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Investigating the moisture content of flax fibre reinforced composite materials

Keten elyaf takviyeli kompozit malzemelerin nem muhtevalarının incelenmesi

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ABSTRACT

Increasing environmental consciousness, triggered by global climate change awareness, has found a response in the composite material industry and has pushed the industry representatives to search for environmentally friendly alternatives to conventional materials. To reduce the carbon footprint and minimize the damage to nature, the preference for natural fibres instead of synthetic fibres can be considered a step taken in this context. Today, it is possible to see natural fibre applications in many industrial products, including automobile interior parts.

The purpose of using flax fibre in composite materials is not different from conventional fibres, however, their hydrophilic characteristics make flax fibre composites sensitive to temperature and the humidity of the surroundings. This study aims to investigate the moisture content of flax fibre composites as well as their hybrids with E-glass fibres at room temperature by using thermogravimetric analysis (TGA). It is observed that flax fibre samples have a moisture content of 4.9%, while E-glass samples have only a moisture content of 0.5%. The hybrid samples lay between these two values having a moisture content of 2.5%.

Keywords: Flax fibres, Moisture content, Hybrid composites, TGA

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ÖZET

Küresel iklim değişikliği fakındalığının tetiklemesiyle artan çevre duyarlılığı, her sektörde olduğu gibi kompozit malzeme sektöründe de karşılık bulmuş ve sektör temsilcilerini çevreci çözümler araştırmaya itmiştir. Karbon ayak izini düşürmek ve doğaya verilen zararı minimuma indirmek için konvansiyonel malzemeler olan sentetik elyafların yerine doğal elyaflar tercih edilmeye başlanması bu bağlamda atılmış bir adım olarak değerlendirilebilir. Otomobil iç parçalarının da dahil olduğu birçok endüstriyel üründe doğal elyaf uygulamaları görmek mümkündür.

Kompozit malzemelerde keten elyafın kullanılma amacı geleneksel elyaflarda olduğundan farklı değildir; ancak keten elyafların hidrofilik karakteristiği, bu elyafın kompozitlerini sıcaklığa ve çevrenin nemine duyarlı hale getirir. Bu çalışma, termogravimetrik analiz (TGA) kullanarak keten elyaf takviyeli kompozitlerin ve bu elyafların cam elyafla yaptığı hibrit kompozitlerin oda sıcaklığında sahip oldukları nem miktarını araştırmayı amaçlamaktadır. Keten elyaf numunelerin nem muhteviyatları %4.9 olarak bulunurken, bu değer cam elyaf numuneler için %0.5 olarak bulunmuştur. Hibrit numunelerin nem muhteviyatları bu iki değerin arasında %2.5 olarak bulunmuştur.

Anahtar sözcükler: Keten elyaf, Nem oranı, Hibrit kompozitler, TGA

1. INTRODUCTION

Fibre-reinforced composite materials, which allow the production of complex structures thanks to their easy workability, have also become the preferred materials in the maritime field thanks to their high corrosion resistance (Bulut and Erdoğan, 2011). However, the energy consumed to produce synthetic fibres used today, and therefore the amount of CO_2 released to nature has begun to be questioned due to increasing global warming awareness. The energy consumed for the production of one kilogram of the most commonly used fibres today, such as carbon fibre and E-glass fibre, is approximately 500 MJ (Zhang *et al*., 2020) and 54.7 MJ (Joshi *et al*., 2004). In terms of the global warming indicators, the corresponding carbon emissions are 36 and 2.7 CO_2 kg/kg, respectively (Boegler *et al*., 2014).

The fact that natural fibres have been used instead of synthetic fibres in this period of increasing environmental and global warming effects shows that environmental awareness has also found a response in the field of composites. The stems of plants such as flax, jute, ramie, and sisal are processed into fibres and used as reinforcement material in composite materials (Ashori, 2008). Especially in the interior panels of automobiles, where lightweight is required to reduce fuel consumption (Khalfallah *et al*., 2014), the applications of flax and jute fibres are increasing day by day. If the values given for carbon fibre and E-glass fibre are compared with flax fibres, the environmental impact of the situation is illustrated more clearly: The energy required to produce one kilogram of flax fibre is 9.55 MJ (Joshi *et al*., 2004) while the corresponding global warming indicator is -1.4 MJ (Boegler *et al*., 2014). This means that while the flax plant is developing photosynthesis, aside from releasing carbon during respiration, it releases O_2 to nature and captures carbon from the atmosphere.

