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Dynamic mechanical and thermal properties of flax/bio-phenolic/epoxy reinforced hybrid composites

Abstract

Objectives:

The influence of flax fibre loading on the dynamic thermal and mechanical characteristics of composites was investigated in this work.

Methods:

Flax composites were fabricated at 30 (F-30), 40(F-40) and 50(F-50) wt% fibre loading. As a control, a bio-phenolic/epoxy polymer blend (P-20) containing 20% bio-phenolic was made.

Results:

The findings suggest that the incorporation of flax into the composites led to an enhancement in the dynamic mechanical properties of the experimental sample. The glass transition temperature (Tg) was identified via dynamic mechanical analysis to be between 60°C to 85°C. Aside from that, the cole-cole plot revealed a heterogeneous mixture of fabricated samples, with strong fibre-to-matrix adhesion. Thermogravimetric analysis (TGA) showed adding flax to the composite reduces thermal stability.

Conclusions:

With the integration of flax as reinforcement, it could be asserted that the composites' dynamic mechanical properties improved while their thermal stability reduced.

Keywords: Flax Fibre, Epoxy Resin, Bio-phenolic resin, dynamic mechanical analysis, Thermal properties.

1. Introduction

Fibre-reinforced polymer composites (FRC) are employed in a variety of applications, like in automobiles, aircrafts, and building parts. This is due its acceptable mechanical properties, lightweight, high impact strength, chemical/corrosion resistance, high fatigue resistance, and long-life expectancy (Chegdani et al., 2020, Sathees Kumar et al., 2021). Natural fibre has a lower environmental impact than synthetic fibre since it is renewable, sustainable, produce fewer greenhouse gases, and decreases dependency on petroleum-based resources (Banik et al., 2017, Potluri et al., 2017).

In 2019, the production of natural fibre alone was approximately 33 million tons, while the combined production of synthetic and natural fibres was estimated to reach 110 million tons which indicated that natural fibre accounted for 30% of the total production fibre (Townsend 2020). Table 1 summarizes several of the natural fibres global production. Ramie, jute and kenaf the examples of plants that grow to harvest fibre, while oil palm, sugarcane bagasse, and flax are obtained from agricultural waste. Natural fibre-based polymer composites are employed in numerous industries, including furniture, construction, automotive, railway coaches, packaging, and aerospace (Mishra et al., 2020, Venkatesan and Bhaskar 2020). In these applications, natural fibres such as flax, bagasse, jute, abaca, coir, kenaf, sisal and oil palm fibre have been employed (Mishra et al., 2020, Venkatesan and Bhaskar 2020). Natural fibres intrigue scientists due to their modest weight, superior specific strength and modulus, nonabrasiveness, minimal cost, and biodegradability (Faruk et al., 2014, Nadlene et al., 2015). Flax was cultivated primarily in France, Belgium and Canada to harvest it fibre and extract the oil from flax seed (Uppal et al., 2022). Flax is a low density fibre with high specific stiffness, cheap and recyclable (Bos et al., 2002, Summerscales et al., 2010). The use of the flax fibre depending on it grade. The high quality of the fibre was used in textile industry while lower quality was used in composite industry (Uppal et al., 2022). Flax fibre can improve the dynamic response of the composite due to its high crystalline structure, which enhances the interfacial adhesion among both adjoining fibre layers as well as consequently enhance polymer motions inside the composites.(Bos et al., 2002). The investigators looked at the viability of flax as a reinforcement on various polymer matrices.

It is critical to understand the maximum temperature that fibres can withstand throughout the production or fabrication process, as natural fibre is temperature sensitive. The thermostability of natural materials can be altered by temperature, which can result in the contraction or expansion of radiation heat, as well as an expansion in the volume and percentage of moisture absorption, which causes the fibres to swell (Wang et al., 2005). Natural fibre degrades at temperatures exceeding 180 °C, hence natural fibre-reinforced polymer composites fabricated at this temperature will have poor mechanical properties (Gassan and Bledzki 2001). Researchers have reported that the flax fibres degraded at a temperature of around 200 °C (Kannan et al., 2013). According to the available literature, no research has been conducted on the dynamic mechanical and thermal characteristics of flax-reinforced bio-phenolic/epoxy composites. This study's primary objective is to assess the influence of flax loadings upon this dynamic mechanical as well as thermal characteristics of composites. The fabricated polymer blend with 20 wt% bio-phenolic was based on the results of the earlier studies. (Ismail et al., 2021, Ismail et al., 2023). The previous study reported the mechanical as well as physical characteristics of flax reinforced bio-phenolic/epoxy composites (Ismail et al., 2022).

