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Investigating the Effects of SiO_2 - TiO_2 Concentration in Epoxy-Based Hybrid Nanocomposites

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KEYWORDS

Hybrid Nanocomposite; Epoxy; SiO_2-TiO_2; TGA; DSC.

ABSTRACT

The hand-lay-up mixing/casting process was used in this study to fabricate epoxy nanocomposites reinforced with SiO_2 and TiO_2 nanoparticles. The produced composites' thermal and thermogravimetric characteristics were examined using the correct characterization methods. Preparation of epoxy/ 2%wt SiO_2 and epoxy/ SiO2-TiO2 hybrid nanocomposites (HNC) was studied and investigated in this paper. The HNC were processed using different TiO_2 nano filler concentrations (i.e. 0, 0.3, 0.6, and 0.9) wt%. The results of DSC showed that The Glass Transition Temperature (Tg) of pure epoxy was (85.5°C) that of increases with the increase in the filler content (wt%), while the results of (TGA) curves showed that the pure (Epoxy) underwent one stages thermos gravimetric analysis process and total weight loss was (98.76%), and the weight ratios of the added SiO_2-TiO_2 hybrid nanocomposites showed one and two stages of thermos gravimetric analysis of the weight loss process and total weight loss were lower compared with pure Epoxy. Because of improved interfacial contact and uniform nanoparticle dispersion, the results showed that the addition of SiO_2 and TiO_2 nanoparticles considerably improved the epoxy matrix's examined characteristics. These results demonstrate how well hybrid nanoparticle reinforcement may enhance the functionality of epoxy-based nanocomposites.

1. INTRODUCTION

. Polymers, such as plastics and rubbers, pervade our lives, and we come across them in many different forms. Their physical properties are therefore of the utmost importance, and only a better knowledge of these properties will allow an appropriate use in technological and engineering applications [1]. Because it is simple to handle, mold, and cure, epoxy is one of the most widely used thermoset resins [2]. Polymers can be filled with particulate organic and inorganic fillers, in composite technology that could be interesting for further improving their mechanical and physical properties [3]. Nanoparticles, including nanoceramic, have been used as fillers in polymer composites for their mechanical and physical properties of the polymer [4]. Due to the combination of superior mechanical and thermal properties of inorganic parts with flexibility and processing ease, which are characteristics of organic polymers, several studies have focused on these organic–inorganic hybrid materials. In this regard, polymer matrix composites with inorganic nanoscale fillers have been developed as prospective advanced materials [5]. The nanocomposites' behavior as a whole depends on various mutual factors such as the inherent properties of the polymer, composite processing approaches, nanoparticle distribution and concentration within the matrix, particle size and extent of interfacial compatibility between nanoparticles and polymer matrix [6].

Inorganic NPs have been found to change the mechanical and thermal properties of polymers [7]. The best studied class of inorganic fillers under present investigation are metal and nonmetallic oxides that serve to regulate stiffness/strength, hardness, viscoelastic properties, as well as glass transition temperature [8]. The most crucial elements for producing homogenous nanocomposites with the required characteristics are the NPs/polymer interface adhesion [9].

The combined effects of SiO_2 and TiO_2 nanoparticles on the thermal stability and glass transition behavior of epoxy systems have received little attention, despite the fact that numerous studies have examined the thermal behavior of epoxy nanocomposites reinforced with individual ceramic nanoparticles. An important method for assessing curing properties, glass transition temperature (Tg), and thermal transitions in polymer nanocomposites is differential scanning calorimetry (DSC). According to earlier studies, the addition of nanoparticles can alter chain mobility and increase heat resistance because the nanofillers and epoxy matrix have better interfacial contacts [19]. However, the synergistic influence of hybrid SiO_2-TiO_2 nanoparticles on the DSC response and thermal performance of epoxy composites remains inadequately known, particularly in terms of nanoparticle dispersion and loading optimization. This study will use DSC analysis to examine how hybrid SiO_2-TiO_2 reinforcement affects the thermal behavior and structural stability of epoxy nanocomposites [20].

