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Biodegradable Polymer Composites Enhanced via Chemical Treatment for Packaging Applications

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ABSTRACT

This study is on the agricultural biomass waste material corn-husk (CH) fibers by blending it into polycaprolactone (PCL), a biodegradable polymer. The polymer served as matrix while the corn-husk fibers were used as fillers. The corn-husk fibers were modified chemically by NaOH treatment to enhance their compatibility with the PCL blend. Unlike previous studies that focused on untreated natural fibers, this work investigates the impact of alkali modification on the fiber-matrix interaction and overall composite performance. The corn husk were treated with different proportions of alkali (5, 10, 15 and 20 mL) of 2 % aqueous alkali solution to improve their compatibility with PCL. The composites were prepared via melt compounding using a twin-screw extruder and compression molding for the different alkali treated fibers. The effect of the treatment ratio on the morphological, mechanical and thermal properties of the CH/PCL composites were investigated. The study reveals a substantial improvement in mechanical properties, with tensile strength increasing from 5.5 MPa (untreated) to 10.73 MPa at an optimal 10 mL NaOH treatment, demonstrating nearly a hundred percent enhancement over conventional untreated biocomposites. Morphological and thermal analyses confirm improved fiber dispersion and interfacial adhesion, contributing to superior composite stability. Notably, excessive alkali treatment led to fiber degradation, negatively impacting mechanical integrity. This research advances the field by establishing an optimized treatment threshold that maximizes reinforcement efficiency, setting it apart from prior works that lacked systematic treatment optimization. The findings highlights the potential of CH/PCL composites in biodegradable packaging and sustainable material applications, offering a viable route toward high-performance eco-friendly alternatives in line with circular economy principles and promoting environmental sustainability.

Keywords: Agricultural waste; alkali; fiber; mechanical properties; polycaprolactone

INTRODUCTION

The environmental issues associated with conventional petroleum-based plastics, including their long-term persistence in ecosystems and contribution to global pollution, have driven the quest for alternative materials with biodegradable and renewable properties [1]. In this regard, biopolymers have attracted considerable interest as sustainable alternatives due to their natural degradability and potential to lessen dependence on fossil-derived stocks. Composite polymeric materials, which combine two or more chemically and physically distinct phases, have emerged as a popular class of innovative materials [2].

Polymeric materials have a lower density compared to metals, allowing them to be molded into various shapes and applications while maintaining reduced weight, enhanced strength, and corrosion resistance [3]. The packaging industry is a vast global sector interconnected with food, medicine, households, and nearly all business domains. Ideal packaging should be safe, cost-effective, antimicrobial, sustainable, and environmentally friendly [4]. However, most conventional packaging materials such as polyethylene, polystyrene, polypropylene, and polyethylene terephthalate are derived from fossil fuels, making them neither sustainable nor eco-friendly.

There is a strong growing adoption of green packaging materials as environmental concerns sours higher, with the market projected to reach 9 billion USD by 2025 [5]. Polypropylene composites (PPCO) are increasingly utilized in packaging, and ongoing research aims to make PPCO components lighter, stronger, and more cost-efficient. The integration of polymers with natural fiber fillers to create biocomposites presents a sustainable and technically viable alternative to conventional non-biodegradable plastics. Many studies had focused on the suitability of agricultural waste materials and whether they are fully biodegradable, environmentally friendly, abundantly available, renewable, and cheap and of low density [6]. Cornhusk fiber (CHF) is a solid waste product from the harvesting process of corn barn having high moisture content of approximately 55% to 65% and high silica content of approximately 25 %, due to these characteristics, fibers for composite preparation should be cleaned of oil and lignin [7].