2. Materials and Method

2.1 Materials

The plain weave flax fabric was procured from commercial textile company in Guangdong, China, and utilized as reinforcement. The moisture content, weight and yarn count for the flax fabric is 7.20%, 160 GSM and 14 × 14 respectively. Composites were made with a polymer combination that included bio-phenolic and epoxy. Bio-phenolic resin came from Bangalore, India. The epoxy resin and hardener came from Selangor, Malaysia. Teflon sheet was supplied by Evergreen Sdn Bhd from Selangor, Malaysia.

2.2 Composites Preparation

The experimental samples were fabricated with a dimension of 150mm × 150mm. As a control, a polymer blend containing 20wt% bio-phenolic was produced. Table 2 shows the flax loading and the number of flax layers used in the fabrications. The fabrication method and materials were illustrated in Figure 1.

3. Testing of the materials

3.1. Dynamic mechanical analysis (DMA)

The specimen measuring 60 mm x 12 mm x 3 mm was evaluated in line with ASTM D 4065 using DMA 800 to determine its mechanical properties. Three-point bending mode and 1 Hz frequency were used. The experimental composites were heated from 30 °C to 150 °C at 10 °C/min.

3.2. Thermogravimetric Analysis (TGA)

Thermogravimetric Analysis was utilized to investigate the thermal decomposition profile of the samples as per ASTM E1131-03 (2003). All these measurements were taken with a Mettler Toledo 851e (Schwerzenbach, Switzerland). At a rate of 50 mL/minute, between 40 and 45 mg of samples were placed in the alumina crucible and subjected to pyrolysis in an environment containing nitrogen. The experimental composites were heated from 30 °C to 800 °C at a rate of 10 °C/min.

4. Results and Discussion

4.1. Dynamic Mechanical Analysis

4.1.1. Storage Modulus

The storage modulus of the samples is depicted within Figure 2. As the temperature rises, the storage modulus gradually decreases as the temperature increases, the composite undergoes three distinct phases: the glassy region, the transition region, and the rubbery region. The polymer molecules behave differently in each region. The compounds are densely packed and have little mobility at low temperatures (glassy region), ensuing in a high storage modulus. As the temperature raises, molecules absorb the energy and undergo intrinsic motion. When the temperature approaches the Tg, the energy is absorbed, causing the chain to slowly detangle which increases the component's free volume and mobility. As a result, dramatically decrease of storage modulus was shown in the glass transition region. While the storage modulus of all composites remains constant in the rubbery region. The graph in Figure 2 demonstrates an improvement in storage modulus by the incorporation of flax. This illustrates that adding reinforcement to the polymer matrix improved the material's ability to bear mechanical constraints while retaining recoverable deformation (Jawaid et al., 2012).

The storage modulus is enhanced by expanding the flax loading up to 40% wt and the storage modulus was reduced when flax loading increased to 50wt%. The trend of the storage modulus in this study showed a similar pattern to the flexural modulus which was reported in the prior research (Ismail et al., 2022). The storage modulus of P-20 was 2210 MPa at 30°C, but it dropped to 8 MPa once it approached the Tg. The results indicated that when a material is passed through Tg, the reinforcement increases the storage modulus of the samples. The findings showed that the reinforcement improved its storage modulus when a material passes through Tg. F-50 noted the uppermost storage modulus at 120 °C which is 496 MPa, while F-30 and F-40 recorded a storage modulus at 120 °C around 199 MPa and 467 MPa respectively.

