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A case study on mechanical enhancement of pineapple leaf fibers treated with lime and sodium bicarbonate

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ABSTRACT

Natural fibers are gradually replacing glass fibers in fiber-reinforced composites, especially for applications where extremely high strength is not required. In Mauritius, pineapple leaves are an abundant agricultural waste, offering an untapped source for bio-based fiber composites. However, achieving the necessary mechanical properties generally demands chemical pretreatment, commonly with environmentally harmful sodium hydroxide (NaOH). This study aims to develop and evaluate alternative, eco-friendly chemical treatments for pineapple leaf fibers (PALF). Two environmentally friendly treatments – lime and sodium bicarbonate – were investigated and compared to conventional NaOH treatment. Both treatments use natural substances and are tested on two different types of PALF with varying soaking times, while solution concentrations and preparation methods were optimized to minimize resource and energy consumption. To identify the most effective treatment, mechanical properties such as tensile strength, diameter, force at break and linear density were measured and FTIR spectra as well as SEM images were analyzed to evaluate both performance improvements and the underlying mechanisms. The results show that sodium bicarbonate treatment for 24 h yields the highest tensile strength increase (> 75%) for both studied PALF types, whereas lime or NaOH treatment results in an approximately 50% increase. Both ecofriendly treatments of PALF perform as well as or better than the commonly used treatment, highlighting their potential for future sustainable composite applications.

1. Introduction

In the course of rising environmental awareness, natural fibers are emerging materials in composites (Komuraiah et al., 2014, p. 360; Cruz and Fanguero, 2016). They are gradually replacing the use of traditional glass fibers, especially in applications that do not require very high load capacity (Karina et al., 2007; Komuraiah et al., 2014, p. 360). They offer many advantages as compared to fiberglass, e.g. lower density, lower cost of production/extraction, renewability, and biodegradability (Soburrun and Ramasawmy, 2022). In African countries and small island developing states (SIDS), like Mauritius, untapped natural resources such as agricultural bio wastes are abundant (Adeleke et al., 2021; Soburrun and Ramasawmy, 2022).

Pineapple leaves, for instance, are often an unused by-product that is either incinerated or buried in the field (Soburrun and Ramasawmy, 2022). Pineapple leaves contain 2.5 – 3.5% comparably fine, white, strong, and silky fibers (Banik et al., 2011, p. 172). This work aims to prepare pineapple leaf fibers (PALF) to utilize them in a recyclable bio high-performance composite. However,

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untreated natural fibers usually have rather low tensile strength and poor adhesion with polymer matrices, which are required to obtain high-performance composites (Cruz and Fangueiro, 2016; Soburrun and Ramasawmy, 2022). Therefore, they are commonly pretreated to enhance their tensile strength and wettability (Hashim et al., 2012). This work focuses exclusively on the development of an eco-friendly method to treat PALF in order to increase the tensile strength (Li et al., 2007; Pankaj and Kant, 2022).

Generally, the chemical composition varies from fiber to fiber (Mwaikambo, 2006). Before chemical treatment, PALF consists mainly of cellulose (roughly 70%), hemicellulose (13–19%) and lignin (4–13%) (Sreenivasan et al., 2011; Komuraiah et al., 2014; Jose et al., 2016; Hazarika et al., 2017; Karimah et al., 2021). The higher the cellulose content and crystallinity, the better the strength properties of a fiber (Karimah et al., 2021). In contrast, hemicellulose and lignin are often referred to as amorphous cementing material hindering the fibers tensile strength (Mohanty et al., 2005; Karimah et al., 2021).

The removal of the cementing components of the fiber allows the microfibrils to rearrange themselves in a better way along the direction of the tensile deformation. (Lopattananon et al., 2008). Moreover, the cellulose content is enhanced, thereby, the crystallinity raised and the diameter reduced (Komuraiah et al., 2014; Jose et al., 2016; Karimah et al., 2021). Simultaneously, a transformation from cellulose I to cellulose II can take place. During this conversion, a better chain ordering within the microstructure can be achieved, which leads to higher crystallinity equaling to a higher tensile strength (Ferreira et al., 2020; Karimah et al., 2021).

The widely studied and commonly used chemical pre-treatment is sodium hydroxide (NaOH) treatment (Baruah et al., 2018; Ravi et al., 2018). In previous studies, increases of up to 150% in the tensile strength of PALF have been achieved by treatment with sodium hydroxide (Ariffin and Yusof, 2017; Asim et al., 2018; Gaba et al., 2021). As each fiber type has different chemical compositions and physical properties, the ideal NaOH concentration for the alkaline treatment can vary (Li et al., 2007). Generally, the higher the concentration, the more impurities are removed (Fook and Yatim, 2015, p. 39). However, if the concentration is too high, cellulose II crystals will begin to degrade, resulting in a reduction in crystallinity and a deterioration in mechanical properties (Soburrun and Ramasawmy, 2022; Pankaj and Kant, 2022). For sodium hydroxide treatment of PALF, several studies have reported the optimal NaOH concentration for achieving the highest tensile strength to be 6% (Ariffin and Yusof, 2017; Ramsewak, 2017; Asim et al., 2018; Gaba et al., 2021). Two other important factors impacting the outcome of the treatment are soaking time and temperature (Hashim et al., 2012; Rajeshkumar et al., 2020, p. 305). The diameter of fibers is significantly reduced with a higher soaking time which implies a constant removal of material (Rajeshkumar et al., 2020, p. 313). Nevertheless, there is an optimal soaking time that should not be exceeded, as degradation can occur after this time (Asim et al., 2018). For PALF, depending on the concentration and temperature of the NaOH solution, it is found to be 5–6 h (Ariffin and Yusof, 2017; Asim et al., 2018). Despite its effectiveness, NaOH is synthetic, relatively expensive, and poses environmental and health hazards, leading to increased efforts to find more environmentally friendly substitutes (Soburrun and Ramasawmy, 2022, p. 257). One promising candidate is **lime treatment**. Only few papers have studied the effect of lime treatment on fibers' chemical composition and structure, and even fewer investigated the treatment's impact on fibers' mechanical properties (Ramasawmy et al., 2021, p. 3). Generally, the goal of lime treatment is identical to sodium hydroxide treatment. There is little evidence of the transformation of cellulose I to cellulose II, which can enhance fibers' crystallinity (Le Troedec et al., 2008; Ramasawmy et al., 2021). However, a characteristic effect of lime treatment is the deposit of calcium nodules on the fiber surface building a stable cross-linked calcium pectinate layer (Sedan et al., 2008; Ramasawmy et al., 2021).

