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Research article

Analysis of thermal decomposition kinetics of chicken feather fiber reinforced Poly-lactic acid composites filament

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ABSTRACT

Derivative Thermogravimetric analysis under air was used to observe the thermal decomposition process of Chicken feather fiber (CFF) reinforced Poly-lactic acid (PLA) composite filament of 2.2 mm diameter. The thermal degradation of the sample was initiated at 140 ̊C. Approximately 75 % of the thermal degradation occurred between the temperature of 357 ̊C and 399 ̊C. The composite's activation energy was established using the Coats-Redfern method. The results showed that the activation energy of 112.06 kJ/mol is utilized for the sample throughout the temperature range of 23 ̊C to 398 ̊C. A low activation energy is indicative of rapid chemical reactions between the CFF and PLA molecules. The results from TGA and DTGA indicate that the addition of CFF in the PLA matrix enhanced the thermal stability.

1. Introduction

Thermogravimetric analysis (TGA)-based thermal degradation of natural fiber composites is a key field of study in materials science and engineering. The heat stability and breakdown behavior of these composites are well-understood by TGA [1]. The activation energy, which is one of the important characteristics derived from TGA analysis, is essential for comprehending the kinetics of decomposition [2–5]. One method that is frequently used to examine the thermal behavior of materials is thermogravimetric analysis (TGA). Researchers can analyze the weight loss of a composite sample as a function of temperature and time under controlled conditions when using TGA on natural fiber composites. For the purpose of their design and use, this technique offers insights into the thermal stability and decomposition behavior of the composites. The process by which a material disintegrates into more basic components as a result of heat application is known as thermal decomposition. Natural fibers are incorporated in a polymer matrix to form natural fiber composites. The sample is heated under controlled conditions in air, nitrogen, or helium during TGA analysis. The sample's weight changes are continuously monitored as the temperature increases, providing insight into the behavior of thermal decomposition. The thermal decomposition is observed in three stages [6,7].

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1. Drying stage
2. Polymer Matrix Degradation
3. Natural fiber Decomposition

Drying Stage: The initial stage observed in the TGA analysis of natural fiber composites is the drying stage. This stage involves the removal of moisture absorbed by the natural fibers and the polymer matrix. The weight loss during this stage corresponds to the evaporation of water present in the composite, and it is typically a small fraction of the total weight. This stage is observed from 110 °C to 120 °C.

Polymer Matrix Degradation: Following the drying stage, the decomposition of the polymer matrix takes place. The polymer chains begin to break down and volatilize, leading to a more significant weight loss compared to the drying stage. The rate and extent of weight loss during polymer matrix degradation depend on the specific polymer used in the composite. Analysing the weight loss profile during this stage provides insights into the polymer's thermal stability and decomposition kinetics.

Natural Fiber Decomposition: Simultaneously with the polymer matrix degradation, the natural fibers in the composite undergo their thermal decomposition. Plant-based natural fibers, such as jute, sisal, hemp, or flax, are composed of cellulose, and hemicellulose while animal-based natural fibers contain keratin and other organic compounds. The decomposition of these components occurs over a range of temperatures, typically starting from around 200 °C. During thermal decomposition, cellulose, hemicellulose, and keratin decompose, releasing volatile compounds such as water, carbon dioxide, and organic vapors.

The activation energy is a fundamental parameter that characterizes the thermal decomposition kinetics of a material. It represents the energy barrier that must be overcome for a chemical reaction, such as decomposition, to occur. By studying the weight loss data obtained from TGA experiments, it is possible to determine the activation energy associated with the decomposition process.

The TGA data can be analyzed and the activation energy can be determined using a variety of kinetic models, including the Arrhenius equation. The breakdown rate constant is related to temperature and activation energy by the Arrhenius equation. The activation energy can be found by graphing the logarithm of the decomposition rate constant versus the reciprocal of the temperature. This will yield a linear connection. The decomposition mechanism and the stability of the composite material can be observed using the activation energy. Since additional energy is needed for breakdown to occur, a composite with a higher activation energy is more stable. The design of composite materials with improved thermal stability and the selection of suitable processing conditions can both benefit from an understanding of activation energy. Understanding the thermal breakdown behavior of natural fiber composites is primarily predicated by TGA in conjunction with activation energy studies. The acquired information helps in forecasting the kinetics of decomposition, creating composite materials with enhanced thermal stability, and optimizing processing conditions. Researchers can assess the stability and applicability of various natural fibers and polymer matrices for particular applications by measuring the activation energy.

