

Research Article

Synthesis, Thermal Adsorption, and Energy Storage Calibration of Polysulfone Nanocomposite Developed with GNP/CNT Nanofillers

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The growth of polymer-based materials is becoming requisite in various industrial applications like energy storage, automobile, membrane, and orthopaedics, due to advantages over conventional metallic metal, such as less weight, superior corrosion resistance, ease of the process, and good chemical stability. The current research work is to synthesize the polysulfone (PSU) nanocomposite consisting of 2 wt%, 4 wt%, and 6 wt% of graphene nanoplatelets (GNP) and 3 wt%, 5 wt%, and 7 wt% of carbon nanotube (CNT) nanofillers via cast solution technique. The synthesized composite microstructural, heat storage, and thermal adsorption characteristics are studied. The scanning electron microscopic examination for both PSU/GNP and PSU/CNT composites illustrates good interfacial bonded PSU structure with the uniform distribution of GNP and CNT nanofillers. Due to the effect of percolation, the thermal adsorption characteristics and heat storage of PSU nanocomposite were increased progressively with the additions of GNP/CNT. The PSU composite contained 6 wt% GNP and 7 wt% CNT nanofillers, which showed effective thermal conductivity of 1.23 W/m.K and 1.52 W/m.K, which is 1.7 times larger than the unreinforced polysulfone. Interestingly, the increased temperature of the glass transition decreased the thermal expansion of the nanocomposite.

1. Introduction

Generally, polymers are employed broadly in various applications due to their suitability for low cost, lightweight, and good corrosion resistance but have deprived mechanical and thermal properties compared to metallic compounds [1]. At the earliest, In 1990's, traditional filler-reinforced clay compounds and polymer-laid silicate composites have applied in various industry (low load) applications [2, 3]. However, the addition of multiple

nanofillers into the polymer layer has good thermal conductivity as well as it has good mechanical properties [4]. Recently, the demand for polysulfone material increased in membrane applications like bioreactors, fuel cells, and food processing and storage due to its superior thermal conductivity [5, 6]. Sohail et al. [7] developed and studied the thermal conductivity effect of ceramic nanofiller bonded polymer nanocomposite. They found the increased thermal on the presence of aluminium nitrides. Weidenfeller et al. [8] synthesized

TABLE 1: Characteristics of polysulfones.

Properties	Density	Hardness	Tensile modulus	Tensile strength	Linear thermal expansion coefficient	Service temperature-air	Deflection temperature at 1.8 MPa	Transmission-visible
Units	g/cc	Hv	GPa	MPa	$\mu\text{m}/\text{m}^\circ\text{C}$	$^\circ\text{C}$	$^\circ\text{C}$	%
	1.25	120	2.48	70.3	55.8	141	174	90

TABLE 2: Characteristics of GNP and CNT nanofiller.

Material/properties	Particle size nm	Density g/cc	Tensile strength GPa	Elastic modulus TPa	Thermal conductivity W/mK
GNP	10-50	0.1	125	1.1	500-600
CNT	10-50	1.74	200	1.7	3000

magnetite (Fe_3O_4) filled polymers for electrical applications. They found increased thermal conductivity on 44 vol% of Fe_3O_4 . The polymer-based materials are bonded with copper, magnetite, barite [9], titanium dioxide (TiO_2) and boron nitride [10], CNT/ TiO_2 [11], and modified graphene oxide [12, 13]. Among the various investigations, the carbon nanotube (CNT) and graphene nanoplatelet (GNP) bonded polymer systems have good adhesive, enhanced thermal properties, and reliability. It has been used in electronic devices [14–16]. Hu et al. [17] developed GNP/CNT layered poly(lactic acid) nanocomposite for electronic sensing (strain) devices. They reported that both layers in poly(lactic acid) nanocomposite have good thermal stability. Takenaka and Ichigo [18] reported that the negative or lower thermal expansion of material was estimated easily, and the zero thermal effect of GNP and CNT sensing has been found at an ambient temperature of 25°C . So, the presence of GNP/CNT in a polymer matrix has good thermal sensitivity and is recommended by thermal energy storage applications. However, adding a larger filler in the polymer matrix affected the agglomerate structure and minimized the aspect ratio of surface volume. So, the present study evaluates the microstructure, thermal adsorption, and heat energy storage of polysulfone (PSU) nanocomposite containing 2 wt% to 6 wt% of graphene nanoplatelets (GNPs) and 3 wt% to 7 wt% of carbon nanotube nanofillers with an interval of 2 wt%.