Thermal gravimetric analysis is a technique for measuring weight loss as a function of temperature, and is one of the most important means used to determine the thermal stability of a polymer and determine the temperature at which it disintegrates [10]. The (TGA) gives direct information about thermal stability and is useful in polymer diagnosis, especially in the case of disintegration and giving of well-known structures products, which have been obtained when increasing heating temperature, which may therefore provide more valuable information, because it displays between the limits of higher thermal stability [11,12].

Previous research has focused on incorporating SiO_2 or TiO_2 nanoparticles into epoxy matrices to improve mechanical or thermal properties, despite significant progress in developing epoxy-based nanocomposites reinforced with ceramic nanoparticles [21].

However, the literature currently in publication does not adequately explain the synergistic strengthening process resulting from the simultaneous integration of SiO_2 and TiO_2 nanoparticles into a single epoxy matrix. Specifically, only a few studies have comprehensively linked the ensuing structural and thermal performance of hybrid epoxy nanocomposites with nanoparticle dispersion, interfacial contact, and temperature transitions. Furthermore, issues with hybrid nanofiller filling optimization and nanoparticle agglomeration are still not well addressed [22,23]. Thus, by examining the combined impact of SiO_2–TiO_2 hybrid nanoparticles on the thermal and structural behavior of epoxy nanocomposites, with a focus on dispersion homogeneity, interfacial compatibility, and thermal characteristics assessed through DSC analysis, the current study advances earlier research. This study aims to advance in that it uses the complementary properties of SiO_2 and TiO_2 nanoparticles to increase overall performance and thermal stability when compared to previously published single-filler epoxy nanocomposite structures.

2. MATERIAL AND METHODS

2.1. Materials used

In the present study, epoxy (EP10) and hardener type (HY-956) were utilized in a 3:1 Forsoc Jordan. SiO_2/TiO_2 Assay 99.9 % made in Barcelona Espana and Company with average size 50 nm, made in USA.

2.2. Sample preparation method

The requisite quantities of hybrid nanoparticles were initially measured with a high-precision analytical balance and subsequently incrementally added to the 30 gm epoxy resin while maintaining continuous mechanical stirring at a regulated speed of approximately 900 rpm for 30 minutes to facilitate uniform dispersion and minimize particle agglomeration. The dispersion was then subjected to ultrasonic agitation for 20 minutes to further enhance the interfacial distribution of the nanoparticles within the epoxy matrix. After adding the curing agent in accordance with the epoxy-to-hardener ratio suggested by the manufacturer, the mixture was gently stirred to reduce air entrapment and guarantee homogeneity. To fabricate specimens with precise geometrical dimensions suitable for testing, the resultant nanocomposite mixture was cast into specially designed molds. After allowing the manufactured samples (thickness 20mm) to cure for 24 hours at room temperature, a post-curing procedure was applied after two weeks to guarantee that the epoxy network had fully cross-linked. The epoxy was mixed resin with the SiO_2 and TiO_2 dispersion in a sanitized mixing container. The mixture was vigorously mixed to achieve homogeneity. The usual range of TiO_2 nanoparticle addition is (0, 0. 3, 0. 6, and 0. 9) wt% of 2% SiO_2 of the weight of the epoxy, as shown in Table 1.

2.3. Characterization Techniques

The measurements were conducted using a DSC analyzer under an argon atmosphere to prevent oxidative degradation during heating. Samples with an average mass of approximately 25 mg were sealed in aluminum crucibles and heated from room temperature up to 600 °C at a heating rate of 10 °C/min. The DSC analysis was employed to determine the glass transition temperature (Tg), thermal transitions, curing behavior, and thermal stability of the developed nanocomposites

Table 1. Sample preparation for the study.