Poly-lactic Acid (PLA) is a non-petroleum-based polymer. It is manufactured by polymerization of lactic acid monomer, obtained from the fermentation of agricultural waste materials. PLA presents better manufacturability and strength performance as compared to petroleum-based polymers such as ABS. Researchers have earlier attempted to prepare animal fiber such as crab shell [8–10], chicken feather fiber [11], wool [12], etc. Chicken feathers are available in abundance as waste products from the poultry industry. Chicken feather fibers have a high aspect ratio and also have high thermal resistance properties. CFF has been reviewed as good reinforcement material by earlier researchers [13]. Biocompatible composites prepared with CFF as reinforcement and PLA as matrix material have been reported in previous research. The composites are prepared using: Hand Layup technique [14,15], Compression Moulding [16–18], Extrusion Moulding [19], Spray Layup [20,21], Sandwich Method [22], Vacuum Assisted Resin Transfer Moulding (VARTM) and Solution Moulding [23–25].

Since the Coats-Redfern technique seeks average activation energies throughout the whole selected reaction period, it is a more representative integral method. But as activation energies range in value with different reaction orders, it is essential to first check the function form of the reaction mechanism. Coats-Redfern technique to obtain activation energy of solids have been found to be appropriate by numerous researchers [26,27]. Earlier researchers have used Coats-Redfern technique to obtain the activation energy and observe the thermal decomposition of Chicken feather [28]. There are no known prior attempts to compute the activation energy during the thermal decomposition of chicken feather reinforced polylactic acid biocomposites. The objective of this work was to estimate the activation energy value by performing a differential thermogravimetric analysis using the modified Coats-Redfern method and to investigate the thermal decomposition process of CFF/PLA composite filament by observing the TGA thermogram.

2. Material

The poultry feathers are gathered from local supplier in Gujarat, India. To maintain the homogeneity in the characteristics contour feathers of white broiler chicken was selected. The feathers were pre-processed as suggested in the research paper by Adil Khan et al. [13,22]. The trimmed barbs in the form of short fibers are mixed with PLA granules and formed into filaments using an indigenous short fiber reinforced composite filament extruder.

3. Test procedure

The Thermogravimetric analysis was carried out on TGA-50 (Make: Shimadzu, Japan) at the Applied Chemistry Department of The Maharaja Sayajirao University of Baroda, Vadodara. The test was carried out in controlled air conditions, in a platinum cell. A sample of 22.93 mg was heated from room temperature to 400 °C at a heating rate of 10.00 °C/min. Using OriginPro the first and second derivative of weight loss concerning the corresponding temperature from the TGA thermogram was obtained. The first and the second derivative is used to obtain the peak temperature of the mass loss [29].

4. Results and discussion

4.1. Thermogravimetric analysis

Fig. 1 presents the thermogravimetric decomposition process of CFF/PLA filaments. The constituent materials CFF and PLA were sun-dried and baked before mixing and extruding them into filaments. As a result, the moisture content in the sample was minimum. The only water molecules present would be in the bonded form. This behavior is reflected in the TGA curve. There is minimal weight loss (less than 0.1 %) till the temperature of 140 °C. till the temperature of 300 °C the sample reflects only another 2.5 % of weight loss.

Between the temperature 300 °C–350 °C PLA the sample losses about 20 % of its weight. This is accredited to the polymer chain breakdown. During this stage, the structure of PLA is degraded. This temperature range corresponds to the one found in previous research. The TGA of PLA is shown in Fig. 2.

At the temperature from 354.6 °C to 355.2 °C a sudden drop in weight is observed which is regained at 355.8 °C. the same is observed from the TGA, DTGA, and D2TGA of the filament as shown in Fig. 4. After regaining the weight, the degradation of the sample follows a curve similar to the one observed in the TGA of chicken feathers as shown in Fig. 3. During thermal decomposition, cellulose, hemicellulose, and keratin decompose, releasing volatile compounds such as carbon dioxide, and organic vapors.

The maximum degradation rate of 15 mg/°C is observed from the peak at 355.48 °C and is determined from the DTGA. Observing the residual weight value W_i and W_f at the onset temperature T_i and offset temperature T_f it can be determined that the major weight loss of 72 % is observed between the temperature range of 358.8 °C and 398 °C at a rate of 0.41 mg/°C rise in temperature. Between the temperature 356.4 °C–359.03 °C a weight gain of about 9 % is observed. This is accredited to the absorbance of oxygen before the breaking of another crystalline structure resulting in endothermic behavior. The curve is indicative of an explosive decomposition followed by a recoil. This seconds the conclusion of the breakage of the secondary crystalline structure near 359 °C. Between 359.03 °C and 398 °C, 75 % of the complete degradation of the material was observed. This loss occurs after the breakage of the crystalline structure. 1 % i.e., about 1.56 g of unburnt residue remained at the end of the test.