Natural fibers are primarily composed of three main natural polymers: cellulose, non-cellulosic carbohydrates commonly known as hemicellulose, and lignin. These components are distributed throughout the cell wall with varying degrees of organization. Interfacial adhesion between ligno-cellulosic fibers and matrices is an essential factor in material selection and this vary on the fiber location, age, and species amongst others [8]. Alkali treatment can remove lignin and other non-cellulosic materials from the surface of fibers, improving their interaction with the polymer. This chemical modifications is expected to enhance the dispersion of CH powder (filler) within the PCL matrix, thereby improving the overall performance of the composite material. While CH has been studied as a filler for various polymer matrices, its treatment potential in CH/PCL blend has not been extensively explored, particularly in relation to surface modification techniques. Cellulose is the main component in lingo-cellulosic fibers and the reinforcing material within the cell wall. Cellulose is a linear crystalline condensation polymer consisting of a hydro glucopyranose units held together by β-1, 4-glycosidic bonds shown in Figure 1. It is a high molecular weight homopolymer of glucose and it is laid down in micro fibrils where extensive hydrogen bonding between the cellulose chains produces a strong crystalline structure [9].

The actual base unit, the cellobiose, consists of two molecules of glucose. The polymer chains are ordered in three-dimensional levels forming the supramolecular structure of cellulose as shown in Figure 1. The linear polymeric chains form sheets that are held together with hydrogen bonds. These are connected by weak Vander Waals bonds generating micro-fibril crystalline structures. The overall structure of cellulose consists of crystalline and amorphous regions while the mechanical properties of cellulose depend on the proportion of each region and the spiral angle of micro-fibrils because of its geometrical conditions.

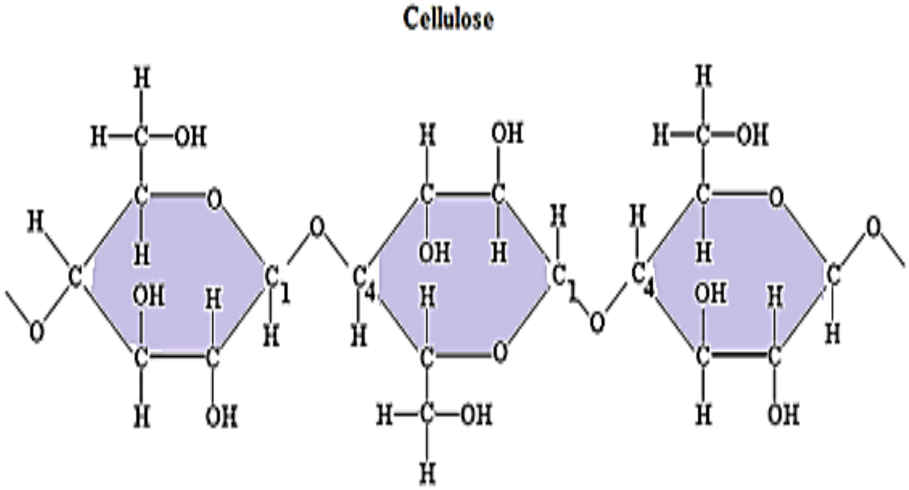
Cellulose

Figure 1. Molecular structure of cellulose [9]

In recent years, extensive research has been conducted on natural fiber-reinforced polymer composites, yet limited attention has been given to optimizing the chemical treatment process to enhance fiber-matrix interaction, particularly for corn-husk (CH) fiber in polycaprolactone (PCL) composites. While previous studies have explored untreated CH, rice husk and kenaf fibers or other lignocellulosic fillers in biodegradable polymers, this study presents a systematic approach to alkali modification. The unique combination of CH and PCL was selected due to their biodegradability, cost-effectiveness, and complementary properties where PCL provides excellent processability and CH fibers offering reinforcement potential from an agricultural waste source. Unlike earlier works, this study optimizes the alkali treatment process and investigates its impact on composite morphology, thermal stability, and mechanical integrity, establishing an optimal treatment threshold for maximizing composite performance. Thus, the objectives of this study are to:

i. Investigate the effect of alkali treatment on the interfacial adhesion and dispersion of CH fibers in a PCL matrix.
ii. Analyze the mechanical, thermal, and morphological properties of CH/PCL composites at different alkali treatment levels.
iii. Identify the optimal alkali treatment concentration that maximizes composite performance without fiber degradation.

MATERIAL AND METHODS

In this study, commercially available e-polycaprolactone (PCL) was purchased from Sigma Aldrich, the melt flow index, density and melting temperature of the PCL are 12.5 g/10 min, 1.145 g/cm^3, and 70 ^{o}C, respectively. The CH stalks were collected from farms within the locality of Aliero.

