


Research Article

Experimental Studies on Fabrication and Thermal Characterization of Shape Memory Polymer Composites with Nanofillers

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Received 13 May 2022; Revised 10 June 2022; Accepted 18 June 2022; Published 12 July 2022

Academic Editor: Ram Prasad

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Shape memory polymers (SMP) have piqued the interest of the research and development sector due to their new features and immense potential. The polymer matrix epoxy was changed using varied weight percentages of aliphatic monomer-neopentyl glycol diglycidyl ether (NGDE), and the optimal weight percentage of NGDE was identified. The weight percentages of NGDE-CNT are 0, 0.3, 0.6, 0.9, 1.2, and 1.5. The use of nanofillers such as carbon nanotubes (CNT) has been promoted to improve the heat conductivity of composites. The thermal conductivity of shape memory polymers has been enhanced through the addition of varying quantities of CNT filler. Mechanical characterizations such as tensile testing, Young's modulus, impact strength, and heat related tests such as form recovery testing, shape fixity, and recovery time were also investigated. From the investigational outcomes, optimal weight percentage of NGDE loading into epoxy is 15%, and maximum tensile strength attains at 1.2 wt. percent of CNT.

1. Introduction

Shape memory polymers are a form of shape memory material (SMM) that can return to their original shape after being altered by an external stimulation. SMP are now part of a new family of materials called as “smart polymers,” and they have the unique ability to be moulded into a temporary shape, store that tempo-

rary shape, and then return to its original shape when prompted by an external signal. However, the SMP actuation speeds are restricted by the poor heat conductivity of polymers, which is typically less than 0.3 W/m-K. [1–4] Polymer shape memory effect is caused by their exceptional molecular structures. A distinctive polymer is a complex combination of several long, intertwined molecular chains linked by distinctive net sites

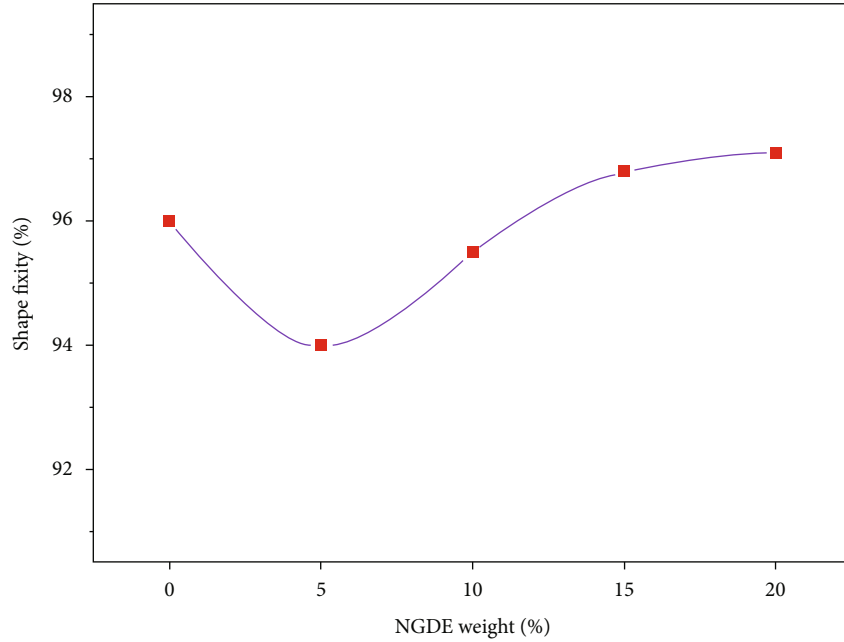


FIGURE 1: Shape recovery values of epoxy with various weight percentages of NGDE.