Switching from synthetic composite materials used in many fields to natural composite materials that are completely environmentally friendly is too optimistic for today (Shah *et al*., 2013; Deka *et al*., 2013). To produce "green" composite materials both the resin and fibre must have such green properties to achieve this goal (Benega *et al*., 2017). However, as of today, natural resins are not able to compete with conventional resins, in terms of mechanical and thermal properties, as well as physical properties, such as viscosity, etc. (Dallons, 2005). Materials based on cashew nut shell liquid hardeners, linseed and soybean oils resin, and UV-cured systems are being developed (Dallons, 2005). However, the best results are found when hybrid systems, comprising synthetic and biobased materials, are used in tandem (Benega *et al*., 2017).

The mechanical properties of flax fibre, which is

one of the most promising natural and sustainable fibres, are lower than the mechanical properties of E-glass fibre, but thanks to their low densities, they can compete with E-glass fibre in terms of specific mechanical properties (Yan *et al*., 2014). However, due to climatic conditions, production processes and environmental factors in which flax fibres are produced, great differences are observed in the mechanical properties of these fibres (Andersons *et al*., 2005). Baley *et al*. (2020) and Blanchard *et al*. (2016) stated that these differences are high at the elementary flax fibre level, but these decrease in the fibre bundle formed by the elementary fibres. Not only the variations that make working with natural fibres hard but also their hydrophilic characteristics, tendency to absorb water. Moudood *et al*. (2019) studied the effect of moisture in flax fibres and its effect on the mechanical properties of their composites. It is reported that humid fabrics lead to poor microstructural quality and deformations on the finished products, such as warpage.

Cheour *et al*. (2016) investigated the effects of moisture absorption on the behaviour of flax/epoxy composites with different fibre orientations. It was stated that the fibre orientation has a significant effect on moisture ingress and the moisture in flax fibres leads to an increase in damping properties.

Lu *et al*. (2022) studied the effect of moisture absorption of both technical and elementary fibres on their flexural properties. It was reported that fibre-matrix debonding occurs when flax fibres swell due to moisture.

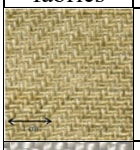
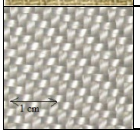
Assaedi *et al*. (2015) studied the thermal behaviour of flax reinforced composite materials by TGA. The degradation of flax fibres was observed in three stages: evaporating of the water absorbed by the fibre, between the temperature of 25 ^{0}C and 250 ^{0}C, decomposition of cellulose between 240 ^{0}C to 365 ^{0}C, and flax fibres decomposition above the temperature of 365 ^{0}C. However, the moisture content of the samples has not been the scope of the study.

The effect of moisture content on mechanical and damping properties of flax fibre composites has been studied by several researchers but the moisture content of flax fibre composites and their hybrids with E-glass fibres have not been studied. This study aims to investigate the moisture content of flax fibre composites and their hybrids with E-glass fibres by employing TGA. To compare the moisture contents, the moisture content of E-glass fibre composites was also studied.

2. MATERIALS AND METHOD

E-glass and flax fibres with different areal weights were used to ensure that the samples were of approximately the same thickness. The physical properties of the fibres used are given in Table 1 (Cihan *et al*., 2019). Gurit Prime 20V epoxy resin and Gurit FAST hardener were used for the composite manufacturing.

Table 1. Physical properties of the fabrics utilized.

Woven fabrics	Fibre type and weave	Areal weight (g/m^2)	Fibre diameter (μm)	Thickness (mm)
	Flax, 2x2	283	23	0.32
	E-glass, 2x2	590	19	0.56

2.1. Sample production

Six-layer symmetrical composite laminates with three different configurations namely, $[G_2F]_s$, $[FGF]_s$ and $[F_3]_s$ were produced by vacuum-assisted resin infusion technique (E denotes E-glass fibres and F denotes flax fibres). This method minimizes the amount of air that can enter the composite material, allowing materials with higher mechanical properties to be obtained than materials produced by the hand lay-up method (Yuhazri and Sihombing, 2010).

