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# Optimization of chemical treatment process parameters for enhancement of mechanical properties of Kenaf fiber-reinforced polylactic acid composites: A comparative study of mechanical, morphological and microstructural analysis

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ABSTRACT

As advancements in sustainable material science continue, kenaf fiber and polylactic acid (KF/PLA) composites have emerged as promising eco-friendly alternatives. The current research article has focused on improving the physical strength of these KF/PLA composites. Methodologies like Taguchi's and Grey Relational Analysis (GRA) have been employed to identify the most effective ways to enhance the chemical treatment process. The composites were manufactured using an injection molding technique while essential variables were modified. These variables comprised the choice of chemical treatments

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(sodium hydroxide, potassium hydroxide, or sodium acetate), the concentration of these chemicals (1%, 2%, or 3% w/v), and the duration of treatment (2, 4, or 6 h). These modifications led to the production of diverse KF/PLA composite variants. The physical strength of these modified composites was evaluated using various methods, focusing on their tensile strength, tensile modulus, elongation under tension, flexural strength, flexural modulus, deformability under bending, and impact resistance. A Scanning Electron Microscope (SEM) was utilized to observe the treated and tested samples in detail. The optimal values identified through GRA and mean plots were same for the composite. Improved mechanical properties of KF/PLA were observed when the optimal conditions of A1-B2-C2 (NaOH, 2%, and 4 h) were applied. Although, the morphology of the PLA matrix-based bio composites reinforced with kenaf fiber with different surface-treatments was recorded using AFM analysis in order to further reveal the surface roughness of fibers upon surface modification and dispersion.



## 1. Introduction

In composite manufacturing, there's been a significant shift towards utilizing biodegradable constituents, prompted mainly by increasing ecological consciousness. Notably, kenaf fibers and polylactic acid (PLA) resin have been recognized as potent options for the production of biocomposites. Derived from the kenaf plant, kenaf fibers are characterized by their robust tensile strength and rigidity, ideal for bolstering polymeric matrices. PLA, a biopolymer synthesized from renewable substrates like corn starch, stands out for its superior mechanical attributes and biocompatibility. The synergy of these materials can yield biocomposites that are not only environmentally benign but also demonstrate desirable functional characteristics Li et al. [1].

The process of fabrication of kenaf/PLA ‘green’ composites starts with the blending of natural kenaf plant fibers and a renewable, biodegradable polymer preferably polylactic acid (PLA), eco-friendly and sustainable composite material which is versatile and has potential applications in several sectors. The preparation of kenaf fibers includes cleaning, drying, and cutting them into small parts before mixing with PLA pellets to form the composite. Green composites have an advantage over typical plastics since they are biodegradable and have a lesser carbon footprint than standard petroleum-based composites. Moreover, the kenaf/PLA composites exhibit enhanced mechanical strength, improved thermal resistance, and superior biodegradability.They represent a potential development in material science because of its adaptability, environmental friendliness, and affordability Kumar et al. [2].

Injection molded composites made from kenaf/PLA have excellent mechanical qualities, heat resistance, and biodegradability, making them useful in many different fields. Potentially leading to eco-friendlier alternatives to conventional petroleum-based plastics, investigating injection molding fabrication for kenaf/PLA composites is an exciting field of study. In order to strengthen the bonding between the fibres or particles and the matrix, a common technique for improving the characteristics of composites is chemical treatment, which entails subjecting the composite to a particular chemical solution. Chemical treatments can also improve element compatibility and eliminate contaminants, making the composite stronger. Chemical treatments, such as those using Sodium Hydroxide (NaOH) or Potassium Hydroxide (KOH), can be very useful for bio-composites fabricated from kenaf and PLA. These treatments improves the bond between the KF and the PLA matrix by removing contaminants and modifying the fibres' surface chemistry. The tensile strength and stiffness of kenaf/PLA composites was noted to rise by 59% and 64%, respectively, after being treated with NaOH, according to research by Yusoff et al. [3] Because of an increase in fibre surface area and improved fibre wetting by the matrix, kenaf fibres were able to better adhere to the PLA matrix, leading to the aforementioned improvement. A similar investigation into KOH treatment of kenaf/PLA composites was conducted by Thakur et al. [4]. A higher tensile strength, bending strength, and impact resistance were all seen in the treated composites. Tensile strength was 31.5 MPa, flexural strength was 60.9 MPa, and impact strength was 4.6 kJ/m2 before treatment. These values rose to 39.2 MPa, 76.3 MPa, and 8.8 kJ/m2 after being treated with KOH, proving the efficacy of chemical treatments. The tensile strength of bamboo/PLA bio-composites enhanced to 58.7 MPa (from 38.9 MPa), the flexural strength enhanced to 106.2 MPa (from 77.3 MPa), and the impact strength increased to 5.7 kJ/m2 (from 3.6 kJ/m2), according to a research by Wu et al. [5]. Using an alkaline treatment, Islam et al. [6] found that the tensile strength of jute/PLA composites enhanced to 44.3 MPa from 25.8 MPa, the flexural strength to 77.6 MPa from 51.7 MPa, and the impact strength to 8.9 kJ/m2 from 6.2 kJ/m2. In addition, Reddy et al. [7] examined how sisal/PLA bio-composites fared after being treated with NaOH. Tensile strength (41.3 MPa, up from 25.6 MPa), flexural strength (80.7 MPa, up from 50.4), and impact strength (11.4 kJ/m2 up from 8.1 kJ/m2) were all measured to be higher. Young and Tsao [8] conducted a study on the mechanical properties of injection-molded BF/PLA composites. They observed that the composite with a 30% fiber loading exhibited the highest tensile strength at 74.53 MPa, indicating an 11% strength improvement compared to untreated composites. Furthermore, flexural loading revealed a maximum strength and modulus of

123.76 MPa and 6.23 GPa, respectively. Long et al. [9] focused on alkali-treated BF/PLA composites, manufactured using twin-screw extrusion and injection molding. The tensile strength of the treated composite with a 30% fiber loading reached 65.46 MPa, showcasing an 11.7% enhancement over pure PLA. The composite with 10% fiber loading demonstrated a peak flexural strength and modulus of 97.94 MPa and 6.25 GPa, respectively. Another study by Tokora et al. [10] investigated various chemical treatment methods during the development of BF/PLA composites via injection molding. Alkali treatment notably improved the bonding between the fibers and the matrix, leading to enhanced strength and modulus. Steam-exploded filaments exhibited superior bending strength. Okubo et al. [11] manufactured a hybrid bio-composite (cellulose/bamboo/PLA) through twin-screw extrusion and injection molding. The inclusion of cellulose established a distinct interface between the polymer and fiber, minimizing abrupt crack propagation at the composite interface. The composite comprising BF/PLA with 2% cellulose demonstrated strength and modulus values of 53.8 MPa and 4.81 GPa, respectively. In a separate study, Zhang et al. [12] explored the properties of injection-molded bamboo particle (BP)/PLA composites. BP-100/PLA displayed superior properties compared to BP-200/PLA and BP-300/PLA composites. Strong interfacial bonding was attributed to hydrogen bond formation between BP and PLA. Fazita et al. [13] assessed the biodegradability and recyclability of injection-molded BF/PLA composites. Tensile strength and modulus initially measured 80.64 MPa and 5.92 GPa, while flexural properties recorded 143 MPa and 4.5 GPa. After recycling, tensile properties slightly decreased, and flexural properties improved. The study concluded that the composite possessed satisfactory mechanical rigidity and thermal stability for recycling purposes. Gamon et al. [14] examined the influence of twin-screw extrusion parameters on bamboo/miscanthus/PLA composite properties. Longer fibers led to more fiber breakages, and the inclusion of long fibers enhanced properties. Short fibers exhibited good chain mobility and crystallinity while resisting deformation under heat. Yang et al. [15] evaluated the mechanical properties of BF/PLA composites produced through hot and cold pressing. A 60% fiber loading resulted in flexural strength and modulus of 56.1 MPa and 7.28 GPa, respectively. Subyakto et al. [16] studied bamboo/sisal/PLA composites through injection molding for automobile applications. Munawar et al. [17] compared the mechanical performance of polypropylene (PP) and PLA matrices reinforced with various PLF types. Using melt mixing and hot pressing, PLA-based composites with 10% Josapine PLF achieved maximum tensile and flexural strengths of 4.2 MPa and 18.12 MPa, respectively. Saikeng et al. [18] investigated individual and hybrid PLF/coir/PLA composites via hot pressing, emphasizing dispersion and matrix-fiber affinity's impact on properties. Addition of PLF enhanced both tensile and flexural properties. Onyekwere et al. [19] employed the hybrid Taguchi-GRA method to optimize properties of BF/polyester composites fabricated through hand layup. Mercerized acetylated treatment and 50% fiber content yielded the best mechanical properties.

The existing literature indicates a lack of comprehensive investigation into the mechanical properties such as tensile, flexural, and impact properties of the KF/PLA green composite fabricated via injection molding machine. Additionally, the influence of chemical treatment parameters on mechanical characteristics through optimization remains unexplored. This study seeks to address these gaps by examining the impact of diverse chemical treatments, involving variations in chemical concentration and treatment duration, on the mechanical properties of the developed green composites. The optimal conditions are determined using the GRA method, while ANOVA is employed to assess the significance of the selected parameters.

## 2. Materials and methods

### 2.1. *Materials*

Raw materials utilized for the fabrication of green composites are kenaf fibers and PLA pellets. PLA is a biodegradable thermoplastic polymer having a glass transition temp. range of 50–80 °C and a melting point range of 155–180 °C. PLA has environmentally friendly properties and very low density, i.e., 1.24 g/cm$^3$. The chopped kenaf fibers are used as a reinforcement material. The kenaf fibers have different favorable properties, such as non-toxic, biodegradable, and lightweight characteristics. The kenaf fibers exhibited a favorable composition with a high cellulose content of 55% and a lignin content of 13%, ensuring their suitability for enhancing the mechanical characteristics of the composite matrix.

### 2.2. *Chemical treatments*

Prior to chemical treatment, kenaf fibers underwent a thorough cleaning and drying process to ensure optimal results. The fibers were initially submerged in a solution comprising distilled water and a gentle detergent for 25 min, effectively removing any dust particles and impurities. Subsequently, the fibers were rinsed with tap water to eliminate residual detergent before being air-dried in preparation for chemical treatment. Table 1 delineates the process parameters and their respective levels used for chemical treatment, while Table 2 specifies the concentrations and types of chemicals in the aqueous solutions with their levels employed for chemical treatment. Following immersion in the designated aqueous

**Table 1 – Comprehensive overview of input parameters and levels for optimizing kenaf fiber chemical treatment.**

| Input Factors | First Level | Second Level | Third Level |
|---|---|---|---|
| Chemical Agents Used | Sodium Hydroxide (A1) | Potassium Hydroxide (A2) | Sodium Acetate (A3) |
| Strength of the Solution (w/v) | 1% (B1) | 2% (B2) | 3% (B3) |
| Duration of Treatment (in hours) | 2 h (C1) | 4 h (C2) | 6 h (C3) |

**Table 2 – Tabular layout for systemic adjustment of chemical agents, strength of solution, and duration of treatment in kenaf fiber treatment.**

| Serial Number | Orthogonal Array | Chemical Agent (A) | Solution Strength (% w/v) (B) | Duration (hours) (C) |
|---|---|---|---|---|
| 1 | 1-1-1 | Sodium Hydroxide | 1% | 2 h |
| 2 | 1-2-2 | Sodium Hydroxide | 2% | 4 h |
| 3 | 1-3-3 | Sodium Hydroxide | 3% | 6 h |
| 4 | 2-1-2 | Potassium Hydroxide | 1% | 2 h |
| 5 | 2-2-3 | Potassium Hydroxide | 2% | 4 h |
| 6 | 2-3-1 | Potassium Hydroxide | 3% | 6 h |
| 7 | 3-1-3 | Sodium Acetate | 1% | 2 h |
| 8 | 3-2-1 | Sodium Acetate | 2% | 4 h |
| 9 | 3-3-2 | Sodium Acetate | 3% | 6 h |

solutions for a predetermined period, then fibers were rinsed in distilled water to wash away the remaining chemicals. The fibers were then air-dried and dried in a oven at 65 °C to eradicate any lingering moisture content. The following equations represent the chemical reactions that transpired during the treatment process:

$$KF - OH + NaOH \rightarrow KF - O - Na+ + H2O \quad (1)$$

$$KF - OH + KOH \rightarrow KF - O - K+ + H2O \quad (2)$$

$$CH3COO- + H2O \rightarrow Na+ + CH3COOH + OH - CH3COO- + H2O \rightarrow CH3COOH + OH- \quad (3)$$

Equations (1)–(3) illustrate the chemically modified kenaf fibers, which in turn improves the interfacial adhesion between the fibers and PLA matrix. This process for the chemical treatment is crucial in ensuring superior performance of the resulting green composites across various applications.

