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Experimental Investigation on the Thermal Behavior of Untreated and Alkali-Treated Pineapple Leaf and Coconut Husk Fibers

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ABSTRACT

The primary objective of this research work is to study the thermal behavior of untreated and alkali treated pineapple leaf (PALF) and coconut husk fibers (COIR). In this context, firstly the cellulosic fibers were treated with an alkali solution of various concentration (2%, 4%, 6%, 8%, and 10 wt%) then secondly the change in their surface chemistry and thermal stability were investigated by means of a Fourier transform infrared spectroscopy (FTIR) and Thermogravimetric analysis technique (TGA) respectively. A significant variation in the chemical composition of cellulosic fibers was observed after an alkaline treatment. Changes in the peak at 1745, 1525, and 1250 cm^{-1} in FTIR spectra corresponds to the partial removal of hemicellulose and lignin components. The thermal degradation of lignocellulosic fibers consists of two major steps i.e. moisture loss (below 150˚C) and the decomposition of hemicellulose, cellulose, and lignin components (between 150˚C to 400˚C). An alkali treatment increases the thermal stability of pineapple leaf and coir fibers through physical and chemical changes. It was observed that the 4 wt% NaOH treated PALF and COIR fibers possess higher thermal stability as compared to other treated and untreated fibers. PALF exhibits a higher rate of decomposition than the COIR fiber but its main decomposition peak lies at a higher temperature of about 40^{0}C.

Keywords: Pineapple leaf fiber, Coir fiber, Alkali treatment (NaOH), Thermal stability, Thermogravimetric analysis (TGA), Natural fiber, Infrared spectroscopy

Recently, most of the industries are looking for those materials which have environment-friendly characteristics, contribute to the lower emission of pollutants, good physical properties, and economical in nature[1-3]. To fulfill the market demands, researchers and technologists are putting their attention towards biocomposite materials. These materials generally made up of lignocellulosic biomass and polymeric resin.

Amongst all the different types of lignocellulosic materials, the coconut husk (COIR) and pineapple leaf fibers (PALF) have high potential to develop high strength biocomposite materials since they possess a large number of advantageous properties such as low cost, lightweight, easy processing, easy availability, high specific strength and stiffness, and non-abrasive nature[4-5]. The COIR and PALF can obtain from the husk of coconut fruit (*Cocos nucifera*) and leaves of pineapple fruit (*Ananas-comosus*) respectively. The inherent properties of COIR and PALF are mentioned in Table 1[6]. COIR fiber contains more amount of lignin as compared to other typically used natural fibers which result it has good resistance to salt water, microbial, and fungus attack. In comparison to COIR, the PALF is a smooth, flexible, thin, and long fiber. PALF can be employed as an electrical, thermal, and acoustic insulator; for feedstock, and biofuel production[7-10]. According to the statistical database (2016) of "Food and Agriculture Organization," the total production of coconut and pineapple fruit in the whole world is 59 and 25.8 millions of tonnes respectively (Table 2)[11]. Inspite of the above mentioned profitable properties, they exhibit some limitations like hydrophilicity, poor wettability, low thermal stability, and poor interfacial adhesion with synthetic polymers which results that all the capabilities of developed biomaterials cannot be exploited to full extent. In this regard, researchers have done a lot of work to impart hydrophobicity in natural fibers by chemical treatment process such as maleic anhydride, peroxide, permanganate, sodium hydroxide, and organosilanes etc. But very little work has been done to improve the thermal stability of lignocellulosic fibers[12-15]. Previous work[16-18] has reported that the natural fibers can be subjected to thermal degradation during the biocomposite processing. So, it is practically significant to understand and overcome the thermal decomposition of natural polymeric materials. Their thermal behavior mainly depends on the chemical composition and internal structure. The constituents of natural fibers such as hemicellulose, cellulose, lignin, pectin, and waxes are not thermally stable even at a relatively low temperature (below 400˚C). The initial decomposition of natural fibers starts at around 200˚C and it was due to the cellular breakdown of hemicellulose. The previous research work[19-21] concluded that the major decomposition of rice husk, natural hemp, and bagasse fibers occur at 351.5˚C, 312˚C, and 327˚C respectively due to the decomposition of α-cellulose.

Thermogravimetric analysis (TGA) is one of the most popular and widely used techniques to analyze the decomposition process of solid material, kinetic analysis of de-volatilization, and to study the effects of heating rate, temperature, pressure, atmosphere gas, gas flow rate, biomass composition, and particles size on mass loss of a sample[22]. Yao Fei *et al.*[23] have studied the decomposition kinetics of 10 types of fibers and found that the activation energy of Bagasse, Bamboo, Cotton Silk, Hemp, Jute, Kenaf, Rice husk, Rice straw, Wood maple, and Wood pine were 161.1, 161,6, 146, 171.1, 165.6, 157.7, 167.4, 176.2, 153.7, 159.3 respectively. The thermal decomposition of natural fibers consists of two to three steps based on their type and chemical composition. Sergio *et al.*[24] stated that the thermal decomposition of NFRC consists of moisture evaporation, hemicellulose degradation, and decomposition of α-cellulose, and lignin. A. Rachini *et al.*[25] reported the order of thermal stability of hemp fiber: NaOH treated fibers> silane-treated fibers> solvent extracted fibers (water/ethanol mixture, 20/80 v/v)> untreated hemp fibers. M. Tajvidi *et al.*[26] studied the thermal degradation of wood flour, kenaf fiber, newsprint, and rice hulls - polypropylene composites. Na Lu *et al.*[27] reported that the hemp fiber was decomposed in two steps, starting with the loss of moisture around 100˚C and followed by successive decomposition of hemicellulose, cellulose, and lignin in the range of 150-400˚C. Jayamani Elammaran *et al.*[28] reported that the thermal decomposition of Betel nut-Polyester composite was shifted to a higher temperature after an alkaline treatment of betel fibers. Alkaline treatment is one of the commonly used cost-effective

