J.Natn.Sci.Foundation Sri Lanka 2024 52 (4): 515 - 525
DOI: http://dx.doi.org/10.4038/jnsfsr.v52i4.12098

RESEARCH ARTICLE

Environmental Chemistry

Optimizing the alkaline concentration for coir fibre treatment and estimation of lifetime

LG Chamath[1,3], LKT Srimal[2,4*], GA Sewvandi[3], R Gallage[2] and J Epaarachchi[5,6]

[1] *Division of Mechanical Engineering Technology, Institute of Technology, University of Moratuwa, Diyagama, Homagama, Sri Lanka.*
[2] *Department of Mechanical and Manufacturing Engineering, Faculty of Engineering, University of Ruhuna, Hapugala, Galle, Sri Lanka.*
[3] *Department of Materials Science and Engineering, Faculty of Engineering, University of Moratuwa, Moratuwa, Sri Lanka.*
[4] *Department of Mechanical Engineering, Faculty of Engineering, South Eastern University of Sri Lanka, Oluvil, Sri Lanka.*
[5] *Centre for Future Materials, Institute for Advanced Engineering and Space Sciences, University of Southern Queensland, Toowoomba, QLD, Australia.*
[6] *School of Engineering, Faculty of Health Engineering and Sciences, University of Southern Queensland, Toowoomba, QLD, Australia.*

Submitted: 18 March 2024; Revised: 13 December 2024; Accepted: 17 December 2024

Abstract: Researchers are developing sustainable insulation composite materials using lignocellulose fibres, particularly coir fibres, which have low thermal conductivity, low density, high moisture resistance, and excellent thermal stability. However, these fibres naturally contain waxy, gummy, oily substances and impurities on their surfaces, necessitating a pre-treatment process to remove these contaminants before manufacturing. Alkaline treatment is an effective method for this pre-treatment, however, selecting the appropriate concentration is critical. Furthermore, thermal decomposition of fibres may occur during composite manufacturing making it essential to assess fibre longevity. In this study, coir fibres were treated with NaOH solutions at concentrations of 0%, 2%, 4%, 6%, and 8%. The effects of alkaline treatment on surface roughness, chemical composition, and thermal stability were analyzed using scanning electron microscopy (SEM), Fourier transform infrared spectroscopy (FTIR), and differential scanning calorimetry (DSC). The activation energy (E_a) for the thermal decomposition of lignocellulose substances was analyzed using the Flynn-Wall-Ozawa (FWO), Kissinger-Akahira-Sunose (KAS), and Friedman methods over a temperature range of 25 °C to 700 °C. The fibre lifetime was estimated using Toop's equation. The results indicated that the 4% NaOH-treated sample exhibited superior surface roughness and thermal stability compared to other concentrations. The decomposition of lignocellulose substances began at a conversion rate of 0.2, with an E_a of 128.88 kJ/mol. The estimated fibre lifetime is approximately 6.55×10^6 hours at 25 °C. The lifetime of coir fibre decreases as temperature increases. Therefore, it is crucial to highlight the necessity of controlling the temperature during the fabrication of composites using coir fibers to prevent thermal degradation.

Keywords: Activation energy, alkaline treatment, coir fibres, lifetime analysis, sustainable insulation materials.

INTRODUCTION

Building insulation materials are primarily categorised into conventional, sustainable, and state-of-art based on their chemical composition, origin, and availability (Kumar *et al.*, 2020). The demand for sustainable composite materials produced from lignocellulose fibres (natural plant fibres) has increased during the last two decades, mainly because they show the least environmental impact among the three main insulation material categories. Furthermore, they offer additional advantages such as low embodied energy, lightweight, biodegradability, low maintenance cost for machine tools due to the non-abrasive nature of fibres, and more available agricultural waste materials (Lahiru *et al.*, 2016; Hao *et al.*, 2018; Santhosh *et al.*, 2020).

* Corresponding author (srimal@mme.ruh.ac.lk; https://orcid.org/0000-0002-3285-3214)

Recently, research activities have focussed on developing sustainable insulation composite materials using lignocellulose fibres such as sugarcane, hemp, pineapple leaf fibres, cotton stalks, coir, and oil palm (Manohar, 2012; Srimal *et al.*, 2015). Among these, coir fibres have shown significant properties than other fibres, including low thermal conductivity (Sathishkumar *et al.*, 2018; Rashid *et al*., 2019; Chamath *et al.*, 2022,2023; Udayakumara *et al.*, 2022), low density (Petroudy, 2017; Hao *et al.*, 2018), high moisture resistance (Ramamoorthy *et al.*, 2015; Rashid *et al.*, 2019), and high thermal stability (Alwani *et al.*, 2014). Hence, coir fibres can be utilized to produce sustainable insulation materials for building insulation.

The main components of coir fibre are cellulose (32%-43%), hemicellulose (0.15%-0.25%), and lignin (40%-45%) (Stokke *et al.*, 2013; Hao *et al.*, 2018; Takagi, 2019). Besides, these fibres contain waxy and oily substances on their surface under natural conditions. These lignocellulose substances help to absorb environmental moistures in natural conditions, causing the fibre to swell throughout the composite (Hao *et al.*, 2018). This swelling reduces the bonding strength between the fibre and the matrix (Hao *et al.*, 2018; Chamath *et al.*, 2020), altering the dimensional stability, mechanical properties, and thermal properties of the composite (Azwa *et al.*, 2013; Petroudy, 2017). It also increases the biodegradability of the fibre due to high microbial attacks in wet conditions (Azwa *et al.*, 2013). The hemicellulose content in the polysaccharide chain mainly contributes to moisture absorption (Petroudy, 2017), while the amorphous region in the cellulose increases the fibres' moisture absorption. This is because the hydroxyl groups in these substances form hydrogen bonds with moisture. However, lignin has the lowest moisture absorption rate than other lignocellulosic substances due to crosslinks and aromatic rings in the molecules (Stokke *et al.*, 2013).

