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Thermal analysis of hybrid composites reinforced with Al₂O₃ and SiO₂ filler particles

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Abstract

This work focuses on the investigation and comparison of thermal analysis of three newly prepared composite materials consisting of jute fiber and glass fiber with nanoparticles (Al₂O₃ and SiO₂). Jute fiber was embedded with glass fiber to improve the mechanical properties. Epoxy resin, particle (Al₂O₃ and SiO₂) and hardener are bonded in three weights (g) 150:0:15, 150:5:15, and 150:15:15 for composite-1, composite-2, and composite-3 respectively. Differential Scanning Calorimetric (DSC), Thermo Gravimetric Analysis (TGA) methods are utilized to identify the thermal behavior of tested composites. The TGA and DSC are utilized to measure the degradation and decomposition of the composite materials. In addition to that, the correlation between theoretical and experimental values was figured out using model equations, and the acceptable level (R²) was presented. The analytical results and the investigational results showed good agreement with them. TGA results showed that the composites-1, composites-2 and composites-3 are thermally stable until 300 °C in nitrogen. Al₂O₃ and SiO₂ enhanced mechanical properties. Most of the composites are approximately 50% of the decomposition fallen within a temperature between 350 °C and 400 °C. The result also showed that the specific heat capacity and total heat absorption during the process of composites are 0.48 J/g °C to 1.4 J/g °C and 135 J to 175 J, respectively. The addition of Al₂O₃, the DSC curve peak temperature of the composites has been diminished. The thermal stability of composite are notably reduced due to the incorporation of Al₂O₃ and SiO₂.

Introduction

New composites are introduced to the engineering field. In recent years, natural fibers reinforced with fillers in thermoplastic composite materials have been widely used [1]. These natural fibers are lighter, economical, and render much greater strength than most inorganic fillers [2–4]. Thus, new materials are indispensable for new demand in industry and face the competition [5, 6]. Glass fiber has better thermal stability which is expensive and not quite environment-friendly. Therefore, natural fibers like jute is embedded in composite due to its cheapness, availability and more environment-friendly [7, 8].

In the context of both energy security and climate change mitigation, biomass has a significant interest as a renewable energy source. As a consequence, Jute is biodegradable as well as recyclable such as it can be used to produce pyro-oil by catalytic pyrolysis [9]. Jute has been grabbed great attention to the researcher. However, the downside of natural fibers is their low thermal processing temperatures, that margin around 200 °C. These products are vulnerable to fire and it is desirable to make them fire-resistant. Thitithanasarn et al [10, 11] found
that thermal degradation resistance of jute fibre can be enriched by coating with flexible epoxy resin. Moreover, natural fibers like jute, coir, sisal, PALF (pineapple leaf fibers), banana have attracted substantial importance as reinforcing materials in polymer matrix composites. However, these fibers are highly hygroscopic in nature and show poor wetting behavior when used with the organic resin matrix [3, 12–14]. Then again, the flame retardant additives with epoxy/glass fiber composites have enriched the fire properties without significantly affecting the mechanical performance [2, 8, 15, 16].

Jute is a low-cost multicellular and lignocellulose fiber. Natural fibers have been tried to modify in many ways like modified jute via graft copolymerization with acrylamide monomer. It was found that the grafted fibers offered more resistance to biodegradation and showed higher water absorbency than their ungrafted counterparts [17–19].