### 2.3. *Green composites fabrication*

Fig. 1 describes the methodological approach employed in the development of composite specimens, emphasizing the pivotal steps in the fabrication process necessary to yield high-caliber green composites. The synthesis KF/PLA eco-friendly composites was executed using an advanced injection molding apparatus (Futura 60), possessing a clamping

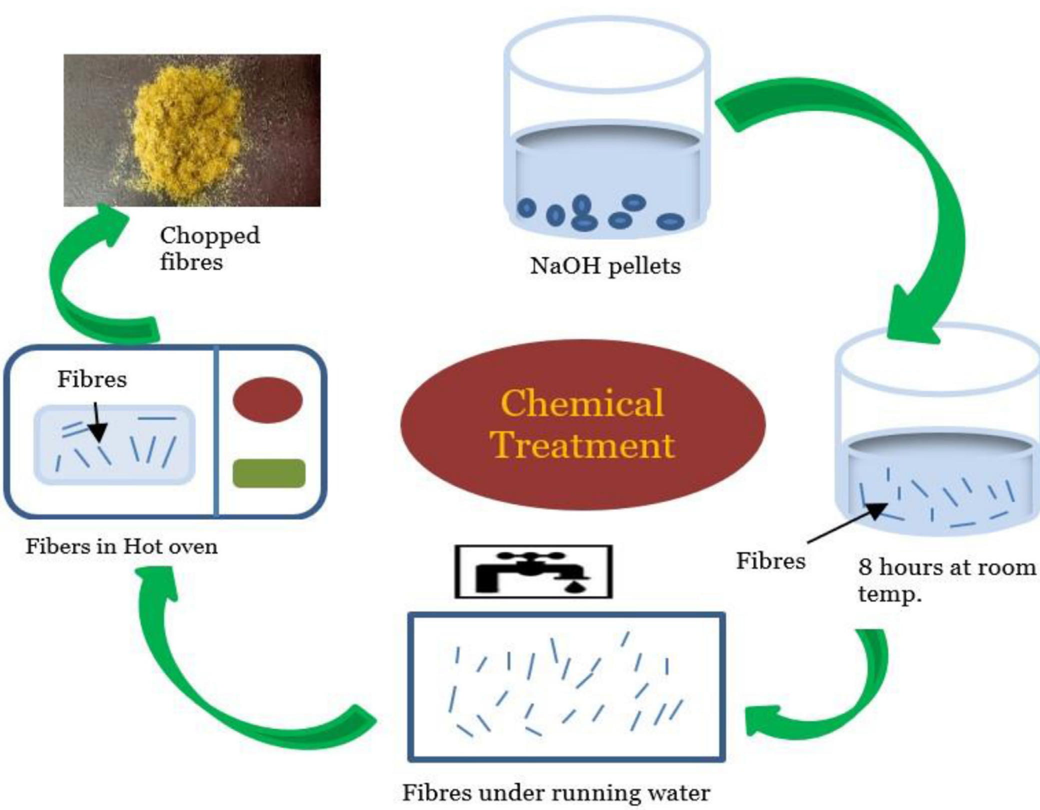



**Fig. 1 – Schematic representation of the kenaf fiber cleaning and chemical treatment process for enhanced composite performance.**

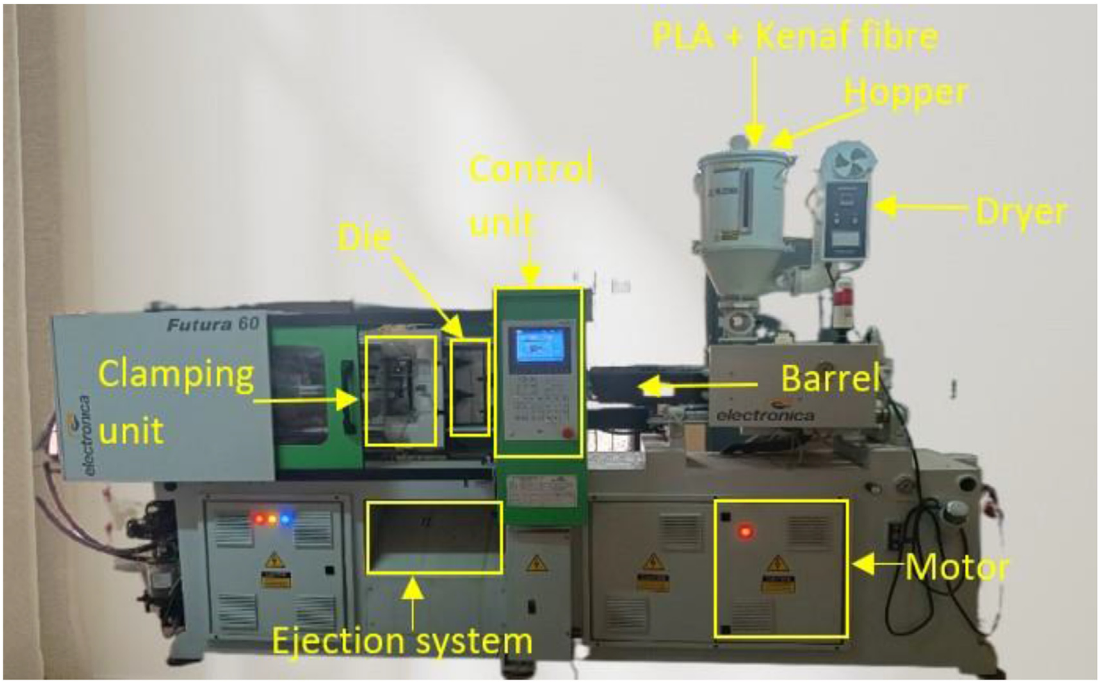



**Fig. 2 – Schematic representation of the Futura 60 injection molding machine, illustrating its key components.**

capacity of 60 tons. Fig. 2 shows the injection molding machine and different components, such as the hopper, heating zones, injection unit, and clamping mechanism, worked in harmony to yield the composites.

Before the composite fabrication process, PLA pellets underwent a preconditioning phase, where they were kept in an electric oven maintained at 60 °C for 7 h, eliminating residual moisture content. A 10% (w/w) kenaf fiber loading was employed to create the KF/PLA composites. The amalgamation of kenaf fibers and PLA pellets was introduced into the injection molding machine's hopper. The barrel's heating zones were meticulously regulated, with temperatures at 160 °C, 165 °C, 175 °C, and 190 °C, to achieve optimal processing conditions. Holding and injection pressures were sustained at 55 MPa and 60 MPa, respectively. Thorough dehumidification of the matrix and fibers ensured the attainment of the desired specimen geometry, mitigating potential defects originating from moisture-related issues. A shot weight of 20 g was employed to fill the mold cavity adequately. The die assembly, as shown in Fig. 3, featured an

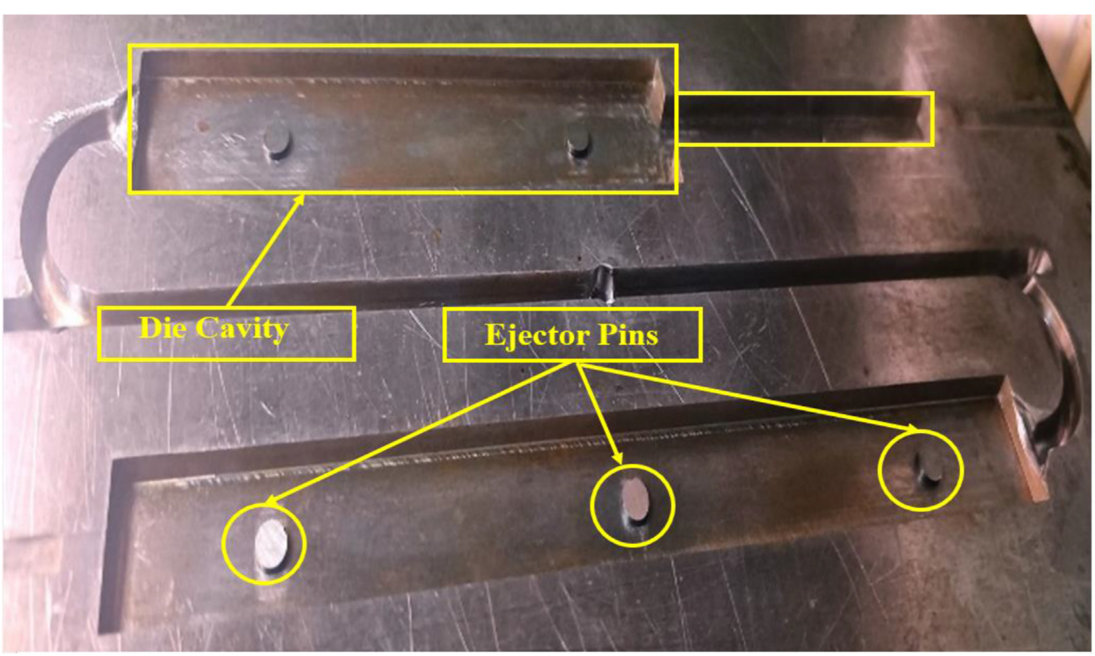



**Fig. 3 – Die assembly of an injection molding machine, featuring a close-up view of the die cavity and ejector pins.**

intricate design consisting of the mold cavity, sprue, runner system, and gate, which collectively facilitated the accurate molding of the composites while minimizing material waste and optimizing part quality. Figs. 2 and 3 emphasize the significance of utilizing a well-designed injection molding machine and die assembly to achieve optimal composite properties and consistent results in the fabrication process.

### 2.4. *Mechanical characterizations*

A thorough evaluation of the mechanical properties of KF/PLA green composites is required to determine their viability as a replacement for non-biodegradable materials in a variety of structural and non-structural applications, such as bowls, table cardboard, door panels, and utensils. We evaluated the material's mechanical properties, such as its tensile and flexural strengths and impact resistance, to determine its effectiveness. ASTM D-638 (165x19 × 4 mm) was used to evaluate tensile strength, ASTM D-790 (125x12.7 × 4 mm) was used to evaluate flexural strength, and ASTM D-256 (63.5x12.7 × 3.2 mm) was used to evaluate impact strength. We utilized an Indian-made UTM (Heico) with a 50 kN load capacity for precise results.The assessment of mechanical qualities required the evaluation of three samples and the calculation of the average value for each. This method ensured the accurate representation of typical material behaviour and minimised the impact of any outliers. Fig. 4 depicts in detail the mechanical characterization process, including the fabrication of composite specimens and the execution of the testing procedures. This meticulous mechanical characterization serves to provide valuable insights into the performance of the developed KF/PLA green composites under various loading conditions. In addition, it contributes to a better understanding of their suitability for diverse applications, thus supporting the advancement of sustainable and eco-friendly materials in the field of composite research.