Thermal degradation can occur during processing of composites and prolonged usage. Therefore, examining the thermal stability of lignocellulose fibre is vital to understand and estimate their composite properties (Alwani *et al.*, 2014). There are several steps in the thermal decomposition of lignocellulose fibres. The first stage of decomposition occurs at a temperature range of 50 °C to 150 °C for the evaporation of mechanically bonded water and decomposition of low molecular extractives (Poletto *et al.*, 2015; Shilpa *et al.*, 2015;). The decomposition of hemicellulose can be identified as the second stage at a temperature range of 200 °C to 350 °C. The third stage of decomposition occurs at the temperature range of 320 °C to 400 °C for cellulose. Finally, the decomposition of lignin is identified as the fourth step of the degradation process (Poletto *et al.*, 2015). However, lignin decomposition can happen in the temperature range of 100 °C to 900 °C due to aromatic rings with various branches and various functions of chemical bonds that affect the high degree of decomposition (Alwani *et al.*, 2014; Poletto *et al.*, 2015).

The moisture absorption resistance and thermal stability can be increased by removing hemicellulose and low molecular extractives. Additionally, removing the surface impurities from the fibre increases the adhesion between the fibre and the matrix due to the improved surface roughness of the fibre (Adeniyi *et al.*, 2019; Chamath *et al.*, 2020). Therefore, it is essential to remove impurities and functional groups from the fibre surface using a pre-treatment process before fabrication (Azwa *et al.*, 2013; Mittal *et al.*, 2018). Bulk treatment or chemical treatment is the most suitable method for fibre treatment (Azwa *et al.*, 2013), which removes the functional groups and increases the fibre surface roughness (Adeniyi *et al.*, 2019).

These chemical treatments can be performed using acid, oxidation, alkaline, and coupling agents modification (such as silane and maleated polypropylene) (Mittal *et al.*, 2018; Adeniyi *et al.*, 2019; Mishra *et al.*, 2020). Alkaline treatment is the most common chemical treatment method used for lignocellulose fibres (Kabir *et al.*, 2013; Shrivastava *et al.*, 2017; Mittal *et al.*, 2018; Adeniyi *et al.*, 2019; Devnani & Sinha, 2019; Chamath *et al.*, 2020). This method partially eliminates hemicellulose, lignin, pectin, waxes, and other contaminants from the fibre surface and is an inexpensive process (Kabir *et al.*, 2013; Mittal *et al.*, 2018). Potassium hydroxide (KOH) and sodium hydroxide (NaOH) are popular substances used for the alkaline treatment (Azwa *et al.*, 2013), with the latter being preferred by many researchers (Kabir *et al.*, 2013; Shrivastava *et al.*, 2017; Mittal *et al.*, 2018; Adeniyi *et al.*, 2019; Devnani & Sinha, 2019).

Impure substances and functional groups are sensitive to react with the alkaline solution, and the following reaction occurs during the treatment process, as shown in Equation 1 (Kabir *et al.*, 2013; Abdellaoui *et al.*, 2019):

$$\text{Fibre-OH} + \text{NaOH} \rightarrow \text{Fibre O–Na}^{+} + \text{H}_2\text{O} + \text{impurities} \quad ...(1)$$

Equation 1 clearly shows that the amorphous region of the fibre can be easily removed from the alkaline solution

by producing Fibre O–Na+. Thereby, treated fibres contain more cellulose and higher surface roughness, which provides better adhesion between the fibre and the matrix (Kabir *et al.*, 2013; Sathishkumar *et al.*, 2018). The increased surface roughness of the fibre could be observed through scanning electron microscopy (SEM), and changes in functional groups' can be confirmed through Fourier transform infra-red spectroscopy (FTIR) analysis (Adeniyi *et al.*, 2019).

However, an excessive concentration of alkaline treatment can cause crack propagation and defibrillation (Mittal *et al.*, 2018). Hence, determining the appropriate concentration of the alkaline solution is crucial for fibre pre-treatment. In addition, the lifetime of the fibre is important because it can be affected by the temperature involved during composite fabrication. However, to the best of our knowledge there are no studies reported on coir fibre lifetime analysis. In this study, the optimal alkaline concentration was determined by treating fibre samples with various NaOH concentrations and the lifetime of fibres treated with the optimal NaOH concentration was also evaluated.

MATERIALS AND METHODS

Brown mature coir fibres were selected from the Minuwangoda area in Sri Lanka for this research study. The density of coir fibres was 1.15 gcm^{-3} (Chamath *et al.*, 2023).

Surface treatment for fibres

Concentrated NaOH solutions with weight to volume ratios of 2%, 4%, 6%, and 8% were prepared from 98% NaOH. The coir fibres were soaked for 24 h at room temperature in these solutions, maintaining the fibre to solution weight ratio at 1:25 (Venkatachalam *et al.*, 2015). After 24 h, the fibres were washed 5 to 6 times using deionized water until a pH value of 7 was reached. The fibres were then dried at 60 °C for 24 h until a constant weight was achieved.

Additionally, coir fibres were soaked in deionized water for 24 h, and the same drying procedure was followed to produce untreated fibres.

Analysing fibre surface properties

SEM images were taken of the treated and untreated fibres with a processing acceleration voltage of 10 kV and ×750 magnification.

FTIR was used to analyse the surface chemical composition of treated and untreated fibres. FTIR values were obtained using a Bruker FTIR instrument with a wave number range of 750 cm^{-1} to 4000 cm^{-1}.

Differential scanning calorimetry (DSC)

Differential scanning calorimetry analysis was conducted by using a TA instruments DSC for both untreated and treated fibres. The experiment was conducted with a heating temperature range of 0 °C to 400 °C and a heating rate of 10 °C/min in a nitrogen atmosphere.

Thermogravimetric analysis (TGA) and activation energy

By examining the SEM, FTIR, and DSC results, an appropriate concentration for the fibre treatment could be selected. Next, TGA was performed on five different fibre samples treated at the identified appropriate concentration using a Thermal analyser SDT 650. The samples (5 – 8 mg) were heated within the temperature range of 25 °C to 700 °C with five different heating rates of 5, 10, 12, 15, 20 °C/min (Alwani *et al.*, 2014) in an argon atmosphere.

