

TGA and Thermal Kinetics of Raw Calotropis Procera Fiber Reinforced PF Composites

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Abstract

Natural fibre-reinforced composites are used in various structurally designed goods, from civil engineering to the production of automobiles, thanks to qualities like minimal density, a favourable aspect ratio, biodegradability, and ease of fabrication. The thermal behaviour of natural fibres and composites has also been researched. The thermal degradation kinetics characteristics of composites made with phenol formaldehyde resin and reinforced with untreated Aak fibre with varying fibre loads have been determined using thermogravimetric analysis (TGA). The Flynn-Wall procedure determined each component and composite material's precise apparent activation energy (E_a). Varying fibre load (5, 10, 15, 20 wt.%) was used to reinforce PF resin, and TGA was taken for all composite samples. By observing TGA data composite with 15 wt% fibre load shows maximum thermal stability, it can also be concluded that the thermal stability of prepared composites increases with increasing fibre load. After 15 wt%, it starts to decrease.

Keywords: Untreated Aak fibre, PF resin, TGA, Chemical kinetics for TGA.

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Introduction

Recent decades have seen a resurgence in interest in natural materials, such as cellulose-rich fibres taken from cultivated plants, often known as lignocellulosic fibres, and their polymer composites due to environmental concerns about world scale pollution and climatic changes [1-4]. In actuality, natural fibres are regarded as environmentally benign due to their biodegradable and renewable qualities and ability to save energy during processing, which is an inescapable issue for synthetic fibres. Suppose a sufficient amount of fibre is added. In that case, a sufficient amount of fibre is added, and both natural fibres and their composites made of polymers are carbon-neutral regarding the CO₂ emissions that result in the earth's greenhouse effect, a key contributor to global warming [5]. In actuality, the content assimilated throughout the growth of the fibre plant will balance off the emission of CO₂ caused by combustion or atmospheric decline at the end of the life cycle of these "green" materials.

Natural fibres, in contrast, have significant limitations a component of to some extent, which also impact their polymer composites [1-4].

The performance of composite materials at different temperatures is a crucial consideration when determining which temperature offers the most appropriate material attributes. Because Calotropis Procera can withstand the harsh climate in western Rajasthan, this ability should be

expected to translate to its fibre and composite. The thermal study is the critical factor that affects how the prepared composite turns out. The TGA analysis is the most widely used method for assessing thermal characteristics. The Flynn-Wall technique determined each component and composite material's precise apparent activation energy (E_a). Varying fibre load (5, 10, 15, 20 wt.%) was used to reinforce PF resin, and TGA was taken for all composite samples.

Materials and methodology

Extraction of Calotropis Procera bast fibre

Calotropis Procera is a desert plant that grows in large quantities in Rajasthan's western region. The corky bark surrounds the steams. Calotropis Procera bark can be used to make strong fibres. Barks of these plants have been collected from the Bikaner region. These collected barks have been cut into 20-30 cm long pieces and then preserved in water for about 1 week until the barks get smooth. Then natural fibres from these barks are extracted manually. These extracted natural Calotropis Procera (CP) fibres were chopped about 10-15 mm for composite preparation.

Composite preparation

To create the standard PF resin, phenol was first dissolved in formaldehyde (HCHO) in the presence of an alkyl medium (NaOH), and then the mixture was heated at 60 °C while constantly stirred for roughly two hours. The resulting

mixture was poured onto a flat, rectangular petri plate and cured at room temperature for three to four days at room temperature to create phenol formaldehyde resin. The procedure for making fibre/polymer composites was the same: chopped fibre was added to the phenol formaldehyde resin (PF) at different loading levels by hand lay-up method. The mixture was then dried in the oven at an even temperature of 60 °C. Prepared composites, then cut into desired shapes and lengths for physical, mechanical and thermal analysis.

Table 1: Sample code according to different fibre load

Fibre load	5 wt%	10 wt%	15 wt%	20 wt%
Sample code	U-5	U-10	U-15	U-20

Analysis using TGA

Samples underwent TGA analysis in the PG research lab at IIT Kanpur. This investigation made use of TA Instruments TGA (SDT Q600). The experiments were performed at temperatures as high as 300 °C. The settings were as follows: Heating rates of 5 °C/min, 10°C/min, 15 °C/min, and 20 °C/min from 25 °C to 300°C in a nitrogen environment were used for prepared CP fibre-reinforced PF composite investigation.