1.1. Selection and Processing of PSU Nanocomposites

1.1.1. Selection of Polymer. The present research polysulfone material is chosen as the primary material due to its transparency, high strength, superior heat resistance, and stability on repeated steam sterilization cycles [19]. It has a suitable glass transition temperature span from 180°C to 250°C . Their characteristics are specified in Table 1.

1.1.2. Selection of Secondary Phase Materials. The graphene nanoplatelets (GNPs) and carbon nanotubes (CNT) were selected as secondary phase materials, and their characteristics are valued in Table 2. Among the various epoxy materials, both effective GNPs and CNT are reinforcements for

improving the material properties like biological and mechanical, thermal, tribological, and electrical [20].

1.2. Processing of PSU Nanocomposites. Figure 1 illustrates the detailed flow processing diagram explaining the concept of pre- and postprocessing of polysulfone nanocomposites containing different weight percentages of GNP/CNT nanofillers.

1.3. Pre- and Postprocessing of PSU Nanocomposites. Based on Stokes' law (equation (1)), the essential quantity of polysulfones is bonding on (2 wt%, 4 wt%, and 6 wt %) of GNP along with varying weight percentages (3 wt%, 5 wt%, and 7 wt %) of CNT nanofillers developed via cast solution technique. The combinations of PSU and its reinforcements are mentioned in Table 3. Initially, GNP and CNT are weighted according to Table 2 composition via a digital weighing machine with an accuracy of ± 0.001 grams. Similarly, polysulfones are dipped in methyl-2-pyrrolidone solution. Correspondingly, the GNP and CNT are blended with the above matrix. The combinations of polymers with reinforcements are solidified for 120 mins via a probe-type solidifier and continued stirrer action for 1 hour to obtain a homogeneous mixture. It helps to minimize the defects on PSU nanocomposites [19]. The stirred PSU/GNP/CNT is mixed with the solution mentioned above to get a homogeneous mixture. The solution phase PSU/GNP/CNT is moulded into a desired specific shape. The temperature flow on PSU/GNP/CNT/solution is maintained at $75^\circ\text{C}/\text{min}$, which leads to the evaporation of the solution phase converted into a solid phase. After dry conditions, the prepared nanocomposites' outer thin film layer (less than 2 mm) is detached for further studies.

$$F_d = 6\pi\mu Rv, \quad (1)$$

where F_d is the frictional forced (Stokes drag), μ is the dynamic viscosity, R is the radius of the sphere, and v is the flow velocity.

2. Results and Discussions

2.1. Influences of GNP/CNT on Microstructure Characteristics of PSU Nanocomposite. Figures 2(a)–2(g) represent the SEM micrograph of pure PSU and GNP/CNT bonded PSU nanocomposites. Figure 2(a) shows the slag-free micrograph of a pure PSU matrix. It was observed from Figures 2(b)–2(d) that the detailed constructional view of GNP and its bonding quality of the PSU matrix had enhanced the composite characteristics. The reason for continuous stir action obtained it for