The efficiency of interfacial interaction in between matrix and the fibres was determined using the effective coefficient, C. Equation 1 was used to calculate the efficiency coefficient based on the storage modulus of the glassy region (30°C) and rubbery region (120°C). Where, E'_G and E'_R are storage modulus at the glass and rubbery regions respectively. The effectiveness coefficient is inversely related to the efficiency of fibre-matrix interfacial interaction. Table 2 shows the storage modulus (at glass and rubbery region) and the effectiveness coefficient of the experimental sample. In comparison to F-30 and F-40, F-50 has the lowest effectiveness coefficient, indicating that F-50 has powerful interfacial interaction among fibres and matrix. The finding is agreed alongside the earlier suggestion that interfacial adhesion is better for F-50 compared to F-30 and F-40(Ismail et al., 2022).

$$C = \frac{\frac{E'_G}{E'_R}(composite)}{\frac{E'_G}{E'_R}(matrix)} \qquad \text{Equation 1}$$

4.1.2. Loss Modulus

The loss modulus versus temperature of the composites was depicted in Figure 3. P-20 exhibited the lowest peak for the loss modulus. The loss modulus of the experimental sample increase as temperature increases until it reaches the Tg, after that the loss modulus of the experimental sample will decrease with increasing in temperature. The accumulation of flax to the experimental polymer caused the peak of the loss modulus curve to rise and the width of the curve to expand. This is because both chain segment and free volume have increased (Woo et al., 1991). Furthermore, the difference in physical condition between the polymer matrix adjacent to the fibres and the remainder of the matrix reduced molecular mobility. Additionally, the reinforcement of fibres increased the internal fraction and enhances energy dissipation (Jawaid et al., 2012, Sharma et al., 2021). F-40 and F-50 had nearly identical peak values for loss modulus, 454 MP and 459 MPa, respectively. While F-30 showed the lowest value for the peak of loss modulus among the composites with reinforcement which is 372 MPa.

4.1.3. Damping Factor (Tan Delta)

Figure 4 illustrates the damping factor of F-30, F-40, F-50 and P-20. Tan delta denotes the association between the storage modulus (E') and the loss modulus (E") properties. The greater the Tan delta peak, more and more energy is lost, resulting in significant non-elastic deformation. A minimal value, on the other hand, indicates that the material is much more elastic. Tan delta increased as temperature increased, reaching an optimum in the transition region and continuously declining in the rubbery region. The low value of tan delta in the glassy region is due to the position of the molecules which close to each other's in the composites and their freezing condition (Anand et al., 2018). The molecule absorbs adequate energy to gradually untangle the chain as the temperature rises until it reaches the transition region, which leads to an increase in free volume and molecular mobility. In contrast, the molecules' chains are disentangled in the rubbery region, allowing the molecules to flow easily and without resistance, leading to a low damping factor. The maximum peak of the Tan delta is shown in Table 3. The highest peak for tan delta was observed in P-20 compared to flax composites, and two peaks were observed. P-20 has the largest peak in the tan delta curved, indicating greater molecular mobility, larger energy dispersion, and greater viscosity. Due to the presence of bio-phenolic in the system, which has a higher Tg than epoxy, there are two peaks in P-20.

However, when reinforcement was added, only one peak was observed due to the reinforcement reducing the molecules' mobility in the composites. The peak for tan delta decreases as the flax loading increases. The decrease in the peak of the Tan delta is explained by the fact that as fibre loading increases, molecular mobility and free volume decrease. According to de Medeiros et al., (2005) the mass fraction and orientation of fibre in composites affected the free volume and molecular mobility. Incorporation of particle in matrix has stop the molecules' segments from moving around, which can be good evidence, reinforcement/filler embedded in polymer chain diminished the mobility of molecule and decreased the friction between them (Mohammed et al., 2017). Additionally, according to Jawaid et al., (2012) reinforcement in polymer composites performed as a deterrent, limiting the polymer's chain mobility, resulting in decreased flexibility and degree of molecular motion, and thus decreased its damping properties. Furthermore, a lower value of the Tan delta peak indicates the contact between fibres and matrix was enhanced. Among all the composites fabricated, F-50 has better fibres and matrix adhesion. A similar trend was shown for the effectiveness coefficient (C) which discuss in the storage modulus.

