Scientific Research Journal,
Vol 22, special issue (2025) 1-18
https://doi.org/10.24191/srj.v22is.10958

Thermal Characterization of Silver-Based Conductive Polymers for Flexible Electronics

Zuraihana Bachok[1], Abdullah Aziz Saad[1,*], Sana Zulfiqar[2], Aizat Abas[1] , Marina Marzuki[3]

[1]School of Mechanical Engineering, Universiti Sains Malaysia, 14300 Nibong Tebal, Penang, Malaysia
[2]School of Mechanical and Manufacturing Engineering, National University of Science & Technology (NUST), Islamabad, 44000, Pakistan
[3]Faculty of Mechanical Engineering Technology, Universiti Malaysia Perlis, 02600 Arau, Perlis, Malaysia

[*]Corresponding Author's E-mail: azizsaad@usm.my

Received: 29 August 2024
Accepted: 3 September 2024
Online First: 25 January2025

ABSTRACT

Conductive polymer composites are increasingly important due to their unique electrical and polymeric properties, with applications in flexible electronics and sensors. Understanding their thermal behavior, especially in PDMS-OH with silver (Ag) nanoparticles, is essential for optimizing performance and reliability. This study investigates the thermal behavior of conductive polymer composites made from polydimethylsiloxane with hydroxyl end groups (PDMS-OH) and varying contents of silver nanoparticles (40%, 60%, and 80%) using thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC). The DSC result revealed that the 60% silver content offers an optimal balance between flexibility and processability, with a Tg of approximately 178.13°C and a delta Cp of 0.130 J/g°C. Meanwhile, TGA results indicate that the 60% silver conductive polymer also achieves superior thermal stability and minimal degradation, making it the most suitable choice for high-performance applications. Although increasing the silver content can improve thermal stability, excessive silver content may lead to adverse effects. Therefore, the PDMS-OH conductive polymer with 60% silver content emerges as the optimal formulation, providing a favorable combination of flexibility, processability, thermal stability, and minimal weight loss, outperforming both the lower and higher silver content formulations, which can lead to

more durable and efficient electronic components, enhancing the overall quality and lifespan of the end products.

Keywords: Conductive Polymer; Silver; TGA; DSC;PDMS-OH

INTRODUCTION

Polydimethylsiloxane with hydroxyl end groups (PDMS-OH) is a widely used elastomeric polymer known for its excellent flexibility, biocompatibility, and ease of processing [1]. However, its low inherent electrical conductivity limits its applications in flexible electronics and conductive materials [2]. To overcome this limitation, silver nanoparticles are incorporated into the PDMS-OH matrix, forming conductive polymer composites [2].

While the addition of silver nanoparticles improves electrical conductivity, it also influences the thermal properties of the composite. Nazarenko study reveals the addition of nanoparticle fillers increased the thermal stability and mechanical properties of the epoxy composites compared to the neat epoxy polymer [3]. Understanding these thermal behaviors is crucial for determining optimal processing temperatures, ensuring device stability during operation, and identifying potential degradation mechanisms. This thermal analysis encompasses a range of techniques, including differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA), to evaluate the behavior of materials as they undergo temperature changes. This study employs TGA and DSC to investigate how varying silver content affects the thermal behavior of PDMS-OH/silver nanoparticle composites. DSC measures the heat flow into or out of a material as it is heated, cooled, or held at a constant temperature, providing information about phase transitions such as melting and glass transitions as described by Ahmad Saidi *et al.* [4], Hu *et al.* [5] and Joseph *et al.* [6]. TGA measures changes in a material's weight as a function of temperature or time, offering insights into thermal stability, composition, and decomposition kinetics as explained by Ng *et al.* [7] and Yussuf *et al.* [8]. These techniques are essential for understanding and optimizing the performance of materials in various applications.

Conductive polymers combine the electronic properties of metals or semiconductors with the mechanical properties and processability of polymers. Extensive research has focused on these materials due to their potential applications in flexible electronics [9-13], bioelectronics [14], electromagnetic interference (EMI) shielding [12,15], and as components in batteries [16] and supercapacitors [17]. Bachok's *et al*. [18] revealed that incorporating silver nanoparticles into these polymers significantly enhances their electrical conductivity and mechanical strength. Conductive polymers, composed of polymer matrices filled with metallic particles like silver, are crucial in the production of flexible electronics, sensors, and other advanced materials. Silver is a preferred filler due to its outstanding electrical conductivity and stability. Ibrahim *et al.* 's [11] research indicates that the silver content in these conductive polymer formulations can vary from 20% to 80% silver. However, maintaining stability, particularly at high silver contents, presents significant challenges [11].