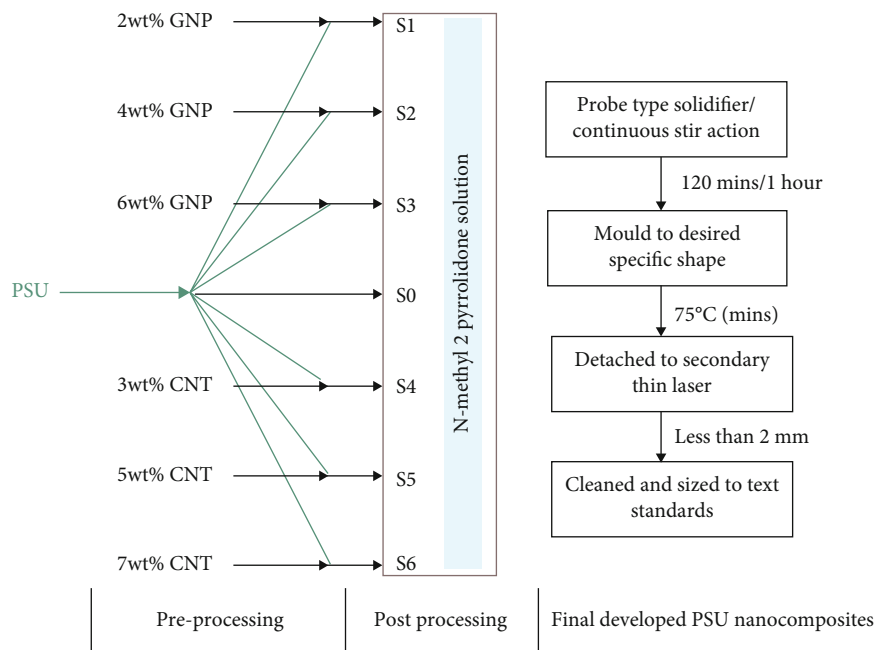


FIGURE 1: Flow processing diagram for PSU nanocomposite fabrication.

TABLE 3: Compositions of PSU nanocomposites.

Elements/sample no.	Units	S0	S1	S2	S3	S4	S5	S6
Composition ratio	wt%	Pure PSU 100/0/0	2 GNP 98/2/0	4 GNP 96/4/0	6 GNP 94/6/0	3CNT 97/0/3	5CNT 95/0/5	7CNT 93/0/7
PSU	Grams	100	98	96	94	97	95	93
GNP/CNT	Grams	0	2	4	6	3	5	7

more than 5 hrs. Similarly, Zhang et al. [13] obtained a fine crystal graphene oxide structure on a PET matrix.

Figure 2(a) indicates the quality connection between PSU and GNP. The fine mesh connection on GNP bonded in the PSU matrix is uniformed. The structure leads to enhance thermal performance as well as reduces the mass during heat storage. Similarly, Figures 2(c) and 2(d) illustrate that the wire mesh connection with good interfacial bonding results in increased thermal conductivity and reduced composite mass loss.

Figure 2(e) shows an effective mesh with PSU composite that resulted in a unique thermal effect on high-temperature storage [5, 6]. Figures 2(e)–(g) show the detailed multilayer mesh connection between PSU and CNT at approximately 15 nm. The fine, coarse multimesh connection was obtained using methyl-2-pyrrolidone solvent at 75°C for 1 hr. The homogenous mixing of the second phase into the primary PSU phase on the solvent medium of methyl-2-pyrrolidone was successfully obtained by continuous stir action under 1 hr at 75°C.

The CNT nanofillers make good interfacial strength and lead to a better conduction path with the enhanced thermal stability of the energy storage system. The multidirectional PSU and CNT network formation was closely observed and illustrated in Figure 2(g). The firm matrix surfaces were

attracted between the PSU and CNT filler on lower rate dispersion during high thermal conductivity [21].

2.2. Effect of PSU/GNP and PSU/CNT on Thermal Properties of PSU Nanocomposites

2.2.1. *Thermal Conductivity.* Figures 3(a) and 3(b) illustrate the thermal conductivity variations of PSU/GNP and PSU/CNT nanocomposites with their nanofiller concentration. The actual thermal conductivity of PSU nanocomposites was evaluated by a thermocouple-based sensor system configured with digital indication. The measured thermocouple results were compared with the theoretical result to identify the enhancement of thermal conductivity [22, 23]. The effective temperature flow was applied for mono and hybrid filler bonded polymer composite. It was observed from Figure 3 graph that filler concentration’s effect on PSU nanocomposite thermal conductivity was closer to the theoretical value. However, the thermal conductivity of PSU/GNP and PSU/CNT nanocomposite was gradually increased with an increase in filler concentration. It was due to the reason that a multiphase interconnection network may lead to enhancing the filler concentration, resulting in increased thermal conductivity. However, the PSU nanocomposite containing 0, 2, 4, and 6 wt% of GNP showed an increased thermal conductivity of 0.45 W/