techniques to develop high-performance biocomposite material. The previous research work reported in Table 3 also assured the researchers about the positive attributes of an alkaline treatment. However, very little work has been done towards the effect of alkaline treatment on thermal stability of natural fibers. Therefore, in the present communication, an experimental investigation has been carried out to study the effect of alkaline treatment of various concentrations (2%, 4%, 6%, 8% and 10%) on the thermal behavior of pineapple leaf and coir fibers. FTIR spectroscopy was also used to characterize the surface chemistry of untreated and alkali treated (NaOH) fibers. The thermo-chemical decomposition reaction of untreated and NaOH treated cellulosic fibers were studied by employing a TGA instrument.

Table 1: Physical properties of pineapple leaf and coir fiber[6]

Physical properties	Pineapple leaf fiber	Coir fiber
Density (g/cm^3)	0.98	1.2
Cellulose (%)	70-82	32-43
Hemicellulose (%)	18.8	0.15-0.25
Lignin (%)	5-12.7	40-45
Pectin (%)	1.1	3-4
Moisture content (%)	11.8	8
Microfibrillar angle (deg)	14	30-49
Diameter (μm)	20-80	100-460
Tensile strength (MPa)	413-1627	131-220
Young's modulus (GPa)	34.5-82.5	4-6
Elongation at break (%)	1.6	15-40

Table 2: Total annual production of pineapple leaf and coir fiber

Pineapple Production-2016		Coconut Production-2016	
Country	**Millions of tonnes**	**Country**	**Millions of tonnes**
Costa Rica	2.9	Indonesia	17.7
Brazil	2.7	Philippines	13.8
Philippines	2.6	India	11.1
Thailand	1.9	Brazil	2.6
India	1.9	Sri Lanka	2.5
Indonesia	1.3		
World	25.8	World	59.0

Table 3: Previous research work reported on the alkaline treatment of cellulosic fibers

Authors	Factors explored by researchers	Results	Reference
M. Mittal *et al.* (2018)	Effect of alkaline treatment on the mechanical properties of PALF/Glass hybrid composite.	The 15/15 (v/v) alkaline treated PALF/Glass composite exhibits 35% higher flexural strength than that of the pure Glass-Epoxy composite.	[29]
R. Ranjan *et al.* (2013)	Effect of alkali treatment on the mechanical properties of Banana/Sisal fiber reinforced PLA composites.	The 2 wt% NaOH treated composite has better mechanical properties (tensile, flexural, and impact strength) than the untreated composites.	[30]

L. Uma Devi *et al.* (2011)	Effect of chemical treatment on the water absorption behavior of PALF/GF fiber reinforced polyester composites.	The alkali treated composites possess higher resistance to water sorption than the untreated composites.	[31]
G. Goud *et al.* (2010)	Effect of alkali treatment on the mechanical properties of *Roystonea regia* fiber reinforced epoxy composites	Alkali-treated fiber reinforced composite showed better tensile and flexural properties than that of the untreated composite.	[32]
P. Anand *et al.* (2014)	Effect of alkali treatment on the mechanical properties of hemp fiber reinforced epoxy composite	Alkali-treated hemp fiber composite yields better mechanical properties than that of untreated composites.	[33]
S.C Venkateshappa *et al.* (2011)	Effect of alkali treatment on the mechanical properties of areca fiber reinforced epoxy composites	The tensile, flexural, and compressive strength of composites were increased after the alkali treatment of areca fiber.	[34]

EXPERIMENTAL DETAILS

Materials

Two different types of lignocellulosic fiber were used in this study, *viz.,* coconut husk (*Coco Nucifera)* and pineapple leaf fiber (*Ananas comosus*). These cellulosic fibers were obtained from M/s Go Green Products, Chennai (India). Table 1 shows the chemical composition of PALF and COIR fibers[6]. Sodium hydroxide (NaOH) used for an alkaline treatment was of laboratory reagent (LR) grade and obtained from a local supplier.

Fiber treatment

PALF and COIR fibers were soaked in various concentrations (2%, 4%, 6%, 8% and 10 wt%) of alkaline solution for 24 hr at room temperature, followed by washing with deionized water and drying in an oven at 60°C for 24 hr. The untreated cellulosic fibers were also washed and dried to remove the surface impurities and absorbed water molecules.

Fourier-Transform Infrared Spectroscopy (FTIR)

Fourier-transform infrared spectroscopy (Perkin Elmer 2000) was used to analyze the surface chemistry of untreated and alkali treated PALF and COIR fibers. FTIR spectroscopy of the cellulosic fibers had done in Analytical Instrumentation Laboratory, CSIR-CSIO, Chandigarh (India).