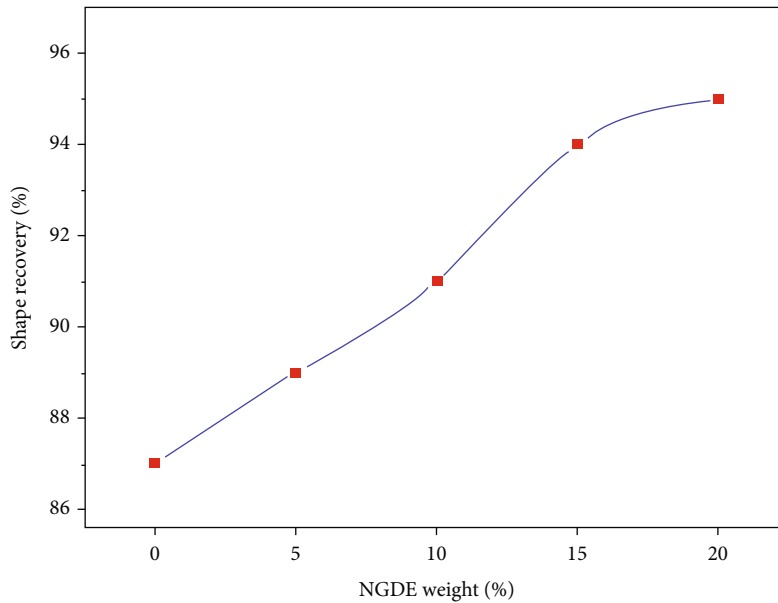


FIGURE 2: Shape fixity values of epoxy with various weight percentages of NGDE.

formed by either chemical or physical crosslinking. The polymer chains relax and become more flexible at temperatures above the glass transition temperature (T_g) and as a result, the polymer softens and becomes rubbery [5, 6]. Once a force is applied to the polymer chains, they may be elongated or constricted to great lengths, and the remaining points can also be relocated. Secondary crosslinks can develop among distorted polymer chains when the temperature is decreased under the glass transition temperature while keeping the predeformed shape. These minor crosslinks aid in the stabilization of the polymer once the external force is withdrawn [7–9]. As soon

as the polymer is heated again above the transition temperature, the secondary crosslinks are loosened, and the original form is restored. However, there are potential uses for SMP in almost every aspect of life, including self-repairing automobile bodywork, kitchenette tackles, switches, radars, intelligent packaging, toys, and tools. Hu and Chen [9] establish to shape memory fibers (SMF) utilized into fabric assembling might make clothing restore their actual level shape when heated above glass transition temperature. However, if the wearing person of a fabric changes, the cloth may quickly adapt to the new wearer's shape without the need for tension. Some of the investigations

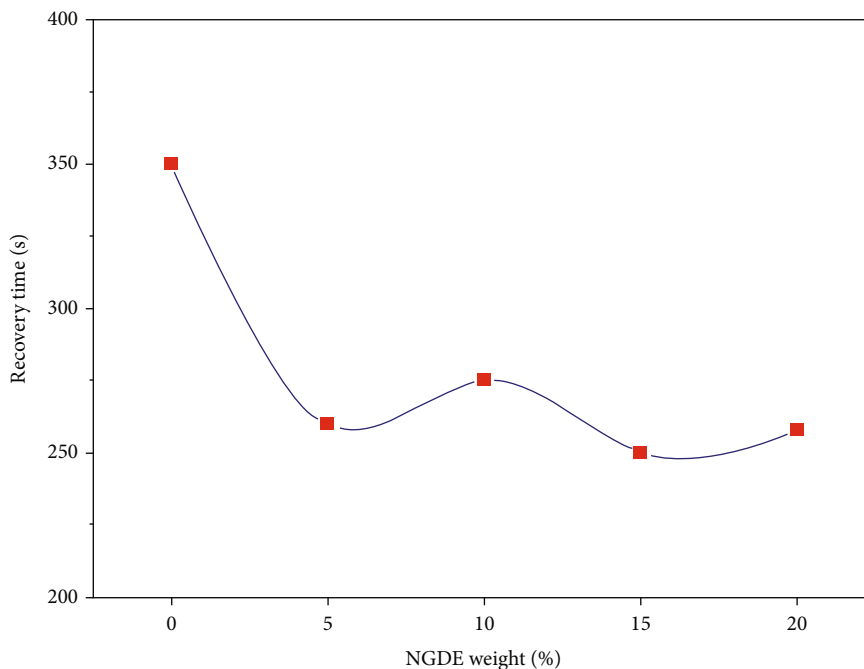


FIGURE 3: Recovery time of epoxy with various weight percentages of NGDE.