Corn-Husk Treatment

The stalks were cut in small pieces, soaked in water for one day. It was then washed and dried for the period of one week. The washed and dried CH stalks was soaked in different proportions of alkali solution of 5, 10, 15 and 20 mL of 2 % alkali aqueous solution for 24 hours. After treatment, the CH fiber was thoroughly rinsed with distilled water until the pH of the rinse solution reached 7, confirming the complete removal of NaOH. Both the treated and untreated CH were grounded using milling machine. The grounded CH powder is then sieved to 200 microns sizes. These sizes were then used for the preparation of CH/PCL composites at different treatment ratio.

Preparation of CH/PCL Composites

To prepare the composites, a total net weight of 30 g was used for the CH and PCL which were mixed in the proportion shown in Table 1. For the preparation, the PCL polymer was placed in the blending machine at a temperature of 80 ^{0}C which allows the granulated PCL to completely melt. After the complete melting of the PCL, the required amount of CH is introduced into the vial containing the PCL and both mixtures are allowed to mix up for 10 minutes in the co-rotating twin-screw extruder machine (model LTE20-40). The extruder's mixing zone was set at the screw speed of 200 rpm. The extruder revolution ensured the homogenous dispersion of the CH into the polymer matrix. The mixture is then removed from the blend and allowed to cool. The cooled mixture is then fabricated into desired shapes for characterization.

Table 1. Composition of samples

S/N	Alkali (ml)	CH (g)	PCL (g)	Total (g)	Treatment
1	0.0	10.0	20.0	30.0	Untreated
2	5.0	10.0	20.0	30.0	Treated
3	10.0	10.0	20.0	30.0	Treated
4	15.0	10.0	20.0	30.0	Treated
5	20.0	10.0	20.0	30.0	Treated

Fabrication of Bio-composite Sheets

Indigenous thermal hot press was used to fabricate the bio-composites sheets. Constructed mold was used for the filling of the samples into shape ensuring that no space was left. Poly sheets were used to cover the mold at the top and bottom to prevent leakage of the material, before compressing in the thermal press. A pressure of 5 MPa was applied at a temperature of 65 ^{0}C for 10 min. This pressure was selected during compression molding to ensure uniform material distribution and effective fiber-matrix embedding, reducing voids and enhancing composite integrity. It has been reported that excessive pressure could lead to fiber damage or polymer degradation, while insufficient pressure might result in poor adhesion and weak mechanical properties. After completion of the compressing time, the mold was cooled under pressure and removed from the thermal press at room temperature. The sheet was taken out from the mold and placed in a sample bag for subsequent characterization.

Material Characterization

The water absorption behavior of the CH/PCL composites was evaluated in accordance with the BS 317:1993 Standard. The samples were weighed (Wi) and soaked in a beaker containing 50 ml of water. The samples were then removed periodically and weighed after 3, 6, 9, 12, 15, 18, and 21 hours. The values recorded are then used to compute the absorption percentage using equation (1), [10]:

$$Water\ Absorption\ Rate\ (\%) = \frac{W_f - W_i}{W_f} x\ 100 \qquad (1)$$

where W_f is the weight of sample after immersion in water and W_i the weight of sample before immersion in water. The samples were submerged in water at a pH of 7 and a temperature of 20 °C. The water absorption test was carried out over a span of up to 21 hours. Five measurements were taken per sample, and the results were averaged.

Tensile and flexural tests was performed using a universal tensile machine RTG1310 with a loading speed of 10 mm/min. The tensile test specimen was shaped in accordance with the ASTM D3039/D3039M17 standard and the flexural specimen in accordance with the ASTM D790 standard.

Functional group changes in the composites were examined using FT-IR spectroscopy. The thermo-gravimetric data was used to determine the weight loss percentage, maximum decomposition temperature (Tmax) and char residue through the DTG curve. The glass transition and melting temperatures of composites were determined via differential scanning calorimetry (DSC) on a Perkin-Elmer DSC.