Samples No.	weight ratio
Epoxy pure	0

EPS	2% SiO2
EPS0.3T	2%SiO2+0.3%TiO2
EPS0.6T	2%SiO2+0.6%TiO2
EPS0.9T	2%SiO2+0.9%TiO2

3. RESULTS AND DISCUSSION

3.1. Thermal Properties "Glass Transition Temperature (Tg) "

The Glass Transition Temperature (Tg) of pure epoxy composites supported using SiO_2 nanoparticles was measured by using DSC. Figures (1 and 2) show the glass-transition temperature (Tg) values for all nanocomposites as shown in Table 2. The glass-transition temperature was important to determine the initial thermal stability of polymers. It was observed that the value of the glass transition temperature of pure epoxy was (85.5°C). With a total weight loss of 98.76%, the pure epoxy (EP) demonstrated a single-stage decomposition behavior in the temperature range of 278–593°C, signifying the full breakdown of the polymeric network. Following the addition of SiO_2 nanoparticles (EPS), the decomposition ranges slightly shifted toward higher temperatures (295–595°C), and the overall weight loss dropped to 87.47%. This suggests that the barrier effect of silica particles, which limits the diffusion of volatile degradation products and enhances thermal stability, is responsible for the improvement in thermal resistance. Depending on the filler content, various degradation behaviors were seen for the hybrid nanocomposites containing nano-TiO_2. Due to the heterogeneous dispersion of nanoparticles and the presence of many degradation processes connected to the interface areas between the epoxy matrix and the nanofillers, the EPS0.3T sample displayed a two-step breakdown process (263–397°C and 467–592°C). On the other hand, with total weight losses of 91.41% and 89.10%, respectively, EPS0.6T and EPS0.9T showed a mostly single-stage degrading pattern. The findings show that the thermal stability of the epoxy matrix is significantly impacted by the addition of nano-TiO_2. When compared to pure epoxy (85.5°C), the greatest glass transition temperature (Tg = 126.7°C) was found in EPS0.6 T. The strong interfacial contact between the epoxy chains and the nano fillers, which limits the mobility of the polymer chains and increases the crosslink density, is confirmed by this significant rise in Tg. In earlier research on epoxy/metal oxide nanocomposites, well-dispersed TiO_2 nanoparticles enhanced the polymer network's stiffness and heat resistance. However, the Tg value decreased to 113.8°C when the TiO_2 concentration rose to 0.9 weight percent. This decrease might be linked to the agglomeration of nanoparticles at greater loading amounts, which results in uneven dispersion and decreased efficiency of chain restriction and stress transmission processes. Consequently, the results indicate that the ideal filler concentration for attaining enhanced thermal performance is 0.6 weight percent TiO_2. The reason for this can be explained as when heating epoxy molecules and secondary molecules of the added material, the heat is gradually absorbed until the temperature reaches the (Tg) temperature, at which the secondary molecules acquire enough energy and begin circling around the primary bond (C-C), which is the backbone of the epoxy chain [13]. The results are in line with earlier studies on epoxy-based hybrid nanocomposites containing SiO_2 and TiO_2 nanoparticles, which showed that while excessive loading led to particle agglomeration and a decline in thermal properties, moderate nanoparticle loading improved thermal stability and Tg. Thus, the current work shows how crucial it is to maximize nanoparticle concentration in order to improve epoxy nanocomposites' thermal and structural performance [13].

Table 2. Values of (Tg) of Epoxy with hybrid nanocomposite

Samples No.	Tg °C
Epoxy pure	85.5
EPS	66.5
EPS0.3T	82.7
EPS0.6T	126.7
EPS0.9T	113.8

3.2. Thermogravimetric Analysis (TGA)

As polymers undergo several changes when heated, they release gases and liquids which were differ in their shapes, colors and molecular weights. The polymer's ability to resist these changes at high temperatures is called thermal stability, so the thermo gravimetric analysis technique (TGA) was used to test the kinetics of reactions and products in the thermal dissolution process stages [14] using differential scanning calorimetry (DSC).

Table 3. The TGA values of Epoxy hybrid nanocomposite

S.NO	Region of Decomposition	Temperature Range (^{O}C)		Weight Loss (%)
		Start	End	Total

EP	1st	278	593	98.76%
EPS	1st	295	595	87.47%
EPS0.3T	1st	263	397	92.48%
	2nd	467	592	
EPS0.6T	1st	279	595	91.41%
EPS0.9T	1st			89.10%

Figures (3-7) show TGA curves for pure epoxy polymer and SiO_2 nanoparticles added with different weight ratios, where we note that the TGA curve of pure epoxy showed a single thermal analysis phase in the temperature range of (278-593) °C with a total weight loss of (98.76) %. After the addition of nanoparticles, it was noted that the EPS0.3T ratio showed two phases of thermal degradation of the weight loss, the first phase occurring from the temperature range of (263-397) °C and the second phase in range of (397-592) °C and can be attributed to the deterioration of the remaining materials [15] or may indicate to the presence of chemical decay resulting from the separation of carbonaceous bond (C-C), in the backbones of epoxy polymer [16,17]. Table 3 presents the improvement of the thermal stability of epoxy after the addition of nanoparticles, down to (89.10%) at a ratio of 0.9.