Thermogravimetric Analysis (TGA)

Thermal analysis of COIR and PALF (untreated and alkali treated) was carried out by using a thermogravimetric analyzer (Perkin Elmer, TGA 4000). A sample of mass 5 mg was evenly and uniformly

distributed in the alumina crucible which was supported by a precision balance. This small amount of fiber was taken for uniformity of temperature throughout the sample. The variation in mass of a sample with respect to temperature and time was monitored and recorded by Perkin Elmer thermal software (Pyris). The temperature was ranged from 25˚C to 700˚C with a heating rate of 10˚C/min under the high purity nitrogen (inert) atmosphere at a flow rate of 20 ml/min. The heating rate of 10˚C/min was selected for better resolution of transition.

RESULTS AND DISCUSSION

Fourier-Transform Infrared Spectroscopy (FTIR)

Fig. 1 and 2 illustrates the FTIR spectra of COIR and PALF respectively. It was observed that the surface chemistry of cellulosic fibers was changed after an alkaline treatment. An increase in intensity around 3300 cm^{-1} and 1000 cm^{-1} after an alkaline treatment corresponds to the higher accessibility of -OH functional group. This was due to the removal of waxy and gummy substances. The increase in peak intensity at around 1600-1650 cm^{-1} and 1250 cm^{-1} after an alkaline treatment also confirmed the removal of wax, adhesives, pectin, and gummy substances from the fiber surface. The absorption peak at 1745 cm^{-1} was observed in raw fibers (PALF and COIR) but disappears after NaOH treatment. It is related with C=O stretch and confirmed the removal of hemicellulose.