Different kinetic parameters obtained from TGA are important to evaluate the thermal decomposition of lignocellulose fibres (Alwani *et al.*, 2014; Poletto *et al.*, 2015; Shilpa *et al.*, 2015). The activation energy (E_a) is especially important for detecting the thermal decomposition of lignocellulose fibres. The activation energy represents the minimum energy required to initiate the reaction (Yao *et al.*, 2008). The model-free method was used to determine the kinetic parameters due to its accuracy and simplicity (Alwani *et al.*, 2014; Shilpa *et al.*, 2015). Flynn-Wall-Ozawa (FWO), Kissinger-Akahira-Sunose (KAS), and Friedman (Alwani *et al.*, 2014) methods were applied to find the activation energy of the coir sample treated with the appropriate concentration.

Equation 2 gives the FWO method,

$$\ln \beta = C - 1.052\, E_a/RT \quad ...(2)$$

Where β is the heating rate in °C/min, C is a constant, E_a is the activation energy for decomposition in J mol^{-1}, R is the gas constant, and T is the temperature in K. The activation energy for decomposition at each conversion rate was determined by calculating the slope of the line that plots ln β against $1/T$ (Alwani *et al.*, 2014; Devnani & Sinha, 2019).

Equation 3 gives the KAS method:

$$\ln(\beta/T^2) = \ln[AR/(E_a x g(x))] + (1/T)(E_a/R) \quad ...(3)$$

Where x is the degree of conversion given by $x = (m_o - m_t)/(m_o - m_f)$, where m_0 is the initial sample mass, m_t is the sample mass at time t, and m_f is the final mass. The decomposition activation energy for each conversion rate was calculated by the slope of the line that plotted $\ln(\beta/T^2)$ versus *1/T* (Alwani *et al.*, 2014).

Equation 4 presents the Friedman method:

$$\ln(dx/dt) = \ln[Af(x)] - E_a/RT \quad ...(4)$$

Where *f(x)* is the reaction model. The decomposition activation energy for each conversion rate was calculated by the slope of the line that plotted $\ln(dx/dt)$ versus *1/T* (Alwani *et al.*, 2014).

Lifetime analysis

The lifetime of natural fibres can be calculated from the E_a value (Batista *et al.*, 2015; Enciso *et al.*, 2021), and the estimated lifetime depends on the activation energy of the material, as shown in Equation 5 (Toop, 1971; Batista *et al.*, 2015; Enciso *et al.*, 2021). Here, Toop's method was used to estimate the lifetime of coir fibres.

$$\log t_f = E_a/2.303RT_f + \log\left((E_a/\beta R).P(X_f)\right) \quad ...(5)$$

Where t_f is the estimated lifetime to failure (min), T_f is the failure temperature (K), and $P(X_f)$ is the function that depends on the activation energy at the failure temperature.

RESULTS AND DISCUSSION

SEM image analysis for untreated and treated fibre surface

SEM images in Figure 1 show a comparison between untreated and treated coir fibres. According to Figure 1a), untreated fibre shows a smooth surface with lignin, hemicellulose, wax, and oily substances (Mittal *et al.*, 2018; Devnani & Sinha, 2019). However, in Figure 1b), the fibre surface roughness of the 2% alkaline treated fibre is increased after the alkaline treatment, with frequently distributed pinholes throughout the fibre surface due to partial removal of lignocellulose substances and impurities. Further, the surface roughness of the 4% alkaline treated fibre in Figure 1c) is higher than in Figure 1b) indicating surface roughness can be increased with alkaline concentrations, which increases the number of pinholes on the fibre surface.

In Figure 1d) the plane view of 6% alkaline treated coir fibre shows vertically distributed cracks on the fibre surface suggesting that high concentrations of alkaline treatment can damage the fibre surface by removing excessive amounts of lignocellulosic substances from the fibre's cell structure. Moreover, in Figure 1e) of the 8% alkaline treated fibre, surface cracks have propagated in vertical and horizontal directions.

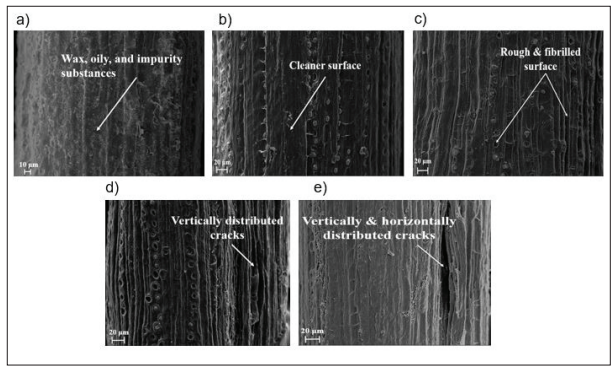

Figure 1: Plane view of coir fibres (×750), a) untreated fibre, b) 2% NaOH treated fibre, c) 4% NaOH treated fibre, d) 6% NaOH treated fibre, e) 8% NaOH treated fibre.

The results above suggest that alkaline treatment can increase the surface roughness of coir fibre, but excessive concentrations can damage the fibre cell structure, causing crack propagation on the fibre surface.

Similar observations were reported for pineapple leaf fibres, coir fibres and kane grass fibres treated with NaOH (Mittal *et al.*, 2018, Devnani & Sinha, 2019). Therefore, it is particularly important to identify the appropriate concentration for fibre treatment.

FTIR spectrum analysis for treated and untreated coir fibres

The FTIR analysis helps to identify various types of functional groups in the coir fibres. Figure 2 shows the FTIR spectrum for untreated and treated coir fibres. The functional groups of carbonyl (-CO), hydroxyl (-OH), carboxyl (-COOH), and methylene ($-CH_2$) in lignocellulosic substances on the fibre surface were identified from this FTIR spectrum (Adeniyi *et al*., 2019; Mishra *et al.*, 2020).