Most previous works use saturated lime solutions with 10%-M or lower concentrations (Le Troedec et al., 2008; Anggraini et al., 2017; Ramasawmy et al., 2021). Soaking times for lime treatments investigated in previous studies range from 1 h to 5 days (Sedan et al., 2007; Le Troedec et al., 2008; Liang et al., 2014; Anggraini et al., 2017; Ramasawmy et al., 2021). The higher the soaking time, the higher the density of the calcium nodules on the fiber surface (Ramasawmy et al., 2021, p. 12). Apart from this work, there is currently no knowledge of any other work dealing with lime treatment of pineapple fibers. Therefore, findings of this work could reveal whether lime treatment is a suitable eco-friendly substitute for NaOH treatment of PALF.

Sodium bicarbonate treatment (s.b.) is a rather new eco-friendly and cost-effective approach and therefore less common and studied than NaOH. Fiore et al. (2016) propose the interaction between fiber and sodium bicarbonate solution is similar to traditional mercerization. Komal et al. (2022) investigate the effect of sodium bicarbonate treatment on PALF. Their results show sodium bicarbonate behaves like mild NaOH and removes non-cellulosic contents. It thereby increases the tensile strength from less than 400 MPa to more than 500 MPa (applying 10% sodium bicarbonate over 72 h). They note the longer the soaking time, the higher the fiber's crystallinity. However, identical to NaOH treatment after a certain time, excessive removal of hemicellulose causes a reduction of the single fiber's tensile strength. According to their work, the ideal soaking time is 3 days when using a 10% sodium bicarbonate solution, as for 2 and 4 days the single fiber tensile strength is lower (Komal and Singh, 2022, p. 9). While previous studies (Fiore et al., 2016; Bakri et al., 2018; Oliveira et al., 2018; Mukhtar et al., 2019; Belaadi et al., 2020; Komal and Singh, 2022) have applied sodium bicarbonate to other natural fibers such as flax or sisal, and lime treatments have mostly been analyzed for chemical composition, their influence on the tensile properties of PALF remains largely unexplored. In particular, no systematic comparison of lime and sodium bicarbonate treatments against conventional NaOH has been carried out at the single-fiber level. This study addresses this gap by evaluating the effectiveness of both eco-friendly treatments in improving PALF tensile strength, supported by FTIR and SEM analysis. The aim is to identify whether these alternatives can provide competitive mechanical performance while avoiding the drawbacks of NaOH treatment.

2. Material and methods

2.1. PALF types

Two different types of pineapple fibers were used: local fibers from Mauritius and fibers imported from Indonesia. The **local** fibers

were mechanically extracted from the leaves of the *Victoria* pineapple variety (the common variety grown in Mauritius). First, all spines on the outer edges of the leaves were manually trimmed and removed. The trimmed leaves were then fed into a mechanical decorticator. Subsequent to this, the extracted fibers were subjected to a drying process via air circulation, followed by a thorough combing procedure.

The imported fibers were procured from Nextevo Pte Ltd., a Singaporean entity, and were sourced from Indonesia. The Indonesian pineapple leaves were obtained from the variety *Ananas comosus* by local farmers, and the fibers were subsequently extracted through Mechanical Extraction at Nextevo's production facilities. Afterwards, they were left to sun-dry. Hence, both extraction processes are identical in nature.

2.2. Fiber treatments

For each treatment batch, 1 g of local and 1 g of imported fiber are separately bundled. The bundles are knotted with PALF itself. The bundles were then inserted into a measuring cylinder and suspended from a wire to reduce entanglement (see Fig. 1). For the treatment, 100 mL of solution was prepared for two fiber bundles (total fiber mass = 2 g), giving a fiber-to-solution ratio of 1:50. This preparation ensured that, even accounting for minor losses (e.g., from evaporation), the effective ratio would not fall below 1:40, which we considered the minimum required for consistent treatment. The prepared solution was poured into the glassware containing the fiber bundles (see Fig. 2).

In the course of the investigation, multiple bundles were treated in a given beaker in a simultaneous manner. Thereafter, the bundles were removed from the beaker one at a time, contingent upon the relevant soaking time as stipulated in the experimental plan. Subsequent to the completion of the designated soaking time, the bundle was extracted from the solution and meticulously washed with tap water until the washing water exhibited a neutral pH of 7. Following this, the fibers were meticulously brushed while in a horizontal position on a pristine and horizontal surface. Finally, the fibers were subjected to an air-drying process for a minimum period of three days. To ensure the completion of the drying process, the fibers were weighed at regular intervals until no further mass loss was observed.