Material performance is often evaluated based on mechanical characteristics such as tensile properties, flexural properties, and impact properties. These attributes play a crucial role in assessing a material's capability, especially under extreme conditions, directly influencing its engineering performance. In recent years, extensive research has been conducted on kenaf fiber reinforced composites to compreh-

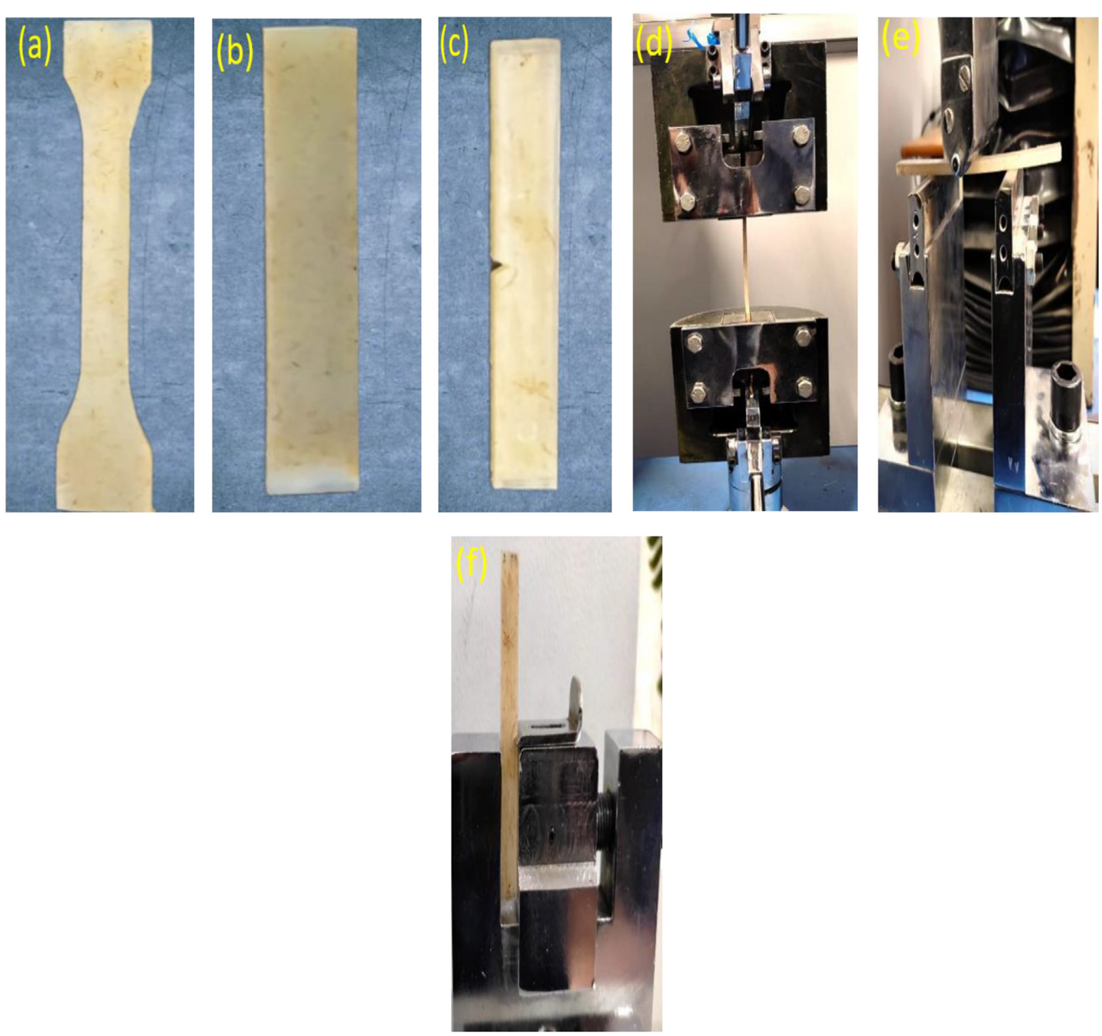



**Fig. 4 – Specimens prepared for (a) Tensile, (b) flexural, (c) impact strength testing and Specimens during (d) Tensile, (e) flexural, and (f) impact strength testing.**

ensively understand their mechanical behavior. Notable mechanical properties of these composites encompass the ultimate tensile strength (maximum tension stress sustainable without fracturing), fracture strain (stress at fracture from bending or flexure), flexural modulus (indicating stiffness during flexion), and impact strength (resistance to impact loading). The tensile and flexural characteristics of kenaf-reinforced composites are influenced by factors like fiber type, orientation, content, and form. Critical factors impacting the tensile properties of KF/PLA bio composites are interfacial bonding, non-cellulosic compounds on fiber surfaces, and fiber surface roughness. Existing literature reveals that untreated fibers and their composites exhibit weak tensile strength due to the presence of non-cellulosic compounds on the fiber surface. To address this, surface treatments are employed to eliminate these compounds. For instance, Bachtiar et al. [20] demonstrated that applying an alkali treatment with 0.25 M concentration and 1-h soaking time to sugar palm fibers led to improved tensile strength in sugar palm fiber-reinforced epoxy composites. This treatment enhanced fiber surface roughness by removing non-cellulosic contents. Flexural strength and modulus are influenced by interfacial adhesion between matrix and fibers, as well as the transfer of tension between them. Studies by Weyenberg et al. [21] highlighted that a 3 wt% NaOH concentration in flax fiber mercerization treatment yielded the greatest enhancement in longitudinal and transverse bending strength of flax fiber composites. This improvement was attributed to enhanced interfacial bonding between fibers and the matrix. Yousif et al. [22] also found that a 6 wt% NaOH-treated kenaf fiber reinforced composite exhibited higher flexural strength compared to a 5 wt% NaOH-treated kenaf/epoxy composite. The impact strength of KF/PLA bio composites, measured using an Izod impact tester, is influenced by interfacial bonding, composition, and matrix material toughness. Venkateshwaran et al. [23] demonstrated that using 1 wt% NaOH in alkali treatment of banana fibers resulted in the greatest increase in impact strength of banana/epoxy composites, compared to treatments with 20 wt% NaOH. In conclusion, characterizing the mechanical properties of kenaf fiber reinforced composites is essential for understanding their performance. Tensile, flexural, and impact properties are affected by factors such as interfacial bonding, surface treatments, fiber content, and matrix material composition.

## 3. Results and discussion

### 3.1. *Grey relational analysis (GRA)*

Grey Relational Analysis (GRA) is a robust multi-criteria decision-making method, particularly effective for handling situations with incomplete or uncertain data. In the context of optimizing chemical treatment process parameters for enhanced mechanical properties of (KF/PLA) composites, GRA offers a systematic approach to evaluate the influence of various parameters and identify the optimal combination that results in superior composite properties. When applying GRA to the optimization of KF/PLA composites, the method begins by gathering experimental data on the mechanical characteristics of the composites produced with varying the different chemical treatment process parameters. The parameters under consideration for chemical treatments are (i) medium of chemical such as sodium hydroxide, potassium hydroxide, and sodium acetate; (ii) chemical concentration such as 1%, 2%, and 3% w/v; and (iii) treatment duration such as 2, 4, and 6 h (Table 1). The mechanical properties of interest, such as tensile strength, flexural strength, and impact strength, are assessed for each combination of parameters. The first step in GRA is to normalize the experimental data using the equations for normalization as in Equation (4), allowing for a consistent comparison of the output responses.

$$X_i(p) = \frac{X_i^{(0)}(p) - Min\ X_i^{(0)}(p)}{Max\ X_i^{(0)}(p) - Min\ X_i^{(0)}(p)} \tag{4}$$

where $X_i^{(0)}$ *(p)* denotes output response in every column, Min $X_i^{(0)}$ *(p)* denotes min. value of the output response, *Max* $X_i^{(0)}$ *(p)* is the maxi. value of output response, and $i = 1,2,3 \ldots n$ denotes number of experiments Following normalization, the deviation sequence (Δij) is calculated to determine the absolute difference between the reference sequence and the comparative sequences for each parameter combination. The value for the deviation sequence ($\Delta_{ij}$) was determined using Eq. (5).

$$(\Delta_{ij}) = |X_{oj} - X_{ij}| \tag{5}$$

where $X_{oj}$ is the ideal normalized value, next, the grey relational coefficients (GRCs) are derived to quantify the degree of association between the reference sequence and the comparative sequences for each combination of process parameters in accordance with Equation (6). High GRC values indicate a strong association between the parameters and the desired mechanical properties, while low values indicate weak associations.

$$GRC_{ij} = \frac{\Delta_{min} + \zeta\ \Delta_{max}}{\Delta_{oi}(k) + \zeta\ \Delta_{max}} \tag{6}$$

Where $\Delta_{min}$ is lowest value of deviation sequence, $\zeta\ \Delta_{max}$ denotes maximum value of deviation sequence, $\Delta_{oi}$ (k) is the individual deviation sequence and ζ denotes coefficient of constant (presumed 0.5). After calculating the GRCs, the grey relational grades (GRGs) are obtained by averaging the GRCs for each alternative as per Equation (7). The GRGs are then ranked, with the highest GRG indicating the most desirable overall performance in terms of mechanical characteristics of KF/PLA bio-composites.

$$GRG = \frac{1}{m}\sum GRC_{ij} \tag{7}$$

Grey Relational Analysis (GRA) utilizes a specific concept of information. It categorizes situations with no information as "black" and situations with perfect information as "white" Chan & Tong [24]. This method was first introduced by Deng in 1982 to handle challenges in dealing with incomplete and uncertain systems Md. Israr Equbal et al. [25]. In recent times, GRA has become a powerful tool for studying processes with multiple performance measures. For instance, A.K. Sood et al. [26] used GRA alongside the Taguchi method to understand how different process settings affect various aspects of creating FDM parts. Similarly, K. Jangra et al. [27] applied GRA to improve both material removal rate and surface roughness in the WEDM of WC co composite using grey relational analysis. Tarang et al. [28] utilized the grey-based Taguchi method to optimize the process parameters of submerged arc welding in hard facing, considering multiple weld qualities.

The next step involves calculating the Grey Relational Coefficient using normalized experimental data. This coefficient shows how well the desired and actual data correlate. By averaging these coefficients for each performance aspect, an overall Grey Relational Grade is obtained. This grade helps us determine the best process parameters using the Taguchi method, aiming for the highest Grey Relational Grade. To simplify, the Grey Relation Coefficient can be calculated from normalized data, and the best parameter level is the one with the highest Grey Relational Grade, Franko Puh et al. [29]. Once we find this level, we can decide on the best parameter settings by looking for the highest Grey Relational Grade, which means better product quality. To do this, we calculate average grade values for different parameter levels and select the one with the highest average grade value, Amlana Panda et al. [30]. After figuring out the best combination, the next step is the Analysis of Variance (ANOVA), which helps us see which parameters significantly affect the multiple aspects we're measuring. ANOVA separates the total variation in responses into contributions from different parameters and error, Datta et al. [31]. The P-value, usually derived from Fisher's F-ratio, tells us if a response is significant (usually when it's less than 0.05). Degrees of freedom (DF) are important for calculating mean squares (MS) and understanding how much independent information we have to evaluate sum of squares (SS). Using grey relational analysis based on grey system theory can be a way to understand complex relationships between multiple responses. This leads to a grey relational grade that helps us assess multiple responses together. In simpler terms, this grade makes it possible to optimize multiple responses as if they were a single grade. So, applying this combination of grey relational analysis and Taguchi method has great potential for optimizing situations with multiple responses.

However, in this study, mechanical characteristics of the produced green composites was accomplished using the Taguchi L9 orthogonal array (OA). In order to assess the efficacy of the composites, a summary of the acquired mechanical parameters is provided in Table 3. Table 5 explains how the deviation sequence for the output answers was computed using the normalized data shown in Table 4. The grey relational grades (GRG) were determined by first calculating the grey relational coefficients (GRC) for each green composite and then taking a weighted average of the GRC over all trials. A brief representation of the relationships between the GRC, GRG, and ranks is shown in Table 6.