For conductive polymers to be effective, they must not only exhibit high electrical conductivity but also possess thermal stability to ensure strong adhesion to substrates, which is essential for creating high-quality flexible printed circuits. While numerous studies have focused on enhancing the electrical conductivity and stretchability of these materials [18–22], there is a noticeable gap in research dedicated to improving their thermal stability. To the best of the authors' knowledge, no studies have specifically addressed the thermal analysis of PDMS-OH/silver conductive polymers.

Understanding the impact of varying silver content on the thermal properties of conductive polymers is vital for optimizing their performance. This study aims to elucidate the relationship between silver content and the thermal properties of PDMS/OH conductive polymers using TGA and DSC techniques. By analyzing the thermal behavior of conductive polymers with different silver contents, this research seeks to determine the optimal composition for enhancing functionality.

METHODOLOGY

Sample Preparation

Table 1 lists the materials used to fabricate conductive polymers with varying silver content.

Table 1: Material used to fabricate conductive polymer

Materials	Properties	Function
PDMS-OH (epoxy resin)	Molecular weight: 110 x 10^3 g/mol, viscosity: 50 x 10^3 cSt	polymer matrix/ binder
ETMS (cross-linking agent)	Molecular weight: 236.34 g/mol, specific gravity: 1.07 g/ml at 25°C, purity > 98%	improve mechanical properties and adhesion
D4 (Organic solvent)	Density: 0.956 g/ml	used for processing the PDMS-OH
Toluene (viscosity controller)	Molecular weight: 92.14 g/mol, purity 99%, density: 0.867 g/ml	be a solvent or viscosity controller
Silver nanoparticles (conductive material)	Size: 2 – 3.5 µm	conductive filler
DBDTL (Catalysts)	Molecular weight: 631.56 g/mol, purity 95%, density: 1.066 g/ml	aid in the curing process
Acetic Acid (Catalysts)	Purity 99%	aid in the curing process

To prepare these conductive polymers, silver nanoparticles were combined with a PDMS-OH (polydimethylsiloxane with hydroxyl end groups) binder to achieve both electrical conductivity and stretchability. A small amount of toluene was added to the mixture to achieve the desired viscosity for processing, and the mixture was stirred for 24 hours using a magnetic stirrer. This step ensured complete dispersion of the silver nanoparticles within the PDMS-OH matrix, which is crucial for achieving uniform electrical conductivity in the final composite.

After the initial mixing, octamethylcyclotetrasiloxane (D4), an organic solvent, and (3-glycidyloxypropyl) trimethoxysilane (ETMS), a cross-linking agent, were added to the mixture to improve the mechanical strength and adhesion of the cured composite. This mixture was then stirred for an additional 3 minutes. Following this, a small amount of acetic acid and dibutyltin dilaurate (DBDTL) were added as catalysts to facilitate the curing process of the PDMS-OH matrix. The final mixture was carefully poured into uniform shapes suitable for DSC and TGA testing.

The curing process was conducted at a room temperature for 24 hours to solidify the PDMS-OH, permanently embedding the silver nanoparticles within the polymer matrix. The process for creating the conductive polymer samples is illustrated in Figure 1. Multiple formulations were prepared by varying the silver nanoparticles in the mixture. The formulations contained silver nanoparticles of 40%, 60%, and 80%, while maintaining the proportions of other components constant. The final samples with these formulations can be seen in Figure 2. The goal was to optimize the formulations to balance stretchability, electrical conductivity and thermal stability.