2.3. Design of experiments

Lime treatment was carried out by dissolving quick lime (also known as calcium oxide powder) in distilled water, yielding a slaked lime solution (Kenny and Oates, 2007). The objective of the solution preparation phase was to minimize the use of resources (energy, quick lime and distilled water), while still ensuring an efficient treatment enhancing the fibers' tensile strength. Preliminary tests were conducted to ascertain the most efficacious method for preparing the slake lime solution with the highest pH while utilizing the minimum of resources. Consequently, the following preliminary experiments were carried out:

1. Test which of the following solution preparation methods achieves the highest pH value:
 - a) Add room temperature water to quick lime powder (reference solution)
 - b) Mechanically stir reference solution for 8 h
 - c) Magnetically stir reference solution for 8 h
 - d) Add hot water (temperature > 80 °C) to quick lime powder
 - e) Add hot water (temperature > 80 °C) to quick lime powder and magnetically stir the solution for 8 h
2. Determine the extent to which reducing the concentration of the solution significantly lowers its pH. At the same time, study how the pH of the solutions changes as a function of the total reaction/solution time

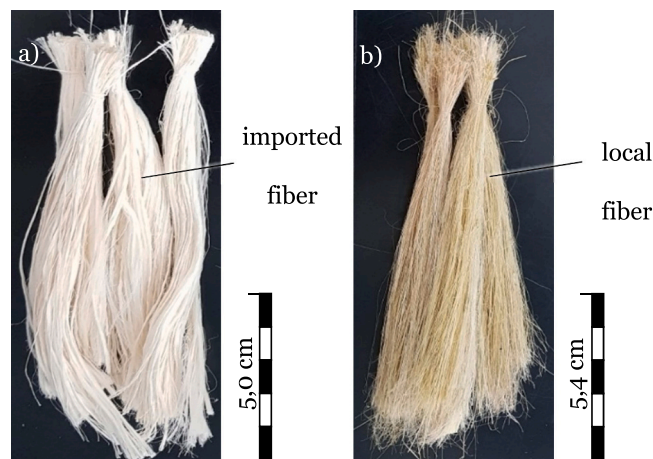


Fig. 1. Bundles of untreated a) imported (origin Indonesia) and b) local (origin Mauritius) PALF.

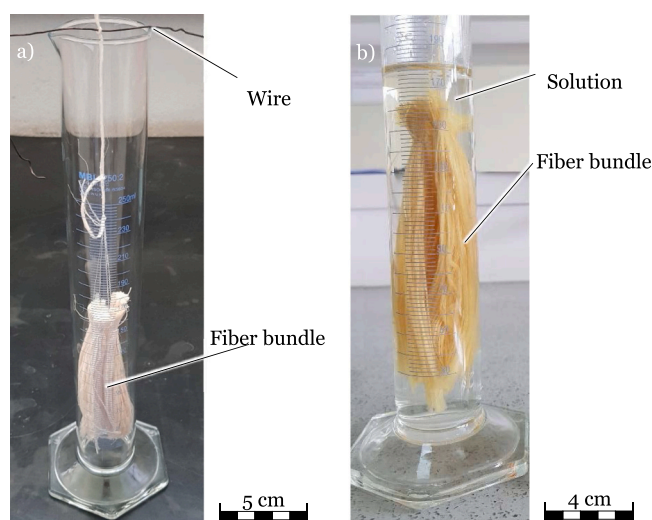


Fig. 2. PALF treatment setup: a) bundles of fibers suspended from a wire into glassware and b) fiber bundles immersed in lime solution.

3. Identify the lowest concentration that still achieves an insignificantly lower pH compared to higher concentrations

Preliminary tests were conducted to identify solution preparation conditions that achieve an effective alkalinity for fiber treatment, expressed as a target pH range. For lime treatment, the most suitable conditions were found by adding hot water ($>80\text{ }^{\circ}\text{C}$) to quick lime powder, followed by magnetic stirring for 8 h. Under these conditions, a calcium oxide concentration of 1 % CaO produced a stable pH between 12 and 13, while minimizing resource consumption. In contrast, for sodium bicarbonate, higher concentrations (around 3 %) were required to reach pH values in the range of 8–9. Thus, while the exact concentrations differ between chemicals, the key parameter for treatment efficiency was the resulting solution pH, which guided the choice of concentration. This approach is consistent with previous studies showing that fiber pretreatment effectiveness correlates with solution alkalinity (Le Troedec et al., 2008; Anggraini et al., 2017; Hussain et al., 2021; Ramasawmy et al., 2021).

The final solution for treatments was prepared one day in advance and left to dissolve overnight. The initial mixture was filtered through standard filter paper to remove any undissolved lime particles before the fibers were immersed. The investigated soaking times for lime treatment were 1 h, 2 h, 4 h, 8 h and 24 h. All soaking times were performed at room temperature. The set of soaking times performed were similar compared to previous studies (see Section 1) (Sedan et al., 2007; Le Troedec et al., 2008; Liang et al., 2014; Anggraini et al., 2017; Ramasawmy et al., 2021).

Sodium bicarbonate treatment (NaHCO_3) was administered via the dissolution of commercially available baking soda powder in distilled water. The treatment was carried out for the soaking durations of 8 h and 24 h at room temperature in order to compare with the effect of lime treatment for the same soaking durations. Preliminary testing indicated that a 3 % sodium bicarbonate solution attained the highest alkalinity, with a pH value ranging from 8 to 9. Shorter durations (≤ 4 h) were initially tested but showed negligible effect due to the mild alkalinity of NaHCO_3 . To ensure resource efficiency and avoid unnecessary use of materials, only 8 h and 24 h treatments were therefore retained, as they were expected to produce measurable effects. In contrast to the filtration requirements for higher concentrations, the 3 % NaHCO_3 solution does not necessitate filtration, as the powder is capable of complete dissolution in distilled water.

The chemistry of these treatments relies on alkaline reactions. NaOH removes hemicellulose and lignin while partly converting cellulose I to cellulose II (mercerization). Lime releases OH^- ions, causing similar effects and forming calcium pectinate nodules on the fiber surface.