The standard GRGs were evaluated to get the optimal value for each input variable. A maximum GRG number indicated the best potential output response. According to the results, the optimal treatment for the KF/PLA composite is a chemical bath of 2% sodium hydroxide (NaOH) for 4 h at the levels A1-B2-C2. Analysis of variance (ANOVA) on the GRG was employed to validate the ideal level, and the results showed that the optimal values for the means and GRGs were compatible with one another. The greatest GRG value was found in the KF/PLA composite, at 0.6958. Using GRA, the information is converted into a grey system, which provides a more flexible and understandable depiction of unreliable or sparse data. Determining the grey relational degree between the grey system and a reference system provides a quantitative assessment of the closeness of the two systems' connections. The mechanical characteristics of KF/PLA composites may be optimised and processing decisions made with the use of this degree of connection by ranking and prioritising factors or generating data-driven forecasts.

### 3.2. *Analysis of variance of GRG*

An extensive ANOVA (analysis of variance) was well thought-out on the GRG to pinpoint the most crucial factors that have a remarkable impact on the mechanical characteristics of KF/PLA composites. The Minitab software was employed to perform the statistical evaluation, carefully inspecting data normality and equal variance assumptions to ensure accuracy in the results. The data normality was meticulously assessed using several graphical representations, such as probability plots (Fig. 5) and histograms. The visual aids facilitated the identification of a normal distribution, as the plotted points closely adhered to the fitted distribution line with a 95% confidence level. This normal distribution is further corroborated by the Anderson-Darling statistic, which stands at 0.62. Additionally, the P-value of 0.070 denotes that there is not significant evidence for the rejection of null hypothesis. enough evidence to reject the null hypothesis, confirming the normality of the data. To examine equal variance, a set of detailed plots was generated (Fig. 6). The P-values obtained from these plots were higher than the predetermined alpha value, implying that there is insufficient affirmation to invalidate the null hypothesis, which presumes equal variances among the data. As a result, any significant differences in variances can be dismissed. Following the completion of the ANOVA, mean plots were constructed (Fig. 7) to provide an all-encompassing perspective on the GRG analysis results. These plots revealed consistent optimal levels when compared with the grade rank data. Table 6 represents the mean responses, emphasizing the higher mean values at optimal level for process parameters for the KF/PLA composite. The most favorable level for the KF/PLA composite was determined from both rank one and meant plots that identified the optimal level as A1-B2-C2. A thorough breakdown of the sum of squares (SS), F value, P value, and percentage contribution of the parameters are provided in Table 8, offering a complete view of the ANOVA analysis outcomes.

In the case of the KF/PLA composite, the null hypothesis was rejected as the P values for chemicals used for treatment, their concentration, and treatment period were determined to be 0.184, 0.134, and 0.433, respectively. All of these values are lower than the established alpha value. Among the parameters, the concentration exhibited the most substantial percentage contribution, accounting for 62.71% of the total variance. Furthermore, values of P for the chosen parameters of KF/PLA composites were found to be lower than the specified confidence value. Table 7 presents the model summary for GRG, where the R-square values for KF/PLA composites were identified to be as high as 99.34%. A high R-squared value

Table 3 – Mechanical properties of KF/PLA composite: Assessment based on selected orthogonal array.

| A | B | C | TS (MPa) | TM (GPa) | PET (%) | FS (MPa) | FM (GPa) | PEF (%) | IS (J/m) |
|---|---|---|---|---|---|---|---|---|---|
| NaOH | 1 | 2 | 56 | 5.20 | 5.82 | 90.00 | 4.52 | 1.59 | 34.00 |
| NaOH | 2 | 4 | 60 | 5.50 | 5.96 | 93.00 | 4.64 | 1.68 | 36.00 |
| NaOH | 3 | 6 | 52 | 4.80 | 5.20 | 96.00 | 4.76 | 1.88 | 32.00 |
| KOH | 1 | 4 | 52 | 5.00 | 4.96 | 102.00 | 5.82 | 1.60 | 30.00 |
| KOH | 2 | 6 | 48 | 4.70 | 4.80 | 109.00 | 5.84 | 1.45 | 32.00 |
| KOH | 3 | 2 | 43 | 4.20 | 4.60 | 83.00 | 4.07 | 1.85 | 28.00 |
| $CH_3COONa$ | 1 | 6 | 54 | 6.20 | 4.72 | 63.00 | 4.82 | 1.20 | 36.00 |
| $CH_3COONa$ | 2 | 2 | 56 | 5.40 | 6.18 | 62.00 | 6.22 | 0.90 | 38.00 |
| $CH_3COONa$ | 3 | 4 | 51 | 6.40 | 3.80 | 49.00 | 6.34 | 0.96 | 34.00 |
| Minimum | 1 | 2 | 43 | 4.20 | 3.80 | 49.00 | 4.07 | 0.90 | 28.00 |
| Maximum | 3 | 6 | 60 | 6.40 | 6.18 | 109.00 | 6.34 | 1.88 | 38.00 |
| Mean | 2 | 4 | 52.44 | 5.27 | 5.12 | 83.00 | 5.23 | 1.46 | 33.33 |

suggests a strong statistical link between the data points and the model. The residual plots for GRG are shown in Fig. 8 to test the constant variance and uniform distribution assumptions as exhibited in Fig. 9.

Fig. 8a shows the examination of the residuals versus fits plot in an effort to confirm these presumptions. If the assumptions of random distribution hold, then the points in this figure should be uniformly distributed on each side of zero. Fig. 8b displays residual against order plots, which were constructed to verify the residuals' independence. Evidence that the residuals are genuinely independent may be seen in the random distribution of residuals and the centre line distribution. This study's in-depth investigation has provided useful information for enhancing the mechanical characteristics of KF/PLA composites by chemical treatment process optimization. The use of statistical methods and graphical displays has proven that the data are normally distributed, have the same variance, and are independent of one another. This all-encompassing method has allowed for a more nuanced comprehension of the aspects that most significantly affect the performance of the composite.

### 3.3. *Confirmation test for GRG*

The ideal set of process parameters was verified through a confirmation test. A comparison was made between the predicted Grey Relational Grade (GRG) and the experimental graded value obtained to evaluate the enhancement in the GRG. The predicted GRG was determined using Equation (8), where rm is the average mean of grades, the mean of grades at the optimal level, and q denotes number of input parameters.

$$r = r_m + \sum (r - r_m)$$

Table 10 shows the experimental and anticipated GRG at the process parameter maximum. For the KF/PLA composite, the experimental and expected GRG values are 0.6958 and 0.695846, respectively. GRG showed a 0.000046988 improvement(see Table 9).

## 4. Structural examination of untreated and chemically Treated Samples

The process of chemically treating kenaf fibers is a common method used to enhance the material's properties and customize it for specific uses. Kenaf fibers possess inherent components like lignin and hemicellulose, which can impede optimal performance in certain applications, such as composites, by disrupting the bonding between the fiber and matrix. Chemical treatments, such as alkali treatment, play a role in eliminating these components, thereby improving the adhesion between the fiber and matrix material. These treatments also have the potential to boost the fibers' tensile strength, enhancing their durability. This quality is especially advantageous in fields like construction materials and automotive components. Another rationale

Table 4 – Normalized mechanical properties of KF/PLA composites.

| TS (MPa) | TM (MPa) | PET (%) | FS (MPa) | FM (GPa) | PEF (%) | IS (J/m) |
|---|---|---|---|---|---|---|
| 0.7647 | 0.4545 | 0.8487 | 0.6833 | 0.1982 | 0.7041 | 0.6000 |
| 1.0000 | 0.5909 | 0.9076 | 0.7333 | 0.2511 | 0.7959 | 0.8000 |
| 0.5294 | 0.2727 | 0.5882 | 0.7833 | 0.3040 | 1.0000 | 0.4000 |
| 0.5294 | 0.3636 | 0.4874 | 0.8833 | 0.7709 | 0.7143 | 0.2000 |
| 0.2941 | 0.2273 | 0.4202 | 1.0000 | 0.7797 | 0.5612 | 0.4000 |
| 0.0000 | 0.0000 | 0.3361 | 0.5667 | 0.0000 | 0.9694 | 0.0000 |
| 0.6471 | 0.9091 | 0.3866 | 0.2333 | 0.3304 | 0.3061 | 0.8000 |
| 0.7647 | 0.5455 | 1.0000 | 0.2167 | 0.9471 | 0.0000 | 1.0000 |
| 0.4706 | 1.0000 | 0.0000 | 0.0000 | 1.0000 | 0.0612 | 0.6000 |
| 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 | 0.0000 |
| 1.0000 | 1.0000 | 1.0000 | 1.0000 | 1.0000 | 1.0000 | 1.0000 |
| 0.5556 | 0.4848 | 0.5528 | 0.5667 | 0.5091 | 0.5680 | 0.5333 |

Table 5 – Deviation sequence analysis for Kenaf/PLA composite - exploring variability in properties.

| TS (MPa) | TM (MPa) | PET (%) | FS (MPa) | FM (GPa) | PEF (%) | IS (J/m) |
|---|---|---|---|---|---|---|
| 0.2353 | 0.5455 | 0.1513 | 0.3167 | 0.8018 | 0.2959 | 0.4000 |
| 0.0000 | 0.4091 | 0.0924 | 0.2667 | 0.7489 | 0.2041 | 0.2000 |
| 0.4706 | 0.7273 | 0.4118 | 0.2167 | 0.6960 | 0.0000 | 0.6000 |
| 0.4706 | 0.6364 | 0.5126 | 0.1167 | 0.2291 | 0.2857 | 0.8000 |
| 0.7059 | 0.7727 | 0.5798 | 0.0000 | 0.2203 | 0.4388 | 0.6000 |
| 1.0000 | 1.0000 | 0.6639 | 0.4333 | 1.0000 | 0.0306 | 1.0000 |
| 0.3529 | 0.0909 | 0.6134 | 0.7667 | 0.6696 | 0.6939 | 0.2000 |
| 0.2353 | 0.4545 | 0.0000 | 0.7833 | 0.0529 | 1.0000 | 0.0000 |
| 0.5294 | 0.0000 | 1.0000 | 1.0000 | 0.0000 | 0.9388 | 0.4000 |

behind chemically treating kenaf fibers is to enhance their resistance to water. Like many natural fibers, kenaf has a tendency to absorb water, which can be problematic in specific contexts. By undergoing chemical treatments, the hydrophobic nature of the fibers can be adjusted, minimizing water absorption. This adjustment is particularly important when working with composite materials, as ensuring a strong bond between the fiber and matrix material is of paramount significance.Chemical treatments can modify the kenaf fibers' surface, enhancing their compatibility with different matrix materials. Finally, chemical treatments can enhance fiber uniformity, which contributes to the optimal performance of the end product and reduces variability during the manufacturing process.

The kenaf fibers underwent a chemical process using a variety of agents such as Sodium Hydroxide (NaOH), Potassium Hydroxide (KOH), and Sodium Acetate ($CH_3COONa$). Before this treatment, contaminants were found on the fiber surfaces. A visible improvement in surface quality was seen in the treated fiber samples. Figs. 10–12 provide a comparative view of the SEM images of kenaf fibers untreated and treated with NaOH, KOH, and $CH_3COONa$. The effectiveness of the chemical process in clearing surface contaminants, insoluble particles, and non-cellulosic elements such as lignin, pectin, waxes, and oils from the kenaf fibers is evident from these images. The chemical process played a significant role in enhancing the kenaf fibers' hydrophilic behavior, wettability, and surface irregularities. Latif et al. [32] mentioned that the chemical processing of natural fibers helps remove non-cellulosic material, thereby improving surface irregularities and wettability. Ahmed et al. [33] suggested that surface contaminants in natural fibers can compromise the bonding between the matrix and fibers, but chemical processing can enhance and encourage better interfacial adhesion as illustrated in Fig. 13.