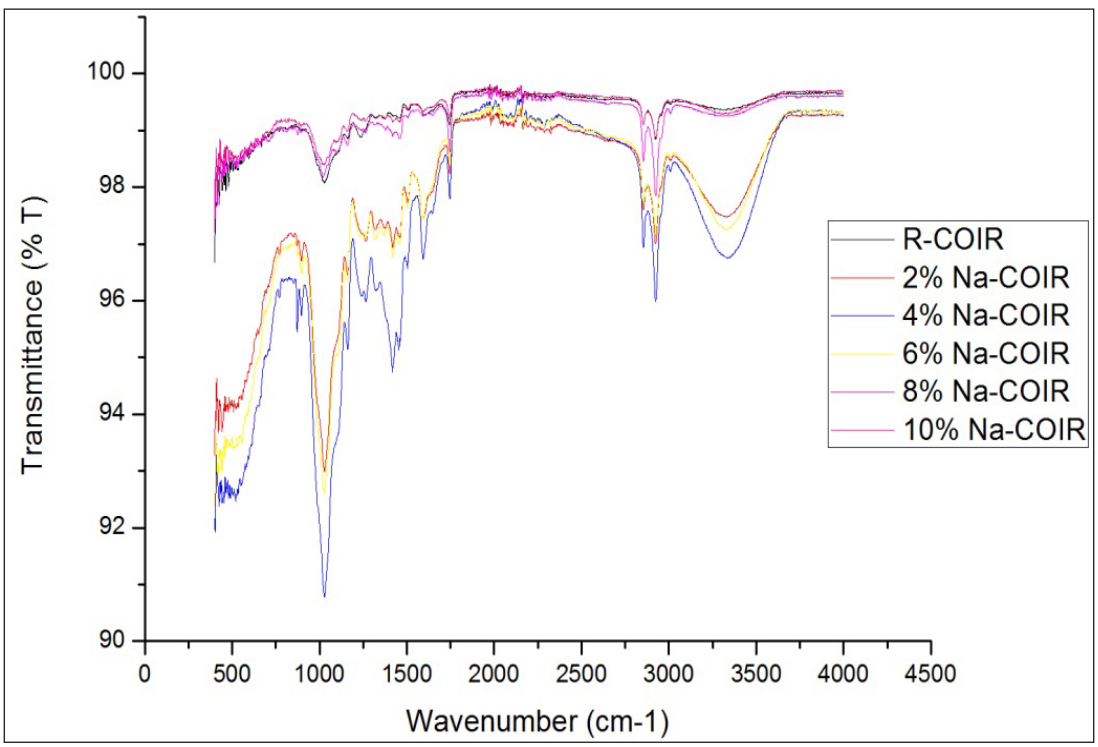

Fig. 1: IR spectra of untreated and alkali treated coir fiber

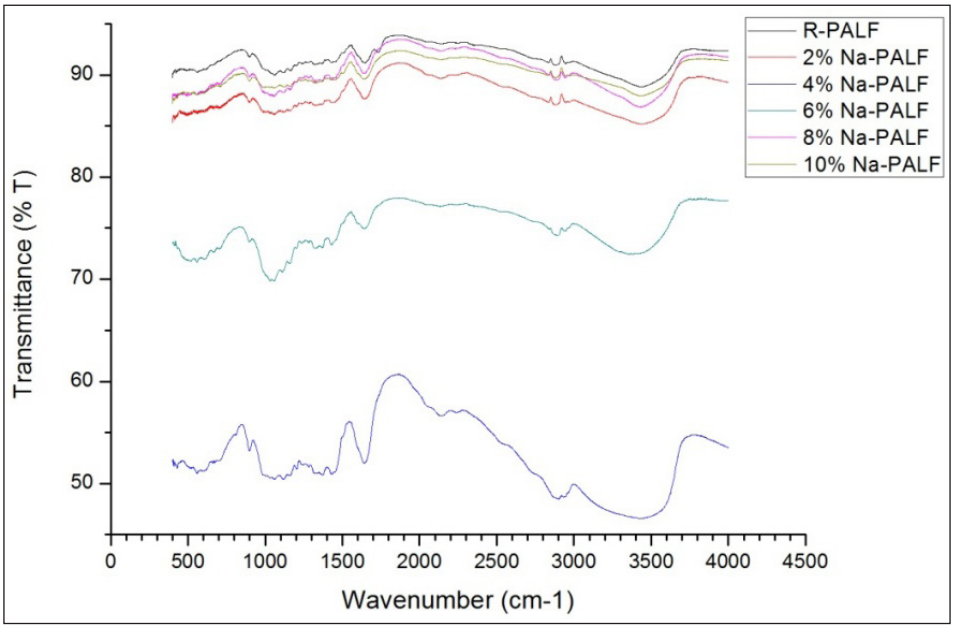

Fig. 2: IR spectra of untreated and alkali treated pineapple leaf fiber

The IR spectra of raw PALF and COIR fibers are shown in Fig. 3. It was observed that the PALF consists a large amount of hydroxyl (-OH) group than that of COIR fiber. The broad peak in the region 3200-3500 cm^{-1} is attributed to the vibration of –OH group. The peak at 1244 cm^{-1} is much smaller in PALF than COIR. This peak corresponds to the C=O stretch of an acetyl group of lignin.

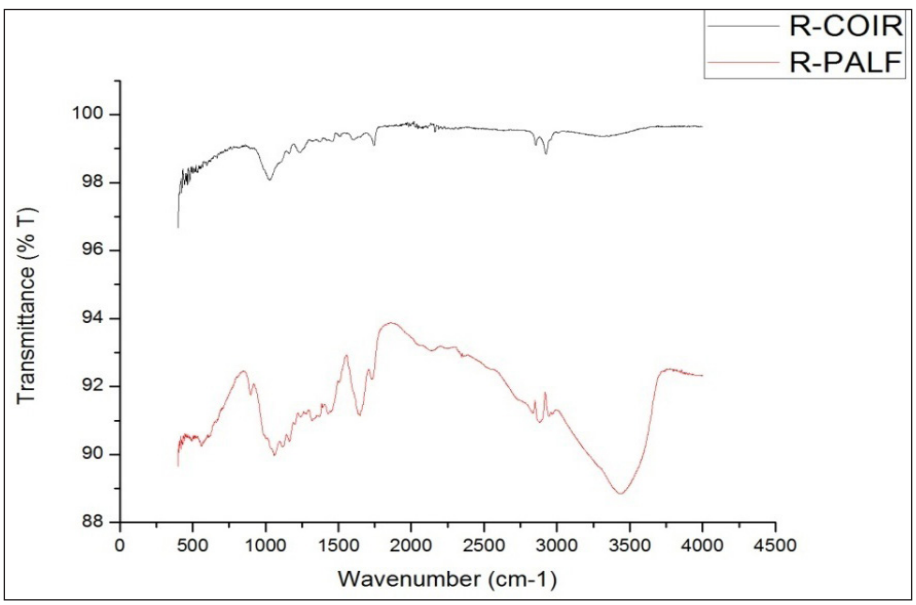

Fig. 3: IR spectra of coir and pineapple leaf fiber

Thermal Decomposition of Untreated and Alkali-Treated Pineapple Leaf and Coir Fibers

TGA and DTG curves of untreated and alkali treated (2%, 4%, 6%, 8%, and 10%) COIR and PALF fibers are shown in Figs 4-9 and the corresponding weight loss as a function of temperature is mentioned in Table 4 and 5. In all cases, the initial stage of decomposition occurs between 30˚C to 185˚C which indicate the release of bound water and volatile extractives. The previous work (Slopiecka *et al.*[16], Ndazi Bwire *et al.*[35]) are also reported that all the types of natural fiber exhibit endothermic decomposition (within 5-10% range) due to an evaporation of moisture, but the rate of evaporation depends on the hydrophilicity of natural fibers. The peak temperature corresponds to the moisture loss appear at 48˚C and 31˚C for untreated COIR and PALF respectively. The PALF exhibits a higher rate of moisture loss than that of COIR fiber. This might be due to the high moisture content in PALF. Table 3 depicts that the 4 wt% NaOH treated PALF exhibits less weight loss (around 48%) due to moisture evaporation as compared to the untreated fiber. This was attributed to the removal of hemicellulose, pectin, and waxy substances after an alkaline treatment. Arifuzzaman Khan *et al.*[36] also reported that the percentage of initial weight loss due to moisture evaporation is higher in untreated COIR fiber than that of NaOH treated one. At 100˚C, 8 wt% alkaline treated COIR fibers possess less weight loss (approx. 40%) as compared to raw fibers. Ndazi *et al.*[35] concluded that the structurally bound water is resistant to complete water removal during drying.