Sodium hydroxide treatment (NaOH) was included as the reference treatment in order to benchmark the efficiency of the eco-friendly alternatives. A 6 % NaOH solution was prepared by dissolving pellets in distilled water, and fibers were soaked for 8 h and 24 h at room temperature. These soaking durations were chosen to allow direct comparison with the lime and sodium bicarbonate treatments, while maintaining consistency with established procedures for PALF mercerization (Ariffin and Yusof, 2017; Asim et al., 2018; Gaba et al., 2021).

2.4. Testing methods

2.4.1. Single fiber tensile test

The tensile testing of single fibers was conducted on a Testometric M 500–50AT Universal testing machine (UTM) equipped with a 10 kgf (kilogram-force) load cell, with a gauge length of 25.4 mm as per ASTM C1557, 2020. The crosshead speed was set at 3.5 mm/min or 5 mm/min so that fiber fracture resulted between 5 and 30 s. This test was repeated for a sample size of 50 fibers for each experimental condition.

The tensile strength was determined by dividing the breaking force by the cross-sectional area of the fiber. The diameter of the fiber was taken as the average value of ten measured points along each fiber. These measurements were taken using a Digimicro-Profi USB microscope equipped with ImageJ software.

2.4.2. Linear density measurements

After examining to what extent the tensile strength increased through the various treatment conditions, it was imperative to investigate the change in the physical property, such as the fineness of the fibers, represented by the linear density in tex [g/km]. Linear density was chosen as it better reflects changes in fiber fineness than actual density, which is difficult to determine for natural fibers with irregular cross-sections. Such alterations can be attributed to the elimination of cementing material, a phenomenon substantiated by a decrease in mass (Zwane et al., 2019, p. 7). The linear density (D) was determined by dividing the sum of the total mass (m) of a given fiber bundle by the sum of lengths of all fibers comprising the bundle [l] (ASTM D1577, 2022):

$$D \text{ [tex]} = \frac{\sum m \text{ [g]}}{\sum l \text{ [km]}} \quad (2.2)$$

2.4.3. FTIR analysis

The presence of the main chemical functional groups in the untreated and treated PALF fibers was assessed through the Fourier-transform infrared spectroscopy (FTIR). It is imperative to acknowledge that FTIR spectroscopy offers relative intensity values exclusively. Consequently, the peak heights observed within an FTIR spectrum are indicative of the proportion of the respective chemical functional groups present within the fiber specimen that has been examined (Gaffney et al., 2012).

The first batch of samples (im untreated, im lime 1 h - 8 h) was FTIR tested using a Bruker Alpha FTIR spectrometer in the range of wavenumbers from 400 to 4000 cm^{-1} with a sample scan time of 24 scans, and a resolution of 4 cm^{-1} . Each fiber sample was directly tested without any sample preparation. The remaining samples were tested with another FTIR spectrometer model, the Bruker Alpha II. The sample preparation procedure involved grinding and compressing the fibers with a mortar into a powder form. The compacted powder was then inserted into a small metallic cup which was subsequently inserted into the FTIR testing machine. Prior to conducting any subsequent analyses on the SpectraGryph software, all FTIR graphs obtained were normalized. While there are numerous characteristic wavelengths that can be examined, the present study exclusively focuses on the assessment of the peaks at the following characteristic wavelengths in order to evaluate the effect of the treatment: 1244 cm^{-1} for lignin removal, 1730 cm^{-1} for hemicellulose removal and for cellulose transformation 893 and 1420 cm^{-1} . In addition, the total crystallinity index (TCI) and lateral order index (LOI) were calculated from the FTIR spectra by taking the absorbance ratios of the characteristic peaks at 1372/2900 cm^{-1} (TCI) and 1420/893 cm^{-1} (LOI), respectively, to provide a quantitative measure of changes in cellulose structure resulting from the treatments. (Nelson and O'Connor, 1964; Biagiotti et al., 2004; Liu et al., 2004; Liu et al., 2014; Ramasawmy et al., 2021; Soburrun and Ramasawmy, 2022).

2.4.4. SEM analysis

Scanning electron microscopy was performed to obtain images of the surface morphology of the fibers using a FlexSEM 1000 by the Hitachi High-Tech Corporation with an accelerated voltage of 5 kV, a working distance of 10.5 mm, and tilt compensation set to zero. Two fibers (chosen at random) of each experimental condition were analyzed, each with a magnification level of 500X, 1000X and 2000X. The different samples were coated with a thin layer of gold powder so that the coatings electron emission can be measured producing a detailed field in gray scale images (Mohammed and Abdullah, 2018).

2.5. Statistical analysis of data

The collected data underwent both qualitative and quantitative analysis using MS Excel, with the statistical evaluation focusing on tensile testing results. Outliers in force at break and diameter were removed using the interquartile range (IQR) method, while a modified approach using the median was applied for tensile strength due to a low IQR. In instances where any of the three related measurements (diameter, force at break, tensile strength) were identified as outliers, the entire fiber sample was excluded, thereby ensuring a minimum sample size of 46 fibers per condition. The statistical significance of differences in fiber properties was assessed using z-tests at a 95 % confidence level, with p-values computed via MS Excel's Analysis ToolPak. A p-value below 0.05 indicated a statistically significant difference. A two-tailed distribution is assumed.

3. Results and discussion

3.1. Tensile strength results

The general increase in tensile strength for almost all treatment conditions is shown in Fig. 3. The only treatment conditions which reduced the TS compared to untreated fibers are im lime 1 h and im lime 2 h. For imported fibers there is no statistically significant difference between the mean TS of untreated and lime 1 h - 4 h treatments. The batches lo lime 24 h and lo NaOH 24 h are the only two treatment conditions not achieving a statistically significant change in TS compared to untreated local fibers. The highest TS for local and imported fibers is achieved through sodium bicarbonate treatment with a soaking time of 24 h: lo s.b. 24 h with 591 MPa and im s. b. 24 h with 575 MPa. It is notable that both equal an increase TS of > 75 % compared to the respective untreated fibers. As mentioned

above, the ideal soaking time for a treatment of PALF with a 6 % NaOH solution is 5 – 6 h (Ariffin and Yusof, 2017; Asim et al., 2018). Due to the much longer soaking time but identical concentration applied, degradation and rupture of fiber surface may have caused a decreased TS for NaOH 24 h compared to NaOH 8 h (Asim et al., 2018).