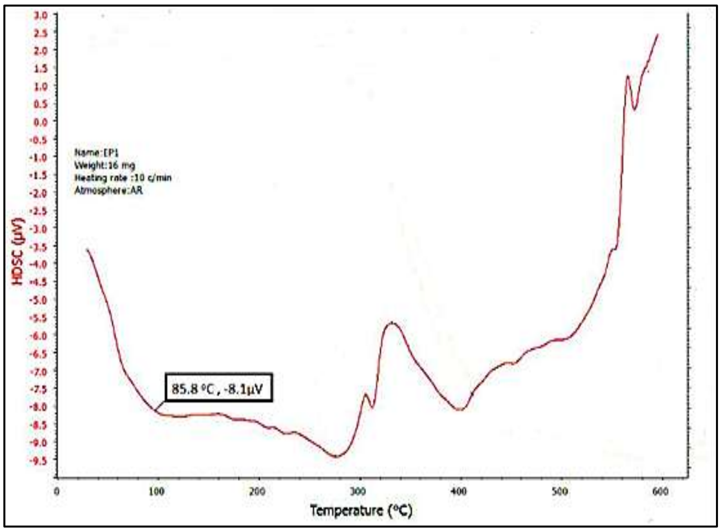
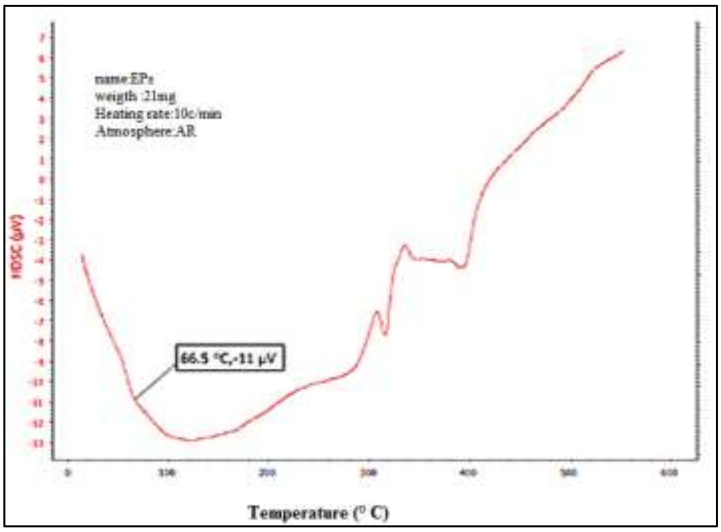
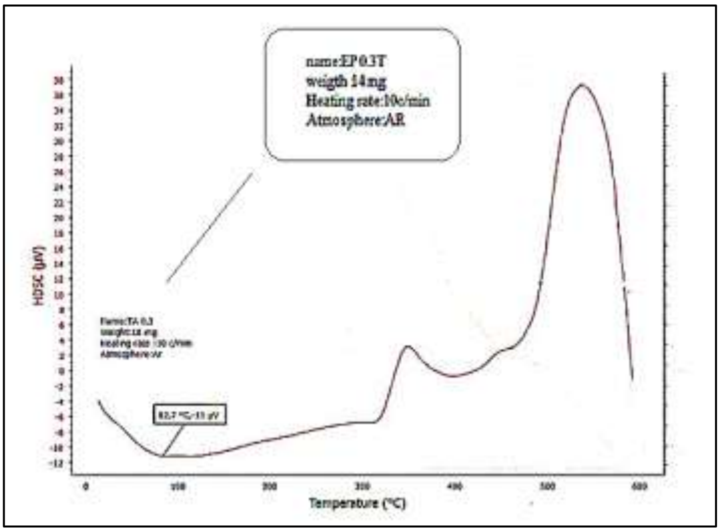