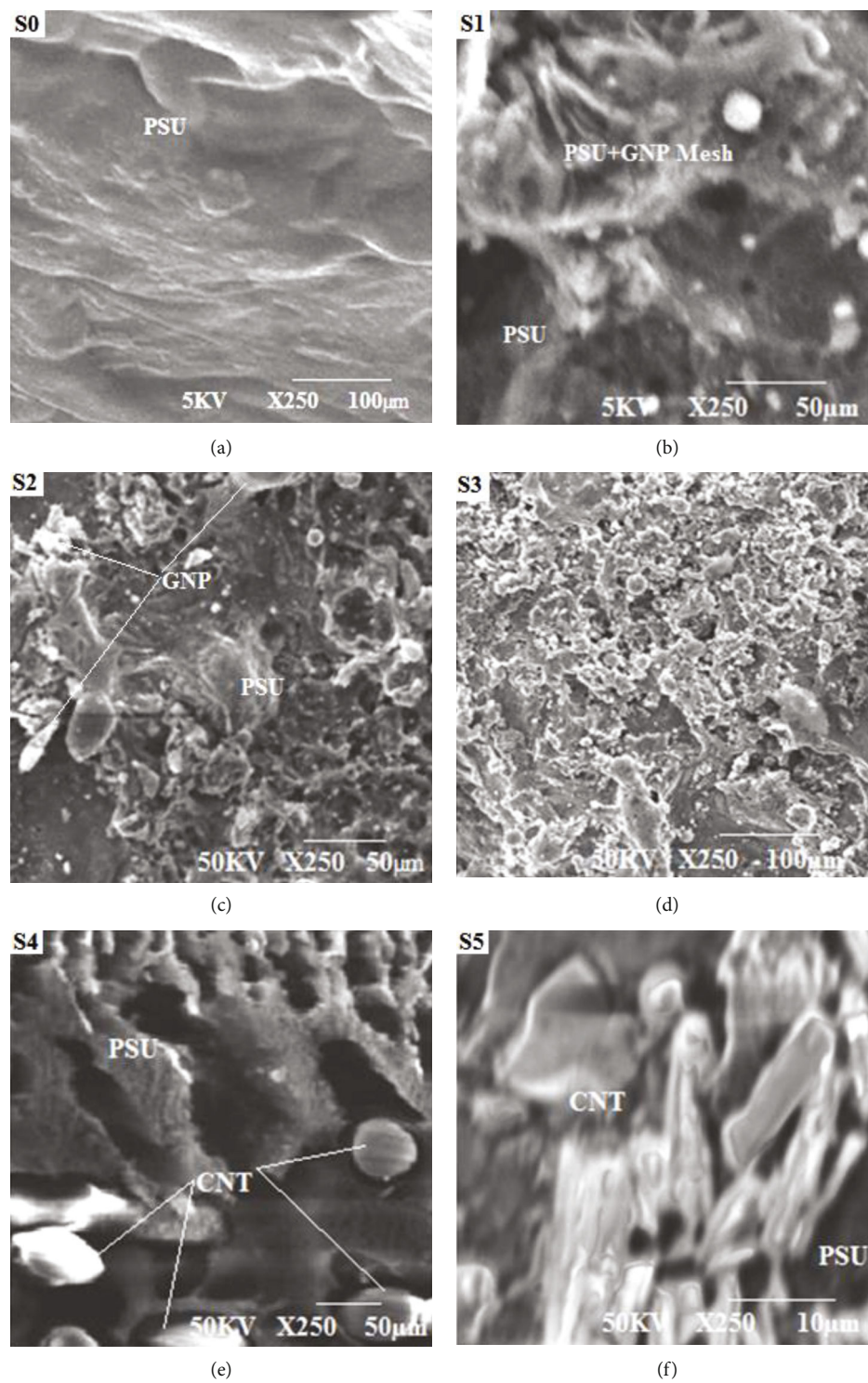


FIGURE 2: Continued.