RESULTS AND DISCUSSION

The FTIR peaks for the CH/PCL composites (Figure 2), showed that all the CH/PCL samples shows distinct FT-IR bands characteristic of lignocellulose material at 3000-3100 cm^{-1} [11]. This peak is likely due to the presence of lignin in raw fibers, which indicates a high concentration of hydroxyl (-OH) groups. Following NaOH treatment, the OH stretching peak become sharper at 1750 cm^{-1}. This change indicates a reduction in hydrogen bonding, as the NaOH treatment partially removes lignin and hemicellulose, which is particularly evident for the 10 mL treatment. The treatment also consume some -OH groups by forming ester bonds, improving fiber compatibility with the matrix, resulting in better dispersion within the composite. The appearance of a strong peak at 1800 cm^{-1} is linked to C=O stretching of hemicellulose and lignin. The absence of a peak near 1750 cm^{-1} further confirms the removal of aromatic rings from lignin [11].

Additional peaks are observed at the 650 to 730 cm^{-1} corresponding to long chain rocking vibrations, and the 1000 to 1200 cm^{-1} peak is due to C-O-C aliphatic ether stretching vibrations [12]. The principal bands of PCL is at 1350 and 1700 cm^{-1} (CH$_2$ and CH bending vibrations), while the bands in the range of 2750 to 3000 cm^{-1} belonged to intermolecular and intramolecular hydrogen bonds due to asymmetric stretching vibrations [12]. The appearance of these bands is facilitated by the tightening of the voids between filler and matrix attributed to the alkali treatment.

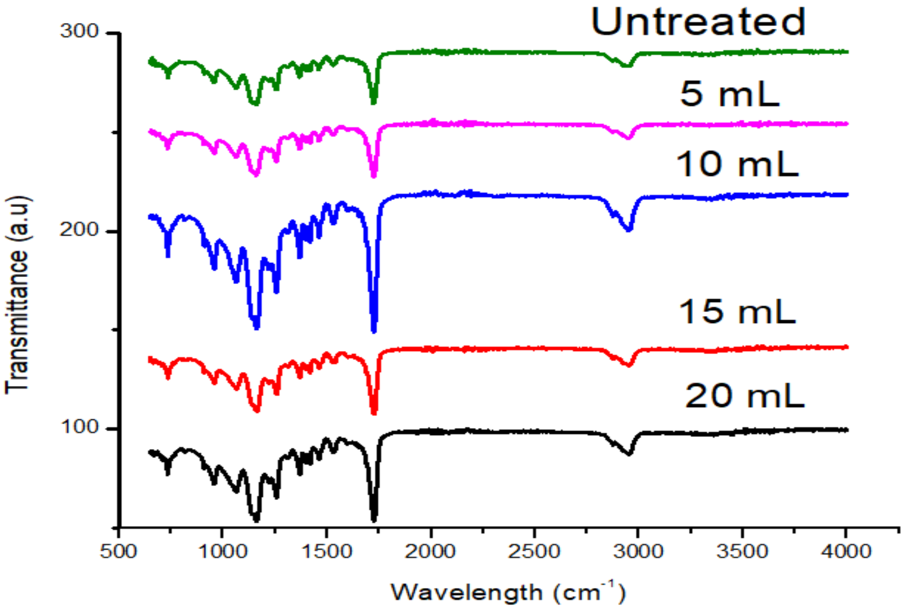

Figure 2. FTIR spectra for the CH/PCL composites

The charge transfer interactions between the conjugated surfaces of the alkali and the particles of CH/PCL significantly modified the properties of the composites [13]. The FTIR analysis conforms that the alkali treatment improve the compatibility between fiber and the matrix , enhancing interfacial adhesion and contributing to better dispersion and mechanical properties in the composite.

The presence of these characteristic peaks associated with cellulose, lignin, hemicellulose and PCL confirms the natural fiber composition with polymer, while shifts in peak intensity and position reflect potential interactions between the fibers and the PCL polymer [14].

Mechanical Properties

The tensile strength of CH/PCL composites shown Figure 3 increased by 51 % and this increment was due to the alkali treatment. The behavior displayed may be attributed to the cleaning and tightening of the fiber void/gaps in the composites [10]. While [7], reported that increase in tensile strength is a manifestation of strong adhesion and interfacial polarization bond between the fiber and the polymer influenced by the treatment. However, further increase of the treatment ratio reduced the tensile strength. There was a decrease in tensile strength from the optimum (10.73 MPa) to a value of 6.33 Mpa, this drop in tensile strength represents 42 % drop in tensile strength.