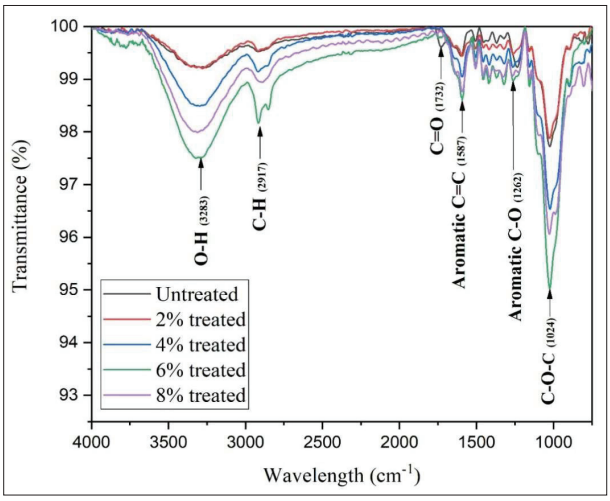

Figure 2: FTIR spectrum for untreated and treated coir fibres

The highest peak at 1024 cm^{-1} is associated with C-O-C stretching of cellulose. The wavelengths 1262 cm^{-1} and 1587 cm^{-1} are associated with aromatic C-O stretching and aromatic C=C stretching in lignin, respectively (Mishra *et al.*, 2020). The C=O stretching of ester and aldehyde is given by lignin, hemicellulose, and pectin with a peak of 1732 cm^{-1} (Mittal *et al.*, 2018). Also, the wavelength 2917 cm^{-1} is related to the C-H stretching vibration in CH_2 and CH groups in hemicellulose and cellulose (Devnani & Sinha, 2019), while the broad peak between the wavelength 3200 cm^{-1} to 3550 cm^{-1} is related to the stretching vibration of OH groups of lignin and cellulose (Mishra *et al.*, 2020).

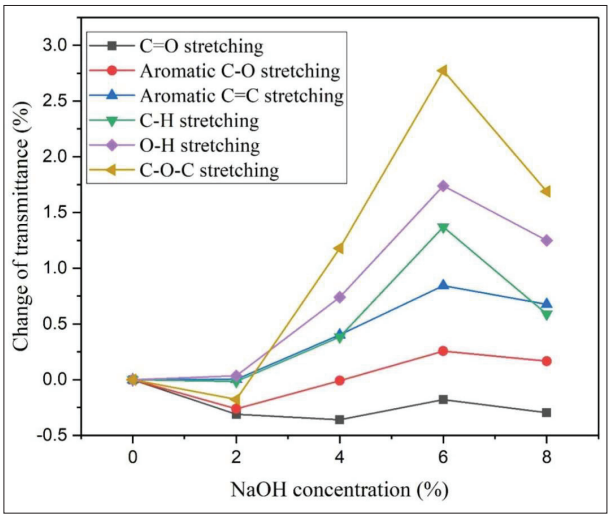

Figure 3: Comparison of the changes of the transmittance for the main peaks.

Figure 3 compares the changes in transmittance for the main peaks of the treated fibres compared to the untreated fibre.

After the treatment process, the peaks at C=O stretching (1732 cm^{-1}) were reduced than in the untreated fibre. This could be due to the removal of C=O functional groups from pectin, hemicellulose, and lignin on the fibre surface. The peak intensity was reduced to 4% in the treated sample, and the peak intensity of the 6% sample being higher than other treated fibres. This increase may be due to the exposure of internal functional groups of the fibre cell after crack propagation. Subsequently, the peak intensity of the 8% sample was less than the 6% treated fibre due to the removal of excess functional groups. This result confirms that a 4% alkaline treated sample contains the minimum amount of hemicellulose, lignin, and pectin on its surface.

After the treatment, the peak intensity at 1262 cm^{-1}, and 1587 cm^{-1} increased in the 6% alkaline treated fibre for aromatic C-O and aromatic C=C functional groups of lignin, which shows the highest intensity. This increase may be due to the high accessibility of infrared energy for removing wax, pectin, and gummy substances from the fibre surface during crack propagation. However, the peak intensity was reduced after the 6% treatment due to the removal of excess functional groups. The same trend was observed for the peak intensity of stretching functional groups of C-H, O-H, and C-O-C.

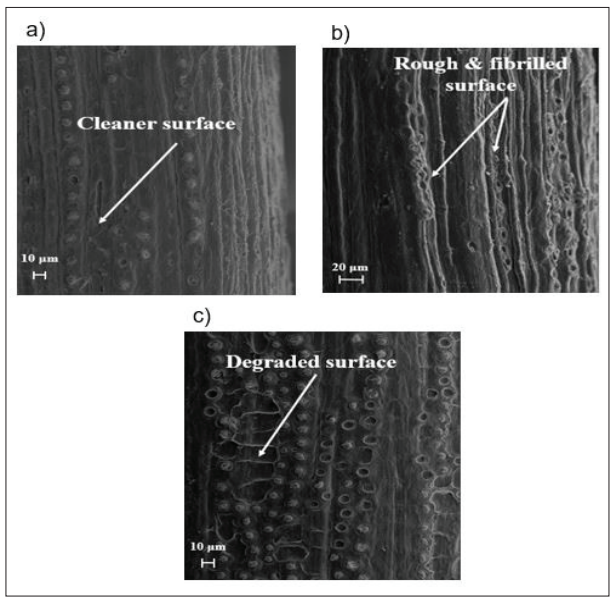

Figure 4: SEM images of coir fibres treated with NaOH (×750). a) 3%, b) 4%, c) 5%

According to the SEM analysis in Figure 1, fibre surface roughness can be increased with higher alkaline concentration. However, excess concentration can damage the fibre surface through crack propagation. The FTIR analysis confirmed the removal of surface impurities. Moreover, excessive concentration can result in the removal of lignin and cellulose from the fibre surface making it necessary to identify the appropriate concentrations for fibre treatment. Based on the above results, an alkaline concentration of 4% can be considered as the most effective for fibre treatment. However, further comparison of the effects of 3%, 4%, and 5% is necessary to select the optimal solution. SEM images of 3%, 4%, and 5% for comparing surface properties are shown in Figure 4.

The surface roughness of the 4% NaOH treated fibre (Figure 4b) is higher than that of the 3% NaOH treated fibre (Figure 4a). The fibre shown in Figure 4c for the 5% NaOH treated fibre has begun to degrade. Therefore, the 4% NaOH treatment shows better surface properties from the SEM analysis. The previous SEM analysis results and FTIR analysis indicate that the 4% NaOH concentration is the best for fibre treatment.

DSC analysis of coir fibres

The location and magnitude of the exothermic and endothermic peaks on the DSC curve show the thermal phase transformation of the material during heating. Endothermic peaks provide information on sample phase transition, evaporation, dehydration, melting, and pyrolysis (Azwa *et al.*, 2013; Ali & Alabdulkarem, 2017). Exothermic peaks offer insights into chemical reactions, oxidation, crystallisation, decomposition, and combustion (Kabir *et al.*, 2013).