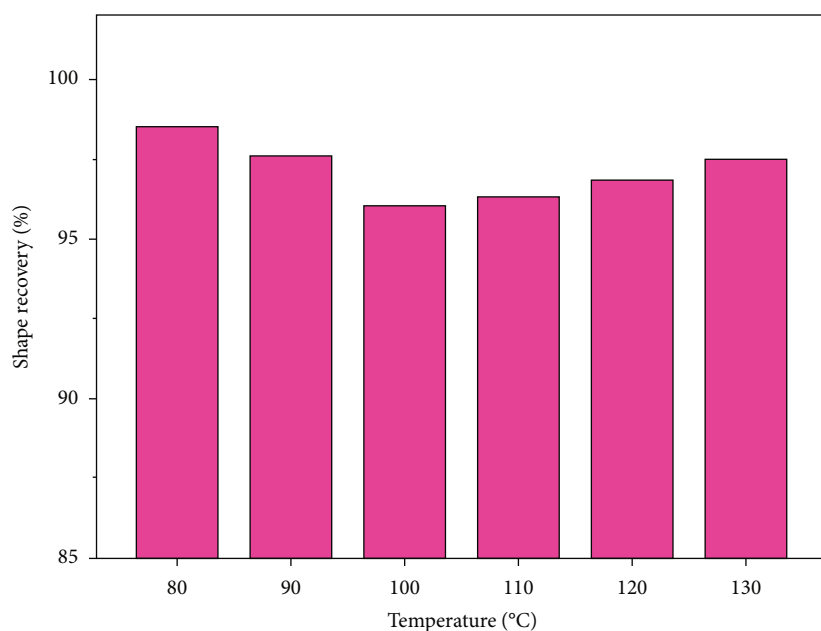


FIGURE 4: Shape recovery values of E-15NGDE sample at different temperatures.

conducted in the literature [10–13] include deployable hinges, glasses, reflectors for antennas, and transforming wings. Nanostructures serve as a link between molecules and larger systems. Clusters, quantum dots, nanocrystals, nanowires, and nanotubes are examples of individual nanostructures, whereas multi-nanostructures are arrays, assemblies, and super lattices of individual nanostructures. Nanoparticle shapes are extremely essential in defining features like reactivity and electronic spectra. Carbon nanotubes (CNTs) have been the subject of intense research and development since their discovery in 1991

[14–16]. By employing different types of monomers or fillers, the thermal transition temperature, which is critical in shape memory qualities, may be altered to meet individual demands. Because of these qualities, epoxy-based polymers are an excellent study material for shape memory investigations. Using extra monomers to change the shape memory properties of epoxy thermosets is a valuable method. The thermal conversion temperature of epoxy polymers can be altered by the presence of monomers. Thus, neopentyl glycol diglycidyl ether is generally utilized related to the diglycidyl ether of bisphenol-an epoxy

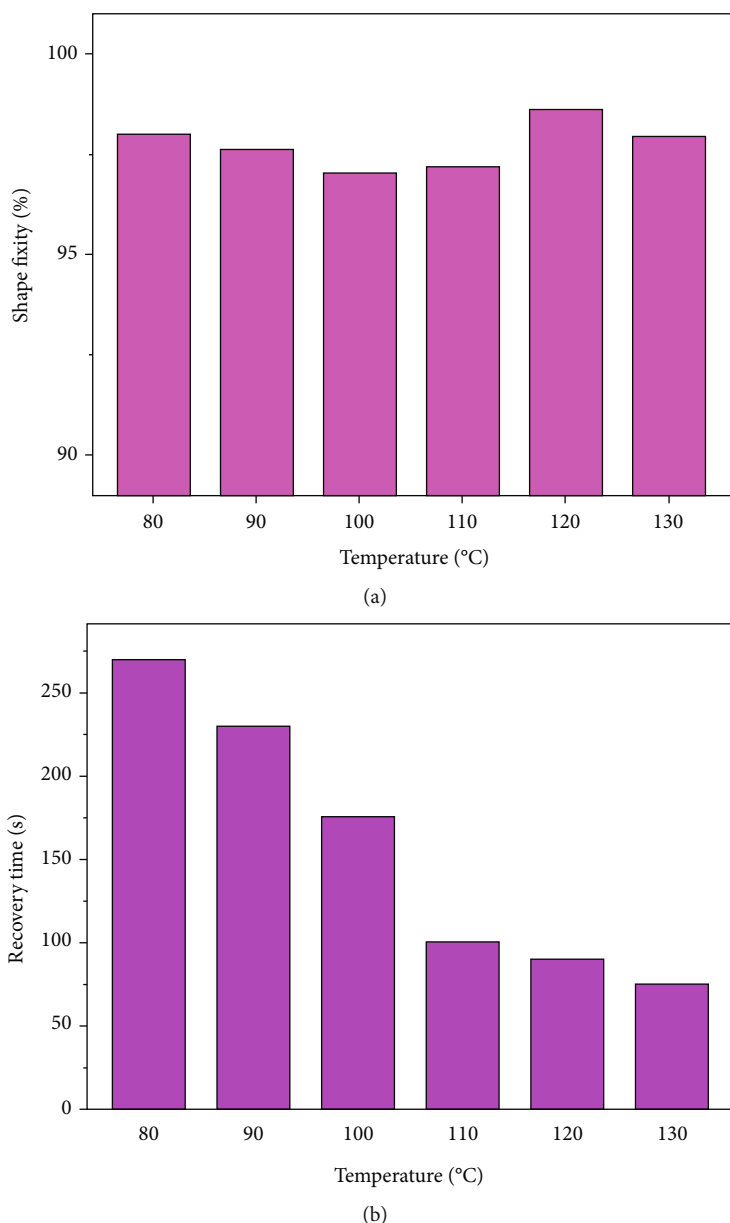


FIGURE 5: (a) Shape fixity. (b) Recovery time of 15% NGDE sample at different temperatures, respectively.

