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# THERMAL PROPERTIES OF NANO-COMPOSITES: A COMPARATIVE STUDY BETWEEN EPOXY/GRAPHENE AND EPOXY/$CaCO_3$

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**Abstract**

Recently, many nano-particles have been involved with the epoxy resin to form unique composites applicable for various applications. In this study, thermal properties of epoxy/graphene nano-composites have been compared with that composed of epoxy/nano-$CaCO_3$. Experimental works produced samples of different loading of nano-particles as: 0.3, 0.6, 0.9, 1.2, 1.5 and 1.8% of the weight. The data of thermal properties of these composites have been obtained experimentally from measurements of glass-transition temperature, thermal stability, thermal conductivity and thermal expansion. Generally, the adding of nano-particles enhanced the thermal behaviour of the epoxy composite, where the loading of 0.6% of nano-graphene showed the optimum thermal properties for nano-graphene composites, while the optimum loading for nano-$CaCO_3$ composites was 1.5%. Furthermore, nano-$CaCO_3$ composites have shown more improvement than that of nano-graphene composites by a range of 5-30% at their optimum loadings. However, some drawbacks may be noticed at high loading rate due to the agglomeration of particles within the composite

Keywords: Composite materials, Epoxy, Nano-particles, Polymer, Thermal.

## 1. Introduction

Recent years have witnessed rapid developments in the use of nano-composites for various industrial applications. Epoxy is one of the most thermosetting materials under consideration of composite for manufacturers. Epoxy has many advantages since it is easily to cure, workability and affordability which makes it suitable as adhesive, coating and sealant if it is used alone, or in the case of composite it can be an alternative material for beams, columns and boards for various applications [1]. Epoxy resin has many molecular groups called Glycidyl groups. Each group has the root $CH_2$-CH-O, which gives the hardness to the polymer. Epoxy resin can be formed by the mixing of many basic polymeric materials [2-4].

Epoxy composites are subjects of researches for numerous studies where many reinforcement materials can be involving such as: fibers, fillers, particles and layers. The newly line of nano-composite has found a wide space in epoxy composites due to their reliable thermal and mechanical properties. Curing and cross linking of epoxy is an essential parameter in the fabrication since it is responsible of oxidizing some sub-groups within the epoxy. Sometimes, fillers added to the epoxy provide unstable crosslinked structures which cause the weakness or the degradation at elevated temperatures, as well as phase separation [5, 6].

Recently, epoxy nano-composites have become a matter of interest since it can be employed to withstand various operational conditions, with the involving of different loading of particles. Nano-composites adopt extreme low size particles (usually around or less than 100 nm), where the reduction of particles' size has a direct role in improving polymer bonding, which reduces the spaces between the monomers, thus enhance the overall thermo-physical properties [7, 8].

Many nano-particles can be used as reinforcements for composite materials, such as: carbon based nano-particles and silica-based nano-particles, as well as metallic nano-particles [9-15]. Graphene is a graphite layer has a hexagonal lattice of carbon atoms bonded in the atomic group. Graphene is denoted by its superior thermal conductivity and excellent effects in improving the mechanical properties. Graphene is usually preferred due to ultra-surface area (around 2500 $m^2/g$) [16-20].

Studies that investigated the thermal features of the composites that involving graphene nanoparticles (GNPs) are mainly looking to improve the thermal performance of these composite by analysing different thermal properties. The studies usually preferred involving low fraction of GNPs usually less than 1% in weight, as in [21-30], or in somewhere, they used rather more that 1% in weight, as in [31-37]. Most of the reviewed studies have preferred the using of small content graphene. The reason behind that is due to the best values of thermal characteristics. The using of nano-$CaCO_3$ is epoxy composites was mentioned by some studies, as in [38-43. The studies have utilized nano-$CaCO_3$ for various diameters, weight contents, processes of preparing and combinations. According to these studies, epoxy/nano-$CaCO_3$ composites show excellent improvement in the thermal characteristics when the weight exceeds 1% of the composite. Some drawbacks may also notice due to the agglomeration of particles within the composite.

The objective of this study is to investigate the thermal properties of epoxy nano-composites and seek to find the optimum loading of nano-particles and the corresponding benefits or drawbacks. The current study is using two common epoxy nano-composites; epoxy/GNPs and epoxy/nano-$CaCo_3$ and comparing between them depending on their thermal behaviours. Different samples with many loadings of nano-particles (0.3, 0.6, 0.9, 1.2, 1.5, 1.8%) are manufactured to measure thermal features of epoxy with and without nano-particles.