According to the results of SEM and FTIR analysis, the 8% NaOH treated sample shows a higher degradation on the fibre surface. Therefore, both the untreated and the treated samples were subjected to thermal analysis using NaOH concentrations of 2%, 4%, and 6% by DSC analysis. The results of the DSC analysis are presented in Figure 5.

Both, untreated and treated samples gave an endothermic peak between 5 °C and 170 °C. This peak is attributed to the evaporation of moisture from the fibre surface (Kabir *et al.*, 2013). The untreated fibre had the lowest moisture evaporation temperature indicating it contained the maximum moisture content.

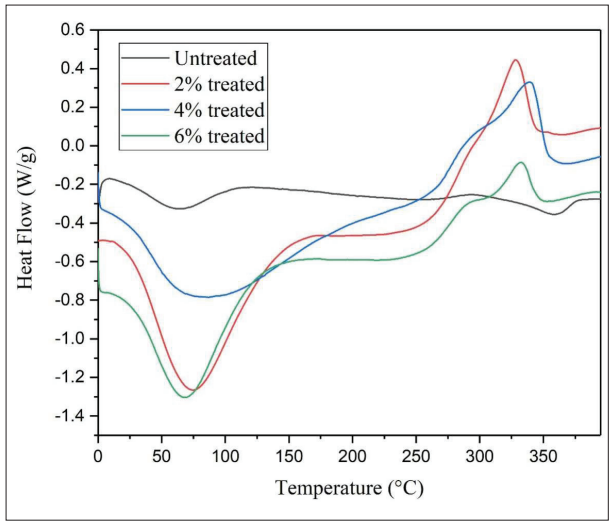

Figure 5: Differential scanning calorimetry (DSC) analysis for untreated and treated coir fibres

Additionally, both untreated and treated samples displayed an exothermic peak in the temperature range of 260 °C to 320 °C related with the degradation of hemicellulose (Kabir *et al.*, 2013). However, the decomposition temperature (272 °C) of hemicellulose in treated samples was higher than that in the untreated sample. The decomposition temperature for the 4% NaOH treated sample shows the maximum value.

Finally, untreated fibres show an endothermic peak in the temperature range of 296 °C to 390 °C, whereas the treated fibre shows exothermic peaks in the region of 300 °C to 360 °C compared to the untreated fibre. These peaks are related to the degradation of cellulose and lignin substances (Kabir *et al.*, 2013). The untreated fibre contains high amounts of lignin and cellulose. This leads to high energy absorbance required to degrade the fibre, which causes an endothermic peak during the decomposition. However, the 4% NaOH treated sample shows the highest temperature (317 °C) for the third stage of decomposition. Therefore, based on the DSC analysis, the 4% NaOH treated sample shows the highest thermal stability due to the removal of impurities and hemicellulose from the fibre surface.

The decomposition of the fibres followed the first, second, and third decomposition stages, as summarised in Table 1.

Table 1: Thermal analysis of untreated and treated fibres

Fibre treatment	1st stage Moisture evaporation temperature (°C)	2nd stage Hemicellulose and lignin decomposition temperature (°C)	3rd stage Cellulose decomposition temperature (°C)
Untreated fibre	17.91	264.43	305.47
2% NaOH treated	25.55	271.76	306.21
4% NaOH treated	20.06	272.73	317.07
6% NaOH treated	24.43	265.91	313.52

The sample treated with 4% alkaline solution shows improved surface roughness, chemical composition, and thermal stability according to SEM, FTIR, and DSC analysis. Further, it is recommended to investigate the activation energy and lifetime of the fibre.

Thermal gravimetric analysis and activation energy

Figure 6 shows the TGA and differential thermal gravimetric (DTG) curves, obtained at a heating rate of 5 °C/min for the 4% NaOH treated fibre. The two main steps of decomposition could be identified as the evaporation of water in the temperature range of 25 °C to 150 °C, and the second stage that follows is the decomposition of lignocellulose substances in the temperature range of 175 °C to 430 °C (Azwa *et al.*, 2013; Alwani *et al.*, 2014).

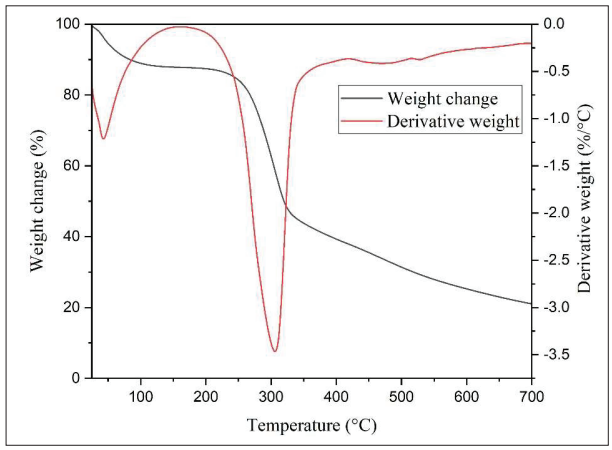

Figure 6: Thermal gravimetric (TG) and differential thermal gravimetric (DTG) curves for the 4% NaOH treated fibre at a heating rate of 5 °C/min