The produced laminates were left to cure at laboratory temperature (20 ^{0}C) for 24 hours. After this process, the laminates were post-cured for 7 hours in an oven at 65 ^{0}C to increase the mechanical properties and environmental resistance of the laminates. Then, samples were prepared by grating the laminates into small particles in a ceramic vessel. Each vessel

contains about 10 mg of grated laminate particles.

2.2. Thermogravimetric analysis

The TGA is performed over a temperature range of 25-800 ^{0}C with a heating rate of 10 ^{0}C /min under a nitrogen atmosphere. The relationship between the residual weight and temperature is plotted for the $[G_3]_s$ and $[F_3]_s$ samples, and the $[G_2F]_s$ layup is tested to find out whether there is a distinct behaviour for the three components. Upon increasing the temperature, moisture in the samples is first evaporated producing information on how much moisture is present in the sample.

3. FINDINGS AND DISCUSSION

Thermo Gravimetric Analysis (TGA) approach provides information on the changes in physical and chemical properties of materials that are measured as a function of constantly elevating temperature. As well as information on the decomposition temperature of components of the composites, which in turn, indicates the fibre volume content of the composite materials, given each constituent has a distinct decomposition temperature and there is enough equipment resolution. Along with information on decomposition temperatures, the TGA also provides information on the moisture content of the composite materials. It can also show the evaporation of other solvents when involved.

The $[G_3]_s$ samples have a moisture content of 0.5% as shown in Figure 1. The epoxy in the samples starts to decompose near 300 ºC and it continues until all the epoxy resin burns out, leaving the unburned fibres and epoxy ash to be weighted. These values are used to determine the component content of the constituents. This approach applies to the $[G_3]_s$ layup laminates as epoxy resin and E-glass fibres have different decomposition temperatures.

Moisture is the first constituent that is subtracted from the composite samples with the increasing temperature as the moisture in the composite is in a weak bond or free state (Assaedi *et al.*, 2015).

The decomposition of epoxy resin occurred between the range of 300 - 400 ^{0}C whereas no decomposition of E-glass fibres is observed between the range of 0 - 800 ^{0}C, as shown in Figure 1. E-glass fibre volume fraction for $[G_3]_s$ layup can then be calculated after removing the remaining ash residue of the epoxy.

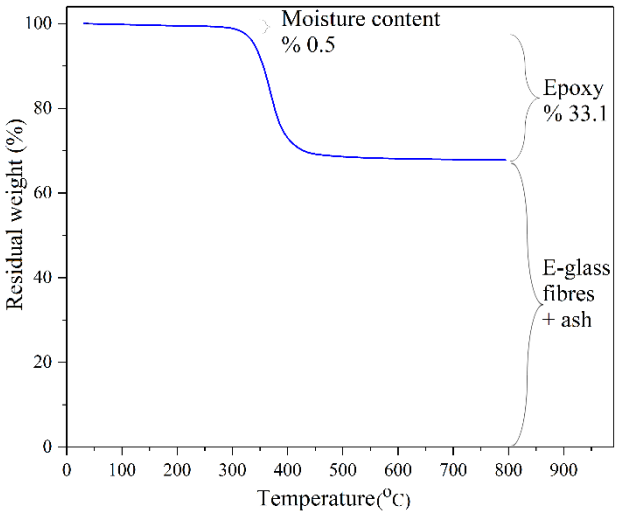


Figure 1. Residual weight vs. Temperature plot of $[G_3]_s$ samples generated by the TGA.

The moisture content of $[G_2F]_s$ was 2.5%, as shown in Figure 2. It was impossible to determine the decomposition temperature of epoxy resin and flax fibres with the parameters used. There is no distinct decomposition behaviour observed in the curve that renders the determination of the flax fibre content.