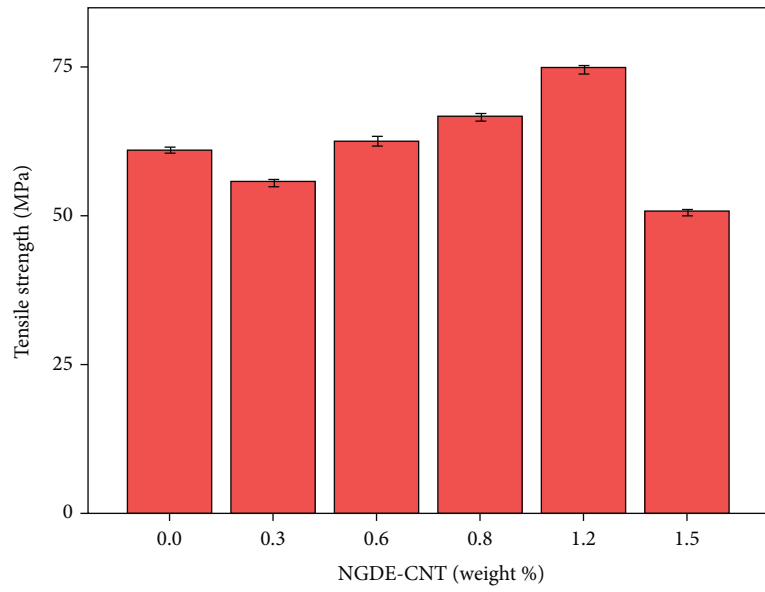
pitch [17, 18]. The essential goal of this work stayed makes shape memory polymers utilizing thermoset-based epoxy resin. Monomers with aliphatic structures were used to modify epoxy resin, and the effects of the monomer on mechanical, thermal, and shape memory qualities were studied. Following that, SMA composites of modified epoxy were created utilizing CNT at different concentrations of 0.3, 0.6, 0.9, 1.2, and 1.5 wt.% by weight. The mechanical, thermal, electrical, and shape memory characteristics were investigated.

2. Experimental Methods

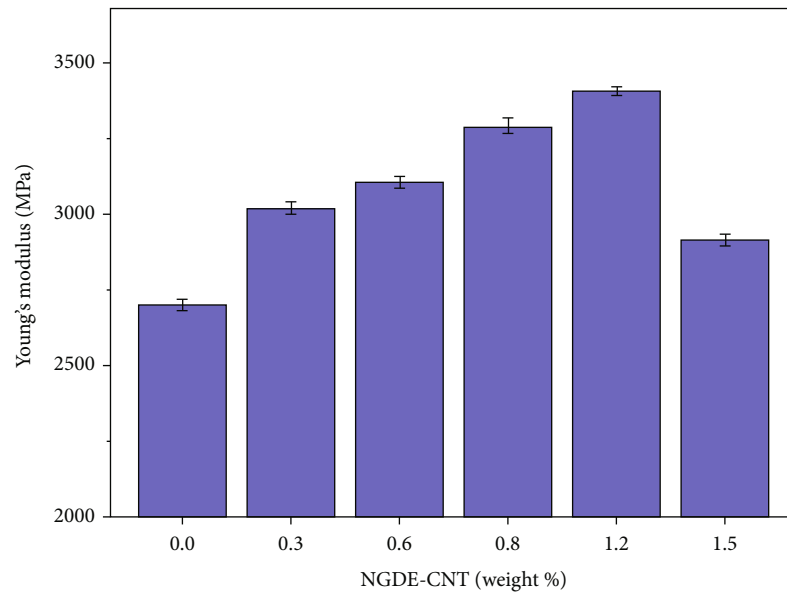
As portrayed in the underlying activity, the epoxy and epoxy curing agent can be kept inside a vacuum oven for three hours at 60°C and -0.8 bar. Following 30 minutes at room temperature, the monomer was blended in with epoxy resin

for 15 minutes at 40 rpm. The polymer's monomer content ranged between 5%, 10%, and 15% by weight. The combination was then degassed for 45 minutes in a stove at 55°C and -0.8 bar prior to being refrigerated for half an hour. The monomer (epoxy) combination and epoxy curing agent were then joined for 20 minutes at 50 rpm prior to utilizing a similar restoring cycle as in the earlier technique. Carbon nanotubes were utilized as conductive fillers, and they likewise consolidated to shape hybrid fillers. For each of them, the identical approach was followed. Carbon nanotubes at concentrations of 0.3, 0.6, 0.9, 1.2, and 1.5 wt.% were used to make the composites.