## 2. Materials and Methods

In the current study, various specimens of epoxy/nano-composites incorporated with several loadings of nano-particles have been fabricated. The matrix used in the composites was SIKADUR-52 epoxy provided by Sika Group. The used epoxy has two parts as the resin and the amine curing agent. The material is hydrophobic and has a limited viscosity value. Nano-materials were purchased from the Chinese manufacturer (XFNANO). The characteristics of the components are shown in Table 1.

**Table 1. Features of the materials*.**

| Material | Properties |
|---|---|
| **Epoxy** | The density is 1.25 g/cm$^3$. The viscosity is less than 490 mPa.s at room temperature. |
| **Graphene nanoparticles** | Black powder of purified particles. The density is less than 0.08 g/cm$^3$ and the average size of the particles is 50 nm. |
| **$CaCO_3$ nanoparticles** | White powder of purified particles. The density is less than 0.38 g/cm$^3$ and the average size of the particles is 100 nm. |

* Data taken from the provider.

Each nano-composite can be prepared by mixing the epoxy with either GNPs or nano-$CaCO_3$ in a careful procedure. In the beginning, the desired quantity of epoxy resin has been heated up to 60 $^oC$ using an electrical heater for reducing the initial viscous fluid which facilitates the dispersion of nano-particles later. Précised quantity of nano-particles then dispersed into the epoxy resin using bath ultra-sonication, followed by mixing the solution in a suitable pot and by an electrical stirring up to 1000 rpm during 30 min for homogeneous dispersion of the nano-materials. Note that GNPs have sizes of less than 50 nm, while nano-$CaCO_3$ particles have sizes of less than 100 nm. Afterward, the solution should be cooled at room temperature. Then, hardener (Amine) has been poured at a concentration of 2:1. Finally, solution has been weighted again in order to ensure the required total mass. The mixture can be casted in a suitable die to cure for one day time. A heating process can be done lately at 80 $^oC$ for four hours to enhance the solidification.

The study aimed to manufacture different epoxy specimens containing nano-particles of 0.3, 0.6, 0.9, 1.2, 1.5 and 1.8% of the weight, beside the neat epoxy specimen. The experimental works including the preparing of the materials, the fabrication of the specimens, and the measurements were done in Nanotechnology laboratory at the University of Mustansiriyah from 2 April to 25 May 2022. Multiple samples are required according to the standards for the measurement (ASTM), as shown in Table 2. The instruments are shown in Fig. 1. The measurements have included the values of glass-transition temperature, thermal stability, thermal conductivity and thermal expansion.

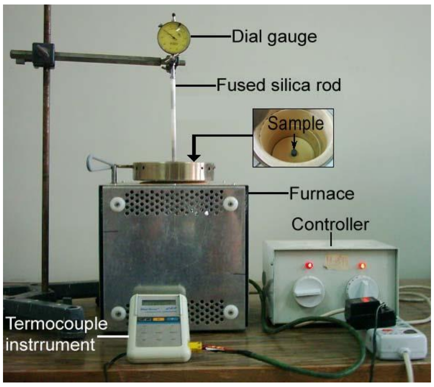
The measured values; glass-transition temperature ($T_g$), thermal conductivity ($k$), thermal expansion ($\alpha$) and mass loss ($M_{loss}$) should be submitted to uncertainty analysis. The analysis was performed according to the resolution and accuracy of the instrument served in the work. The functions mentioned in Table 3 are mathematical functions based on statistical processes that are well defined in the reference [44]. As seen, the maximum uncertainty value has appeared in thermal conductivity measurements (±12.2) and this is due to large domain of readings.