The changes in TS are to a large extent different when comparing the findings for imported and local fibers. For imported fibers, the ideal soaking time (of all tested: 1 h, 2 h, 4 h, 8 h, 24 h) for lime treatment of PALF is 24 h with an increased TS by 56 % which is even higher than the reference treatment *im* NaOH 8 h with 49 %. For local fibers, the optimal soaking time for lime treatment of PALF is 1 h with an increased TS by 49 % which is almost identical to the reference treatment *lo* NaOH 8 h. As illustrated in Fig. 4, the data set presents the percentage change (relative to untreated fibers) in average tensile strength, force at break, diameter, and linear density for both imported and local fibers, categorized by treatment type.

The soaking times that resulted in the highest respective tensile strength increase are depicted without a pattern filling. The results indicate that both ecological treatments (lime and sodium bicarbonate) effectively increase the breaking load while reducing fiber diameter, often outperforming the conventional NaOH treatment. Of particular note is the finding that sodium bicarbonate treatment of imported fibers resulted in the most significant increase in force at break (73 % for 24-hour soaking), while local fibers exhibited a less pronounced increase in force but a more substantial reduction in diameter. The effectiveness of these treatments, particularly at low concentrations (1 % lime and 3 % NaHCO₃), is noteworthy. To further elucidate these findings, FTIR analysis will be conducted in the following sections to investigate the chemical modifications responsible for these improvements.

3.2. Linear density

For local fibers, all treatments result in a reduction in linear density, indicating mass loss due to the removal of cementing materials. A similar trend is observed for most imported fibers, except for *im* NaOH 8 h and *im* lime 2 h, which show higher linear density values. This variation can be attributed to the natural variability of fiber diameter, as these fibers were taken from different bundles, leading to a higher mean diameter after treatment. In general, longer soaking times (excluding lime treatments) correlate with greater mass reduction, consistent with previous studies (Zwane et al., 2019; Rajeshkumar et al., 2020, p. 305). The most significant decrease in linear density is evident for *lo* lime 4 h (-43 %), with *lo* lime 24 h also showing a notable reduction in both diameter and linear density. While there is partial alignment between the linear density results and the observed diameter changes, a clear correlation is not supported, suggesting potential discrepancies between mass reduction and diameter reduction. The greater percentage decrease in linear density compared to diameter across most batches suggests a correlation between mass reduction and diameter reduction, though not on a 1:1 scale, implying a decrease in density. This phenomenon can be attributed to the alkaline treatment penetrating the fiber’s cell walls and microfibrils, leading to the partial removal of amorphous components such as hemicellulose and lignin, reducing the fiber’s net mass. Furthermore, the treatment eliminates surface impurities, such as waxes, and removes hemicellulose and lignin from the fiber surface, resulting in a decrease in both effective diameter and mass. Consequently, the combined mass loss from internal and surface component removal leads to a more substantial decrease in mass compared to the reduction in fiber diameter. These

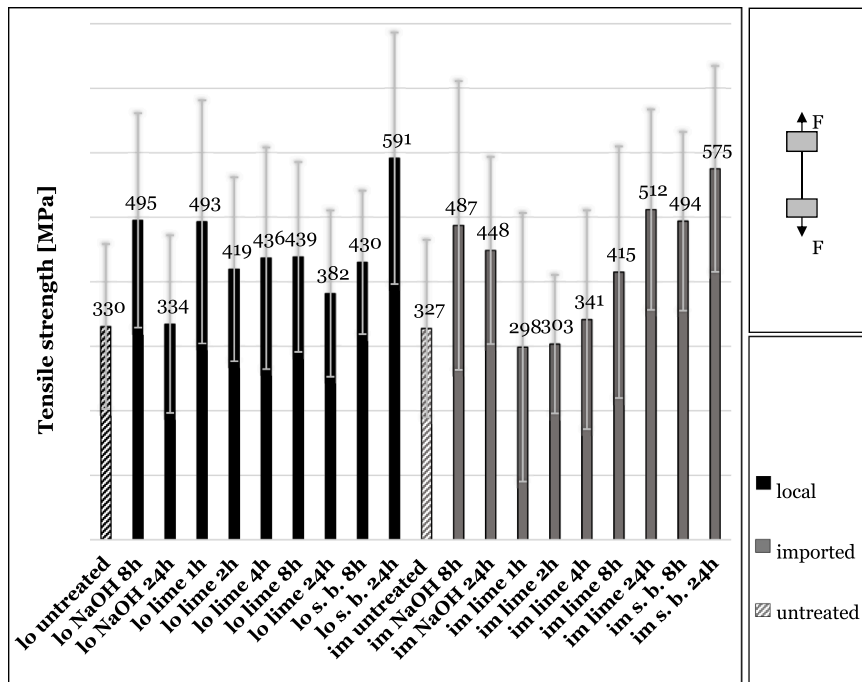


Fig. 3. Tensile strength summary of all specimens (treated and untreated) for imported (*im*) and local (*lo*) fibers.