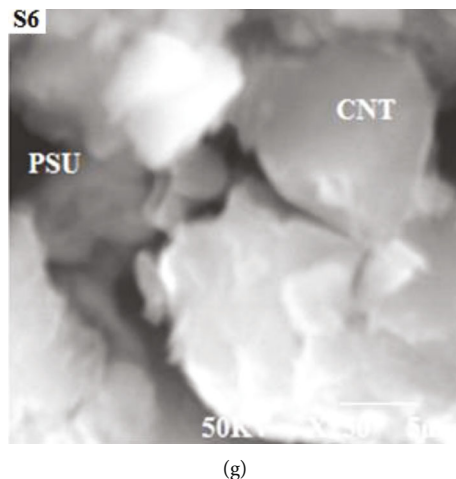


FIGURE 2: (a) SEM micrograph of pure PSU. (b) SEM micrograph of PSU/2 wt% GNP. (c) SEM micrograph of PSU/4 wt% GNP. (d) SEM micrograph of PSU/6 wt% GNP. (e) SEM micrograph of PSU/3 wt% CNT. (f) SEM micrograph of PSU/5 wt% CNT. (g) SEM micrograph of PSU/7 wt% CNT.

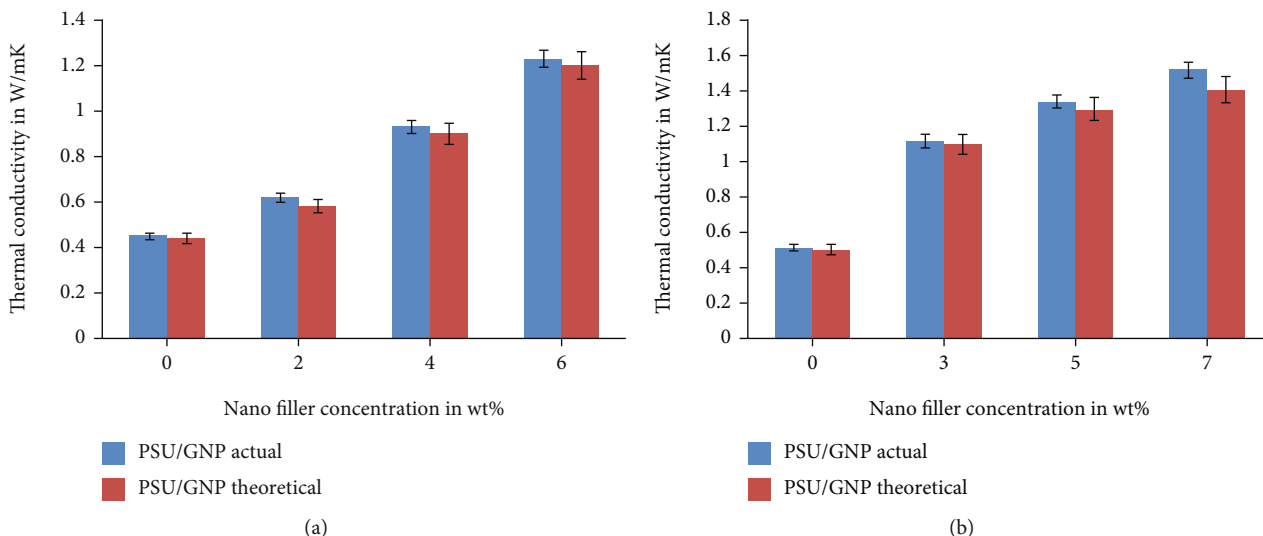


FIGURE 3: (a) Effect of GNP nanofiller concentration on thermal conductivity of PSU composite compared with the theoretical value. (b) Effect of CNT nanofiller concentration on thermal conductivity of PSU composite compared with the theoretical value.

mK, 0.62 W/mK, 0.93 W/mK, and 1.23 W/mK, respectively. It was because GNP can withstand high temperatures [12, 17].