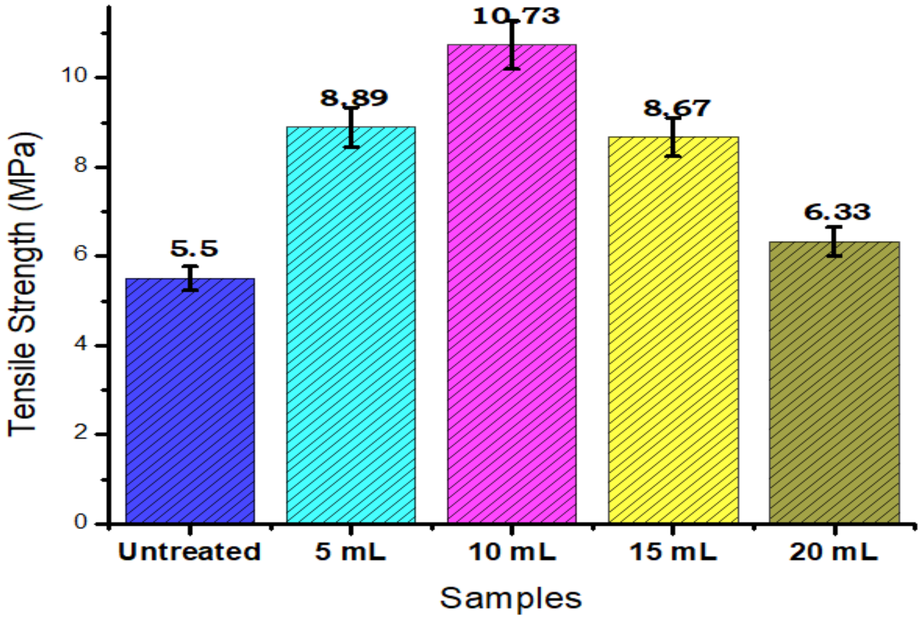

Figure 3. Tensile strength of the samples

The same response pattern was observed for the flexural strength as shown in Figure 4. There is a close agreement in the results obtained for the flexural and tensile strength in this work, and this may be due to good preparation technique free from impurities. The behavior for the tensile and flexural strengths are in agreement with studies where the mechanical properties of fiber/polymer composites decreased with increased treatment ratio over a certain optimum point [15].

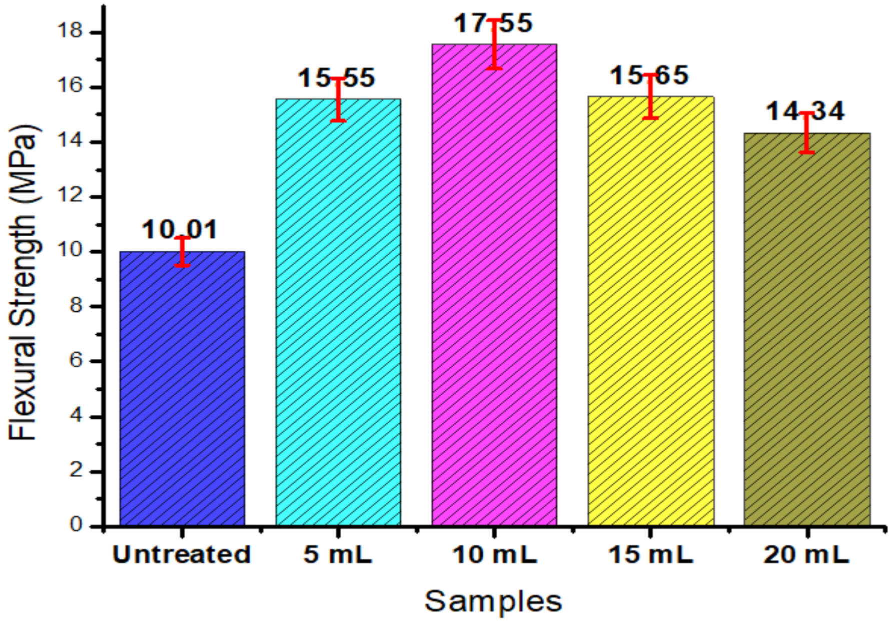

Figure 4. Flexural strength of the samples

The factor contributing to this behavior is the reduction in the density of the composites at higher treatment content causing the interfacial adhesion between fiber and matrix to weaken. This phenomenon is similar with previous studies, where the treatment with 15 mL produced the highest tensile strength in rice husk/PCL composites [6]. Based on the results obtained for the tensile and flexural strengths, it is therefore posited that, fiber/polymer composites can be treated with alkali but should not exceed the optimum break point to maximize mechanical properties**.**

