. This implies that the nanofibers are evenly distributed and retain their structural integrity throughout the creation of the composite. The incorporation of these crystalline nanofibers into the mostly amorphous PVA/CMC blend enhances the bio-composite's mechanical and thermal stability.

The crystallinity index (CrI) of PCF-B was determined to be 93.2% according to the Segal equation (equation 1), reflecting a highly organized cellulose structure in the composite. This value is significantly greater than the figures noted for untreated pineapple leaf fibers, which generally fall between 35 to 55% Jain et al. (2019); Moreno et al. (2019); Saha et al. (2021), and surpasses the 58–85% range usually seen for alkali-treated pineapple fibers (Gaba et al., 2021; Saha et al., 2021; Sasikala & Umapathy, 2018) . Additionally, the CrI achieved in this study is somewhat greater than the 80–89% values noted for nanocellulose sourced from pineapple and processed via combined alkali, bleaching, and acid hydrolysis methods (Huda et al., 2008; Lee et al., 2020). The increased crystallinity indicates that the treatment given to the pineapple crown fibers successfully eliminated amorphous elements like lignin, hemicellulose, and pectin while maintaining the cellulose I crystalline structure. The elevated crystalline content is anticipated to improve stiffness, tensile strength, thermal stability, and stress-transfer effectiveness in the PVA/CMC matrix. As a result, the crystallinity attained in PCF-B is favorable when compared to earlier reported pineapple fiber-reinforced materials and showcases the efficacy of pineapple crown nanofibers as reinforcement for biodegradable polymer composites.

3.8 Soil Degradation Test

Synthetic films have negative impacts on the environment; it is vital to look into their biodegradation (Amaregouda et al., 2022). The biodegradability of the produced films was assessed using a moisture-controlled soil burial degradation test. Samples of PVA-CMC-PCF have demonstrated good biodegradation activity. In a similar method by Rumi et al. (2024), the films were weighed and left in the ground for 15 to 30 days, and the rate of film degradation was assessed after the intended time. There was a noticeable weight drop and a reduction. In line with the earlier research on the PVA/CMC system Channa et al. (2022), films with 5wt.% and 7wt.% PCF content demonstrated the best degradation values of 71 and 65%, respectively. The chemical compositions of PVA and CMC are rich in hydroxyl groups and oxygen-containing connections that increase hydrophilicity and enable microbial and enzymatic actions, thus encouraging biodegradation (Rahman Khan et al., 2024; Tyagi & Thakur, 2023). However, a high rate of disintegration for the 3wt. % film was noted, which may be related to the CMC's high carbohydrate content, which gives bacteria a significant carbon source.

However, a high rate of disintegration for the 5wt.% film was noted, which may be related to the CMC's high carbohydrate content, which gives bacteria a significant carbon source. Soil moisture stimulates bacterial activity, which is a key factor in the breakdown process. The films expanded as a result of the soil's water and moisture diffusing into them, improving their biodegradability. Compared to earlier research that reported degradation rates at 36 and 40%, the current study's results are outstanding (Hussain et al., 2023)

4.0. Conclusion

The SEM result shows a more uniform dispersion of fibers within the polymer matrix and an enhanced interfacial contact. The surface morphology is characterized by a rough and unconventional texture, which is suggestive of the natural fiber content

The tensile strength, elongation at break, and modulus of elasticity showed that PVA, CMC, and PCF blended films all affect the tensile strength, while the PVA/CMC/PCF Bio Nanocomposite film with 3wt.% PCF indicated the highest effects on the tensile, elongation at break, and modulus of elasticity. The observation of the morphological, thermal and the dynamic mechanical properties showed that the PVA/CMC film reinforced with 3 wt.% PCF had the best interaction and cohesive structure, and the thermal stability was increased from 200 ^{o}C to 400 ^{o}C on the addition of PCF. The improvement in the thermal properties indicates that PCF acted as an insulator to the volatile products generated during decomposition. Additionally, it was observed that the final residue percentage > 450 ^{o}C indicating a more thermally stable char formation due to the presence of the filler. The Lignin, cellulose, and silica components of the pineapple crown are responsible for the stability of the film structure upon heating

The presence of highly organized cellulose crystallites formed from the treated pineapple crown fibers is specifically confirmed by the high-intensity peak at the (002) plane (≈ 26.50).

The chemical structures of PVA and CMC contain heterobonds, which aid in the biodegradation process. However, a high rate of disintegration for the 5wt.% film was noted, which may be related to the CMC's high carbohydrate content, which gives bacteria a significant carbon source.

Finally, the obtained results indicated that PCF reinforced PVA/CMC films can be considered as a potential alternative for packaging materials such as food wrappings.

5.0 Recommendation

The researcher recommends that more studies can be carried out on the microbial activities on PVA/CMC/PCF nanocomposites film for sustainable packaging applications.

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Declaration of Generative AI and AI-assisted technologies in the writing process

During the preparation of this manuscript, the authors used Deep Seek AI to compare and analyse some sections of the results with related literatures in line with the results obtained. After using this tool, the authors reviewed and edited the content as needed and take full responsibility for the content of the publication.

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