The structural analysis of the tested samples was assessed based on the Grey Relational Grade (GRG) rank, where Rank 1 signifies superior characteristics, and Rank 9 suggests poor attributes. For the (KF/PLA) composite, the highest mechanical attributes were seen when fibers underwent a 4-h treatment with 2% NaOH (Rank 1 with parameters A1-B2-C2). Conversely, the least mechanical attributes were observed when fibers were processed with 3% KOH for 2 h (parameters A2-B3-C1). The chemical process enhanced the fiber surface by modifying the fibers' hydrophilic behavior and removing unwanted residuals [34,35]. This led to improved mechanical attributes due to alterations in the fiber surface properties. The 4-h treatment duration contributed to the formation of enhanced mechanical properties in the prepared samples. However, prolonging the treatment duration may make the fibers more rigid and brittle [36,37]. Treatment with KOH resulted in lesser mechanical performance as the hydrophilic hydroxyl groups were not effectively cleared. Alkaline treatment resulted in a cleaner fiber surface with fewer imperfections and voids in the composite [38]. The KF/PLA composites exhibited the best mechanical properties when fibers were treated chemically with $CH_3COONa$. Treating the fibers with a lower concentration of $CH_3COONa$ led to better mechanical properties. Higher concentrations of $CH_3COONa$ treatment removed cellulose content, resulting in fiber damage and a negative effect on mechanical properties. However, a 6-h treatment with $CH_3COONa$ improved the fiber surface, but the formation of grooves and cracks led to weaker mechanical properties of the specimens [39].

Table 6 – GRC, grade, and rank evaluation of KF/PLA composite - A multi-criteria comparison.

| TS (MPa) | TM (MPa) | PET (%) | FS (MPa) | FM (GPa) | PEF (%) | IS (J/m) | Grade | Rank |
|---|---|---|---|---|---|---|---|---|
| 0.6800 | 0.4783 | 0.7677 | 0.6122 | 0.3841 | 0.6282 | 0.5556 | 0.5866 | 3 |
| 1.0000 | 0.5500 | 0.8440 | 0.6522 | 0.4004 | 0.7101 | 0.7143 | 0.6958 | 1 |
| 0.5152 | 0.4074 | 0.5484 | 0.6977 | 0.4180 | 1.0000 | 0.4545 | 0.5773 | 5 |
| 0.5152 | 0.4400 | 0.4938 | 0.8108 | 0.6858 | 0.6364 | 0.3846 | 0.5666 | 6 |
| 0.4146 | 0.3929 | 0.4630 | 1.0000 | 0.6942 | 0.5326 | 0.4545 | 0.5646 | 7 |
| 0.3333 | 0.3333 | 0.4296 | 0.5357 | 0.3333 | 0.9423 | 0.3333 | 0.4630 | 9 |
| 0.5862 | 0.8462 | 0.4491 | 0.3947 | 0.4275 | 0.4188 | 0.7143 | 0.5481 | 8 |
| 0.6800 | 0.5238 | 1.0000 | 0.3896 | 0.9044 | 0.3333 | 1.0000 | 0.6902 | 2 |
| 0.4857 | 1.0000 | 0.3333 | 0.3333 | 1.0000 | 0.3475 | 0.5556 | 0.5794 | 4 |

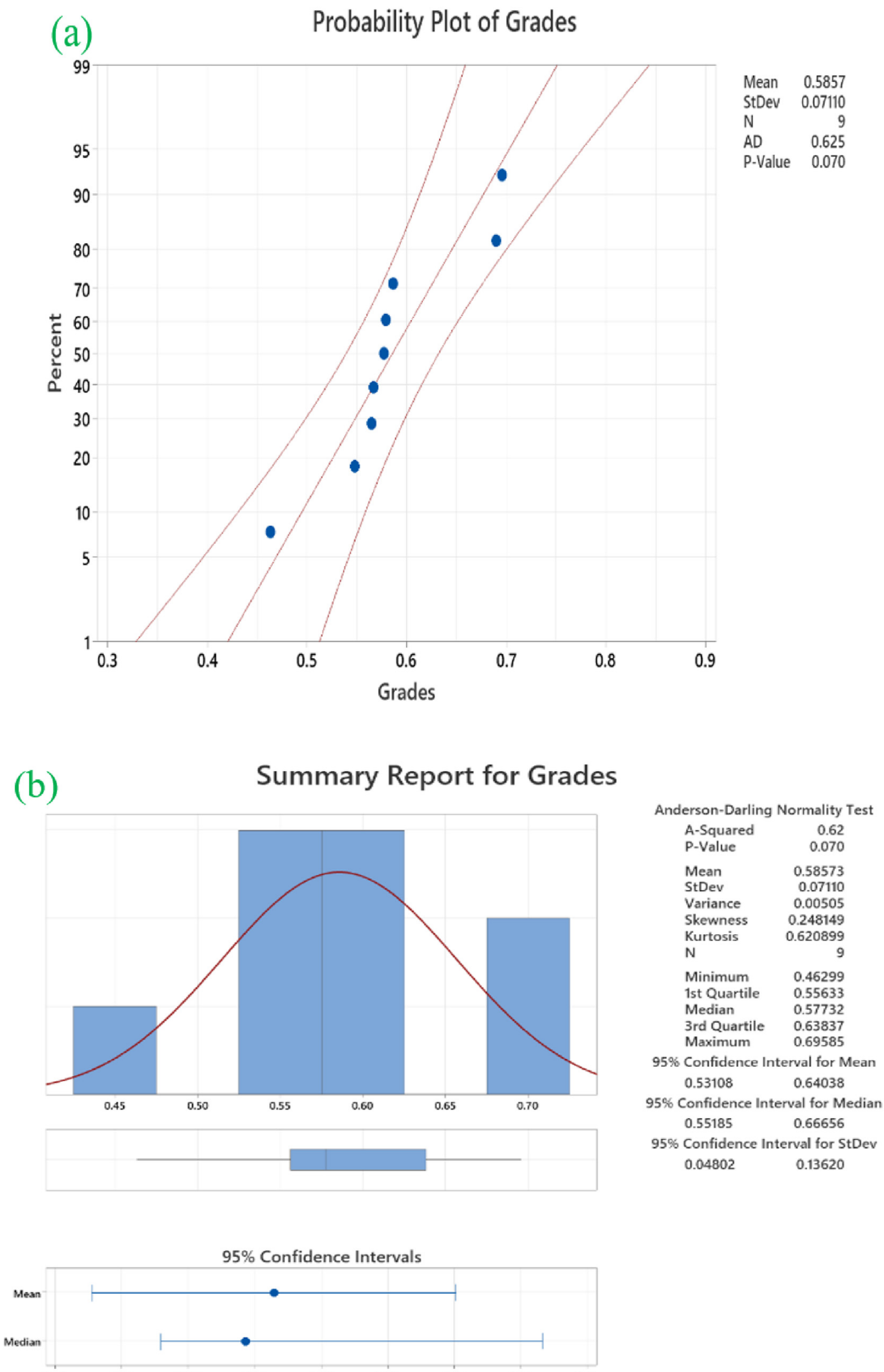



**Fig. 5 – Comprehensive normality test results for KF/PLA composite - (a) probability plot of grades, visualizing data distribution and deviation from the normal curve, and (b) summary report of grades, highlighting key statistical measures and assessing data normality.**

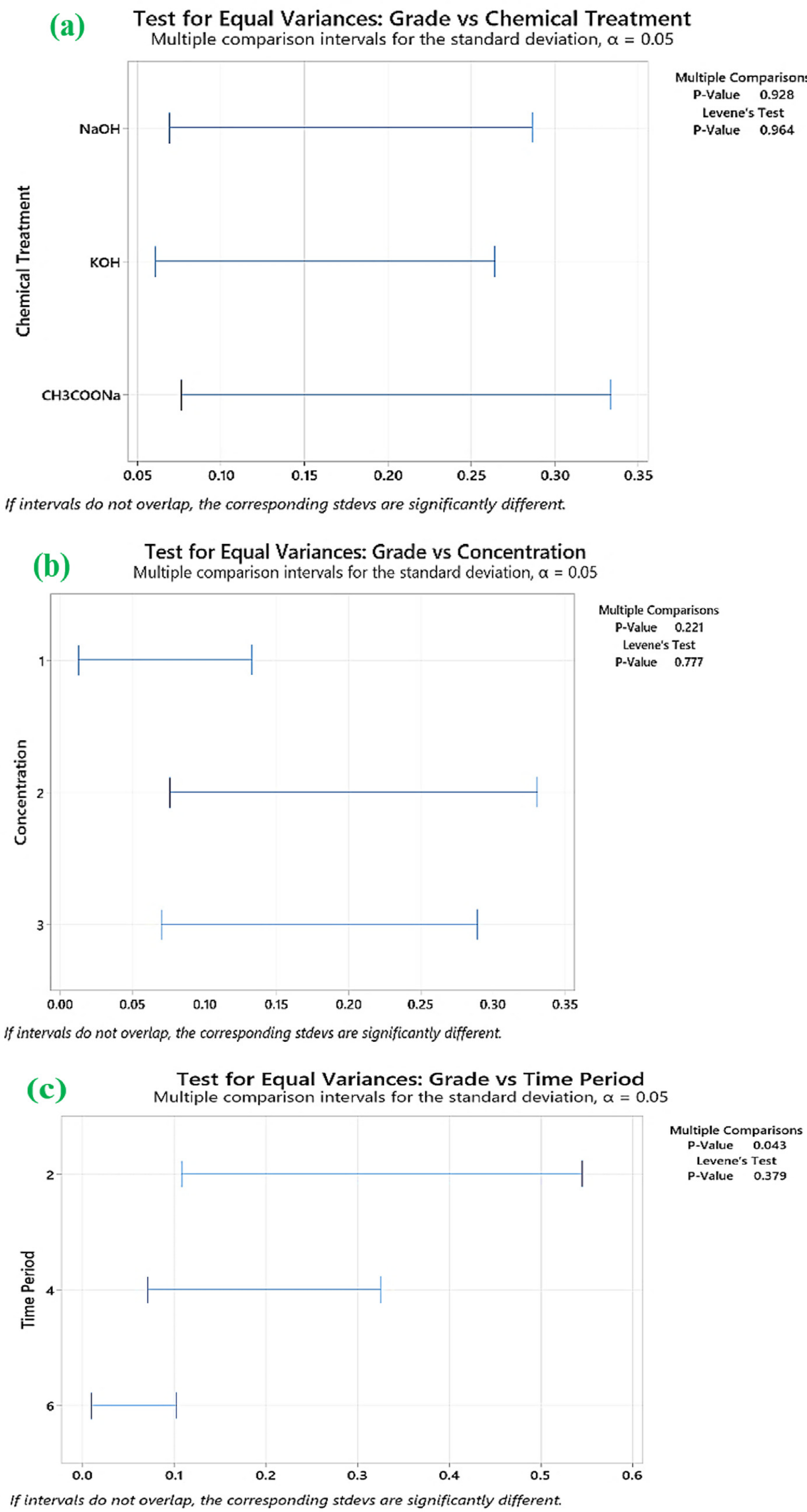



**Fig. 6 — Test Results for Equal Variance in KF/PLA Composite - Analyzing the Influence of Three Input Parameters: (a) Grade vs. Chemical Treatment, (b) Grade vs. Concentration, and (c) Grade vs. Time Period - Evaluating Variability and Consistency.**