It was observed from Figure 3(a) that the thermal conductivity of PSU/GNP nanocomposite gradually increased with an increase in GNP nanofiller. The transmission of higher heat energy requires good interfacial connection [20, 23]. The maximum thermal transient effect of 1.23 W/mK was found on 6 wt% GNP bonded PSU nanocomposite. It was 1.7 times higher than the thermal conductivity of the pure PSU matrix. It happened due to the uniform distribution of GNP connection in the PSU matrix, and effective interfacial bonding may be stable in higher temperatures. The proof of multi-interfacial connection is illustrated in Figures 2(b)–2(d).

The thermal conductivity of PSU/CNT nanocomposite was increased by 12.5% approximately as compared to

PSU/GNP composite. It was due to their combination of PSU and CNT. Generally, PSU is stable on repeated seam sterilization and has high heat resistance with a glass transition temperature span from 180°C to 250°C, and CNT has good thermal conductivity in different modes [24]. It is noted in Figure 3(b) that the composite’s thermal conductivity increased from 0.52 W/mK to 1.52 W/mK on 0, 3, 5, and 7 wt% of CNT. The effective connection of CNT in the PSU matrix accumulated the heat energy effectively. A similar trend was reported by the researcher [22]. The effective connections of PSU and CNT are evidenced in Figures 2(e)–2(g).

2.2.2. *Thermal Effusivity.* Figure 4 shows the thermal effusivity of PSU/GNP and PSU/CNT nanocomposite. The GNP and CNT represent a gradual improvement in thermal effusivity on the PSU matrix. The additions of GNP in the PSU

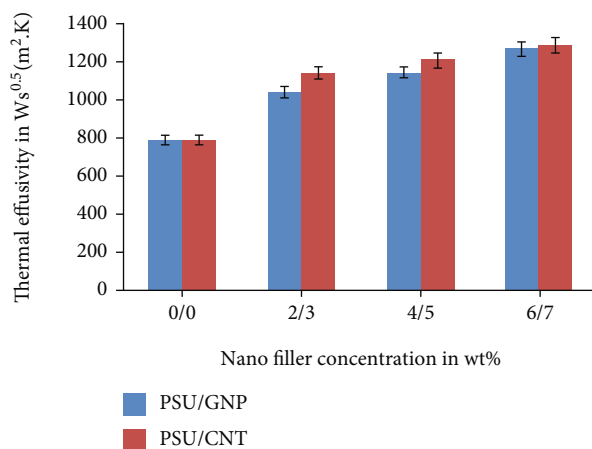


FIGURE 4: Effect of GNP and CNT nanofiller concentration on thermal effusivity of PSU composite.

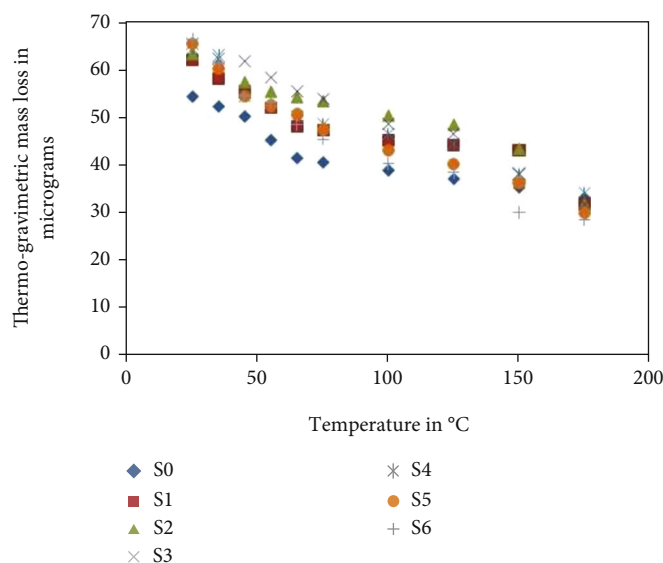


FIGURE 5: Mass loss during thermal adsorption effect of PSU nanocomposite.