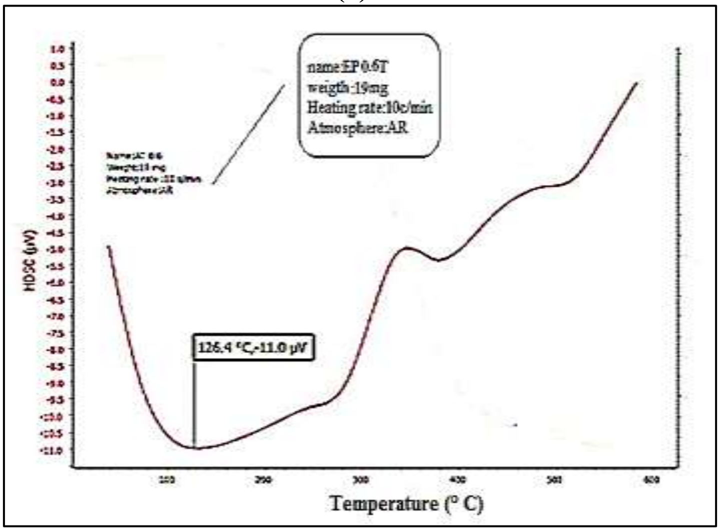
Fig. 1. The DSC diagram thermal weight for pure (Epoxy)



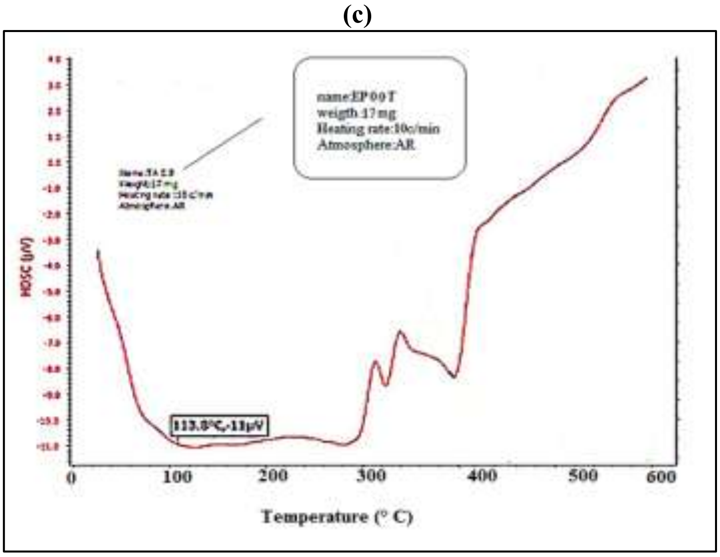
(a)



(b)



(c)



(d)

Fig. 2. The DSC thermal weight of Epoxy-hybrid nanocomposite (a) Eps, (b) EPS0.3T, (c) EPS0.6T, and (d) EPS0.9T

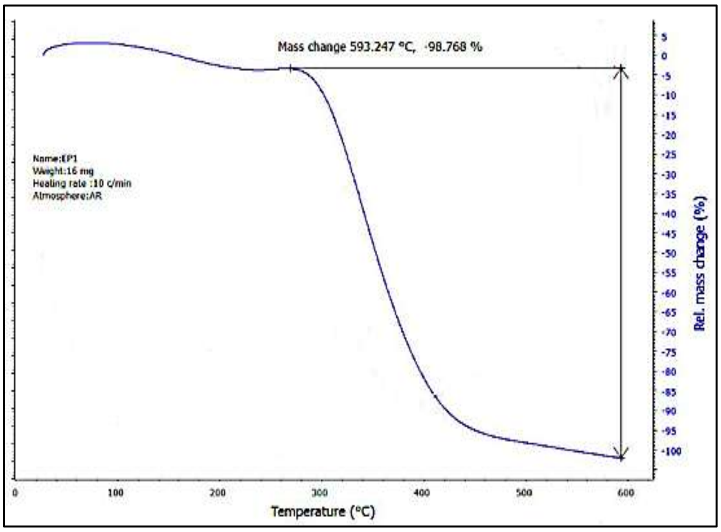


Fig. 3 .TGA curve of the pure (Epoxy).