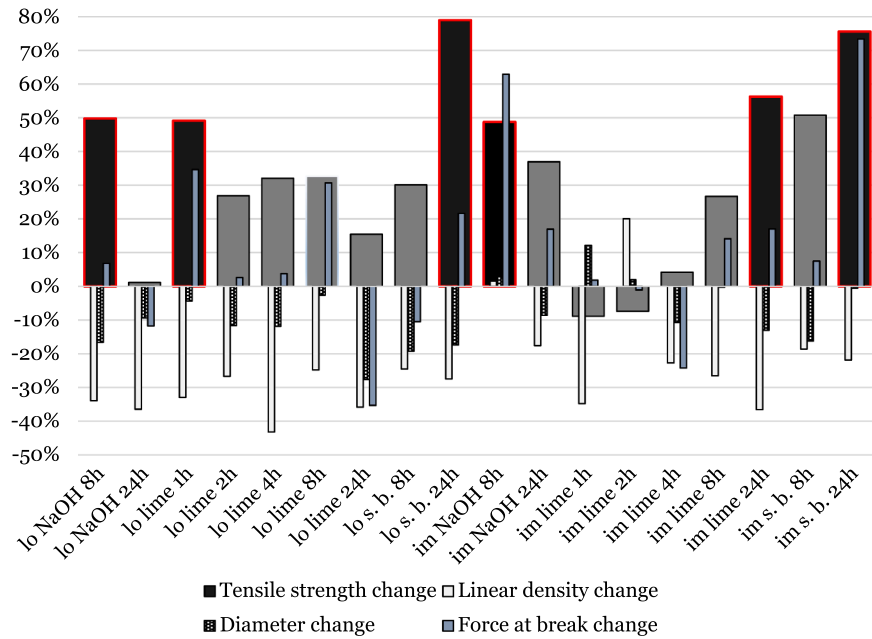


Fig. 4. Average tensile strength, force at break, diameter and linear density change of each treatment type of imported (*im*) and local (*lo*) fibers.

findings are supported by studies that have reported similar effects of alkaline treatments on natural fibers. (Chen et al., 2017)

3.3. FTIR analysis

The obtained FTIR spectra (see Fig. 5) showed that the *lime* and *NaOH* treatments of imported and local PALF led to the partial removal of lignin and hemicellulose. For both untreated and treated fibers at various treatment times (see Table 1), the TCI generally decreased while the LOI increased following alkaline treatment. This trend indicates a conversion from cellulose I to cellulose II and a

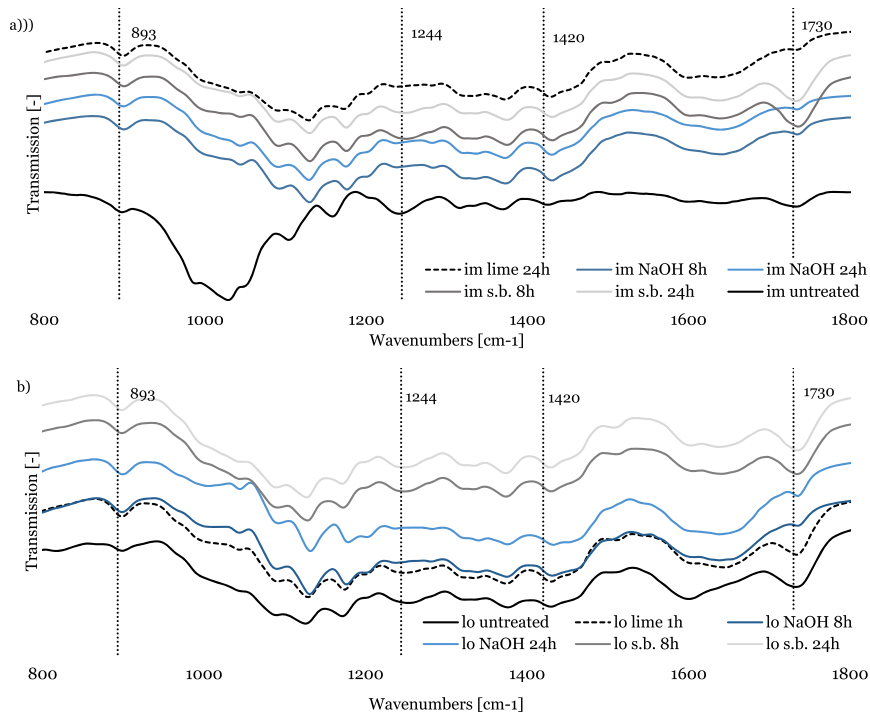


Fig. 5. FTIR stacked transmission spectra of a) imported (*im*) and b) local (*lo*) fiber samples.

reorganization of the cellulose structure, consistent with the findings of Oh et al. (2005).

In addition to these index changes, peak shifts in the spectra further support this interpretation: the cellulose I band near 898 cm^{-1} shifts toward $\sim 893\text{ cm}^{-1}$ in treated samples, a well-established marker for cellulose II formation (Oh et al., 2005; Yue et al., 2013). Together with the increased intensity at $\sim 893\text{ cm}^{-1}$, this provides strong evidence that the applied treatments induced cellulose transformation.

A notable exception in this trend is the lo lime 24 h sample, which shows an unexpected increase in TCI alongside a lower LOI. This deviation is likely attributable to sample inhomogeneity or local treatment effects. Nevertheless, the value was retained in the dataset but marked as a possible outlier.

To evaluate hemicellulose removal, the peak area around $1725\text{--}1735\text{ cm}^{-1}$ was quantified. A marked decrease is observed after NaOH treatment, confirming effective hemicellulose removal. Lime treatment also reduced the peak area but to a lesser degree, while sodium bicarbonate treatment produced little to no reduction within the tested soaking times. This limited effect is in line with the lower alkalinity of NaHCO_3 , which is insufficient for rapid removal of amorphous components. Fiore et al. (2016) reported that effective hemicellulose removal with sodium bicarbonate requires soaking times of up to five days. Since the present study only tested up to 24 h, the relatively minor changes in peak intensities are consistent with expectations.