**Table 2. Samples served in the work.**

| **Measurement** | **Sample** |
|---|---|
| **Glass-transition temperature & Thermal stability** | Particles of mesh 8 (diameter < 2.36 mm).   |
| **Thermal conductivity (ASTM C177)** | Disc of 40 mm diameter and 5 mm thickness.   |
| **Thermal expansion (ASTM E831)** | Rectangular of 80 mm length, 10 mm width and 4 mm thickness.   |

**(a) DSC-TGA analyser (SDT-Q600 V20.9)**

**(b) Thermal conductivity apparatus**




**(c) Push rod dilatometer**

**Fig. 1. Instruments used in the measurements.**

**Table 3. Uncertainty analysis of the measurements.**

| Measurement | B | $\bar{X}$ | $\sigma_x$ | $\sigma_{\bar{y}}$ | $P_x$ | $U_x$ | $U_x/\bar{X}$ % |
|---|---|---|---|---|---|---|---|
| **Tg(°C)** | 0.06 | 102 | 5.1 | 1.92 | 5.57 | 5.57 | ±5.4 |
| **k (W/m.K)** | 0.05 | 0.48 | 0.03 | 0.01 | 0.03 | 0.06 | ±12.2 |
| **α (1/K) × $10^{-4}$** | 0.08 | 1.41 | 0.05 | 0.02 | 0.06 | 0.1 | ±7.1 |
| **$M_{loss}$** | 0.03 | 70.6 | 2.4 | 0.91 | 2.64 | 2.64 | ±3.7 |

## 3. Results and Discussion

The reaction of epoxy is affected by many aspects including the type of the resin, curing agent [45], temperature and heat transfer of the mixed resin [46, 47], as well as the viscosity of the resin [48]. For a proper curing process, it should control the viscosity and the temperature to satisfy a harmonious process of gel forming. The size of nano-particles and the procedure of dispersion have also influenced the product and its corresponding features. If extreme small particles dispersed in the matrix and there is a homogeneous distribution, an excellent product will form. The involving of nano-particles within the matrix may improve chemical stability and enhances dimensional stability and corresponding thermal performance [49].

The data obtained from the measurements are shown in Figs. 2-5 for epoxy/nano-graphene and for epoxy/$CaCO_3$. The data revealed the impact of nanoparticles to on the values of glass-transition temperature, thermal stability and thermal conductivity that have increased by the increasing of nano-particles content. This is because the reinforced nano-particles cause a higher density of the composites, and thus improve the overall thermal properties. In addition, the interacted particles affect to generate high interaction energy.

It was noticed that the thermal properties have improved effectively for an extreme could be denoted as the optimum amount. This amount of nano-loading satisfies best combination with the matrix. Beyond this limit, the properties went done gradually. The optimum value for GNPs loading was 0.6%, while it was 1.5% for $CaCO_3$. The reason of decreasing of the features beyond the optimum value is mainly as a result of poor dispersion of nanoparticles which causes the agglomeration effect. Agglomeration of nano-particles leads to insufficient attracts between the reinforcements and the matrix [30, 50].

Glass-transition temperature is the point when the material goes from the glassy state to the rubbery state. Several aspects influence the glass-transition temperature of nano-composite like the configuration of the polymer, rigidity and crosslinking of the chains and the molecular weight resulted by the incorporation of nano-particles. Measured data have stated the values of epoxy/GNPs are higher at its peak by 9 $^oC$ comparing to the values obtained for the referenced epoxy. The data revealed also that the glass-transition temperature was even more by 14 $^oC$ when carbon calcium nanomaterials are used as maximum. Increasing of glass-transition temperature is due to the restriction of movement of polymer chains due to the increasing of crosslinked structure as a result of nano-particles role. This is also mentioned by [25, 26, 27, 32, 42], where they explained that the rise in glass-transition temperature was attributed to the limited mobility of moles in well mixed composites, which decreases the molecular free volume, as a results of excellent dispersed particles. Decrease in glass-transition temperature at high loading and beyond a certain value can be attributed to the agglomeration and poor dispersion of nano-particles within the composites that have reduced the crosslinking sites [30] and also to the higher thermal conductivity of these samples [36].

The thermal stability is usually indicated by the ability of the material to save its mass at elevated temperatures. The results of the thermo-gravimetric analysis obtained by DSC-TGA analyser has shown that thermal degradation of epoxy composites starts beyond 200 $^oC$ with a mass loss less than 5%, and then accelerates rapidly when the temperature exceed 350 $^oC$ with a mass loss more than 20%. The majority of the mass has been lost when the temperature closest to be 500 $^oC$. Hence, this temperature has been depended to compare the degradation between different epoxy composites, which is based also by many references [21-27]. It can be seen from the mass loss behaviour (Fig. 3), that the thermal stability of epoxy/GNPs composites is better than that of neat epoxy, where the mass loss was 64% in comparison to 78% for the neat epoxy. On the other hand, the mass loss was 55% for carbon calcium nano-composite. This behaviour is essentially due to the retarding impact of nano-particles in the epoxy (crosslinked structure), as well as good interfacial bonding between particles of the reinforcement and the polymer as denoted by [21, 24, 25, 31, 38, 39, 43] as well. The reduction in the thermal stability at high loadings can be attributed to the agglomeration which devastates the chemical stability for the matrix [41].