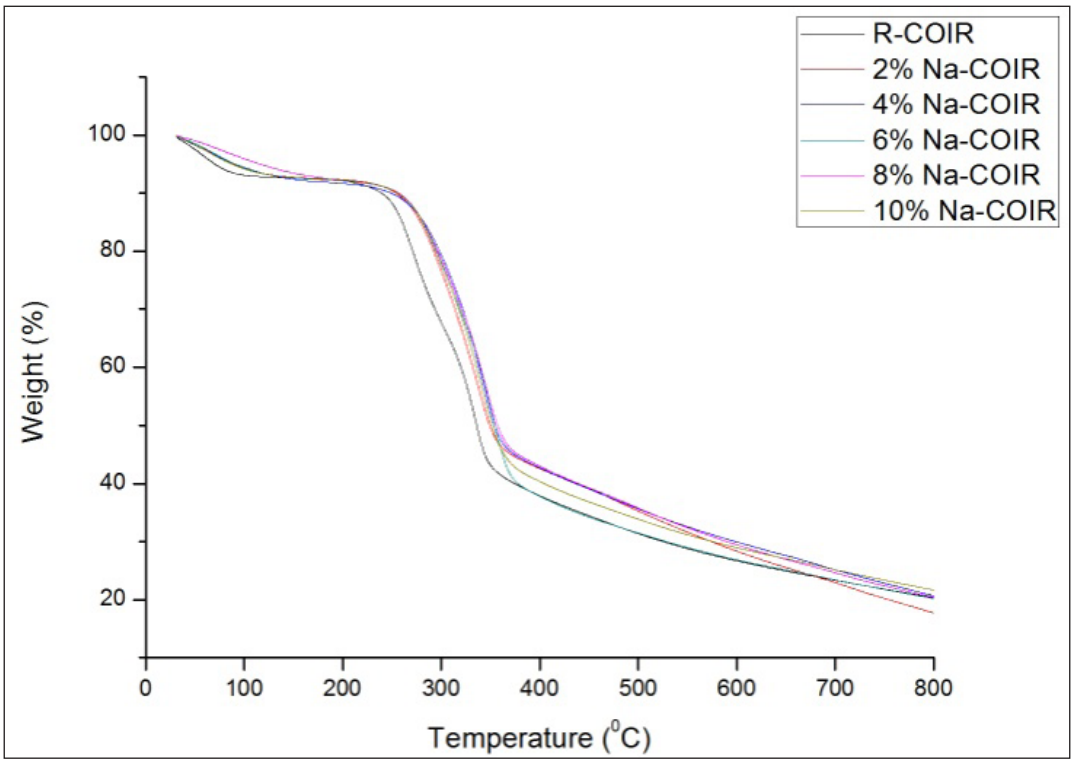

Fig. 4: TGA curves of untreated and alkali treated coir fibers

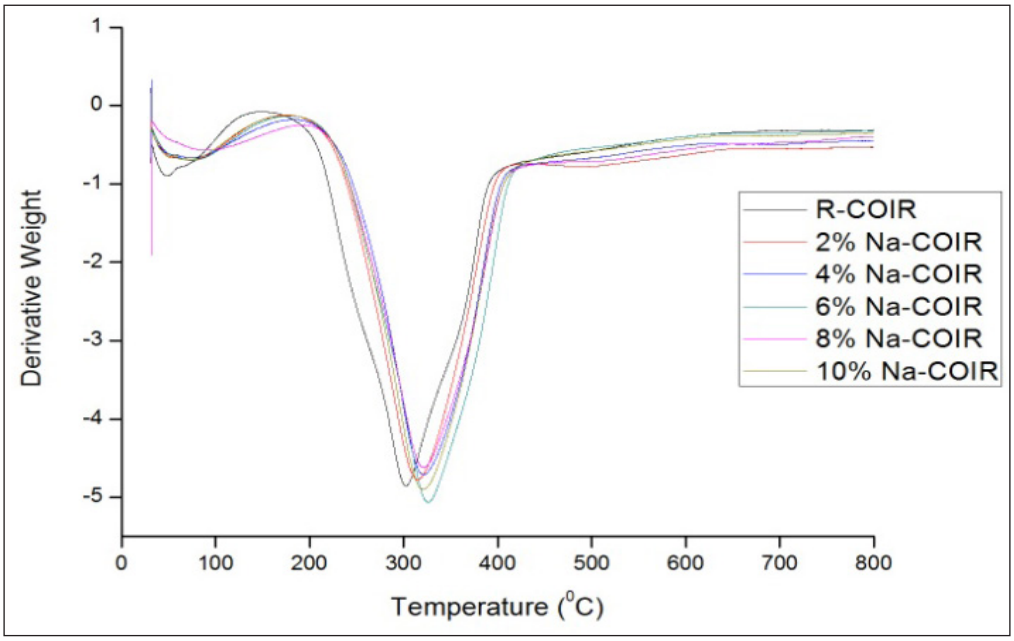

Fig. 5: DTG curves of untreated and alkali treated coir fibers

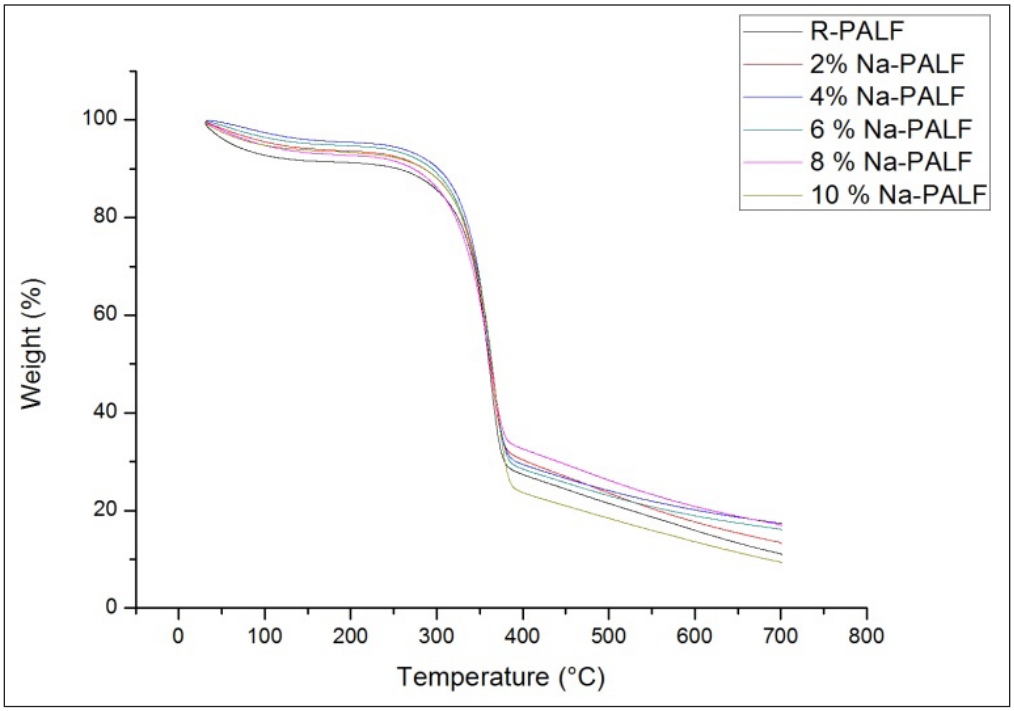

Fig. 6: TGA curves of untreated and alkali treated pineapple leaf fibers

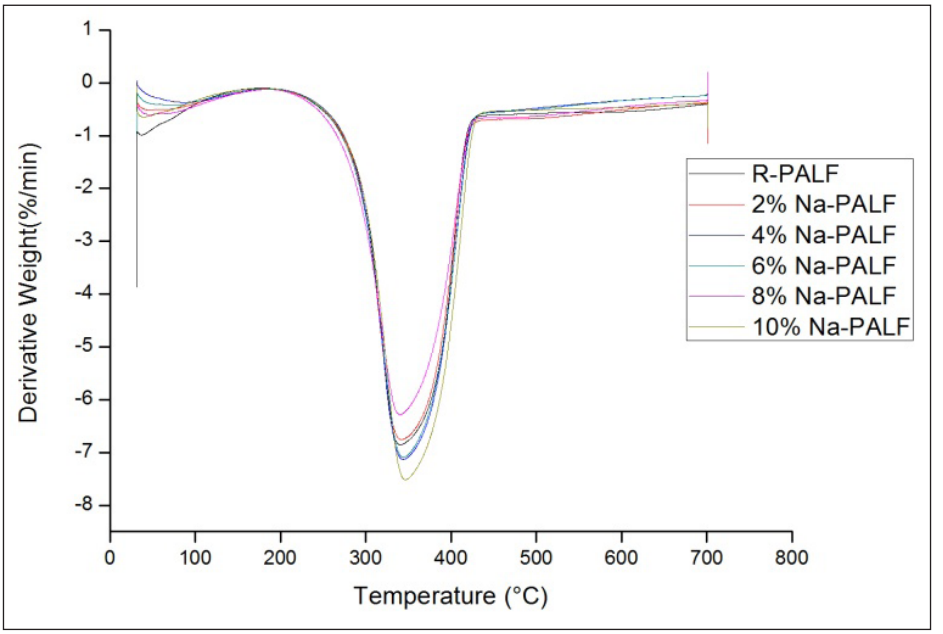

Fig. 7: DTG curves of untreated and alkali treated pineapple leaf fibers

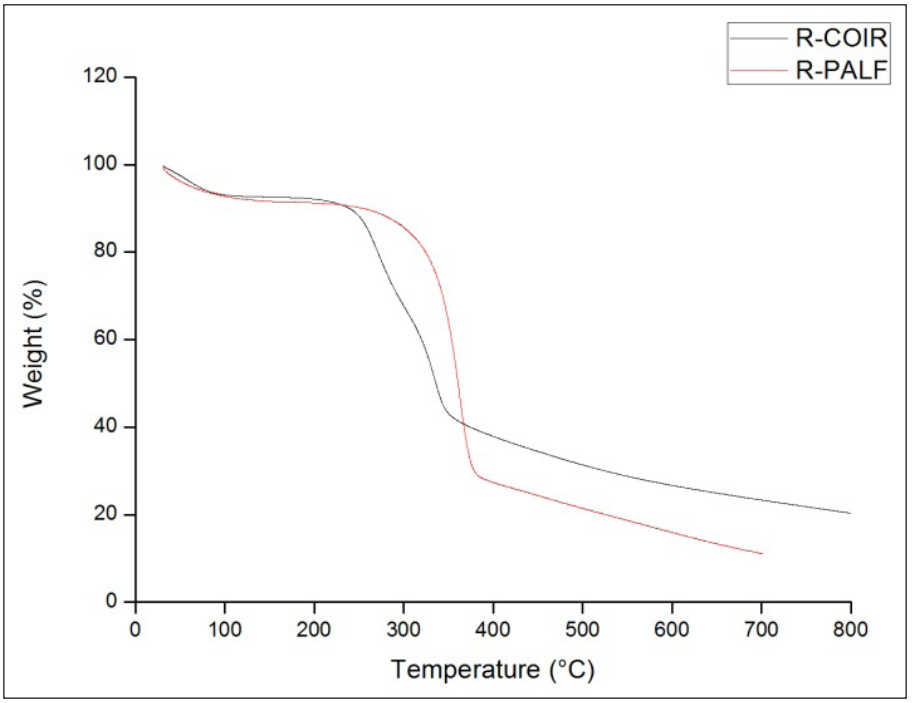

Fig. 8: TGA curve of untreated coir and pineapple leaf fiber

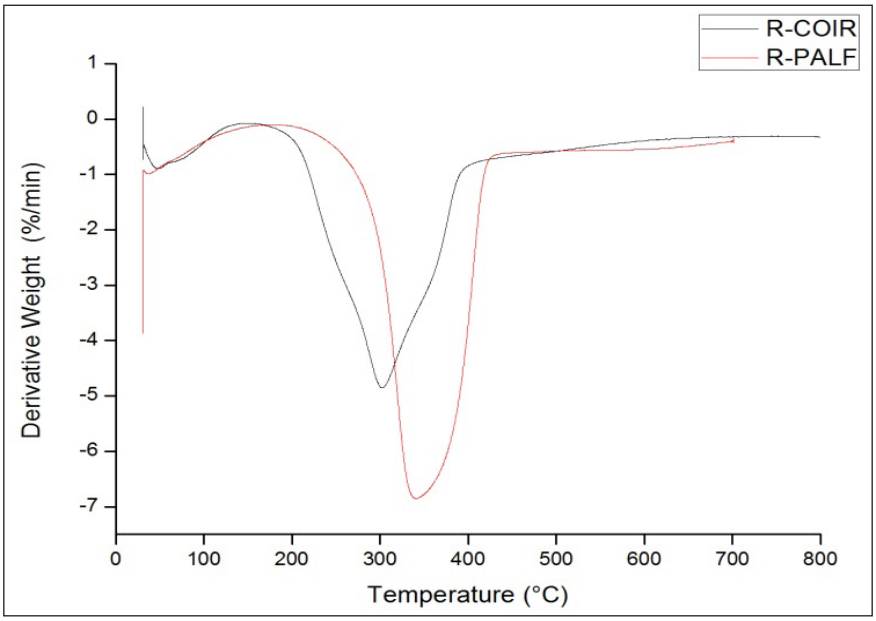

Fig. 9: DTG curve for untreated coir and pineapple leaf fiber

Table 4 and 5 depicts that the weight loss in all cases (Untreated and NaOH treated PALF and COIR fibers) is negligible below 290˚C; major mass loss (30-50 wt%) occur above this temperature and it was due to the degradation of crystalline cellulose. M. Siti. Alwani *et al.*[37] also reported that the major mass loss (30-50%) in SCB, PALF, COIR, and BPS fibers occurred at a temperature above than 300˚C. Hemicellulose decomposition is the first stage of fiber degradation. As the temperature increases, the cellular breakdown of hemicellulose takes place. The coconut husk fibers started to decompose at 150˚C; however, the