Thermogravimetric analysis (TGA) is a mostly used thermal analysis technique for the characterization of both organic and inorganic materials including fibers. TGA provides quantitative results regarding the weight loss of a material’s sample as a function of temperature as well as it gives fundamental information about thermal properties of the material. It is thus of thermal characteristic to understand a new composite has an indispensable role in predicting the possible application. The glass transition interprets a couple of aspects such as change of size which is due to change in the amount of amorphous material. While jute also works as a thermal insulation. In addition to that glass fiber’s insulation capacity is enhanced more [20, 21]. It is also found that the adding of jute in the composite ameliorate the storage modulus (tan delta) which is a positive mechanical property [22–24]. Using jute enhance the composite’s glass transition temperature [25]. Glass fibers also have good thermal insulation [26]. In a certain ratio, Al2O3 and SiO2 have been used in the glass industry from a long time to improve the melting heat loss [27]. Rajaee et al. [28] showed that incorporation of additive enhances the flame retardancy, thermal stability of epoxy resin and glass fabric reinforced composites. However, TGA results demonstrate that the introduction of additives improves the thermal stability of epoxy resin at high temperatures [8, 26, 29]. After that, the addition of flame retardants to epoxy resin increases the tensile and flexural moduli [30]. The materials degradation and decomposition depending on temperature have been tested at different heating rate (5 °C/min, 10 °C/min) [1, 2, 11, 31, 32]. However, findings showed that the weight loss fraction was not affected by heating rate since it was plausibly the intrinsic property of fibers, which was not affected by heating rate [33, 24].

In this study, we used TGA to investigate the loss of weight of hybrid composite reinforced with Al2O3 and SiC to determine the thermal stability. The previous results and assumption prompted to investigate the thermal properties of jute fiber and glass fiber reinforced epoxy hybrid composites at a certain ramp (5 °C/min).

**Experimental procedure**

**Materials**

The structure of four lamina unidirectional jute and glass fiber was used to fabricate the composites reinforced with Al2O3 and SiO2 nanoparticles. Jute fibers were taken from the jute research institute of Bangladesh. Epoxy Resin, Hardener, Al2O3 nanoparticles and SiO2 nanoparticles (21 nm) were locally purchased from Dhaka, Bangladesh. All reagents were analytical grade. The following amounts (shown in table 1) of each ingredient are needed to fabricate the jute fiber and glass fiber composite.

**Fabrication process**

Two pieces of 4 m2 fiber of jute and two pieces of 4 m2 of glass were cut from the sample. After that, place the first piece of jute fabric on the table surfaced and add sufficient amount of resin and hardener (table 1), then put the glass sheet, again repeat the whole process (jute + resin + glass + resin + jute + resin). For the samples of 2 & 3, a certain amount of Al2O3 and SiO2 nanoparticles (table 1) were mixed. Precisely, the first one is made of jute and glass reinforced with epoxy resin and hardener as a binder while other two composites added the various amount of Al2O3 and SiO2 nanoparticles with the first composite.

Thermal analyzer SDT 650 (Simultaneous TGA and DSC device of TA instrument) was used to measure the thermal endurance of the three composites. Figures 1(a) and (b) show the schematic diagram and TGA machine

| Composite no.: | Reinforcement composition | Epoxy resin (g) | Hardener (g) | Al2O3 (g) | SiO2 (g) |
|----------------|---------------------------|----------------|-------------|-----------|----------|
| 1              | J + G + J + G             | 150            | 15          | 0         | 0        |
| 2              | J + G + J + G             | 150            | 15          | 5         | 5        |
| 3              | J + G + J + G             | 150            | 15          | 15        | 15       |

Table 1. Fiber orientation and amount of binders and particles.
photograph of the experiment and specimens for testing are shown in figure 1(c). For the experimental procedure, TGA and DSC tests were performed on all the investigated materials by using SDT 650 equipment in nitrogen from 50 °C to 800 °C under dynamic conditions, 5 °C/min.

Figure 1. (a) Schematic diagram of the TGA, (b) photograph of the TGA Machine (c) specimens.
Results and discussion

Thermo gravimetric analysis

Figure 2 shows TGA thermographs of the hybrid composites. It is found that composites start degrade at around 325 °C. It is revealed from TGA thermographs that the degradation temperatures of the composite-1, composite-2, and composite-3 are close to each other. From figure 2, at the first stage, the material loses H₂O, gas desorption and molecular weight solvent such as additives, crystallization water. At the second stage, the onset temperature under inert gas (Nitrogen) environment, a certain amount of thermo oxidative (a certain amount of O₂) decomposed. At the last stage, under pyrolysis process when hydrothermal carbonization occurred, there is no formation of volatile gases. At the end of this experimentaion, the tested samples are in the form of exhaust metallic oxides or inorganic salt ashes.