The crystalline structure is more stable than the parent materials. The different slope of denaturing and degradation, as compared to that of CFF and PLA, substantiates the claim of the formation of a rigid polymorph formed at the interfaces. For a better understanding of the CFF/PLA filament thermal degradation kinetics, it is suitable to quantify their decomposition activation energy.

4.2. Activation energy

The fundamental equation in all the kinetic studies is [32].

$$d\alpha / dt = k f(\alpha) \quad (1)$$

Where,

$f(\alpha)$ = reaction model

For polymeric compounds

$$f(\alpha) = (1-\alpha)^n \quad (2)$$

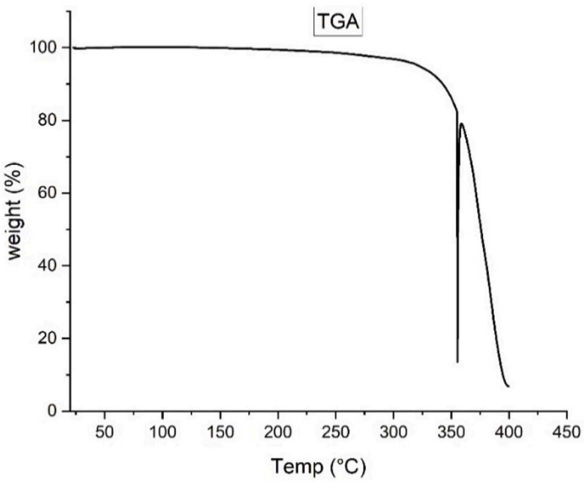

Fig. 1. TGA of CFF/PLA filament.

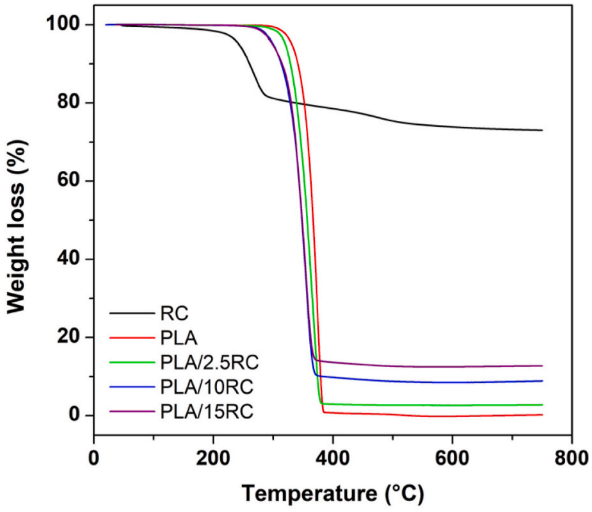

Fig. 2. TGA of PLA [30].

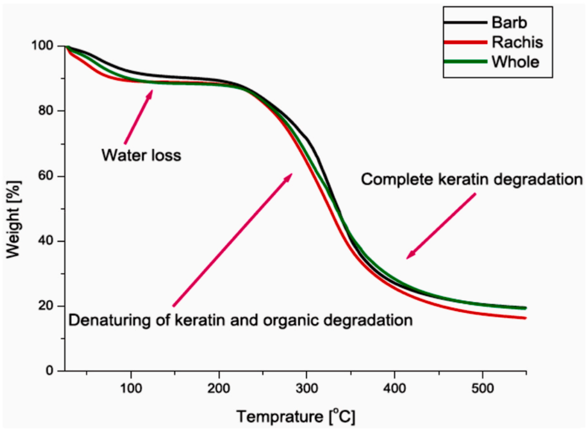

Fig. 3. TGA of various parts of Chicken feather [31].

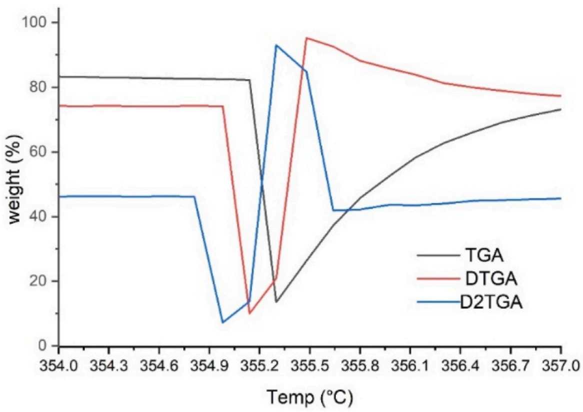

Fig. 4. TGA, DTGA and D^{2}TGA of CFF/PLA filament.

n = order of the equation.

For solid compounds value of n is taken as 0 [33].

dα/dt = rate of conversion

$$\alpha = \frac{(W_i - W_t)}{(W_i - W_f)} \text{ at time t.} \quad (3)$$

k = rate constant.