pineapple leaf fibers decompose at 185˚C. In both cases, the peak temperature of maximum degradation was shifted to a higher temperature after an alkaline treatment. The 4 wt% NaOH treated COIR fiber exhibits higher peak temperature of thermal degradation (about 20˚C) than that of untreated one. This was due to the removal of hemicellulose after an alkaline treatment of cellulosic fibers. Yang *et al.*[38] reported that the presence of a carbonyl group (C=O) in hemicellulose be the cause of its lower thermal stability than that of α-cellulose, and lignin. Ndazi Bwire S *et al.*[19] reported the reduction in a temperature of maximum weight loss after an alkaline treatment of rice husk fiber and it was due to the excess removal of cementing materials like hemicellulose and lignin. The main degradation peak lies within 290˚C-380˚C temperature with 50% degradation. The PALF possess higher mass loss (around 10%) as compared to COIR fiber. This was because of the high cellulose content in PALF. However, the degradation of PALF occurs at a higher temperature than that of COIR fiber. M. Siti. Alwani *et al.*[37] reported that the sugarcane bagasse fiber exhibits higher mass loss than that of pineapple leaf, banana, and coconut fibers. This was attributed to the presence of high cellulose content in SCB fiber.

Lignin was the most thermally stable component of a cellulosic fiber due to its highly cross-linked complex aromatic structure. Mackay and Nassar[39] reported that the thermal stability of lignocellulosic fibers is in the order: lignin>α-cellulose>hemicellulose. Lignin starts to decompose at a lower temperature (typically 160-175˚C) as compared to cellulose but it decomposes slowly under the whole temperature range and its

temperature extends as high as 900˚C. According to Paiva *et al.*[40], the decomposition of lignin occurred in a wider temperature range as compared to the cellulose and hemicellulose components. Table 4 and 5 clearly revealed that the remaining mass of COIR was higher than PALF fiber after 700˚C. This was attributed to the presence of a large amount of lignin compound in COIR fiber. Williams *et al.*[41] reported that the high lignin content in lignocellulosic fibers leads to a high amount of residues during pyrolysis. It was observed that the 4 wt% NaOH treated COIR and PALF fibers possess higher residual mass than that of untreated fibers. This was due to the slow rate of chemical decomposition reaction and dominance of the formation of condensed cellulose as the solid residue.