Overall, the FTIR analysis demonstrates that the most pronounced tensile strength increases cannot be explained by hemicellulose and lignin removal alone. Instead, they correlate more strongly with cellulose I into II transformation (TCI/LOI changes and 893 cm^{-1} band intensification). This explains why sodium bicarbonate treatment, despite its limited removal of hemicellulose and lignin, still yields the highest tensile strength: the strengthening effect is primarily driven by cellulose transformation and fiber densification, rather than by extensive elimination of amorphous components.

It should also be acknowledged that differences between local and imported PALF may partly stem from their natural origin, as climatic and soil conditions influence fiber morphology and chemical composition (Komuraiah et al., 2014; Karimah et al., 2021). Such inherent variability could contribute to differences observed in treatment response.

The wavelength sections characteristic for cellulose II revealed the high tensile strength results for *s.b. 24 h* could be caused by a pronounced cellulose transformation increasing crystallinity. Compared to the other treatment conditions, the indication of cellulose II content of *s.b. 24 h* samples are not as superior as tensile results. Moreover, the FTIR spectra indicate that treatment conditions with second highest tensile strength results (*im lime 24 h* and *lo lime 1 h*) are also characterized by strong cellulose II formation. Hence, (except for *im lime 24 h*) the FTIR analysis suggests that the most distinct tensile strength increases can rather be explained by cellulose transformation than by removal of cementing material.

3.4. SEM analysis

The main goal of the SEM analysis is to reveal how the treatments removed impurities from the surface as well as verified whether the lime treatment of the PALF led to the formation of calcium nodules on the fiber surface (observed by previous studies undertaking lime treatments of natural fibers (Sedan et al., 2008; Ramasawmy et al., 2021)). These nodules can have a reinforcing effect, potentially improving the tensile strength of the fibers (Sedan et al., 2008). Furthermore, it is of interest to compare the surface morphologies of treated and untreated fibers, since increased surface roughness is important for the manufacturing of natural fiber reinforced composites, as it can enhance matrix bonding between the fiber surface and the polymer (Zwawi, 2021). As can be seen from the SEM images obtained, the anticipated calcium nodule reinforcing structure is present to a modest extent in the lime-treated local PALF sample compared to the imported PALF sample (see Figs. 6 and 7). Fig. 7d shows distinct ridges and nodules on the surface, which are absent in Fig. 7a. This suggests that extended soaking periods not only effectively remove significant surface impurities, such as wax — thereby exposing the fiber wall structure — but also lead to an increased presence of calcium nodules, as observed in the SEM images and consistent with (Ramasawmy et al., 2021). While impurity removal is notable for both imported and local fibers, the observed increase in tensile strength in lime 8 h aligns with the SEM findings. However, the lower tensile strength of local lime-treated fibers at a 24h-soaking time compared to lo lime 1 h remains unexplained, as no specific difference in surface morphology could be identified.

Table 1

Determined total crystallinity index (TCI), lateral order index (LOI) and peak area of wavelength for hemicellulose in different treatment conditions on local PALF.

Treatment Condition	TCI (I_{1372} / I_{2900})	LOI (I_{1420} / I_{893})	Peak area at $\sim 1730\text{ cm}^{-1}$
lo untreated	1.045	3.123	0.553
lo lime 1 h	0.942	4.574	0.489
lo lime 2 h	0.940	4.367	0.336
lo lime 4 h	0.924	4.861	0.342
lo lime 8 h	0.893	5.806	0.332
lo lime 24 h	1.083	1.986	0.340
lo NaOH 8 h	0.878	5.633	0.199
lo NaOH 24 h	0.849	5.696	0.288
lo s.b. 8 h	0.949	5.535	0.530
lo s.b. 24 h	0.999	4.665	0.512

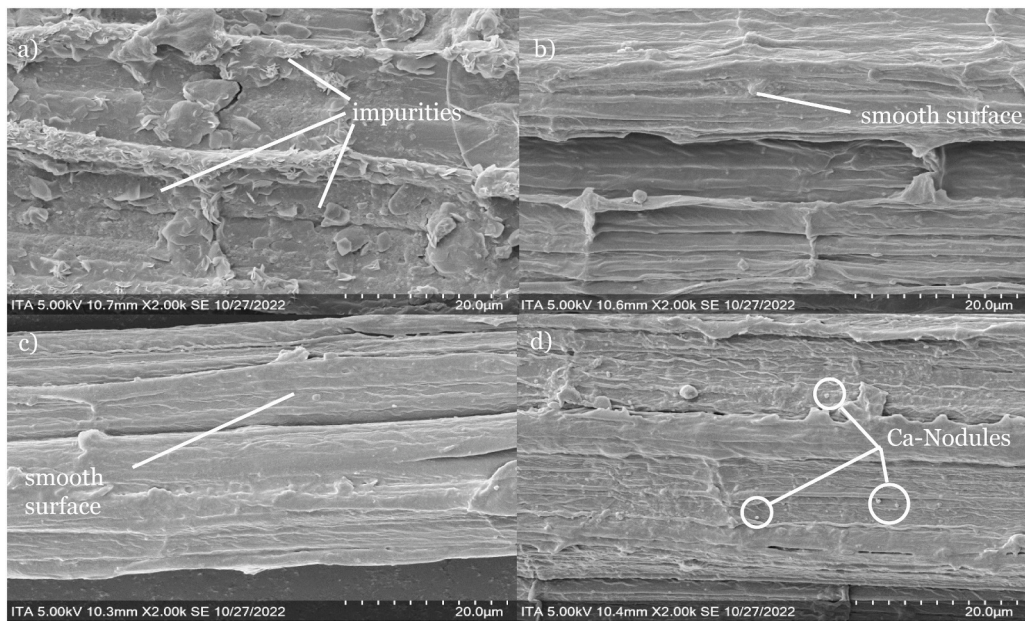


Fig. 6. SEM images at 2000 × magnification of local PALF: a) *untreated fiber* showing surface impurities, b) *lime-treated (1 h)* fiber with a relatively smooth surface, c) *lime-treated (4 h)* fiber with smooth surface morphology, and d) *lime-treated (8 h)* fiber displaying some calcium nodules.