The water absorption rate for the composites is shown in Figure 5. The untreated composite had the highest level of water absorption, this behavior is attributed to the higher content of cellulose and hemicellulose in the corn husk, creating higher percentage of hydroxyl (-OH) and acetyl (C_2H_3O), the principal contributor to moisture absorption [16]. The property of the hemicellulose and cellulose in the corn husk was the principal contributor to the high level of water absorbed by the composites and this is in agreement with works reported in [17]. The absorption characteristics of the composites was also influenced by alkali content, through reinforcement of the bond between fiber and matrix [17]. The strong homogenous dispersion of the fiber creates stronger bonding resulting to absence of voids/fiber pull-outs and cracks in the fabricated pellets, resulting in less weakness to water damage.

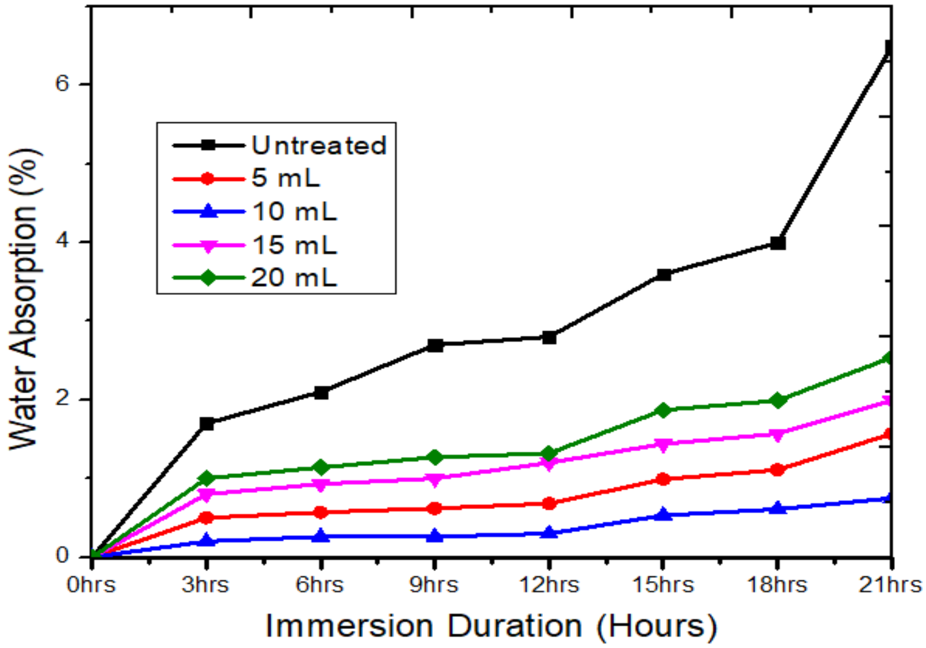

Figure 5. Water absorption rate of the prepared CH/PCL composites

The suitability of polymeric materials for engineering purposes based on water absorption capacity have been reported to be between than 0.7 to 1.7 % [18]. It is thus, convenient to state that the 5 mL and 10 mL are suitable for the fabrication of industrial packaging materials.

Thermal Properties

The DTG thermograph shown in Figure 6 reveals two distinct endothermic peaks at approximately 420 ^{0}C and 580 ^{0}C. The composite's degradation facilitated structural changes that contribute to the material's thermal behavior and stability under high-temperature conditions [19]. The DTG curves showed that raw fiber reached its peak degradation temperature at 410 ^{0}C, while for the alkali treated, they were observed in the range of 420 ^{0}C to 580 ^{0}C. These findings suggest that the alkali treatment improved the thermal stability of the composites, which likely resulted from the removal of non-cellulosic materials [20]. Evidently visible is the peak at the 430 ^{0}C thermal range, for the 10 mL and 15 mL treated composite. This peak suggests enhanced thermal stability, possibly due to improved fiber-matrix interaction.