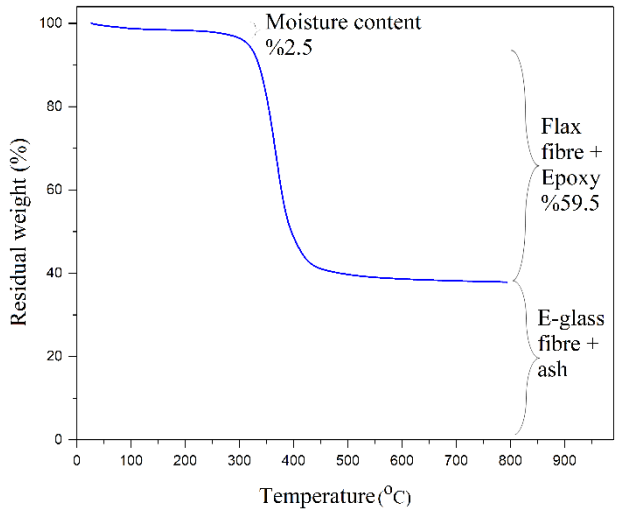


Figure 2. Residual weight vs. Temperature plot of $[G_2F]_s$ samples generated by the TGA**.**

In Figure 3, the first significant reduction in the weight is observed between 80-120 ^{0}C, where

water in the sample evaporates. This stage is followed by the degradation of the epoxy resin and flax fibres between the temperature of 300-400 ^{0}C. After about 500 ^{0}C, the curve flattens out, no constituent left to be burnt out. The remaining is the ash of the epoxy resin and the flax fibres.

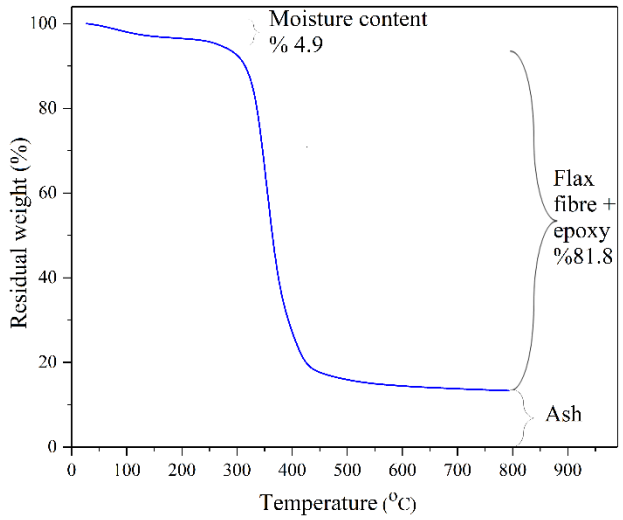


Figure 3. Residual weight vs. Temperature plot of $[F_3]_s$ samples generated by the TGA.

The moisture content grows as the flax fibre content increases as shown in Table 2.

Table 2. Mean moisture content of the samples.

	$[G_3]_s$	$[G_2F]_s$	$[F_3]_s$
Moisture Content (%)	0.5	2.5	4.9

This behaviour can be attributed to hydrophilic characteristics of flax fibres that cause degradation in the mechanical properties, causing stress at the fibre/matrix interface region resulting in weak matrix/fibre interfaces (Azwa *et al*., 2013).

4. CONCLUSION

TGA results show that the moisture content increases as the flax fibre volume fraction of the laminates increases. Since the moisture content has a significant effect not only on the mechanical properties but also on the damping properties, the moisture content of the samples needs to be determined and taken into account for a reliable reading. By this means, the interpretation of the experimental results will be more reliable. For further work, slower heating ramps may be employed to increase the graph resolution and to indicate distinct decomposition temperatures.

AUTHORSHIP CONTRIBUTION STATEMENT

Mehmet CİHAN: Conceptualization, Methodology, Validation, Formal Analysis, Resources, Writing - Original Draft, Writing-Review and Editing, Data Curation, Visualization. **Marcos Antonio Gimenes BENEGA:** Review and Editing, Data Curation, Visualization, Methodology, Validation, Formal Analysis. **Hélio RIBEIRO:** Review and Editing, Data Formal Analysis.

CONFLICT OF INTEREST

The author declares that for this article they have no actual, potential or perceived conflict of interests.

ETHICS COMMITTEE PERMISSION

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