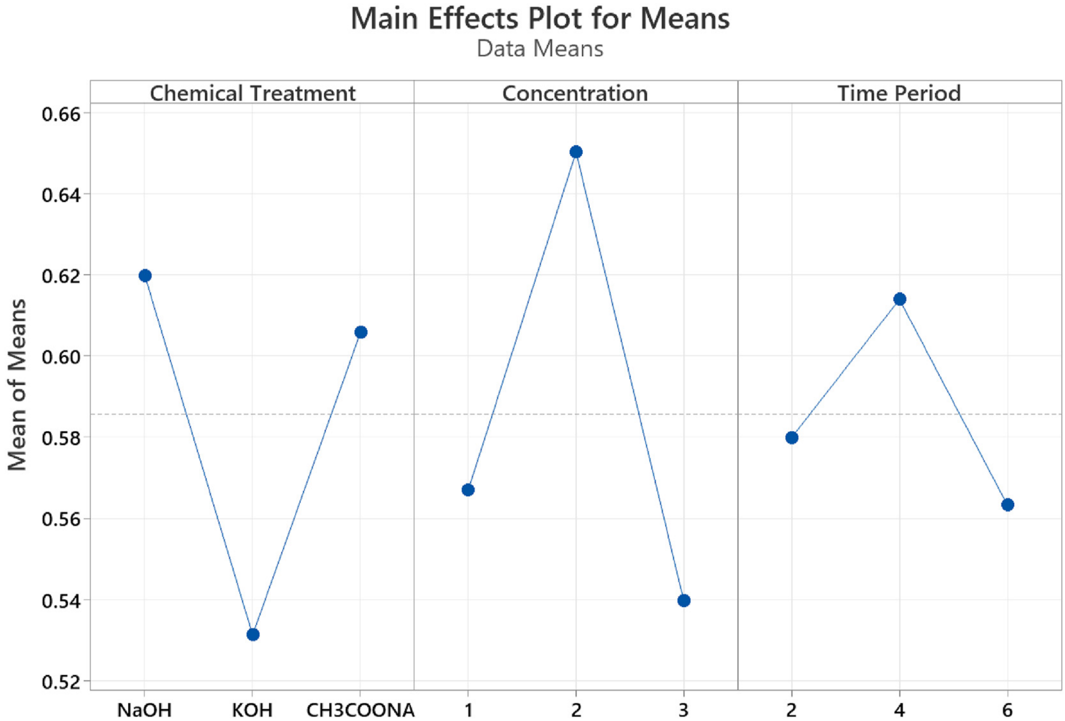



**Fig. 7 – Comparative mean plots for KF/PLA composite - visualizing the central tendencies of key mechanical properties and their interactions.**

## 5. FTIR spectroscopy

Fourier transformation infrared spectroscopy (FTIR) was performed of untreated and treated kenaf fibers and KF/PLA based bio-composite. FTIR was perfomed by scienticfic FTIR spectrometer (PerkinElmer). FTIR spectrometer was obtained in the range of 4000 $cm^{-1}$ to 40 $cm^{-1}$ in the tramsmittance mode at a scanning rate of 2 mm/s. The FTIR analysis of untreated and treated kenaf fibers is depicted in Fig. 10The spectral analysis reveals significant insights about the fiber treatment process. Notably, a wide peak centered around 3400 $cm^{-1}$ signifies the existence of hydroxyl (OH) groups in both treated and untreated fibers. However, the intensity of this peak is notably lower in treated fibers due to the removal of hemi-cellulosic and lignin components. The utilization of alkali treatment results in a reduction of hydrogen bonding as a consequence of the interaction with sodium hydroxide, leading to the removal of hydroxyl groups (-OH). These hydroxyl groups are known to engage in hydrogen bonding with carboxyl groups, as evident from the broadened and diminished peaks around 3400 $cm^{-1}$.Moreover, the absorption peaks around 2900 $cm^{-1}$, associated with C–H stretching in cellulose and hemi-cellulose, experience attenuation, indicating the removal of a portion of hemicellulose. This observation is consistent with the findings by Liu et al. [40]. The presence of a peak at 1630 $cm^{-1}$ in untreated fiber becomes absent in treated fiber. Likewise, the intensity of the peak at 1550 $cm^{-1}$ untreated fiber, linked to C=C aromatic symmetrical stretching in lignin, diminishes following the alkaline treatment process. Additionally, the peak in the 1030 $cm^{-1}$ range arises from the stretching of C–O and O–H groups, as reported by Le et al. [41]. The hydroxyl groups within cellulose possess the capability to interact with carboxyl and carbonyl groups within PLA, facilitating hydrogen bond formation. However, the existence of non-cellulosic impurities such as pectin and waxy substances in untreated kenaf fiber masks a portion of these cellulose-derived –OH groups [42–44]. Consequently, only a restricted number of –OH groups can establish bonds with the chemical groups of PLA.

**Table 7 – Response table for means.**

| Levels | Chemical treatment | Concentration | Time period |
|---|---|---|---|
| 1 | 0.6199 | 0.5671 | 0.5799 |
| 2 | 0.5314 | 0.6502 | 0.6139 |
| 3 | 0.6059 | 0.5399 | 0.5633 |
| Delta | 0.0885 | 0.1103 | 0.0506 |
| Rank | 2 | 1 | 3 |

**Table 8 – ANOVA of GRG for KF/PLA composites.**

| Source | DOF | Adj SS | Adj MS | F-value | P-value |
|---|---|---|---|---|---|
| Chemical treatment | 2 | 0.013579 | 0.006790 | 4.45 | 0.184 |
| Concentration | 2 | 0.019809 | 0.009904 | 6.49 | 0.134 |
| Time period | 2 | 0.003996 | 0.001998 | 1.31 | 0.433 |
| Error | 2 | 0.003052 | 0.001526 | | |
| Total | 8 | 0.040436 | | | |

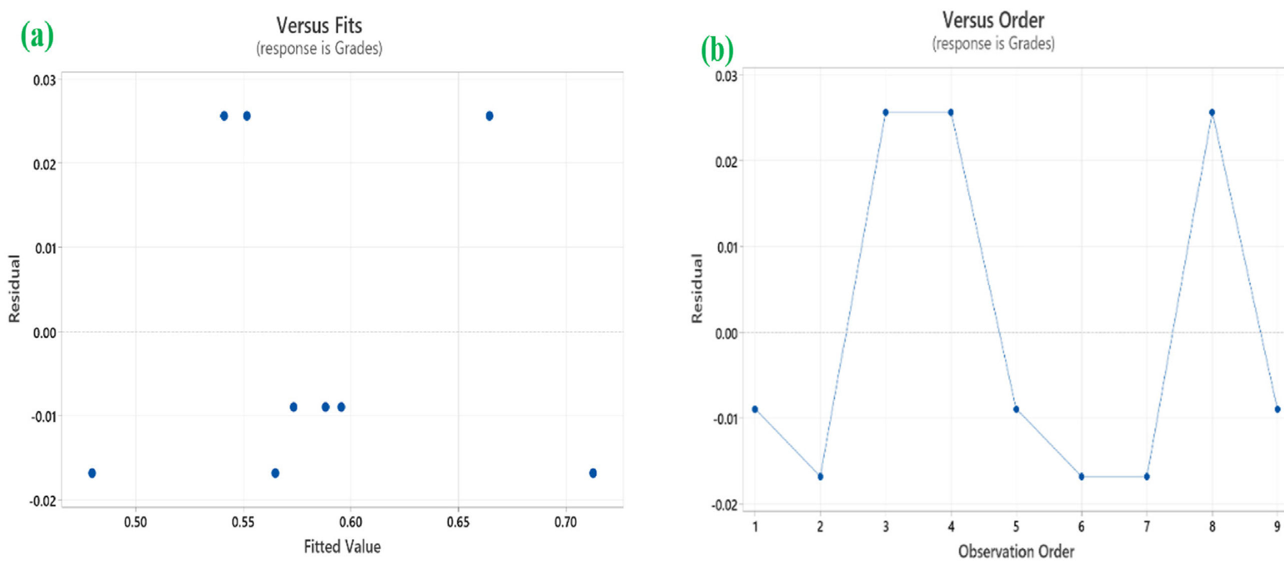



**Fig. 8 – Residual plots for KF/PLA composites - analyzing model residuals (a) versus order and (b) versus fits to assess model adequacy and identify potential outliers.**

**Table 9 – Summary of the grey relational grade model for the kenaf fiber/polylactic acid composite.**

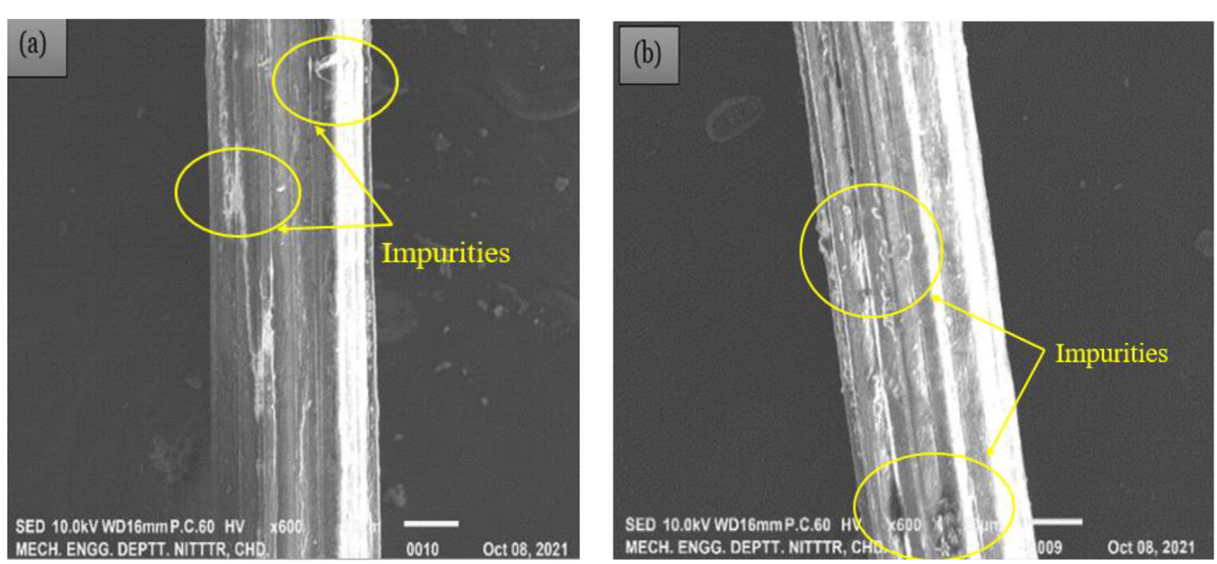
| S-Value | R-Squared | Adjusted R-Squared |
|---|---|---|
| 0.0390644 | 99.45% | 97.12% |

**Table 10 – Evaluating the differences in grey relational grade for the kenaf fiber/polylactic acid composite.**

| Best Parameter Set | GRG Value | Progress |
|---|---|---|
| Experimental Results (A1-B2-C2) | 0.6958 (Rank 1) | 0.000046988 |
| Estimated by Mean Plot (A1-B2-C2) | 0.695846 (Forecasted) | – |

## 6. Morphological analysis of the tested specimens

Failure analysis of KF/PLA-based composites is rather difficult. The failure of natural fiber reinforced composites is influenced by fiber loading, orientation, and fiber adhesion. Bio-composites fail due to a range of factors such as fiber breakage, pullout, delamination, and insufficient interfacial adhesion [45–47]. The failure of KF/PLA bio-composites under tensile and flexural loads was examined in this work. Tensile loading caused the most matrix cracks, fibre breakage, and fiber pullout as shown in Fig. 14 (a,b). Meanwhile, due of the weak interface, matrix cracks and fiber




(a) (b)

**Fig. 9 – (a,b) SEM images of single strand neat kenaf fiber (untreated).**

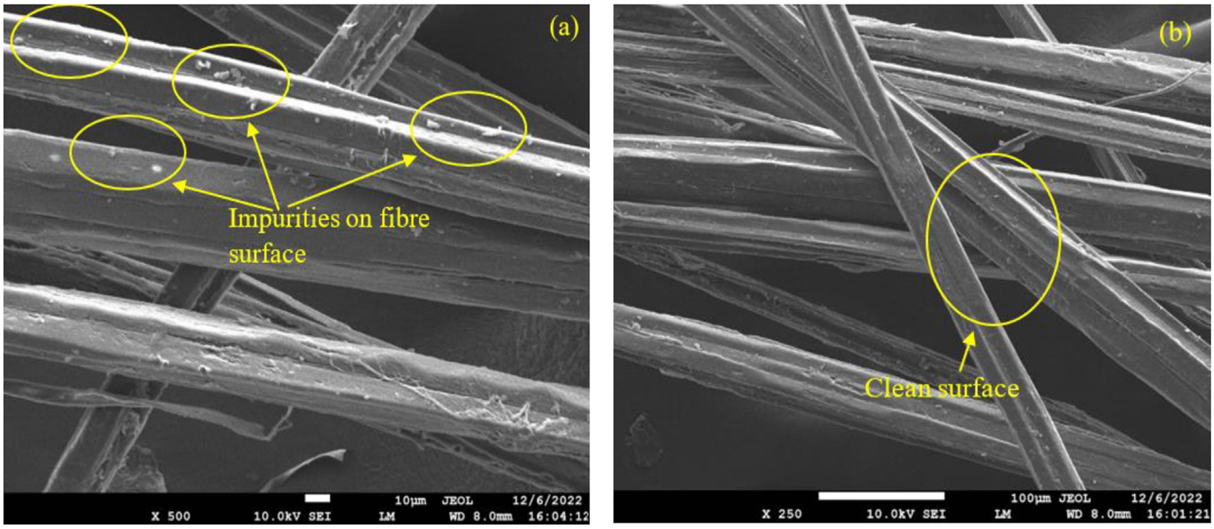



**Fig. 10 – Sem images of kenaf fibers - a comparison between untreated and NaOH-treated samples to examine surface morphology and the effects of alkaline treatment.**

pullout were seen under flexural loading [48–50] as shown in Fig. 15 (a,b).