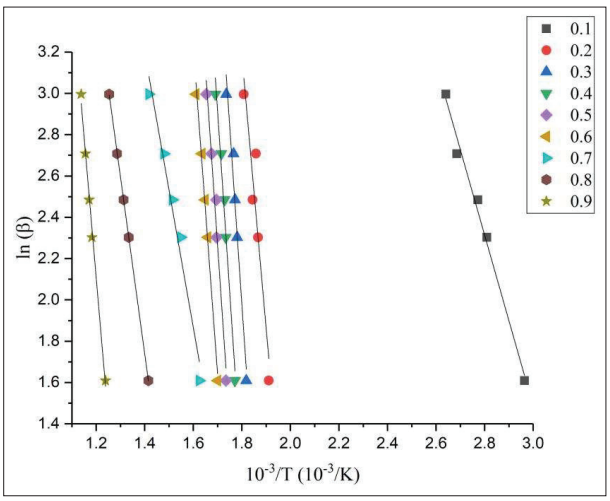

Figure 7: Linear plot of FWO method

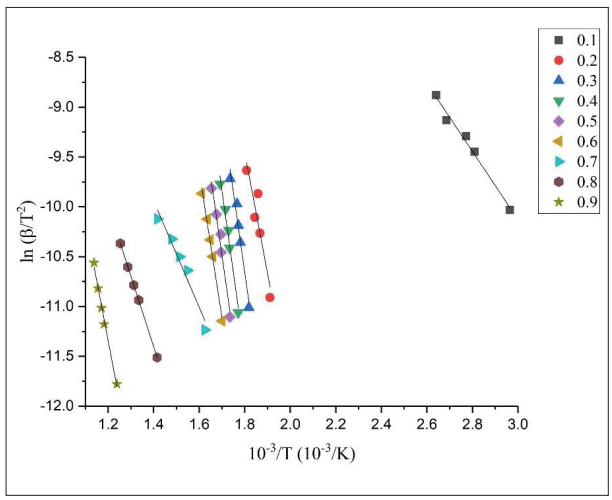

Figure 8: Linear plots of KAS method

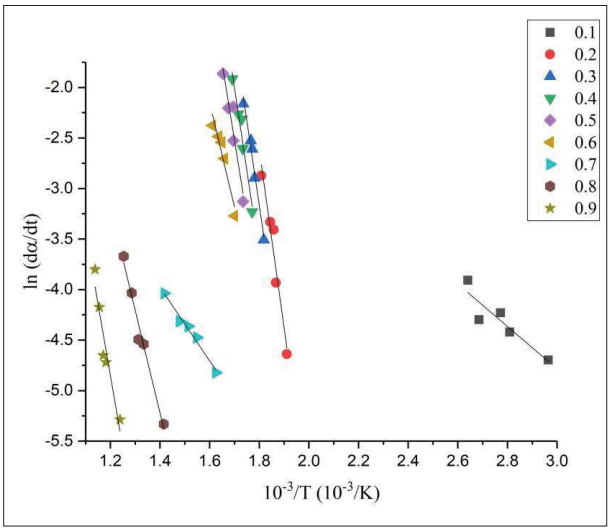

Figure 9: Linear plots of Friedman method

Next, the kinetic study was conducted with different heating rates of 5 °C/min, 10 °C/min, 12 °C/min, 15 °C/min, and 20 °C/min to further investigate the thermal decomposition of the 4% NaOH treated fibre sample. The FWO, KAS, and Friedman methods were employed to determine the activation energy. Figures. 7, 8, and 9 show the plots for the FWO method, KAS method, and Friedman method, respectively.

The conversion rate of 0.1 provides the activation energy for the first stage decomposition of moisture evaporation. The lines between the conversion rates of 0.2 to 0.6 are parallel to each other in the above three methods, which implies the possibility of a single reaction mechanism occurring at that conversion rate. The second stage of decomposition begins at the conversion rate of 0.2 (Alwani *et al.*, 2014). However, the reaction mechanism changes after the 0.6 conversion rate due to the complex reactions of the fibre constituents at high temperatures (Alwani *et al.*, 2014).

Table 2: Activation energies (kJ/mol) calculated from the three methods for conversion rates of 0.1-0.9

Conversion rate	FWO method		Kissinger method		Friedman method	
	Ea	R^2	Ea	R^2	Ea	R^2
0.1	34.39	0.9901	28.45	0.9852	17.45	0.8420
0.2	109.67	0.8916	100.74	0.8738	146.22	0.9490
0.3	143.22	0.9708	133.86	0.9666	138.54	0.9791
0.4	145.95	0.9811	136.35	0.9783	136.34	0.9612
0.5	143.08	0.9730	133.27	0.9689	126.81	0.9088
0.6	131.69	0.9933	121.64	0.9921	85.89	0.9256
0.7	55.31	0.9743	44.37	0.9986	30.28	0.9830
0.8	71.32	1.0000	58.88	1.0000	84.45	0.9794
0.9	113.65	0.9952	99.68	0.9940	118.41	0.9179

Table 2 summarises the activation energies obtained from the above three methods for the conversion rates ranging from 0.1 to 0.9.

The activation energy for decomposition is changing from 109.67 to 145.95 kJ/mol, 100.74 to 136.35 kJ/mol, and 85.89 to 146.22 kJ/mol in the FWO method, KAS method, and Friedman method, respectively, at a conversion rate of 0.2 to 0.6. The average activation energy (134.72 kJ/mol) obtained from the KAS method was higher than the other methods at a conversion rate of 0.2 to 0.6. The same observation can also be found in Alwani's work, where the activation energy was 200 kJ/mol, obtained from the Kissinger method (Alwani *et al.*, 2014).

Lifetime analysis for the 4% NaOH treated fibre

The main decomposition begins at the 0.2 conversion rate. Therefore, it is better to determine the lifetime of the 4% alkaline treated coir fibre at 25 °C. The average activation energy obtained from the three methods is E=128.88 kJ/mol at the 0.2 conversion rate. The slowest heating rate of 5 °C/min, was selected for this study (Bogdanov *et al.*, 2014). The temperature was T_c = 500

K at this heating rate and the conversion rate 0.2. Then the E/RT value was calculated and utilized to find the log P(Xf) from the numerical integration table given in the reference (Toop, 1971). The antilog value of P(Xf) was calculated next, and the lifetime of the fibre was calculated using Toop's equation (Toop, 1971; Bogdanov *et al.*, 2014).

Figure 10 shows the estimated lifetime versus failure temperature for the 4% alkaline treated coir fibres obtained by using Toop's method. The lifetime value of the 4% treated coir fibre sample was 6.55×10^6 hours (749 years) at 25 °C. However, the estimated lifetime was determined based on the activation energy value taken from the decomposition curves obtained in an inert atmosphere, without considering mechanical loading and weather conditions (Batista *et al.*, 2015). Nevertheless, the above results indicate that the 4% alkaline treated coir fibres have an improved thermal stability at room temperature.