Furthermore, the thermal conductivity of nano-composite samples was higher in comparison with the neat epoxy. The improving of thermal conductivity is due to the superior thermal conductivity of nano-particles. This behaviour was also referred by [24, 28, 29, 35]. They also revealed the improving in conduction heat transfer due to the super features of GNPs. Note that there is no limitation for the increasing of the thermal conductivity value by the increase of nano-fillers [31, 32, 34]. However, the thermal conductivity of optimum epoxy/GNPs composite rised up to 57% of the value recorded by the controlled epoxy sample. Moreover, the heat conductivity of optimum epoxy/nano-calcium carbonate composite rised up to 86% of the value recorded by referenced sample.

Thermal expansion describes the dimension stability of the material. The involving of nanoparticles in the polymeric matrix significantly will decrease the elongation in the material and ensure good dimensional stability. The coefficient of linear thermal expansion of optimum GNPs composite was lower by 12% comparing to the reference sample, and it was lower by 15% for optimum epoxy/nano-$CaCO_3$. Reduction in the thermal expansion value is a result of the increasing of the rigidity due to the influence of reinforced particles [30]. The improvement in the dimensional stability of the nano-composite can be attributed to the uniform dispersion of nano-particles in the polymer, thus good bonding with the resin. Moreover, agglomeration is expected to have an influence on the values of thermal expansion coefficient at high loadings.

The distribution of nano-particles and their sizes are important factors to consider in the fabricating of nano-composites. The noticed points revealed that features of the reinforced polymer with the nanomaterials have shown excellent enhancement. The improving due to the use of graphene nanoparticles was even better comparing to the nano calcium carbonate, especially at low amount of weight content. This is due to the tiny diameters of graphene particles served in the current study that was less than 50 nm in average. The small particles can enrol better action in enhancing the overall thermo-physical features of the composite [51]. For higher weight content, epoxy/$CaCO_3$ composite has witnessed better features comparing to the graphene composite and that because of the adhesion of calcium carbonate nanoparticles to polymer efficiently at high weight concentration [38, 52]. Results show that the improvement in thermal properties of optimum epoxy/calcium carbonate nanoparticles was better by 5-30% comparing to that of optimum graphene composites.