Thermal decomposition parameters were found from the TGA. First derivatives (DTG) and second derivatives (D²TG) curves were described below using composite-1 data at a heating rate of 5 °C/min for instance (figure 3). Yao et al [33] mentioned in their study that the extrapolated onset temperature of decomposition (Tₒ) was obtained by extrapolating the slope of the DTG curve in correspondence with the first local maximum D²TG curve and down to the zero levels of the DTG axis.

From TGA, first derivatives (DTG) and second derivatives (D²TG) curves were used to examine thermal decomposition parameters. For Composite-1, data was described at a heating rate of 5 °C/min as shown in figure 4. The onset temperature of decomposition (Tₒ) was found from this extrapolated curve. The peak temperature (Tₚ), where the maximum decomposition rate attained was calculated by the first derivative of TG (DTG).

Maximum decomposition rate was obtained at DTG peak which is denoted with Tₑ. The weight loss related to Tₑ, denoted with Wₑ, is shown in table 2. The final, tailing section means the rest of the cellulose decomposition. The last reactions were mostly the decomposition of lignin and tar or char from main components decomposition. The end transition temperature is Tₑ and Wₑ is the weight loss corresponding to the Tₑ.

Tₒ = Onset temperature, Wₒ = Weight loss at onset Temperature, Tₑ = Peak temperature, Wₑ = Weight loss at peak temperature, Tₑ = End temperature of transition, Wₑ = Weight loss at Tₑ, 

Tₑ − Tₒ = Transition period, Wₑ − Wₒ = Weight loss at transition period.

The decomposition of selected composites is represented in table 2. The onset decomposition temperature (Tₒ) specifies a range about 325 ± 5 °C for all composites. Where the highest value is found for the composite-3. Weight loss until onset temperature (Wₒ) was detected around 10% for all composites, while composite-1 exhibited higher percentage which signifies a comparatively faster primary decomposition than other samples. The peak temperature (Tₑ) shows the supreme decomposition rate of composites occurred in a range of 342 ± 3 °C, while composite-1 has the highest value (402.97 °C) due to the presence of metal particles reduced the peak temperature in the composite. The weight loss by decomposition (Wₑ) at this period, reached nearly
22 ± 2% for composite-2 and composite-3 while composite-1 reaches 68.73%. Perhaps the presence of an inorganic substance in the surface layer of composite-2 and composite-3 which experienced smaller decomposition than composite-1. From peak to the end of the transition temperature for composite-2 and composite-3 have rapid decomposition in a short temperature range. Most composites finished almost 55%
Higher residue weight due to higher Al and Si contents. The temperatures and weight loss difference between end and onset points, $T_i$ to $T_e$ and $W_i$ to $W_e$ respectively, showed again that main thermal decomposition fraction (around 60%) happened in a temperature range of about 60 to 80 °C (325 to 395 ± 10 °C in terms of extrapolated temperatures) for the most composites.

For Composite-1 (figure 4(a)), this composite decomposes in three stages; in the first stage it has lost a significant amount volatile. In the first derivative, we observed that the peak transition was at 402.97 °C. Most of the composite weight has been lost in a glass transition period, which starts at 323.4 °C. After the burning, in the third stage there only remains the ash content (carbon black, metallic particles were inserted into composite during fabrication).

The new composition decomposes in three stages, there was high devolatilization in the first stage, which can be observed from time derivative at 137 °C. Due to presence of Al2O3 and SiO2 nanoparticles, the glass transition temperature at 327 °C is observed which is earlier than composite-1. In addition to this, the residual product is much more than composite-1.