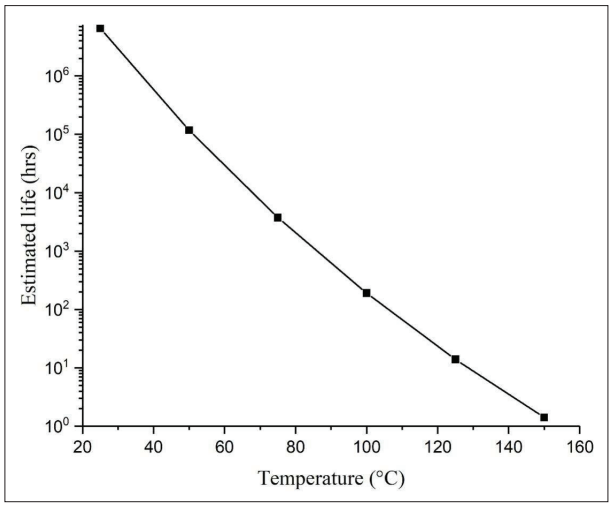

Figure 10: The estimated lifetime of the 4% NaOH treated coir fibres

CONCLUSION

The alkaline treatment effectively removes lignocellulose substances, oil, and wax from the surface of untreated coir fibres. This process alters surface roughness, the chemical composition and thermal stability of the fibres. Surface roughness changes can be observed through SEM analysis showing an increase with higher alkaline solution concentrations. However, excessive alkaline concentration can damage the fibre surface. FTIR analysis confirmed the changes in the chemical composition of the fibre surface including significant reductions in hemicellulose, impurities, and partial removal of lignin with an insignificant removal of cellulose.

The removal of lignocellulose substances from the fibre surface increased with higher alkaline concentrations. However, excessive concentrations caused surface damage particularly above 4% NaOH indicating the delicate balance required in the alkaline treatment processes. Additionally, improved thermal stability of the fibres is seen with alkaline concentrations up to 4% NaOH, but further increase resulted in reduced thermal stability. These findings emphasize the need for careful optimization of alkaline treatment to enhance fibre properties. Therefore, according to the above results, the 4% alkaline treatment was identified as the appropriate concentration.

Model-free methods such as FWO, KAS, and Friedman methods provided information on the activation energy of the 4% treated fibre, providing insight into the minimum energy requirement of the first and second stages of fibre decomposition. The first stage decomposition occurs at the 0.1 conversion rate and the second stage decomposition occurs at the 0.2 to 0.6 conversion rate. Lifetime analysis of the fibre at 0.2 is important before the fabrication process with the 4% treated fibre showing a lifetime value of 6.55×10^6 hours at 25 °C, which reduces with increasing temperature. Therefore, it is especially important to maintain the fabrication temperature and the time to avoid fibre degradation. Further analysis of lifetime considering biological and chemical degradation factors is recommended.

Acknowledgement

The Division of Polymer and Chemical Engineering, Institute of Technology University of Moratuwa and the Instrument Centre of the Faculty of Applied Sciences, University of Sri Jayewardenepura facilitated the instrumental analysis for this work.

REFERENCES

Abdellaoui, H., Raji, M., Essabir, H., & Bouhfid, R. (2019). Mechanical behavior of carbon/natural fiber-based hybrid composites. In *Mechanical and physical testing of biocomposites, fibre-reinforced composites and hybrid composites*, (pp. 103-122). Woodhead Publishing. https://

doi.org/10.1016/B978-0-08-102292-4.00006-0

Adeniyi, A. G., Onifade, D. V., Ighalo, J. O., & Adeoye, A. S. (2019). A review of coir fiber reinforced polymer composites. *Composites Part B: Engineering, 176,* 107305. https://doi.org/10.1016/j.compositesb.2019.107305

Ali, M. E., & Alabdulkarem, A. (2017). On thermal characteristics and microstructure of a new insulation material extracted from date palm trees surface fibers. *Construction and Building Materials, 138,* 276-284. https://doi.org/10.1016/j.conbuildmat.2017.02.012

Alwani, M. S., Khalil, H. A., Sulaiman, O., Islam, M. N., & Dungani, R. (2014). An approach to using agricultural waste fibres in biocomposites application: Thermogravimetric analysis and activation energy study. *BioResources, 9*(1), 218-230. https://doi.org/10.15376/biores.9.1.218-230

Azwa, Z. N., Yousif, B. F., Manalo, A. C., & Karunasena, W. (2013). A review on the degradability of polymeric composites based on natural fibres. *Materials and Design, 47,* 424-442. https://doi.org/10.1016/j.matdes.2012.11.025

Batista, N. L., Costa, M. L., Iha, K., & Botelho, E. C. (2015). Thermal degradation and lifetime estimation of poly(ether imide)/carbon fiber composites. *Journal of Thermoplastic Composite Materials, 28*(2), 265-274. https://doi.org/10.1177/0892705713484740

Bogdanov, D. A., Subbotin, E. V., Ershov, S. V., & Kazakov, A. V. (2014). Estimation of polymer lifetime by TGA decomposition kinetics. *PNRPU Bulletin: Electrotechnics, Informational Technologies, Control Systems, 1*, 57-65.

Chamath, L. G., Srimal, L. K. T., & Sewvandi, G. A. (2020). Effect of alkaline concentration on the surface properties of coir fibers. *Proceeding of the National Engineering Research Symposium, 1*, 10. https://doi.org/10.13140/RG.2.2.36167.37283

Chamath, L. G., Srimal, L. K. T., & Sewvandi, G. A. (2022). Evaluating the thermal conductivity of three-phase insulation composite using analytical and numerical methods. *Proceedings of Moratuwa Engineering Research Conference Sri Lanka*.