The rate constant k in the present study is obtained using the Arrhenius equation:

$$k = A \exp(-E/RT) \quad (4)$$

A = Arrhenius frequency factor.

E = Activation Energy.

R = gas constant.

T = Process temperature.

A combination of Equations (1), (2) and (4) gives

$$d\alpha/dt = 〚(1-\alpha)〛 \hat{n} \; A \exp(-E/RT) \quad (5)$$

According to the Modified Coats-Redfern method the slope of the plot $ln\,(1 - 〚(1-\alpha)〛\,(1 \hat{-} n))/((1-n)T^2)$ ie ln k vs 1/T is equal to (-Ea/(2.3 R)). From the TGA it is clear that till the temperature of 140 °C there is an insignificant change in the weight of the sample. Considering this fact, the Arrhenius plot shown in Fig. 5 is drawn for a temperature range of 140 °C–400 °C only. The slope of the plot is −5.86. The y-intercept of the plot gives the value of ln A and it is found to be −5.273. From the intercept, the value of A is 5.13 e−6.

Equating the Slope with (E_a/2.3 R) the value of activation energy is calculated as 112.06 kJ/mol. The activation energy obtained is very low compared to composites of other natural fibers, which range from 155 kJ/mol to 197 kJ/mol [2]. The activation energy of the composite is lower than the activation energy observed during thermal degradation of chicken feather (200 kJ/mol to 210 kJ/mol) [28]. Chicken feather contains various proportions of different types of proteins [13]. Each component has different decomposition reaction rules with different activation energies. This would make it difficult to model the decomposition of CFF/PLA composite filament behavior considering each component, crystal structure, or chemical composition. Also, different methods of calculating activation energy for the behavior of each component should be used to predict the activation energy. When number of activation energies are less it is easier to calculate various thermal kinetics parameters [34]. For natural fibers such as Chicken feathers, the approach should be to group components with similar behavior and obtain the activation energy of the group to prepare a satisfactory degradation model.

5. Conclusion

TGA and Derivative TGA were used to investigate the thermal degradation of CFF-reinforced PLA composites. The modified Coats-Redfern method was used to determine the activation energy of the composite. The TGA and DTGA exhibit different behavior: the cellulose breaks down first, then the keratin breaks down, and finally the keratin-cellulosic compound breaks down at the end. Higher thermal stability can be confirmed by the higher temperature deterioration observed, as compared to the constituent material. A lower activation energy suggests that the constituent compounds of PLA and CFF are interacting at higher temperatures and lower energy. This feature is advantageous for the relatively low energy required creation of rigid and thermally stable polymorphs at the interfaces. A more straightforward method for manufacturing and using CFF/PLA composite filament could be developed with an understanding of thermal degradation and the effects of activation energy.

Additional information

No additional information is available for this paper.

CRediT authorship contribution statement

Adil Khan: Writing - original draft, Visualization, Validation, Resources, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Saleh Yahya Alghamdi:** Writing - review & editing, Validation, Supervision, Resources, Project administration, Funding acquisition, Formal analysis. **Ali Saeed Almuflih:** Writing - review & editing, Supervision, Resources, Project administration, Investigation, Funding acquisition. **Amal Abdulrahman:** Writing - review & editing, Supervision, Resources, Project administration, Investigation, Data curation. **Karishma M. Qureshi:** Resources, Investigation, Data curation. **Naif Almakayeel:** Visualization, Supervision, Resources, Project administration. **Mohamed Rafik N. Qureshi:** Writing - review & editing, Validation, Supervision, Resources, Project administration, Investigation, Funding acquisition, Formal analysis, Data curation, Conceptualization.

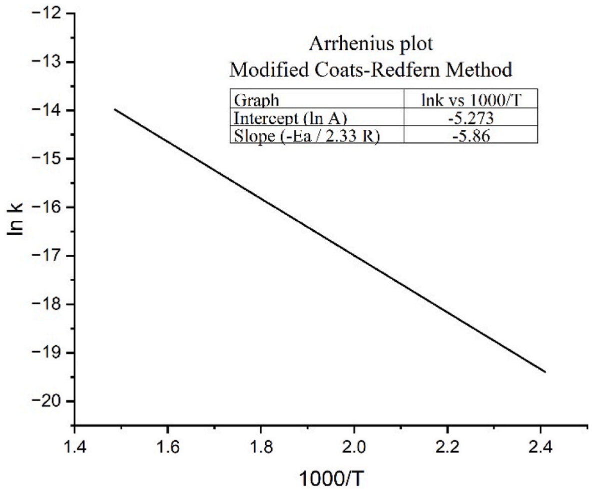

Fig. 5. Arrhenius plot using Modified Coats Redfern method.

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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