## 7. Topographic analysis of the fabricated specimens

The morphological-topographic analysis of the KF/PLA bio-composites was further investigated through the utilization of atomic force microscopy (AFM) provided by Bruker. As AFM is a contemporary technique for imaging notable for its ability to capture high-resolution images and generate quantifiable numerical measurements of surface topography at the nanoscale. AFM employs a finely pointed tip that is thoroughly moved across the surface of the specimen, all the while ensuring that a uniform force is maintained among the tip and the sample [51–53]. The measurement of the cantilever's deflection is documented, resulting in the generation of topographical photographs. The equipment was utilized in tapping operation function under ambient conditions at standard room-temp., employing a scan rate of 1 Hz, scan-size of 10 μm, and a scan angle of 0°. Simultaneous recording was conducted for the topography, amplitude, and phase pictures for the representative sample of KF/PLA bio-composites (2% NaOH treated with 4 h).

As unveiled from Fig. 16(a,b,c), when Kenaf fibres undergo treatment with a 2% Sodium hydroxide (NaOH) solution for a duration of 4 h, numerous types of physicochemical changes have been observed.

### 7.1. *Chemical reactions*

Sodium hydroxide, a strong alkali, has the ability to undergo hydrolysis of the hemicellulose and lignin constituents present in Kenaf fibres. The process of hydrolysis

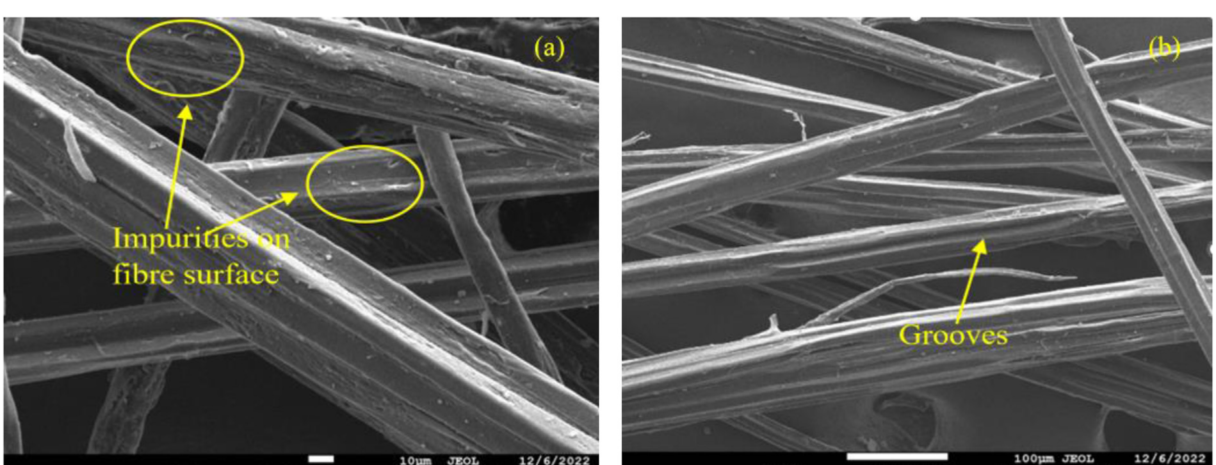



**Fig. 11 – Sem images of kenaf fibers - a comparison between untreated and KOH-treated samples to examine surface morphology.**

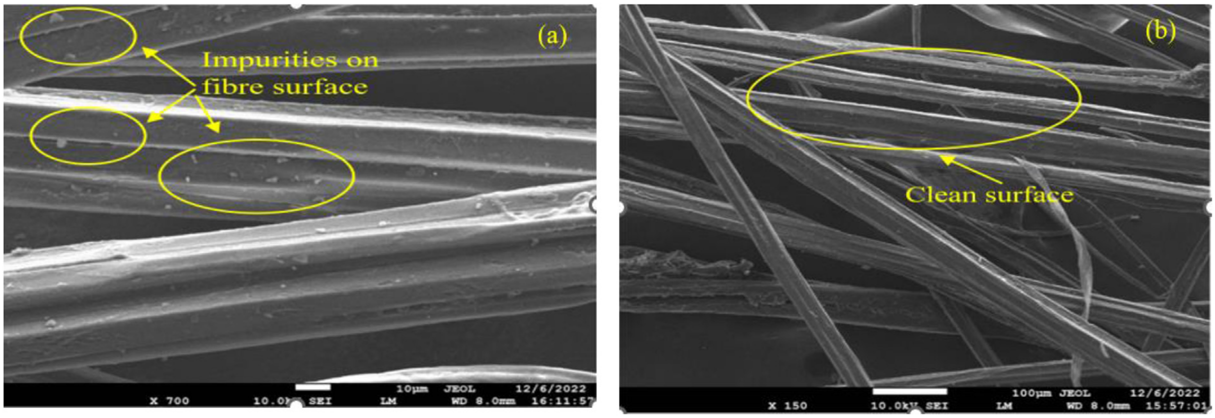



**Fig. 12 – Sem images of kenaf fibers - a visual comparison of untreated and $CH_3COONa$ -treated samples to explore surface morphology and the influence of organic salt treatment.**

results in the cleavage of chemical bonds present in these constituents, ultimately causing their disintegration/decomposition/breakdown, and dissolution in the NaOH solution [54–56]. The aforementioned method has the capability to efficiently eliminate contaminants and enhance the accessibility of fibres throughout subsequent treatments or the manufacture of composites [57,58].

### 7.2. *Swelling of fibers*

The use of sodium hydroxide (NaOH) treatment has the potential to induce swelling in Kenaf fibres. The alkali solution infiltrates the fibre matrix, resulting in its expansion [59–61]. The phenomenon of swelling leads to an expansion in the surface area of the fibres, hence exposing a greater amount of cellulose [62–64]. Cellulose is the principal element that is accountable for enhancing the strength of the PLA matrix in bio-composites [65,66].

### 7.3. *Surface activation*

Additionally, the treatment has the capability of introducing functional groups, such as hydroxyl groups, onto the surface of the fibre. These groups have the potential to strengthen the compatibility among the Kenaf fibres and the PLA matrix, hence enhancing the adhesion between the two materials [67–69].

All in all, the mechanism underlying surface roughness alterations.

### 7.4. *Chemical treatment*

Upon subjecting Kenaf fibres to sodium hydroxide (NaOH) treatment, a series of chemical reactions ensue.

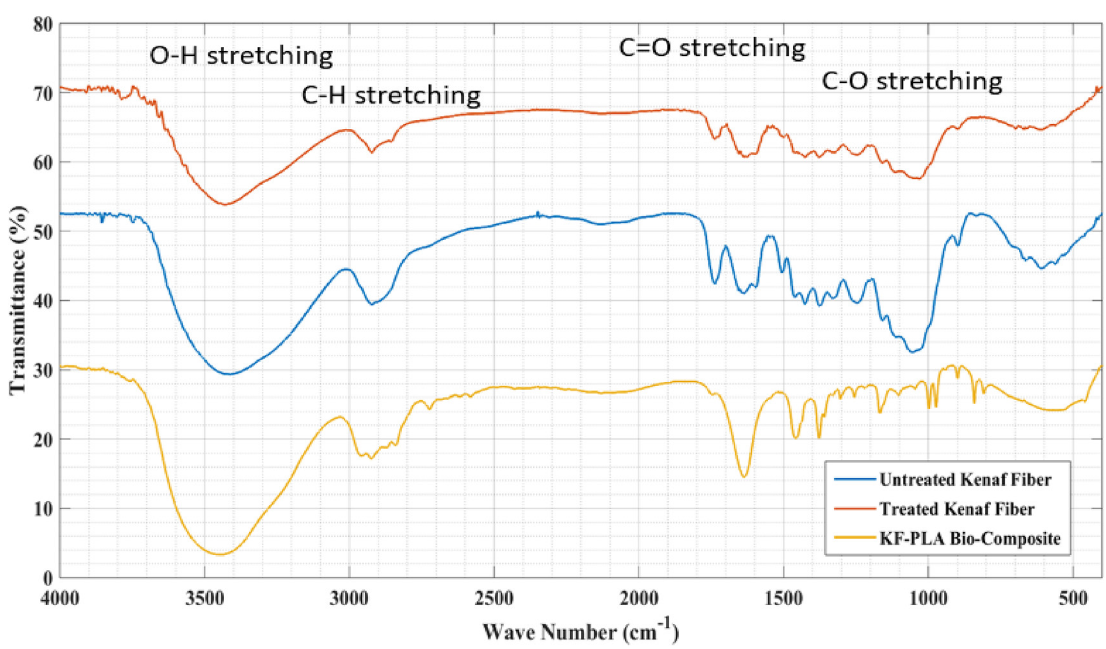



**Fig. 13 – FTIR traces of untreated and treated kenaf fibers and KF-PLA based bio-composite.**

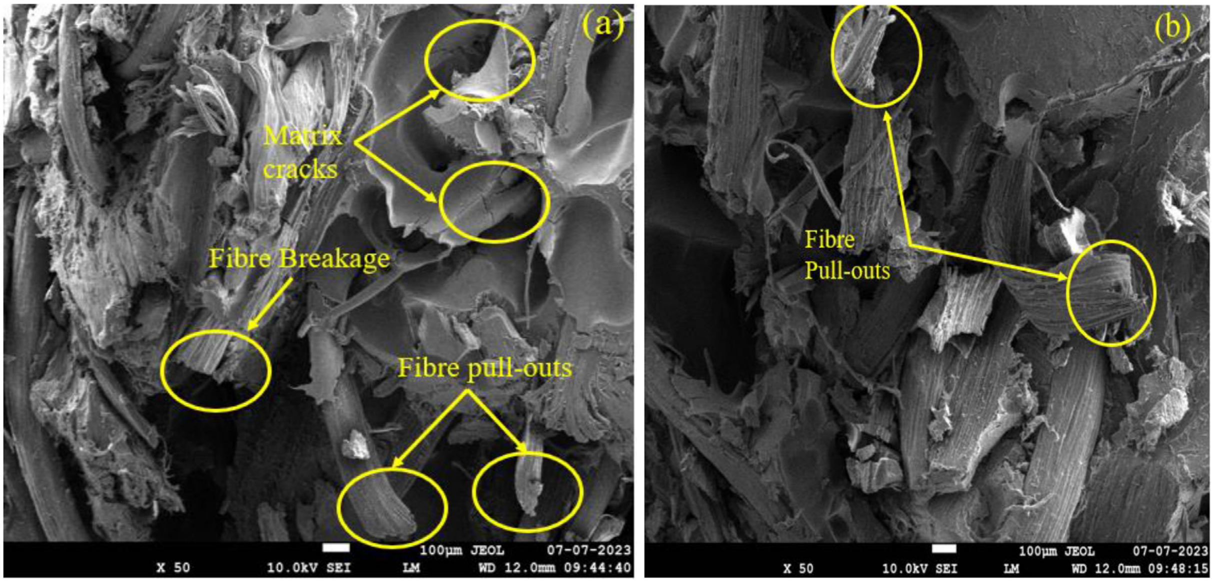



**Fig. 14 – (a,b) SEM Images of KF/PLA bio-composites under Tensile loading.**

### *7.5. Hydrolysis*

Hydrolysis is a chemical process in which NaOH is capable of breaking down the hemicellulose and cellulose constituents prevalent in the Kenaf fibres. The aforementioned technique involves the degradation of polysaccharides into smaller molecular constituents, hence facilitating the elimination of impurities, amorphous areas, and surface contaminants [70–72].