Chamath, L. G., Srimal, L. K. T., & Sewvandi, G. A. (2023). Assessment of transverse thermal conductivity of coir fibre using experimental, analytical, and numerical methods. *Journal of the National Science Foundation of Sri Lanka, 51*(1). http://dx.doi.org/10.4038/jnsfsr.v51i1.10870

Devnani, G. L., & Sinha, S. (2019). Extraction, characterization and thermal degradation kinetics with activation energy of untreated and alkali treated *Saccharum spontaneum* (Kans grass) fiber. *Composites Part B: Engineering, 166,* 436-445. https://doi.org/10.1016/j.compositesb.2019.02.042

Enciso, B., Abenojar, J., Aparicio, G. M., & Martínez, M. A. (2021). Decomposition kinetics and lifetime estimation of natural fiber reinforced composites: Influence of plasma treatment and fiber type. *Journal of Industrial Textiles, 51*(4), 594-610. https://doi.org/10.1177/1528083719886046

Hao, L. C., Sapuan, S. M., Hassan, M. R., & Sheltami, R. M. (2018). Natural fiber reinforced vinyl polymer composites. In *Natural fibre reinforced vinyl ester and vinyl polymer composites*, 27-70. Woodhead Publishing. https://doi.org/10.1016/b978-0-08-102160-6.00002-0

Kabir, M. M., Islam, M. M., & Wang, H. (2013). Mechanical and thermal properties of jute fibre reinforced composites. *Journal of Multifunctional Composites, 1*(1), 71-77. https://doi.org/10.12783/issn

Kumar, D., Alam, M., Zou, P. X., Sanjayan, J. G., & Memon, R. A. (2020). Comparative analysis of building insulation material properties and performance. *Renewable and Sustainable Energy Reviews, 131*. https://doi.org/10.1016/j.rser.2020.110038

Lahiru, A. M., Srimal, L. K. T., Karunasena, H. C. P., & Sumith, B. (2016). Value addition for coir fiber as an oil absorbent material. *Proceeding of the 13th Academic Sessions, Faculty of Agriculture, University of Ruhuna,* Mapalana, Sri Lanka. https://doi.org/10.13140/RG.2.2.10582.11846

Manohar, K. (2012). Experimental investigation of building thermal insulation from agricultural by-products. *British Journal of Applied Science & Technology, 2*(3), 227-239. https://doi.org/10.9734/bjast/2012/1528

Mishra, L., & Basu, G. (2020). Coconut fibre: Its structure, properties and applications. In *Handbook of natural fibres*, 231-255. Woodhead Publishing. https://doi.org/10.1016/B978-0-12-818398-4.00010-4

Mittal, M., & Chaudhary, R. (2018). Experimental study on the water absorption and surface characteristics of alkali treated pineapple leaf fibre and coconut husk fibre. *International Journal of Applied Engineering Research, 13*(15), 12237-12243. ISSN 0973-4562.

Petroudy, S. D. (2017). Physical and mechanical properties of natural fibers. In *Advanced high strength natural fibre composites in construction* (pp. 59-83). Woodhead Publishing. https://doi.org/10.1016/B978-0-08-100411-1.00003-0

Poletto, M., Júnior, H. L. O., & Zattera, A. J. (2015). Thermal decomposition of natural fibers: Kinetics and degradation mechanisms. In *Reactions and mechanisms in thermal analysis of advanced materials* (pp. 515-545). https://doi.org/10.1002/9781119117711.ch21

Ramamoorthy, S. K., Skrifvars, M., & Persson, A. (2015). A review of natural fibers used in biocomposites: Plant, animal and regenerated cellulose fibers. *Polymer Reviews, 55*(1), 107-162. https://doi.org/10.1080/15583724.2014.971124

Rashid, K., Haq, E. U., Kamran, M. S., Munir, N., Shahid, A., & Hanif, I. (2019). Experimental and finite element analysis on thermal conductivity of burnt clay bricks reinforced with fibers. *Construction and Building Materials, 221,* 190-199. https://doi.org/10.1016/j.conbuildmat.2019.06.055

Santhosh, K. S., & Hiremath, S. S. (2020). Natural fiber reinforced composites in the context of biodegradability: A review. In *Encyclopedia of Renewable and Sustainable Materials.* Elsevier Ltd. https://doi.org/10.1016/b978-0-12-803581-8.11418-3

Sathishkumar, S., Naveen, T. K., Jeevarathinam, A., Karthik, V., & Dhandapani, N. V. (2018). Thermal conductivity of natural fiber reinforced plastics. *International Journal of Pure and Applied Mathematics, 118*(20), 43-51. ISSN: 1314-3395.

Shilpa, D., & Manavendra, G. (2015). A study of thermal stability and activation energy on natural fibres for bio composite applications. *International Journal for Scientific Research & Development, 3*(9), 304-310. ISSN (online): 2321-0613.

Shrivastava, R., Telang, A., Rana, R. S., & Purohit, R. (2017). Mechanical properties of coir/glass fiber epoxy resin hybrid composite. *Materials Today: Proceedings, 4*(2), 3477-3483. https://doi.org/10.1016/j.matpr.2017.02.237

Srimal, L. K. T., Fernando, M. A. R. V., & Sumith, B. (2015). Value addition for coir fiber as thermal insulation materials (Master's thesis, University of Moratuwa). Institutional Repository University of Moratuwa. http://dl.lib.mrt.ac.lk/handle/123/10957.

Stokke, D. D., Wu, Q., & Han, G. (2013). *Introduction to wood and natural fiber composites.* John Wiley & Sons. https://doi.org/10.1002/9780470711804

Takagi, H. (2019). Review of functional properties of natural fiber-reinforced polymer composites: Thermal insulation, biodegradation and vibration damping properties. *Advanced Composite Materials, 28*(5), 525-543. *h*ttps://doi.org/10.1080/09243046.2019.1617093

Toop, D. J. (1971). Theory of life testing and use of thermogravimetric analysis to predict the thermal life of wire enamels. *IEEE Transactions on Electrical Insulation, 1*, 2-14. https://doi.org/10.1109/TEI.1971.299128

Udayakumara, D. G. G. V., De Silva, I. R., Chamath, L. G., Srimal, L. K. T., & Gallage, R. (2022). Design and fabrication of a system to measure thermal conductivity of compressible materials. *Proceeding of the 19th Academic Sessions, University of Ruhuna,* Matara, Sri Lanka. http://ir.lib.ruh.ac.lk/xmlui/handle/iruor/5788.

Venkatachalam, N., Naveen, E., & Maheswaran, N. (2015). Alkali chemical treatment on the surface of natural fiber. *International Journal of Innovative Research in Science, Engineering and Technology, 4*(4), 172-178. ISSN: 2319-8753.

Yao, F., Wu, Q., Lei, Y., Guo, W., & Xu, Y. (2008). Thermal decomposition kinetics of natural fibers: Activation energy with dynamic thermogravimetric analysis. *Polymer Degradation and Stability, 93*(1), 90-98. https://doi.org/10.1016/j.polymdegradstab.2007.10.012