4.1.4. Glass Transition (Tg)

With the help of dynamic mechanical analysis, the peak of the tan delta curve, the loss modulus, or the half height of the storage modulus curve can be used to figure out the Tg of a polymer composite (Venkategowda et al., 2021). The Tg determined by loss modulus is more convincing and realistic than the value determined by the Tan delta's peak. The Tg is illustrated in Table 3 using the peak of tan delta and loss modulus. P-20 exhibited two peaks for tan delta but only one for loss modulus. The Tg calculated from the loss modulus peak was found to be lower than the tan delta peak. The Tg was enhanced by raising the flax loading to 40wt%. However, increasing the flax loading to 50wt% reduced the Tg, which is now lower than the Tg of P-20.

4.1.5. Cole-cole

The homogeneity of the polymer matrix and reinforcement can be determined using cole-cole plots (Karaduman et al., 2014). The graph can be used to explore how structural changes in polymers occur when reinforcement is introduced (Harris et al., 1993). In order to investigate the correlation between the storage modulus and the loss modulus, a cole-cole plot was constructed and analyze. The shape of the cole-cole plot can be handled to predict the uniformity of the polymer matrix and reinforcement. An even and semi-circular arc indicates a homogeneous polymer composite, whereas an imperfect or elliptical curve indicates nonhomogeneous dispersion and heterogeneity in polymer composites (Mohammed et al., 2017, Chee et al., 2019). Additionally, ellipsoidal curves show a insistent adherence concerning the fibre and matrix. The cole-cole plots for F-30, F-40, F-50 and P-20 are shown in Figure 5. Cole-cole plots revealed imperfect semi-circles for all composites, indicating the system's heterogeneity. The shape of the curve P-20, on the other hand, it indicates that the bio-phenolic and epoxy molecules is a homogeneous mixture. Cole-cole plots demonstrated a broad curve as the fibre loading increased. This indicate that the fibre and matrix adhered to one another reasonably well. According to the curve, fibre and matrix adhesion is optimal at a fibre loading of 40 wt%. This can be seen with the curved of F-40 is broader compared to other composites. The finding was agree with the finding on the flexural properties of the same composite reported in previous study (Ismail et al., 2022) which showed that F-40 has the highest flexural strength. Similar findings were reported in Kumar et al., (2020) investigation which showed after fibre loading raise up to 45wt% the cole-cole curved become broaden and indicate the effective interfacial interaction amongst fibres and matrix.

4.2. Thermogravimetric Analysis (TGA)

The effect of various flax loadings on the thermal properties of bio-phenolic/epoxy was determined using thermogravimetric analysis. The relationship between weight loss and temperature of F-30, F-40, F-50 and P-20 is depicted in Figure 6(a), While Figure 6(b) depicts the first derivative curve of thermogravimetric (DTG) of F-30, F-40, F-50 and P-20. According to the curves in Figure 6(a) all composites degrade in a single step between 300°C and 550°C. This can be proved with a single peak shown in the DTG curve for all composites. The temperatures at which the experimental sample lost 5%, 25% and 50% of their total weight are shown in the table, as well as the temperature at which the residue decomposed at 800°C. The 5% total weight loss of flax-reinforced composites was observed to occur below 300°C. While the P-20 reached temperatures of over 300°C. This occurred as a result of the evaporation of inherent moisture and water absorbed physically by the fibres (Monteiro et al., 2012). When the temperature of decomposition was measured at various weight losses, it was discovered that increasing the flax loading lowers the temperature of decomposition. This indicates that the supplement of flax has caused a massive drop in the thermal resilience of composites. The decrease in thermal stability was caused by the significantly lessened thermal stability of natural fibres correlated to the polymer matrix (Chee et al., 2019).

5. Conclusion

To assess the dynamic mechanical and thermal properties, different loadings of flax-reinforced bio-phenolic/epoxy were fabricated. The addition of flax to experimental sample polymer blends demonstrated advancement in storage modulus, loss modulus and damping factor. The cole-cole plot denoted that the samples are heterogeneous, and the fibres adhered well to the polymer matrix. When flax was added as reinforcement, the good interfacial interaction among fibres and polymer matrix was derived in intensification in the storage modulus and loss modulus of the samples. However, the use of flax as reinforcement decreased the composites' thermal constancy. After reinforcing with flax, the decomposition temperature of the experimental sample decreased compared to a P-20. The findings of this study as well as a previous findings were used to assess the qualities of flax composites at various fibre loadings, and the optimal fibre loading will serve as the basis to explore the characteristics of flax/carbon/kevlar hybrid composites with the intention of developing a ballistic helmet.