Theoretical methods to study chemical kinetics for TGA

TGA experiments are frequently used to forecast the thermal stability of a material. Additional information on the kinetics of heat disintegration and an in-use lifetime can be predicted using a modified experimental setup. By heating the sample steadily, Flynn and Wall's model [6] calculates the breakdown kinetics of breakdown. This takes less time than other models that have been suggested for the same goal. This is true for single-step decomposition and first-order kinetics.

The Flynn and Wall model can be applied when the sample is heated gradually and at least three different heating rates are used the sample is heated gradually. The Flynn and Wall model can be applied if at least three different heating rates are used

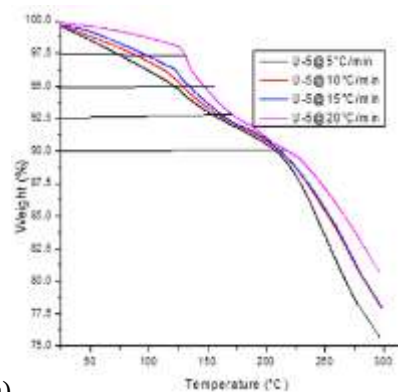
$$E_a = \left(-\frac{R}{b} \right) \frac{d(\ln \beta)}{d(1/T)} \quad (1)$$

The numerical value of b is ascertained through an iterative process. The initial b=0.457 is acquired, and equation (1) is then used to calculate the value of Ea. Ea /RT is calculated using this Ea value, where T is the temperature at which constant mass loss occurs. T is the temperature at which the continuous mass loss occurs and is used to calculate Ea /RT. The technique is repeated until Ea stops changing before calculating the value of b corresponding to Ea /RT using

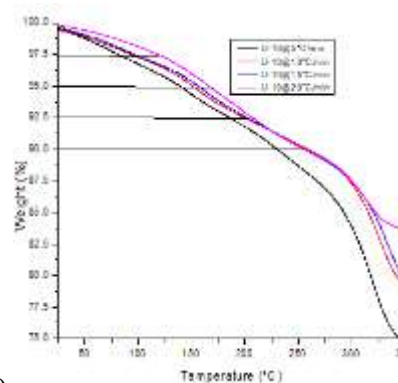
Appendix A [7].

Result and Discussion

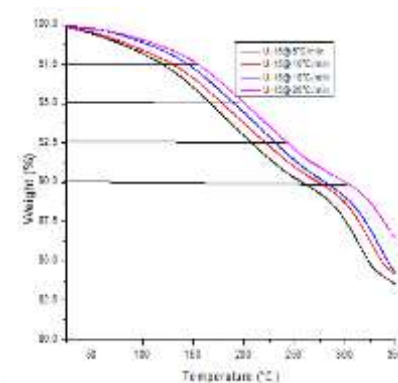
TGA provides information on weight loss at a specific temperature. The TGA curve for Untreated Aak fibre PF composites is shown in Fig 1 (a-d) with the above heating rates.



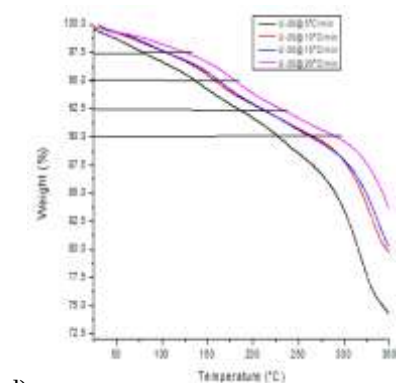
a)



b)



c)



d)

Figure 1 (a-d): TGA curve of untreated Aak fibre PF composite (U-5, U-10, U-15 and U-20) with different heat rates.

The 5% mass loss temperature for all composites is displayed in Table 2. The TGA curve can be used to determine the sample's thermal stability. The decomposition temperature has risen with increasing fibre content up to 15% fibre weight.

This leads to the conclusion that composites thermal stability significantly increased. The applied heating rate controls the reaction time available at a given temperature. Hence kinetics degradation is directly dependent on the heating rate. The time available for the reaction is typically thought to be longer if the heating rate is lower. A lower temperature with a slower heating rate follows a typical trend in the TGA curve, while it heats up more quickly and decomposes at a higher temperature. In this approach, the heating rate significantly impacts thermal stability.