matrix increased progressively from 790, 1041, 1145, and 1273 $W s^{0.5}/m^2.K$. The heat flow path of PSU/GNP nanocomposite compared with PSU/CNT nanocomposite exposed a superior effusivity value of 1290 $W s^{0.5}/m^2.K$ on 7 wt% of CNT nanofiller. It was due to the number of CNT nanofiller links increased in the PSU matrix. Kim et al. [25] reported that the increased interaction between the polymer matrix and secondary phase leads to enhanced thermal properties. Adding a higher nanofiller in the PSU matrix increases the thermal storage and transfer of charge electrons. So, it was proved that the thermal performance of composites was decided by the secondary phase volume, adhesion characteristics, and aspect ratio. The GNP and CNT are talented in high mixing ratios, resulting in high energy storage and good thermal conductivity. However, the concentration of both GNP and CNT in the PSU matrix increased by 61% and 63%, respectively.

2.3. Effect of Thermal Adsorption. The thermal behaviour of GNP/CNT with their composition effect for thermal adsorption properties of PSU nanocomposites were evaluated by thermo-gravimetric apparatus configured with the temperature range from 25°C to 700°C. The present work on the thermal adsorption effect on mass loss on PSU nanocomposites was estimated by 25°C to 175°C at 30 mins, which is shown in Figure 5. During this period, each stage of temperature adsorption behaviour on mass loss of PSU nanocomposite was measure via thermocouple sensor assisted with a digital system. It helps to identify the physical change of the composite [26]. The developed PSU nanocomposite (S0, S1, S2, S3, S4, S5, and S6) mass loss effect during the thermal adsorption study result is shown in Figure 5. It was observed in Figure 5 that the mass loss of PSU and its nanocomposites is showed downtrend with increased temperature. However, the mass loss curve of PSU and its

GNP/CNT nanocomposite shows minor decomposition that occurs during the high temperature of 175° at 25°C/min constant heat flow. The PSU composite contains 6 wt% GNP and 7 wt% CNT, showing enhanced thermal stability with minor mass loss of 31.8 µg and 28.4 µg, so that composite may damage the PSU matrix at low-level decomposition which may not be directed to reduce the properties of composite that are proved.

3. Conclusions

The effort of the current research work is forced to successfully develop the polysulfone nanocomposites containing 2 wt%, 4 wt%, and 6 wt% of GNP and 3 wt%, 5 wt%, and 7 wt% of CNT via cast solution technique to obey Stokes' law. The following conclusions are made below.

- (1) The scanning electron microscope micrograph of both PSU/GNP and PSU/CNT composites was studied, and their micrographs are illustrated in Figures 2(a)–2(g). It reveals that the GNP and CNT nanofillers were uniformly distributed and made good interfacial bonding between PSU/GNP and PSU/CNT
- (2) The CNT: the various/multiple links identified between GNP/PSU and CNT/PSU nanocomposite. PSU found good thermal behaviour as compared to GNP/PSU and PSU
- (3) The composite containing 7 wt% of CNT nanofiller found superior thermal conductivity of 1.41 W/m K and high thermal effusivity of 1290 Ws^{0.5}/m².K. It increased 1.7 times and 23.5% of the heat storage system compared to the PSU matrix and PSU/6 wt% GNP nanocomposite
- (4) The mass loss during the effect of thermal adsorption during the evaluation of PSU and its nanocomposite under 25°C–175°C at 25°C heat flow showed a minimum decomposition rate with little physical change. The composite contained 6 wt% of GNP and 7 wt% of CNT, showing 31.8 µg and 28.4 µg
- (5) Hence, the experimental research on PSU nanocomposite found that CNT has good thermal stability and may be applied to energy storage applications like food storage

Data Availability

All the data required are available within the manuscript.

Conflicts of Interest

The authors declare no conflicts of interest.

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