Conflict of Interest

Authors declared no conflict of interest.

Data Availability

This article contains all data generated or analysed during the course of this study.

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Figure Caption

Figure 1. Fabrication method

Figure 2: Storage modulus of F-30, F-40, F-50 and P-20.

Figure 3: Loss modulus of F-30, F-40, F-50 and P-20.

Figure 4: Damping factor of F-30, F-40, F-50 and P-20

Figure 5: Cole-cole plot of F-30, F-40, F-50 and P-20.

Figure 6: TG and DTG plot of F-30, F-40, F-50 and P-20.

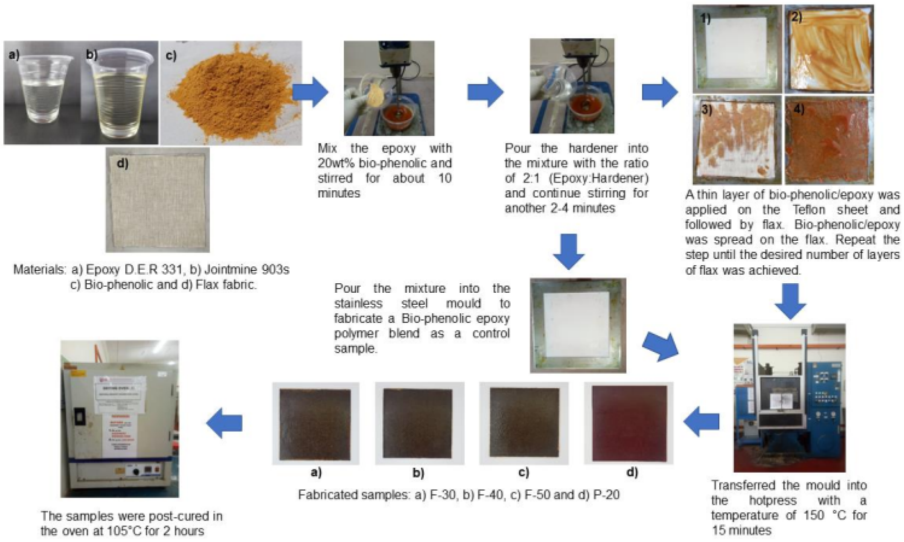
Table Caption

Table 1: Natural fibre manufacturing on a global scale(Townsend 2020)

Table 2: Flax loading, storage modulus (at the glass and rubbery region) and effectiveness coefficient of the composites

Table 3: Maximum peak of tan delta and glass transition temperature

Table 4: Decomposition temperature for 5%, 25% and 50% of total weight loss of the composites and the residue at 800°C



Materials: a) Epoxy D.E.R 331, b) Jointmine 903s c) Bio-phenolic and d) Flax fabric.

Mix the epoxy with 20wt% bio-phenolic and stirred for about 10 minutes

Pour the hardener into the mixture with the ratio of 2:1 (Epoxy:Hardener) and continue stirring for another 2-4 minutes

A thin layer of bio-phenolic/epoxy was applied on the Teflon sheet and followed by flax. Bio-phenolic/epoxy was spread on the flax. Repeat the step until the desired number of layers of flax was achieved.

Pour the mixture into the stainless steel mould to fabricate a Bio-phenolic epoxy polymer blend as a control sample.

Transferred the mould into the hotpress with a temperature of 150 °C for 15 minutes

Fabricated samples: a) F-30, b) F-40, c) F-50 and d) P-20

The samples were post-cured in the oven at 105°C for 2 hours

Figure 1: Fabrication method

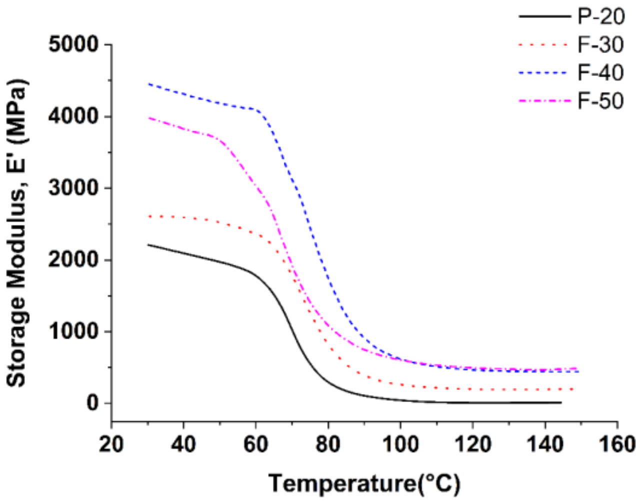


Figure 2: Storage modulus of F-30, F-40, F-50 and P-20.