Table 2: The temperature in °C for 5% mass loss at different heating rates

Sample	Heat rate 5°C/min	Heat rate 10°C/min	Heat rate 15°C/min	Heat rate 20°C/min
U-5	116	128	135	145
U-10	139	154	160	168
U-15	168	181	191	197
U-20	134	157	163	185

A similar observation of thermal degradation of banana-fibre reinforced phenol formaldehyde composite by several researchers [8,9].

Chemical kinetics for TGA

Plotting the multiple TGA curves for all samples in Figure 2 provides an example of this approach. The TGA curve is displayed as coloured lines at various heating rates. Straight black lines represent a constant mass loss. The temperature value for the relevant heating rates can be found at the intersection of the horizontal black lines and the TGA curve. The gradient of this graph is then determined after plotting the line between $\ln\beta$ and $1000/T$. Using the equation below, $b=0.457$ calculates an approximate value for E_a . Activation energy can be determined by the equation (1).

Now the value of E_a/RT is calculated, here E_a is the value of activation energy obtained by the equation (1), and T is the temperature at a constant mass loss (In this work, chemical kinetics of all samples are measured for 5% constant mass loss with 10 °C/min heating rate) by this value corresponding value of b can be got by appendix A. We get the new value of E_a . This value of E_a will match the

obtained value by eq. (1), which is repeated until our value of E_a remains unchanged. In the present work, TGA kinetics for all the samples are calculated for only 5% mass loss.

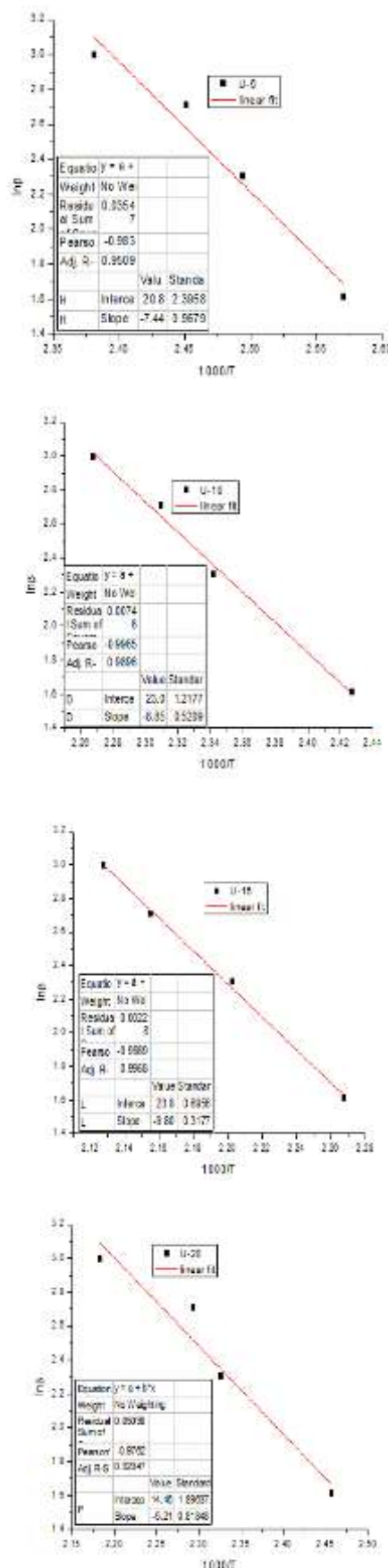


Figure 2(a-d): log heating rate vs. $1000/T$ for untreated CP fibre-reinforced PF composite

Table 3: Activation energy of thermal decomposition for untreated CP fibre-reinforced PF composite.

Sample	$\frac{d \ln \beta}{d(\frac{1}{T})}$ at 5% mass loss	b(constant)	Ea (KJ/mol)
U-5	7.44	0.5281	114.59
U-10	8.85	0.5281	139.3
U-15	9.80	0.5187	157.07
U-20	5.21	0.491	88.05

Table 3 shows the chemical kinetics of TGA for all samples. The maximum activation energy is obtained by sample U-15.

Conclusion

TGA was used to study the thermal kinetics of CP fibre-reinforced PF composite. According to TGA U-15, the sample showed better thermal stability than other composites. The value of activation energy for U-15 is 157.07 KJ/mol, the maximum among all composites. The quantity of energy needed to execute a particular transition is referred to as the Ea of any reaction. The higher the Ea value, the more complex the transition. It signifies that the transition took longer since there was more incredible activation energy. A slower transition rate indicates more excellent thermal durability. As a result, the thermal durability of composite increases with increasing fibre loads up to 15wt%.

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