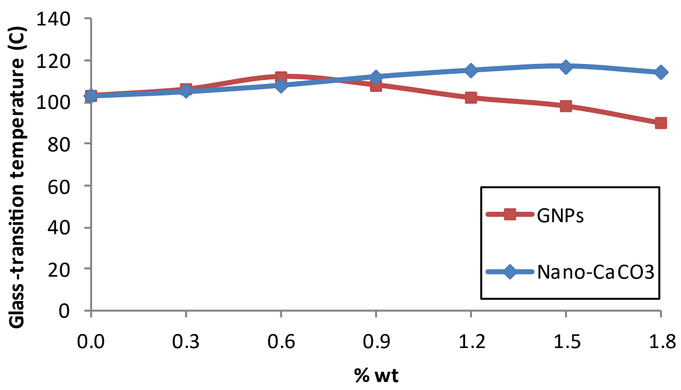



**Fig. 2. Glass-transition temperature of epoxy nano-composites.**

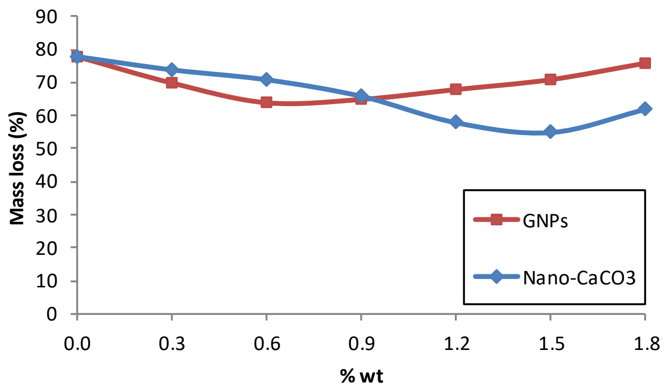



**Fig. 3. Thermal stability of epoxy nano-composites (at 500 ºC).**

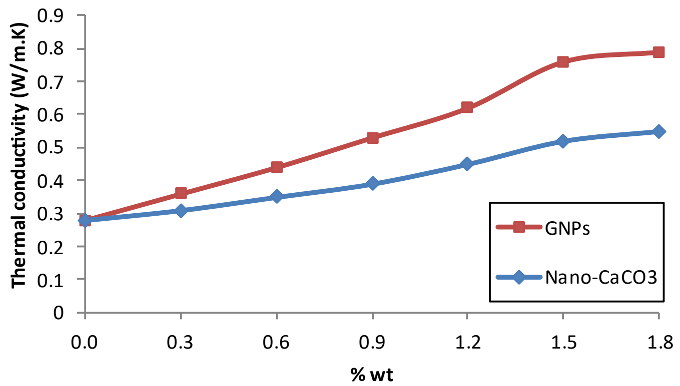



**Fig. 4. Thermal conductivity of epoxy nano-composites.**

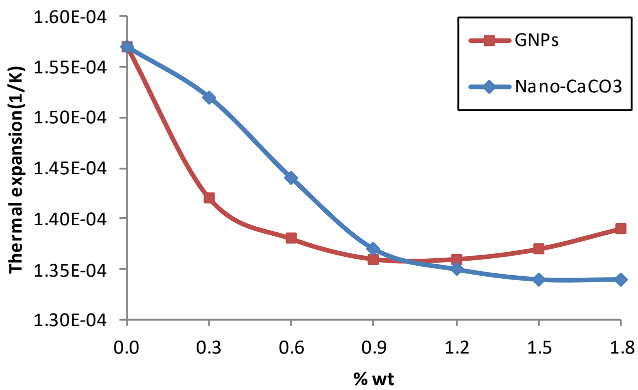



**Fig. 5. Thermal expansion of epoxy nano-composites.**

Data collected during current study can be compared as well with those obtained by previous studies for validation purpose. See Table 4.

**Table 4. Comparison of results between current study and previous studies.**

| Source | Materials and loading | Features |
|---|---|---|
| **Current study** | Epoxy/GNPs (0.3-1.8% wt) and Epoxy/Nano-$CaCO_3$ (0.3-1.8% wt) | Enhance glass-transition temperature, thermal stability and thermal expansion by 10-30%. Enhance thermal conductivity by more than 50%. |
| **[21-30]** | Epoxy/GNPs (< 1% wt) | Enhance thermal stability between 300-600 °C by 5-10%. Control the glass-transition temperature between 45-160 °C and increase by 2-5%. Enhance thermal conductivity by 10-15%. There is a reduction in thermal expansion by 10-30%. |
| **[31-37]** | Epoxy/GNPs (> 1% wt) | Enhance thermal stability between by less than 5% with agglomeration drawbacks. Decrease glass-transition temperature for several degrees due to low dispersion of GNPs. Enhance thermal conductivity by more than 100%. |
| **[38-43]** | Epoxy/Nano-$CaCO_3$ (0.5-10% wt) | Good enhancement for thermal stability and glass-transition temperature at low loadings but it is not useful at high loadings. Enhance thermal conductivity in general. |

## 4. Conclusions

This study has investigated the thermal properties of epoxy nano-composites and found the optimum loading of nano-particles to ensure best performance. The study has served two common epoxy nano-composites; epoxy/GNPs and epoxy/nano-

$CaCo_3$ with a range of loading between 0.3-1.8% wt. The results have shown a reliable enhancement in the thermal properties due to the incorporation of nano-particles, where the values have increased significantly until a certain number of nano-particles, which is the optimum value. The study also has shown some drawbacks at high loading content due to the agglomeration effect. In general, the epoxy/GNPs composite of 0.6% wt presented best overall thermal values, and the optimum values for nano-$CaCO_3$ can be satisfied at 1.5% wt. Furthermore, values of thermal properties of nano-composites at their optimum loadings were better by 10-20% in comparison with that of neat epoxy, except for the thermal conductivity which was about 50%.

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**Abbreviations**

| | |
|---|---|
| ASTM | American Society for Testing and Materials |
| DSC | Differential Scanning Calorimetry |
| GNPs | Graphene Nano-Particles |
| TGA | Thermo-Gravimetric Analysis |

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