2.1. Characterization of Shape Memory Properties. Initially, any stimulation (heat, electricity, etc.) raises the temperature of the specimen over the transition point, and a positive



(a)



(b)

FIGURE 6: Continued.

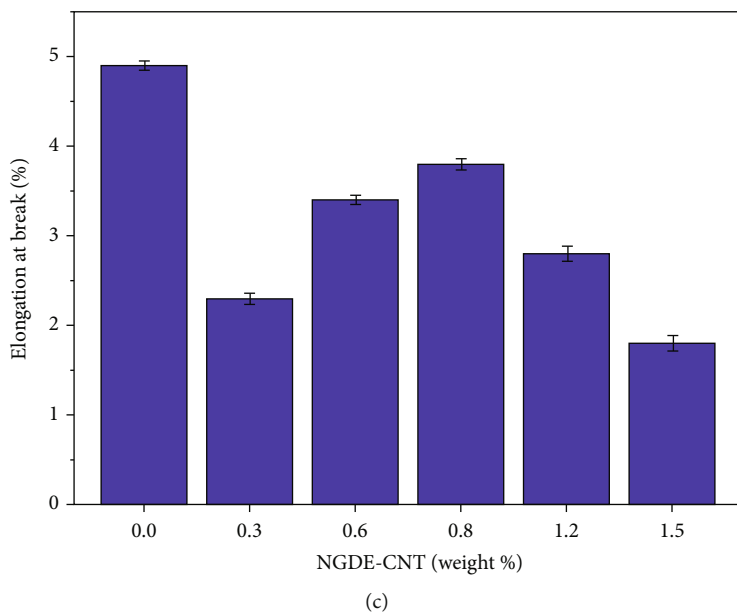


FIGURE 6: (a) Tensile strength of CNT reinforced NGDE polymer composites. (b) Young's modulus of CNT reinforced NGDE polymer composites. (c) Elongation at break of CNT reinforced NGDE polymer composites.

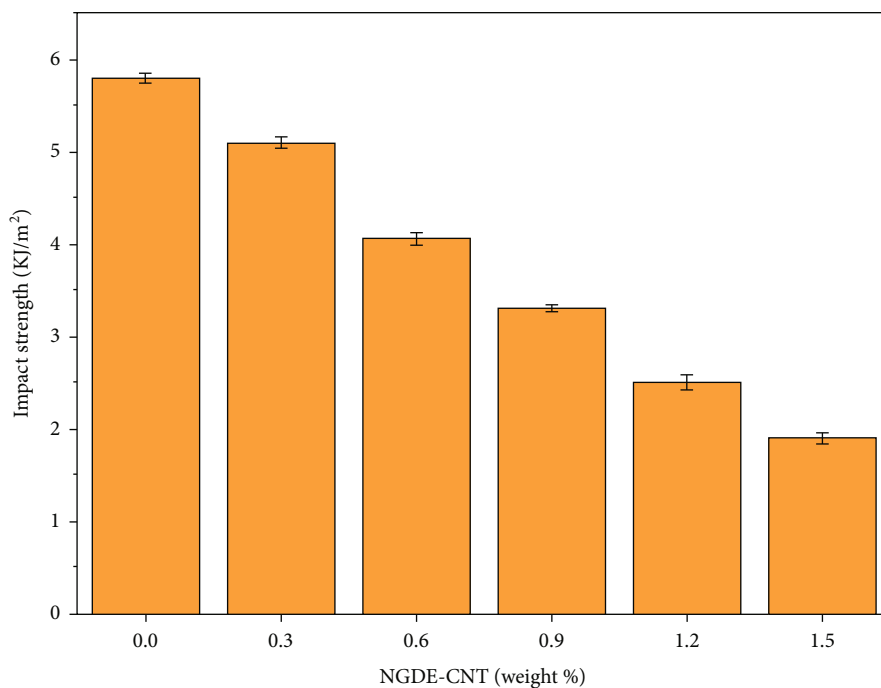


FIGURE 7: Impact strength of CNT reinforced NGDE polymer composites.

distortion is given to the specimen outwardly. The stimulus is then removed, and the strain is recorded as L_i after the temperature of the sample returns to room temperature. Additional physical crosslinks are produced when the sample is cooled due to the solidification of switching domains. Stress is exposed at this moment, depending on the material's form fixity feature. L_f is the name of a new strain. Shape fixity is determined as the proportion of the over two strain values, i.e., L_i/L_f . The deformation angle is

denoted by the symbol (θ_f) . The sample's final angle is denoted as θ_r , and the shape recovery proportion may be computed as the ratio of $(\theta_f - \theta_r)/\theta_f$ [15].