Composite-3 was made of 15 g Al2O3 and 15 g SiO2. Because of extra alumina and silica, the heat flow was higher, and as a result, the decomposition rate was faster as shown in figure 4(c).

Composite with jute, glass, and resin shows better performance against heat. However, composites absorb heat faster and resulting loss of weight faster due to the use of particles. It can be concluded that the number of particles increases, the weight loss was quicker and the amount of residue is higher.

These polynomial equations in table 4 represent the prediction of heat flow through the mass at any temperature $x$. The differences between the theoretical and experimental value are 0.9711, 0.9633 and 0.9633 for composite-1, composite-2, and composite-3, respectively.

**Differential Scanning Calorimetry (DSC) analyses**

Until the end of volatilization whereby dissolved constituents are vaporized, the three composites have taken the heat at almost similar rate though they are different in compositions. While heat flow trend was lower for composite-1, after the first stage (around 325 °C), the heat flow of composite-2 and 3 is lower than composite-1.

Figure 5 represents the DSC curve of the composites. The addition of compatibilizer into the composites, with different weight concentrations of Al2O3 and SiO2, decreased the glass transition and melting point of the composites. As shown in tables 5 and 6, the glass transition temperature ($T_g$) of the composite-2, composite-3 decreased to 356.99 °C, and 360.43 °C from 362.11 °C. The material loss at this point of composite-1,

### Table 2. Weight loss at various transition temperatures.

| Materials    | $T_m$ (°C) | $W_m$ (%) | $T_e$ (°C) | $W_e$ (%) | $T_i$ (°C) | $T_f$ (°C) | $W_i$ (%) | $W_f$ (%) |
|--------------|------------|-----------|------------|-----------|------------|------------|-----------|-----------|
| Composite-1  | 323.38     | 14.406    | 402.97     | 68.73     | 403        | 69.76      | 79.64     | 54.76     |
| Composite-2  | 327.26     | 11.903    | 345.78     | 24.96     | 393.89     | 58.901     | 66.63     | 46.998    |
| Composite-3  | 328.29     | 9.099     | 345.46     | 21.308    | 389.89     | 51.852     | 61.6      | 42.753    |

### Table 3. Weight loss at transition period.

| Transition temperature | $T_m$ (°C) | $T_e$ (°C) | $T_i$ (°C) | Total weight loss at the process (%) | Residual weight (%) at 800 °C |
|------------------------|------------|------------|------------|-----------------------------------|-------------------------------|
| Composite-1            | 323.4      | 382        | 403        | 82.5                              | 17.5                          |
| Composite-2            | 327.3      | 358        | 393.9      | 76.0                              | 25.9                          |
| Composite-3            | 328.3      | 359        | 389.9      | 63.7                              | 36.3                          |

### Table 4. TGA curve fitting.

| No. of composite | Model equation                                                                 | Weight loss at $T_i$ (%) | R²   |
|-----------------|--------------------------------------------------------------------------------|--------------------------|------|
| Composite-1     | $y = 4e^{-11.5x} - 6e^{-0.8x} + 4e^{-5.3x} - 0.0112x + 12.98x + 48.631$         | 68.73                    | 0.9711|
| Composite-2     | $y = -2e^{-11.5x} + 3e^{-0.8x} - 2e^{-5.3x} + 0.0042x^2 - 0.383385 + 106.54$   | 24.96                    | 0.9633|
| Composite-3     | $y = -2e^{-11.5x} + 3e^{-0.8x} - 2e^{-5.3x} + 0.0042x^2 - 0.383385 + 106.54$   | 21.308                   | 0.9633|

* $y$ = Heat flow (mW.min), $x$ = Temperature, $R^2$ = Regression variance, $T_i$ = Glass transition temperature.
composite-2, and composite-3 is 3.5161 %/min, 4.4151 %/min, 3.95 %/min respectively. The peak degradation temperatures are 368.3 °C, 345.78 °C, 345.46 °C. The material losses at peak temperature (Tp) are 5.1334, 5.6546 and 4.9839 %/min respectively. Padal et al [23] showed that the transition temperature of jute nano-fiber composites was at 70 °C to 75 °C. The glass transition (Tg) of composite-1 at 363.18 °C. However, the addition of filler particle to reinforced composites decreased the Tg value of the composites. It is noted that Tg of composite-3 was at 123 °C, indicating that the temperature Tg of the composite decreased around 3 °C as compared to a neat composite-1 of jute fiber and glass fiber.