Table 6 and 7 illustrate the DTG results of untreated and alkali treated fibers. In case of untreated COIR fiber, the two decomposition peaks at 228˚C and 302˚C at the rate of 1.475 and 4.852 %/min correspond to the decomposition of hemicellulose and cellulose components. It was observed that the rate of hemicellulose decomposition is more prominent in untreated fibers (PALF and COIR) as compared to alkali treated fibers. Pineapple leaf fiber exhibits a higher rate of decomposition than coconut husk fiber. Thus from DTG results, we can conclude that the alkaline treated fibers possess degradation peak at a higher temperature than that of untreated fibers.

Table 4: TGA of untreated and treated coir fibers

Fiber	Temperature Range ˚C	Weight Loss (%)	Residual mass (%) at (700˚C)
R-COIR	0-152.4	7.396	23.359
	152.4-242.85	3.05	
	242.85-352.94	46.49	
2% Na-COIR	0-161.9	7.4	22.94
	161.9-262.01	3.68	
	262.01-361.86	42.73	
4% Na-COIR	0-176.06	8.07	25.135
	176.06-277.08	5.7	
	277.08-363.15	39.39	
6% Na-COIR	0-176.19	7.66	23.409
	176.19-271.44	4.8	
	271.44-376.18	47.29	
8% Na-COIR	0-152.38	6.65	24.64
	152.38-266.72	4.7	
	266.47-376.25	43.43	
10% Na-COIR	0-171.52	7.55	25.15
	171.52-276.12	5.69	
	276.12-361.95	40.92	

Table 5: TGA of untreated and treated pineapple leaf fibers

Fiber	Temperature Range ^{0}C	Weight Loss (%)	Residual Char (%)
R-PALF	0-184.78	8.63	11.103
	184.78-330.4	13.63	
	330.4-382.66	48.75	
2% Na-PALF	0-164.31	6.071	13.356
	164.31-261.85	1.97	
	261.85-361.97	41.98	
4% Na-PALF	0-184.04	4.465	17.34
	184.04-324.15	11.18	
	324.15-376.7	49.59	
6% Na-PALF	0-175.1	5.12	16.14
	175.1-321.7	11.16	
	321.7-378.32	50.57	
8% Na-PALF	0-186.88	7.162	17.01
	186.88-315.15	10.54	
	315.15-378.23	46.56	
10% Na-PALF	0-186.95	6.545	9.386
	186.95-326.09	12.03	
	326.09-382.54	54.058	

Table 6: DTG of untreated and treated coir fibers

Fiber	Temperature ^{0}C	Rate of Weight Loss (%/min)
R-COIR	48.43	0.897
	228.43	1.475
	302.56	4.852
2% Na-COIR	72.47	0.697
	243.35	1.162
	314.3	4.774
4% Na-COIR	73.61	0.67
	250.13	1.156
	321.74	4.708
6% Na-COIR	74.43	0.428
	252.74	0.565
	325.9	5.061
8% Na-COIR	90.80	0.571
	252.96	1.413
	321.09	4.62
10% Na-COIR	72.77	0.693
	258.48	1.719
	321.95	4.894

Table 7: DTG of untreated and treated pineapple leaf fibers

Fiber	Temperature ˚C	Rate of Weight Loss (%/min)
R-PALF	31.16	1.47
	310.82	3.54
	341.58	6.85
2% Na-PALF	46.37	0.52
	247.81	0.49
	341.52	6.75
4% Na-PALF	85.79	0.37
	308.69	3.39
	343.43	7.13
6% Na-PALF	70.83	0.43
	302.33	2.73
	343.12	7.091
8% Na-PALF	46.49	0.60
	300.08	2.71
	339.72	6.28
10% Na-PALF	39.7	0.65
	313.05	3.66
	346.85	7.51

CONCLUSION

In this work, an experimental investigation was carried out to study the thermal behavior of untreated and alkali treated PALF and COIR fibers. From this study, the following conclusions can be made:

- The thermal decomposition of PALF and COIR fibers proceeded in two steps. In 1st step, a minor weight loss occurs due to an evaporation of water and extraction of volatile compounds and in the 2nd step, major degradation occurs due to the decomposition of hemicellulose, cellulose, and lignin components.
- The alkaline treatment of PALF and COIR fibers leads to a lower weight loss and shifting of degradation peak to a higher temperature.
- Amongst all the samples, the 4 wt% NaOH treated PALF and COIR fibers exhibit maximum thermal stability. This improvement was due to the removal of less thermally stable hemicellulose component after an alkaline treatment. The temperature of maximum decomposition was shifted from 302˚C to 321˚C after the 4 wt% alkaline treatment of COIR fiber.
- Pineapple leaf fiber exhibits a higher rate of decomposition (6.85%/min) than coir fiber (4.85%/min). It was due to the presence of more cellulosic content in PALF.
- After 700˚C, the remaining mass of COIR (23.35%) was higher as compared to PALF (11.10%). This might be attributed to the presence of high lignin content in COIR fiber.

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