3. Results and Discussion

3.1. Thermal Study in Shape Memory. Shape memory samples and thermal study experiments were performed on

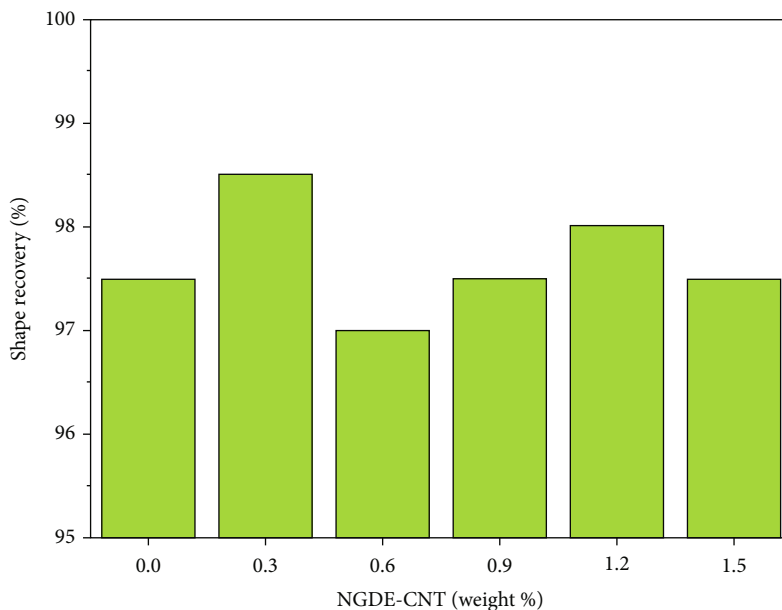


FIGURE 8: Shape recovery of NDGE with various weight percentages of CNT.

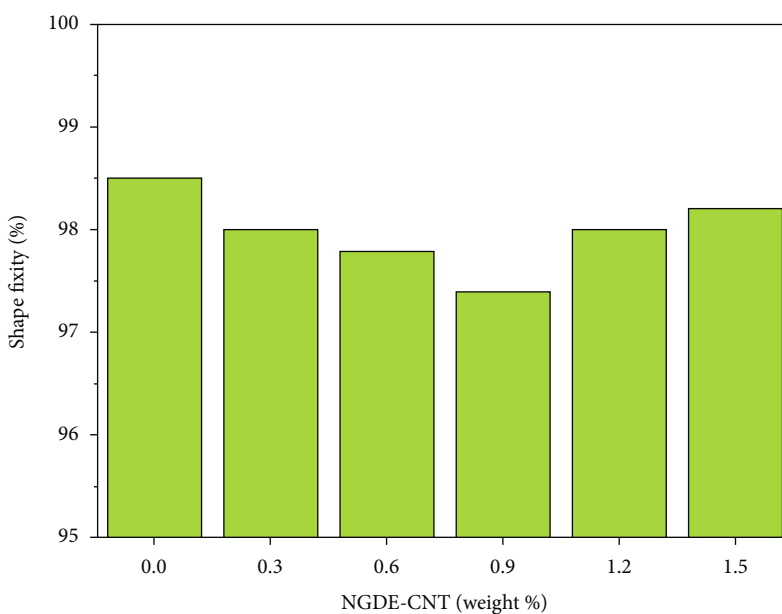


FIGURE 9: Shape fixity of NDGE with various weight percentages of CNT.

monomer-containing samples, and the findings are displayed in Figures 1–3.

Figure 1 depicts the form recovery for epoxy samples with varied quantities of monomer. With the addition of NGDE monomers, a growing tendency for shape recovery is noticed, and it can be noted that the rate of rise of shape recovery is determined to be low after the 15 wt. percent. To increase the shape memory capability of the brittle epoxy matrix, the monomer NGDE was introduced to the epoxy matrix. By incorporating low atomic weight and carbon link

molecules into the matrix, the structural was able to achieve the required deformability by forming a delicate section.