Because of the presence of inorganic particles in the composites, the heat capacities (shown in table 6) decreased significantly for composite-2 and composite-3. It is noticeable that the heat capacity of the same composites are enhancing with the rise of temperature which is symbolized Tg shown in table 4. The heat capacity of new composites has overlapped with few common engineering materials such as Concrete, Timber (alder), Potassium, Paper, summarised in table 7. Thus, the new composites can be a substitute for conventional usable materials which have similar heat capacity and thermal properties with composite. Those are the endothermic reaction. Due to continually phase change, the specific heat has also changed. From the column (table 6) of specific heat capacity, it is observed that increasing the amount of alumina and silica dioxide reduces the specific heat capacity. Also, for the same composite, it can be concluded that specific heat capacity is

![Figure 5. DSC curves, weight loss and total heat absorption variation with the variation of temperature for composite-1, 2 and 3.](image)

| Table 5. Temperature at 5%, 10% weight loss and glass temperature. |
|-------------------------|-------------------------|-------------------------|-------------------------|-------------------------|
| Materials               | aT5% (°C)               | bT10% (°C)              | bTp (°C)                | cWp (%/min)             |
| Composite-1             | 227                     | 297                     | 402.3                   | 362.1                   |
| Composite-2             | 279                     | 320.8                   | 345.8 °C               | 357                     |
| Composite-3             | 301.5                   | 327.5                   | 345.5 °C               | 360.4                   |

- a T5% = Temperature at 5% weight loss, T10% = Temperature at 10% weight loss.
- b Tp = Peak Temperature, Wp = Weight loss at peak Temperature.
- c Tg = Glass Transition Temperature, Wg = Weight loss at glass transition point.

| Table 6. Heat capacity of the composites at Tg and 50 °C. |
|---------------------|---------------------|---------------------|
| Materials           | Heat capacity (J/g °C) at 50 °C | Glass transition temperature, Tg (°C) | Heat capacity at the glass transition (J/g °C) |
| Composite-1         | 1.48                | 362.11              | 23.52                 |
| Composite-2         | 0.66                | 356.99              | 09.17                 |
| Composite-3         | 0.48                | 360.43              | 07.12                 |
augmented noticeably with raising the temperature. Total heat consumption by the individual composites during degradation is mentioned in Table 8.

Non-linear equations (Table 9) have been drawn to predict the lifetime of composites in the SDT 650 machine. The satisfactory levels of those equations of composite-1, composite-2, and composite-3 are 0.9955, 0.9938, and 0.9735, respectively.

Conclusion

TGA and DSC investigation reveals that the glass fiber and jute fiber reinforced with filler particles have given the expected results. The presence of Al₂O₃ and SiO₂ nanoparticles in the composite shows thermal stability. However, if the recycling ability is considered, then the shorter transition period is better. The experimental data is used to compare the three composites made of the different amount of alumina oxide and silica dioxide. Due to adding the Al₂O₃ and SiO₂ nanoparticles transition and melting point have been decreased significantly that means alumina is inversely proportional to thermal stability. Therefore, it can be concluded that if the composites are desired to use beyond certain temperature or thermal stability condition, then some non-metal particle should use rather than metal particle like alumina. Thus, TGA and DSC results revealed that additives alter the degradation profile of the hybrid composites. From this study, researcher will identify characterization of phase behavior and chemical stability of the hybrid composites. This analysis provides valuable information regarding their capacity to identify structural components.

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