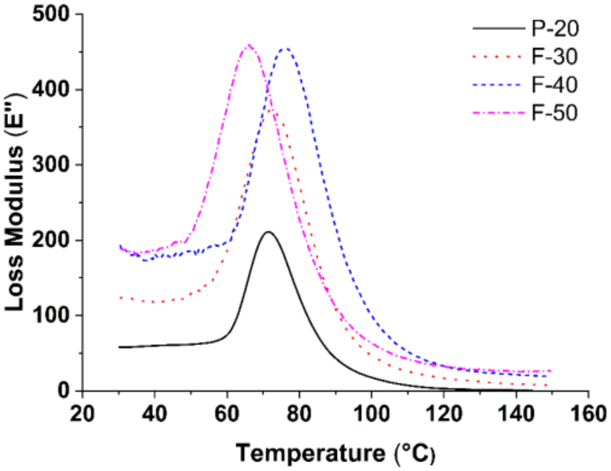


Figure 3: Loss modulus of F-30, F-40, F-50 and P-20.

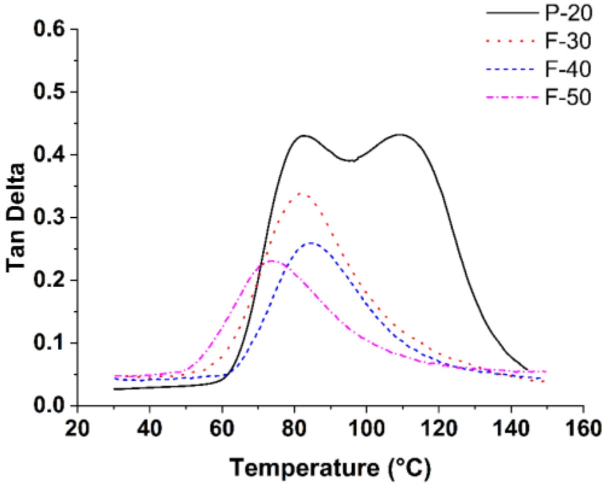


Figure 4: Damping factor of F-30, F-40, F-50 and P-20.

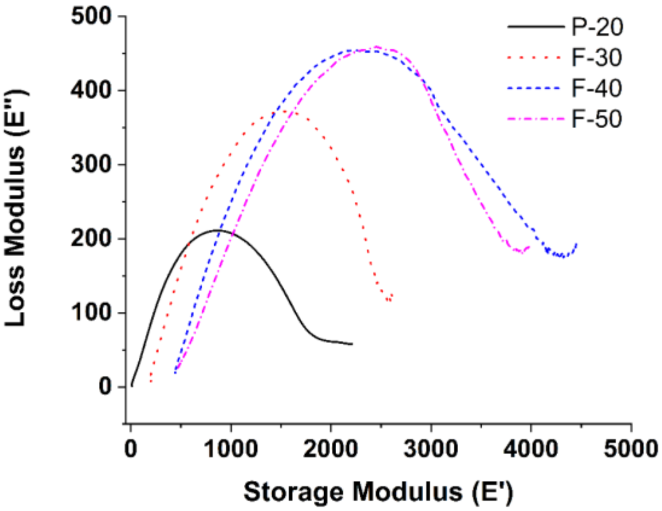


Figure 5: Cole-cole plot of F-30, F-40, F-50 and P-20.