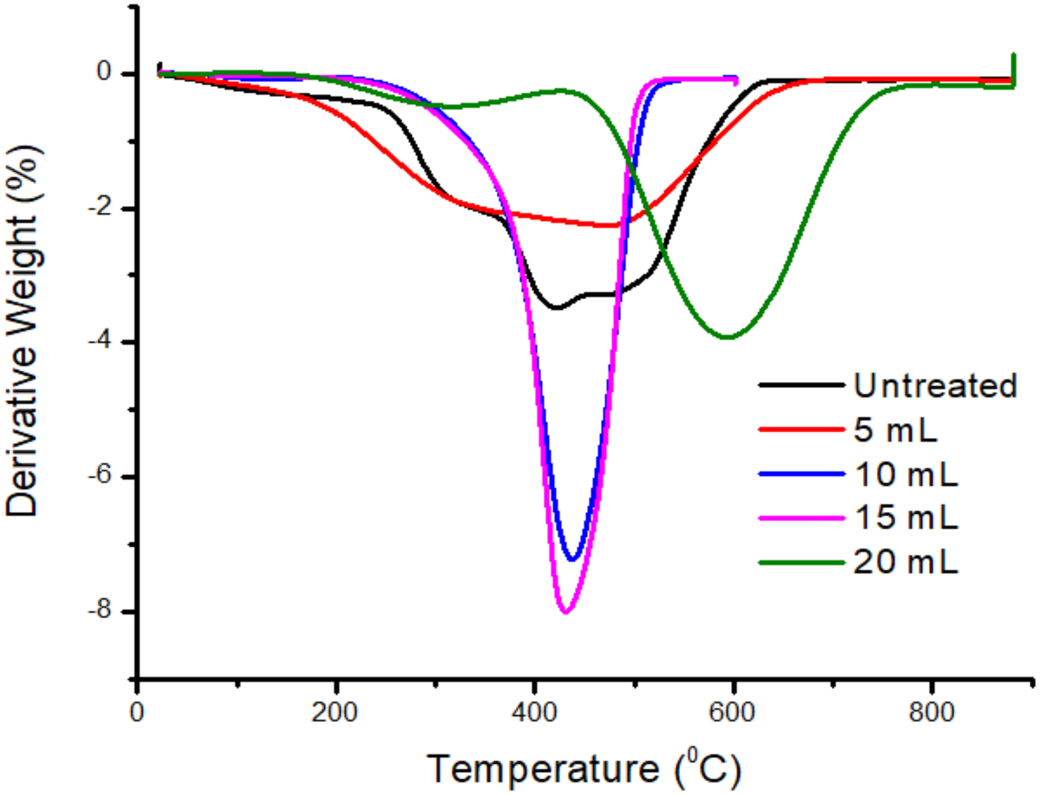

Figure 6. DTG thermograph of composites

This action contributes to the stability of the composites by forming stronger bonds within the composites structure, making it more resistant to thermal degradation. The endothermic peaks are due to phase transitions or material decomposition at the temperature investigated. It is thus posited that the findings supports the use of the composites for industrial packaging.

Figure 7 shows that all composites that are treated demonstrated enhanced thermal stability compared to the untreated composite. The reduced residual weight observed in the NaOH-treated composites suggests that the alkali treatment effectively removed substantial portions of hemicellulose and lignin, resulting in lower char formation. Conversely, the increased residual weight in the composites treated with 10 mL of NaOH indicates enhanced thermal stability, likely due to improved fiber–matrix interaction and structural integrity, which contributed to greater resistance to thermal decomposition.