Figure 2 depicts the values of shape fixity for epoxy samples having varied quantities of monomer. The form fixity of the epoxy sample with 5 and 10 wt. percent NGDE is found to be lower than that of the clean epoxy, and when loaded beyond 10 wt. percent, the shape fixity increases. According to prior research, adding linear monomer reduced crosslink density while increasing elastic ratio. It was argued that a high elastic ratio resulted in high form fixity during cooling,

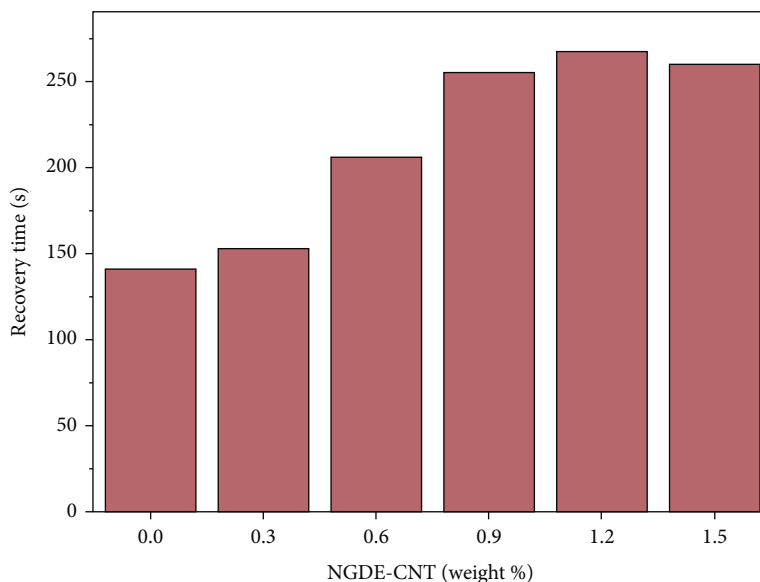


FIGURE 10: Recovery time of NDGE with various weight percentages of CNT.

and that it may perhaps fix a significant quantity of strain with a little amount of tension. As per this contention, it is conceivable to expect an enhancement in fixity property with bringing extra structural into the framework.

Figure 3 depicts the recovery time for epoxy samples having increasing quantities of monomer. As can be observed, the recovery time decreases as the monomer quantity in the structure increases. This impact is brought about by a prevailing delicate fragment because of low crosslink solidity. The presence of monomers, particularly aliphatic structural, in the construction lessens crosslink density. Among the expansion of extensive chains, the space between crosslink areas becomes more noteworthy, bringing about a drop in crosslink density. At high temperatures, a material with a lower crosslink thickness has more powerful organizations, and free volume change is less constrained. Furthermore, tests with low T_g started recuperating before, and subsequently, full recuperation happened in a more limited period. Due to these variables, it is conceivable to infer that recuperation is considerably quicker for monomer-added tests.

3.2. Effect of Temperature in Recovery. Based on prior research, the optimal weight proportion of NGDE to epoxy was established to be 15%. Shape memory tests were performed at 80°C to 130°C to analyze the influence of temperature on shape memory. In the following investigations, epoxy samples containing 15% NGDE (E-15NGDE) were evaluated in order to determine the best temperature for future shape memory testing.

Figure 4 displays the general form recover percent, illustrating that shape recovery did not do very much in the temperature range of 80°C to 130°C. Shape fixity values were stable over 98 percent in Figure 5(a). Other investigations have demonstrated that as long as the application temperature is beyond T_g , there are no significant changes in shape

recovery and fixity. Further, the test was conducted on materials containing 15% monomer, which demonstrated improved recovery and fixity even at 90°C. During this climate, further advancement of these qualities was impractical. Furthermore, this is related to the fact that the hard to soft segment ratio is the most important determinant of these two attributes. The use of different temperatures had no influence on the hard to soft segment ratio, but it did affect how rapidly the temperature of the sample was raised during heating. Figure 5(b) demonstrates that the form recovery time has greatly lowered, which is consistent with the prior observations. As the application temperature increased, the polymer heated up faster and began to recoup faster than other samples, resulting in significantly faster recuperation as compared to experiments conducted at lower application temperatures.

The most appropriate application temperature was decided based on the findings of the prior inquiry. It appeared to be normal to work at higher temperatures to accomplish the speediest recuperation time; in any case, delivering a more useful strategy was likewise a point of this review, and extremely high application temperatures would not help with building a straightforward procedure for shape recuperation activity.