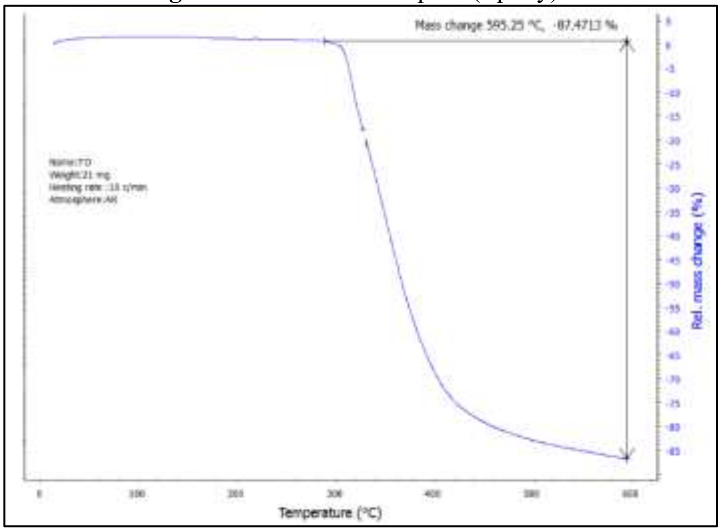


Fig.4.TGA curve for EPS sample.