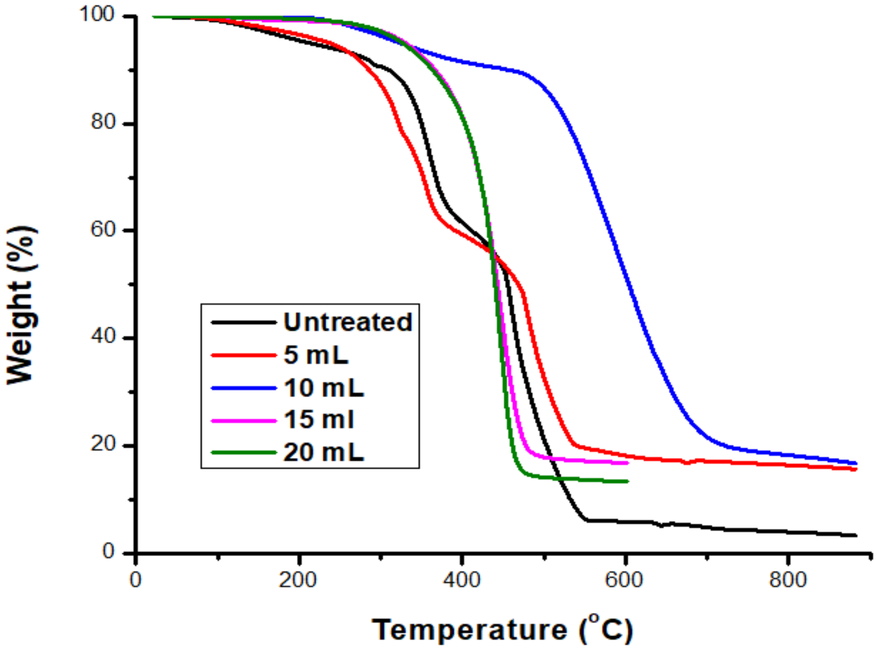

Figure 7. TGA thermograph of composites

The residual weight measured at the maximum temperature provides insight into the material's thermal endurance and its capacity to withstand thermal stress without significant mass loss. Generally, the degradation of the composites at about 360 ^{0}C was not unexpected as lignocellulose fibers are sensitive to temperature, and complete thermal degradation is expected above 400 ^{0}C [21].

For brevity, a summary of the values for the residual weight loss (%) is shown in Table 2. Based on the result, it is concluded that the treated composites performed well than the untreated after degradation. This confirms the suitability of the treated composites for industrial packaging.

Table 2. Summary of Residual Weight of the CH/PCL Composites

Sample	Residual Weight (%)	Type
CH/PCL	3.0	Untreated
CH/PCL5	16.5	Treated
CH/PCL10	17.0	Treated
CH/PCL15	15.7	Treated
CH/PCL20	12.4	Treated

CONCLUSION

A bio-composite with enhanced multifunctional properties was developed by incorporating chemically treated corn husk fibers, an agricultural waste, into a polycaprolactone (PCL) matrix. The fibers were treated with varying ratios of alkali solution to investigate the effect of chemical modification on composite performance. Alkali treatment effectively modified the fiber surface, resulting in significant improvements in the mechanical and thermal properties of the CH/PCL composites. Among the treatment levels, the 10 mL alkali-treated composites exhibited the most pronounced enhancement, followed by the 5 mL treatment, while the 20 mL treatment showed the least improvement. The modified composites demonstrated improved water absorption behavior, thermal stability, and structural integrity, indicating enhanced overall functionality. However, at higher alkali ratio, the performance of the composites began to decline, particularly in tensile and flexural strength, likely due to fiber degradation or excessive removal of binding components. These findings emphasize the potential of alkali-treated agricultural fibers in producing sustainable biocomposites suitable for packaging and related applications.

The systematic optimization of alkali treatment concentrations for CH fibers, distinguishing it from previous works that primarily focused on untreated or arbitrarily treated natural fibers. Unlike prior studies, which often overlooked the threshold beyond which chemical treatment becomes detrimental, this research establishes an optimal NaOH treatment level (10 mL) that achieves nearly double the tensile strength of untreated CH/PCL composites. Furthermore, a controlled melt-compounding and compression molding process, ensured superior fiber dispersion and enhanced interfacial bonding in this research. The findings demonstrate that alkali-treated CH fibers can serve as effective reinforcements for biodegradable PCL composites, offering a sustainable alternative for eco-friendly packaging applications. This research not only supports waste utilization but also paves the way for high-performance, biodegradable material development, aligning with environmental sustainability goals.

DECLARATION OF CONFLICT OF INTERESTS

The authors declare that there is no conflict of interest. They have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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