3.3. Addition of Carbon Nanotube Effects in Mechanical Properties. Tensile testing revealed that the material containing 1.2 wt.% carbon nanotube had the highest tensile strength, as shown in Figure 6(a). The stress focused areas created by the heavily agglomerated particles in the matrix may explain why tensile strength declined substantially as the amount of carbon nanotubes increased. Additionally, the carbon nanotube composite was predicted to have the better tensile strength due to its high aspect ratio.

Figure 6(b) shows that increasing the amount of carbon filler in the composites resulted in a small improvement in modulus. The carbon nanotube fillers' high aspect ratio

contributed to the polymer's hardness, leading to a high elastic modulus results for carbon nanotube-filled composites, particularly at 1.2 weight percent loading. At fixations more than 1.2 wt. percent, the epoxy blend turned out to be really thick, and agglomerations forestalled further increment of the flexible modulus upsides of these specimens. Figure 6(c) shows an overall decrease in elongation at break values as filler loading increased. This reduction in impact was induced by the extremely brittle nature of carbon-based composites.

Figure 7 displays the impact strength values of CNT reinforced NGDE polymer composites; notably, a decline in impact strength was seen in this work for binary carbon nanotube composites. The greatest one was composites' high brittleness, which decreased the material's durability to affect crack. Second, the presence of air bubbles in the incredibly thick epoxy matrix decreased the actual cross-sectional region of the effect tests, bringing about lower impact strength values for the binary composites.

3.4. Thermal Shape Memory Tests of Composites. The addition of carbon nanotubes to the matrix as a filler had no influence on the form recovery percentage. Because of the significant change in modulus, the form recovery effect is observed during the glass transition for epoxy-based polymers [15]. As a result, the impact of carbon particles on this property was not readily discernible. The thermal properties of the polymer matrix had the most influence on shape recovery properties as shown in Figure 8. The maximum recovery is attained at the weight percentage of 0.3 NGDE-CNT.

Figure 9 shows the shape fixity values for all composites. Composites comprising solely carbon nanotubes showed a modest reduction. The soft section of the polymer influences the shape fixity characteristic. Carbon-based chemical substances oftentimes add to the polymer matrix's hard part, making the delicate area less predominant. For this situation, a slight decrease is not out of the ordinary. Hybrid composites, on the other hand, showed just a little increase in structural fixity, indicating that the hard component of these samples was not as viable as the composites containing solely carbon nanotubes. This contention is confirmed by the more limited recuperation time of hybrid composites. Figure 10 depicts the recovery time of shape memory samples. Samples with a greater concentration of carbon nanotubes recovered more slowly. However, at 1.2 percent CNT loading, there was a significant reduction in recovery time. The crucial step of the matrix was upheld via carbon-based parts, and recuperation time was connected with the hard portion's stability.

The highly developed hard domain of carbon nanotube composites resulted in a decrease in soft segment activity, leading in an increase in shape recovery time. The decrease in NGDE-CNT (0%) might be due to structural inhomogeneity generated by air bubbles. In this composition, carbon nanotubes were unable to display their entire effectiveness. Carbon black composites recovered quicker than carbon nanotube composites due to weaker hard portions in their structure, as shown in Figure 10.

4. Conclusion

NGDE monomer was employed to modify the epoxy matrix, and carbon nanotubes (CNT) were used to strengthen the plain epoxy polymer. The mechanical, thermal, and shape memory qualities were studied, and the following findings were reached:

- (i) The addition of NGDE monomers to the epoxy improved form recovery and shape fixity values while decreasing recovery time
- (ii) It has been discovered that the optimal weight percentage of NGDE loading into epoxy is 15%
- (iii) Tensile testing revealed that the material containing 1.2 wt.% carbon nanotube had the highest tensile strength
- (iv) Carbon nanotube fillers with a high aspect ratio increased polymer hardness, resulting in high elastic modulus values for carbon nanotube filled composites
- (v) Impact strength was lowered owing to the comparatively brittle nature of carbon-based composites
- (vi) A composite containing 1.2 wt. percent CNT and 15 wt. percent NGDE monomer reinforcement performed well in all shape memory qualities

Data Availability

The data used to support the findings of this study are included within the article. Further data or information is available from the corresponding author upon request.

Conflicts of Interest

The authors declare that there is no conflict of interest regarding the publication of this article.

Acknowledgments

The authors appreciate the supports from Mizan Tepi University, Ethiopia, for providing help during the research and preparation of the manuscript. The authors thank the University of Manchester for providing assistance to complete this work. This work was funded by the Researchers Supporting Project Number (RSP-2021/265), King Saud University, Riyadh, Saudi Arabia.

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