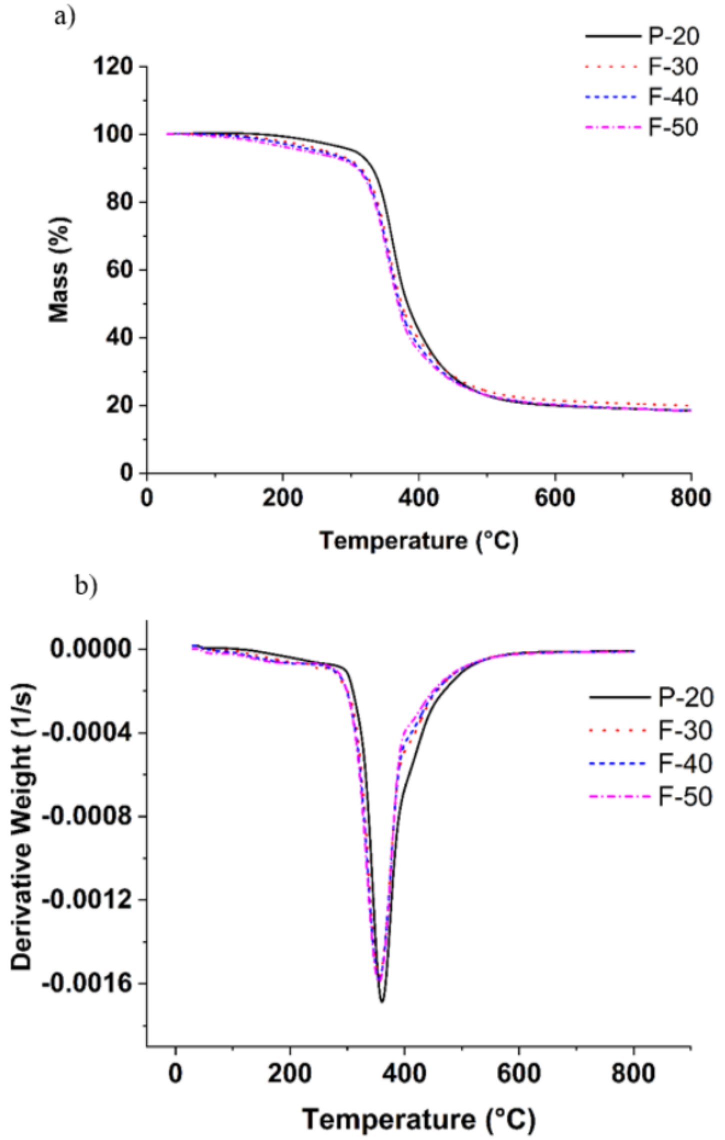


Figure 6: TG and DTG plot of F-30, F-40, F-50 and P-20: a) TG, b) DTG.

Table 1: Natural fibre manufacturing on a global scale(Townsend 2020)

Natural Fibre	World Production (×10^3 Tons)
Flax fibre and tow	790
Ramie	100
Jute-Kenaf-Allied fibres	3200
Cotton lint	25,960
Coir	1,000

Table 2: Flax loading, storage modulus (at the glass and rubbery region) and effectiveness coefficient of the composites

Label	Flax loading (wt%)	Number of Layers	Storage modulus at Temperature 30°C, E'_G (MPa)	Storage modulus at Temperature 120°C, E'_R (MPa)	Effectiveness coefficient, C
P-20	0	-	2205	8	-
F-30	30	6	2609	199	0.048
F-40	40	8	4450	467	0.034
F-50	50	10	3978	496	0.029

Table 3: Maximum peak of tan delta and glass transition temperature

Type of Composites	The peak of Tan delta	Tg from Tan delta (°C)	The peak of loss modulus (MPa)	Tg from Loss Modulus (°C)
P-20	0.43/0.43	82.55/ 109.23	210.97	71.33
F-30	0.34	82.92	372.07	73
F-40	0.26	84.58	454.27	76.33
F-50	0.23	73.92	459.22	66.08

Table 4: Decomposition temperature for 5%, 25% and 50% of total weight loss of the composites and the residue at 800°C

Type of composites	T (°C) at 5 %	T (°C) at 25 %	T (°C) at 50 %	Residue at 800 °C (%)
P-20	306.12	354.5	383	18.45
F-30	265.17	345.67	374.5	19.92
F-40	254.5	343.5	371.33	18.4
F-50	235.83	341.68	369	18.32

Note* Temperatures are given based on total weight lost at 5%,25% and 50%

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