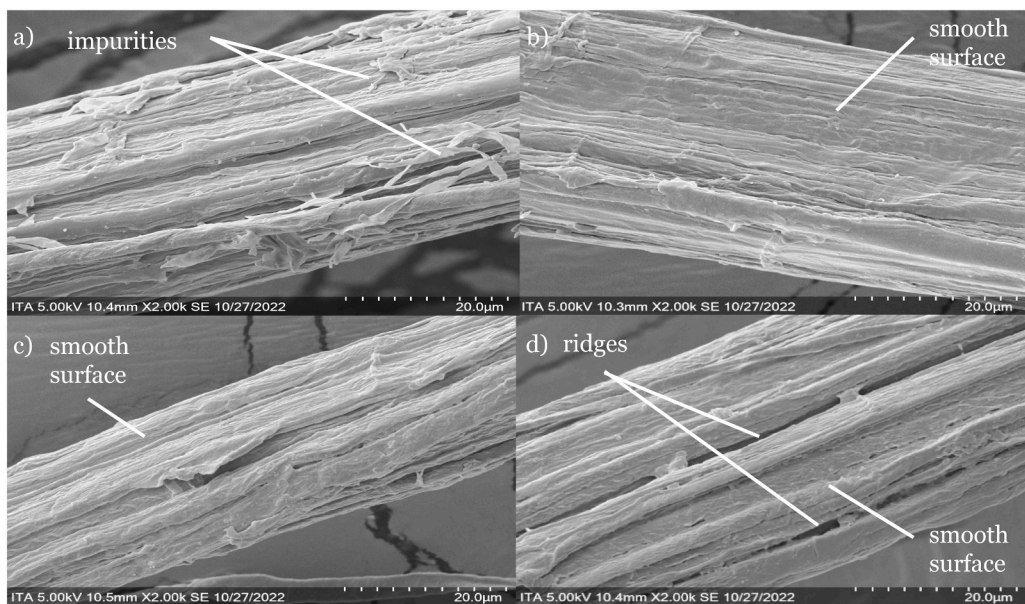


Fig. 7. SEM images at 2000 × magnification of imported PALF: a) *untreated fiber* with surface impurities, b) *lime 1 h* with smooth surface, c) *lime 4 h* with smooth surface, d) *lime 8 h* showing ridges and smooth surface morphology.

4. Conclusions

This study developed and tested two environmentally friendly chemical treatments — sodium bicarbonate and lime — for improving the tensile strength of pineapple leaf fibers (PALF). Sodium bicarbonate treatment (24 h) produced the highest tensile strength increase (over 75 % for both fiber types), while lime treatment achieved about 50 %, both outperforming the conventional sodium hydroxide (NaOH) mercerization. Remarkably, the substantial strengthening from sodium bicarbonate treatment did not coincide with major removal of hemicellulose or lignin, as indicated by FTIR spectra. Instead, the observed mass reduction (>20 %) and diameter reduction (-17 % for local fibers) indicate that fiber densification and structural reorganization, rather than extensive

chemical modification, are primarily responsible for the improved mechanical performance.

FTIR results further indicated that lime treatment removes lignin and hemicellulose similarly to NaOH, and both lime and sodium bicarbonate treatments promote cellulose II formation. SEM analysis also revealed that calcium nodule formation, potentially contributing to further reinforce the fibers, was evident in local PALF after prolonged lime soaking.

While this work focuses primarily on tensile strength, it is worth noting that the wettability of the fibers is also a key factor for their application as reinforcement in polymer composites (Soburrun and Ramasawmy, 2022, p. 255). Future studies could further enhance our understanding by exploring how these treatments affect the fibers' surface structure and compatibility with various polymer matrices (wettability, fiber-matrix adhesion, and performance in actual composite systems). These represent important limitations of the present study. Finally, differences in fiber origin (local vs. imported) must also be considered, as natural growth conditions can inherently affect PALF properties and may influence the outcome of chemical treatments.

In conclusion, this study demonstrates that sodium bicarbonate treatment (24 h) is a viable, environmentally friendly alternative to NaOH, achieving tensile strength gains of > 75 % for both PALF types. Both eco-friendly treatments not only proved to be competitive but in some cases surpass the performance of conventional alkali mercerization, highlighting their promise for advancing sustainable materials in future applications.

Abbreviations

CaO	Calcium Oxide (quick lime)
FTIR	Fourier Transform Infrared Spectroscopy
im	Imported fibers (origin Indonesia)
lo	Local fibers (origin Mauritius)
LOI	Lateral Order Index (I_{1420}/I_{1893})
MPa	Megapascal (unit of tensile strength)
NaHCO ₃	Sodium Bicarbonate
NaOH	Sodium Hydroxide (alkali mercerization)
PALF	Pineapple Leaf Fiber
SD	Standard Deviation
SEM	Scanning Electron Microscopy
SIDS	Small Island Developing States
TCI	Total Crystallinity Index (I_{1372}/I_{2900})
tex	Linear density unit (g/km)
TS	Tensile Strength
UTM	Universal Testing Machine

CRedit authorship contribution statement

Hareenanden Ramasawmy: Writing – review & editing, Supervision, Resources, Methodology, Investigation. **Thomas Gries:** Supervision, Resources, Project administration. **Annalisa Nolte:** Writing – original draft, Visualization, Validation, Investigation. **Ben Vollbrecht:** Writing – review & editing, Supervision, Resources, Project administration, Methodology, Investigation, Funding acquisition, Conceptualization.

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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Data Availability

Data will be made available on request.

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