### *7.6. Swelling*

Swelling can occur as a result of the treatment with NaOH, leading to a swelling of the fibres [73]. This might lead to the expansion of the fiber's surface, unveiling additional surface characteristics [74,75].

### *7.7. Deacetylation*

Deacetylation can occur as a consequence of NaOH treatment, leading to modifications in the molecular-structure and chemical composition of the hemicellulose and subsequently affecting the surface characteristics of the fibre [76,77].

In addition, the alterations in surface roughness subsequent to the application of NaOH treatment can be ascribed to.

### *7.8. Microscopic texture*

The eradication of impurities, amorphous areas, and contaminants can expose the inherent microtexture of the fibres, resulting in disparities/variations in surface height [75–77].

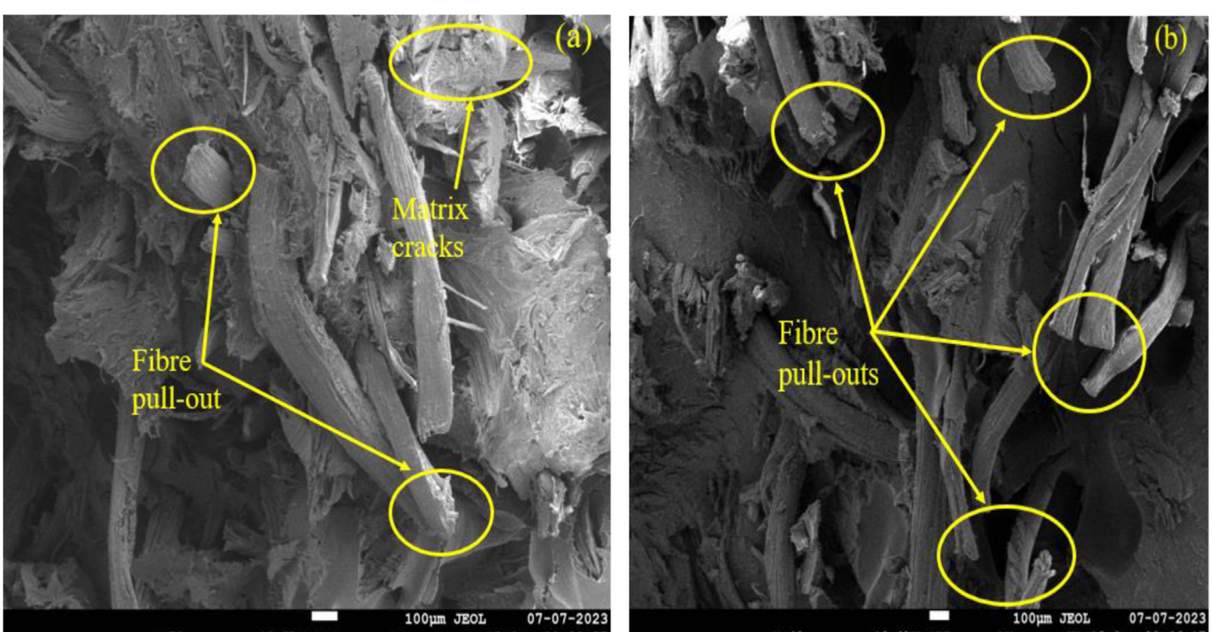



**Fig. 15 – (a,b) SEM Images of KF/PLA bio-composites under Flexural loading.**

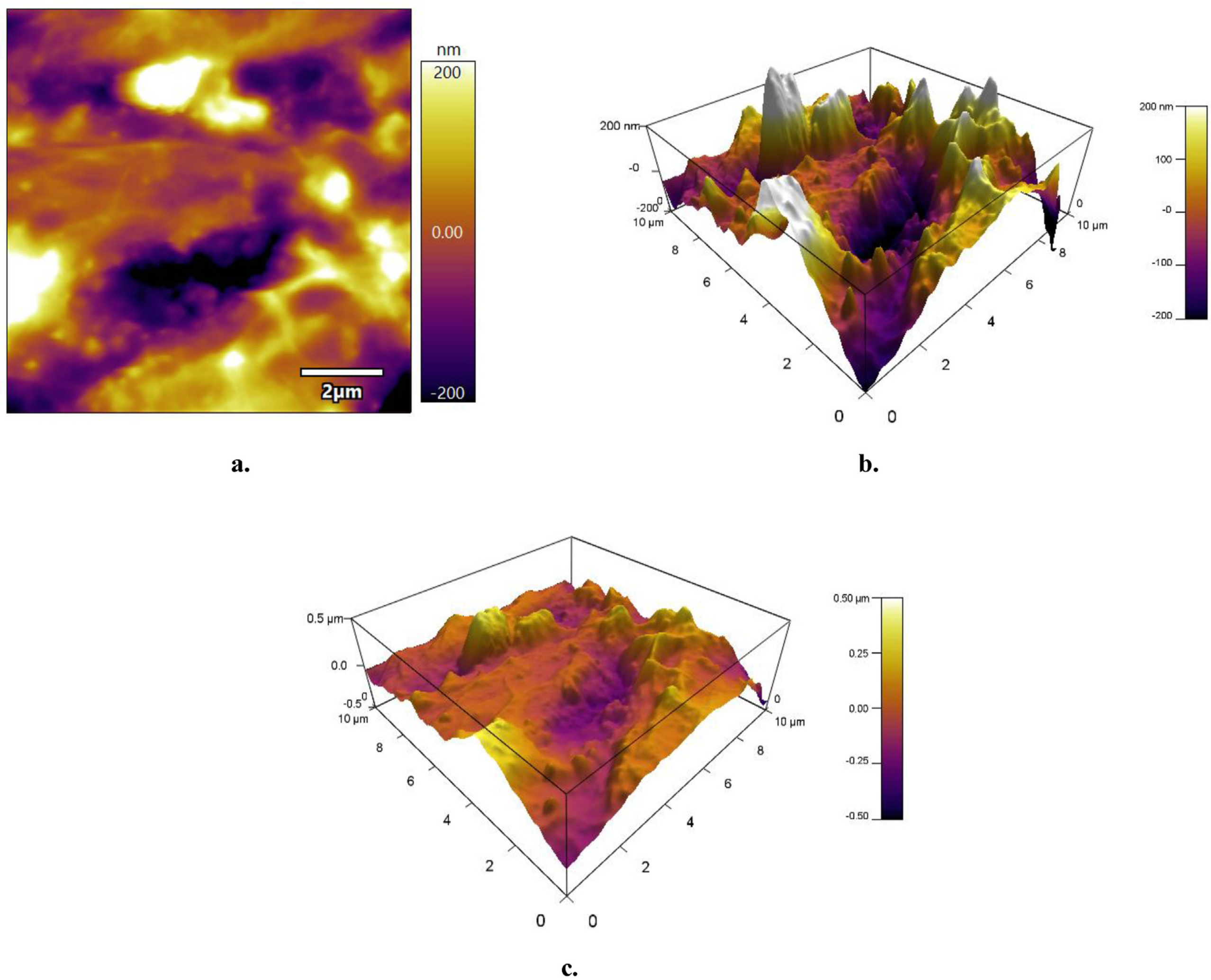



**Fig. 16 – (a,b,c) AFM Images of representative KF/PLA bio-composites sample (2% NaOH treated with 4 h).**

### *7.9. Swelling effects*

The occurrence of swelling in fibres can result in the development of pores, perforations, openings, voids, and surface-irregularities [75–77].

### *7.10. Chemical alterations*

Alterations in the chemical composition, such as the process of deacetylation, have the potential to induce modifications in the surface chemistry. Consequently, these modifications can influence the interaction between the atomic force microscopy (AFM) tip and the surface [75–77].

Hence, it is anticipated that the application of NaOH as a chemical treatment will eventually result in an enhancement of the surface roughness of Kenaf fibres. This may be attributed to the elimination of hemicellulose and lignin during the treatment process. The presence of a rougher surface facilitates enhanced mechanical interlocking and adhesion between the fibres and the PLA matrix, hence yielding superior composite characteristics [75–77].

## 8. Conclusions

This research explored green composites' fabrication and chemical treatment based on Kenaf fiber and Polylactic Acid (PLA). These composites were treated with chemicals like Sodium Hydroxide (NaOH), Potassium Hydroxide (KOH), and Sodium Acetate ($CH_3COONa$). The study aimed to understand the impact of variables such as the type of chemical used, its concentration percentage, and the duration of continuous treatment on the mechanical characteristics of the KF/PLA bio-composites. After analyzing the results, several conclusions were drawn:

i. The statistical analysis validated the experimental data acquired from the KF/PLA composite specimens. The data distribution within the established statistical models was normal, and the optimal levels identified via the Grey Relational Grade (GRG) rank and mean plot were consistent.

ii. The R-square values for the KF/PLA composite were as high as 99.45%, which indicates a substantial fit between the collected data and predicted model.

iii. Most favorable conditions for achieving the highest mechanical properties in the KF/PLA composite were determined to be NaOH chemical treatment, a 2% concentration, and a treatment duration of 4 h (A1-B2-C2). The experimental and anticipated GRG values for the KF/PLA composite were found to be 0.6958 and 0.695846, respectively, indicating an improvement of 0.000046988 in the GRG.

iv. Analysis of Variance (ANOVA) of the process parameters verified the significance of all selected parameters. The chemical concentration emerged as the most influential factor, contributing to 62.71% of the overall effect on the KF/PLA composite among all the chosen parameters.

v. Scanning Electron Microscopy revealed that impurities on the fiber surfaces played a key role in matrix cracking, fiber breakage, fiber pullout, and surface cracks. The chemical treatments of the fibers effectively removed these impurities. The morphology of the PLA matrix-based bio composites reinforced with kenaf fiber with different surface-treatments was recorded using AFM analysis in order to further reveal the surface roughness of fibers upon surface modification and dispersion of kenaf-fiber within the PLA-matrix.

vi. Several sort of chemical treatments were applied to boost the mechanical performance of the green composites. With their enhanced mechanical properties, the chemically treated green composites present potential alternatives to synthetic polymer-based composites. They could be used in a variety of structural and non-structural applications, such as door panels, cardboard tables, bowls, spoons, and food packaging.

## Informed consent statement

Not applicable.

## Availability of data and materials

It is purely experimental research work; all data acquired during experiments and analyses are included in this paper.

## Ethics approval

Compliance with ethical standard.

## Consent to participate

N.A.

## Consent for publication

N.A.

## Authorship contributions

Conceptualization, SK, RD, AM, NKD; methodology, SK, RD, AM, NKD; formal analysis, SK, RD, AM, NKD; investigation, SK, RD, AM, NKD; writing—original draft preparation, SMSAK, KAM, ASB; writing—review and editing, SK, RD, AM, NKD, SS; supervision, SS, SPD, AK, CL, EMTE, MA; project administration, SS, SPD, AK, CL, EMTE, MA; funding acquisition, SS, SPD, AK, CL, EMTE, MA. All authors have read and agreed to the published version of the manuscript.

## Declaration of competing interest

The authors declare that 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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## Nomenclature

PLA – Polylactic Acid
KF – Kenaf Fiber
NF – Natural Fiber
SEM – Scanning Electron Microscopy
UTM – Universal Testing Machine
AFM – Atomic Force Microscopy
GRA – Grey Relational Analysis
FTIR – Fourier Transformation Infrared

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