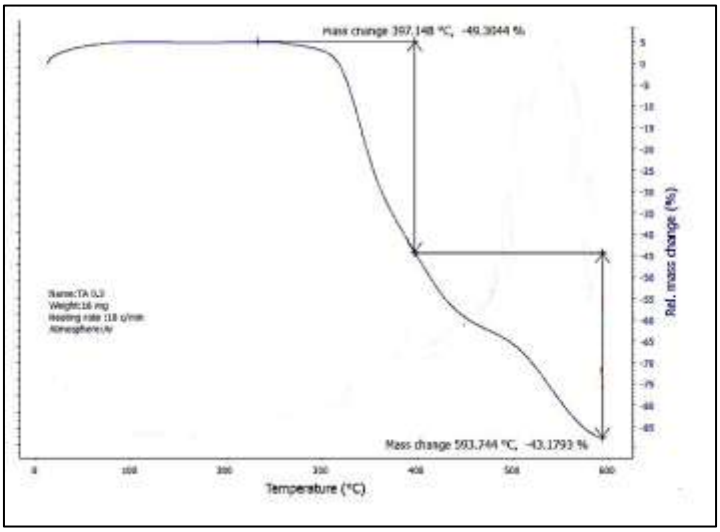


Fig.5. TGA curve for EPS0.3T sample

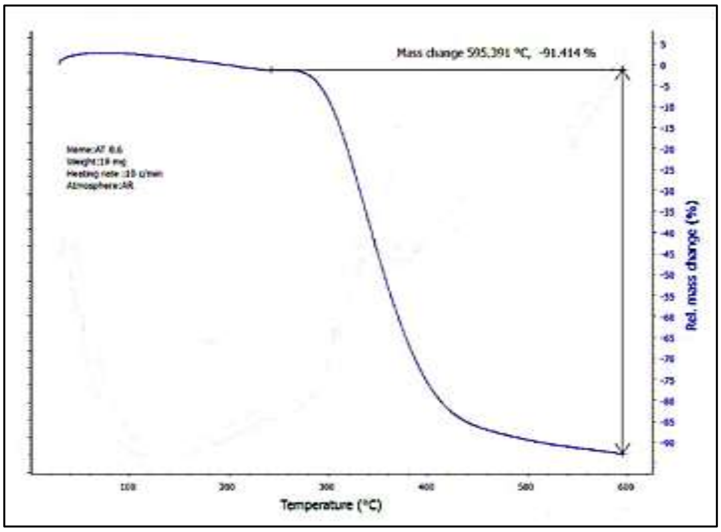


Fig.6.TGA curve for EPS0.6T sample

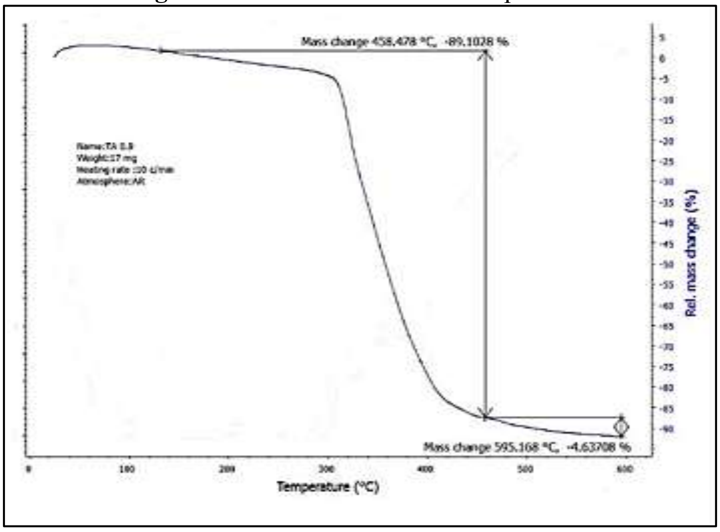


Fig.7.TGA curve for EPS0.9T sample

4. CONCLUSIONS

The current study showed that adding SiO_2 and TiO_2 nanoparticles greatly enhanced the epoxy nanocomposites' thermal performance. Stronger interfacial contacts between the epoxy matrix and the nanoparticles and limited polymer chain mobility are shown by the thermal analysis results, which showed a discernible increase in the glass transition temperature (Tg). Additionally, the composites showed better thermal resilience, especially at higher TiO_2 additive ratios, where less weight loss during thermal deterioration was seen. Overall, the results demonstrate the potential of SiO_2 and TiO_2 nanoparticles for advanced applications requiring high heat resistance and structural stability, such as protective coatings and electronic packaging, and validate their efficacy as reinforcing additions for epoxy resin.

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Conflict of interest

"The authors declare that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest."

Consent for Publication

"Not applicable. This manuscript does not contain any individual person's data in any form (including individual details, images, or videos), and therefore consent for publication is not required."

Availability of Data and Materials

The data supporting the findings of this study are included within the article.

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دراسة تأثير تركيز SiO_2 - TiO_2 في المركبات النانوية الهجينة القائمة على الايبوكسي

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الملخص

استخدمت عملية الخلط/الصب اليدوي في هذه الدراسة لتصنيع مركبات نانوية من الايبوكسي مُدعمة بجزيئات نانوية من ثاني أكسيد السيليكون (SiO_2) وثاني أكسيد التيتانيوم (TiO_2) وتم فحص الخصائص الحرارية والوزنية الحرارية للمركبات المُنتجة باستخدام طرق التوصيف المناسبة. وتناولت هذه الورقة البحثية دراسة تحضير مركبات نانوية هجينة من الايبوكسي/2% وزناً من ثاني أكسيد السيليكون، ومركبات نانوية هجينة من الايبوكسي/ثاني أكسيد السيليكون وثاني أكسيد التيتانيوم. وتم تصنيع هذه المركبات النانوية الهجينة باستخدام تراكيز مختلفة من حشو ثاني أكسيد التيتانيوم النانوية (0، 0.3، 0.6، و0.9%) وزناً. أظهرت نتائج التحليل الحراري التفاضلي (DSC) أن درجة حرارة التحول الزجاجي (Tg) للإيبوكسي النقي تبلغ 85.5 درجة مئوية، وتزداد مع زيادة نسبة الحشو (وزنًا). في حين أظهرت نتائج منحنيات التحليل الحراري الوزني (TGA) أن الايبوكسي النقي خضع لمرحلة واحدة من عملية التحليل الحراري الوزني، وبلغ إجمالي فقدان الوزن 98.76%. أما نسب الوزن للمركبات النانوية الهجينة المضافة من SiO_2-TiO_2، فقد أظهرت مرحلتين من التحليل الحراري الوزني لعملية فقدان الوزن، وكان إجمالي فقدان الوزن أقل مقارنةً الايبوكسي النقي. وبفضل تحسين التلامس البيني وتوزيع الجسيمات النانوية بشكل متجانس، أظهرت النتائج أن إضافة جسيمات SiO_2 وTiO_2 النانوية حسّنت بشكل ملحوظ خصائص مصفوفة الايبوكسي المدروسة. تُبيّن هذه النتائج مدى فعالية تعزيز الجسيمات النانوية الهجينة في تحسين وظائف المركبات النانوية القائمة على الايبوكسي.

الكلمات المفتاحية

مركب نانوي هجين، إيبوكسي، SiO_2-TiO_2، TGA، DSC.