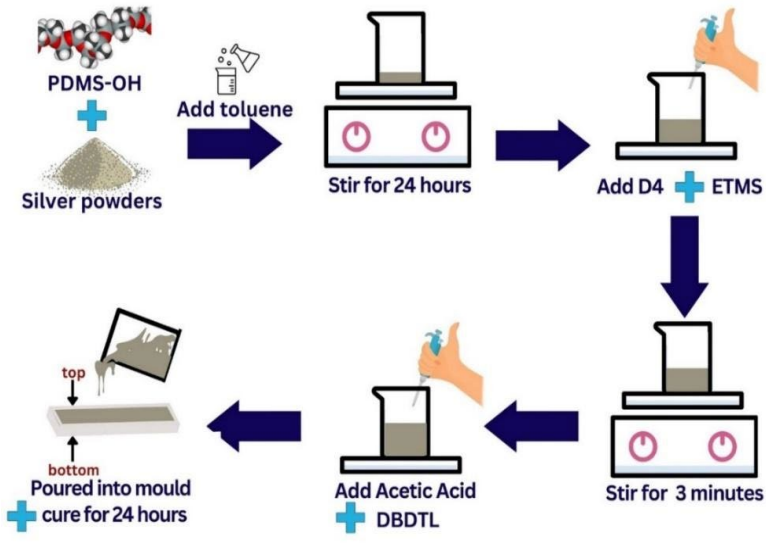

Figure 1: Conductive polymer sample preparation procedure

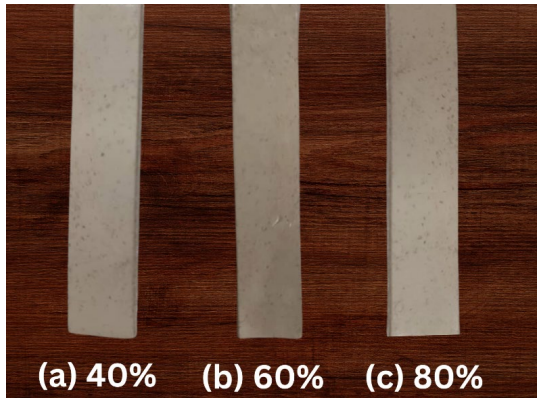

Figure 2: Conductive polymer samples of 40%, 60%, and 80% silver contents

Thermal Measurement

To evaluate the thermal transition and degradation behavior of the conductive polymer, Differential Scanning Calorimetry (DSC) and Thermogravimetric Analysis (TGA) have been used.

DSC-Q20 instrument was used to investigate the performance of the conductive polymer during thermal transitions. Approximately 10 mg of the sample was placed on a heating span, with the temperature range set from 25°C to 300°C at a heating rate of 10°C/min. The results were obtained as a graph plotting heat flow against temperature.

TGA was employed to evaluate the material's behavior by measuring its weight change relative to temperature and time. During TGA, a sample is heated in a controlled environment (usually an inert gas atmosphere), and the weight of the sample is evaluated as a function of temperature or time. We used a TGA, QS-500 (TA Instruments Inc., New Castle, DE, USA), to conduct the thermal degradation analysis of the silver-based conductive polymer. A small sample, weighing between 1 to 100 mg, was placed on a sample pan, and the balance device measured the weight before and after heating. The temperature range was set from 30°C to 900°C, with a heating rate of 10°C/min. Nitrogen gas was used at a flow rate of 70 ml/min due to its inert properties. The resulting weight loss profile of the conductive polymer was then analyzed. TGA provides valuable information about thermal stability and decomposition temperatures.

RESULTS AND DISCUSSION

Thermal analysis techniques, such as Differential Scanning Calorimetry (DSC) and Thermogravimetric Analysis (TGA), are utilized to examine the material properties of silver conductive polymers as they change with temperature. DSC provides insights into phase transitions and heat flow characteristics, while TGA offers information about thermal stability and decomposition behavior. By understanding both the thermal and mechanical responses to temperature changes, we can gain a comprehensive understanding of the material's performance.

Differential Scanning Calorimetry (DSC) Analysis

The Differential Scanning Calorimetry (DSC) analysis of conductive polymers with varying silver content (40%, 60%, and 80% by weight) reveals distinct thermal behaviors and transitions. A 10 mg weight of conductive polymer sample was placed on the aluminum pan of DSC analyzer in the nitrogen gas environment with the expulsion rate of 70 ml/min. The working temperature range was set as 25°C to 300°C with the heating rate of 10°C/min and heat flow of 20 mW.

Figure 3 depicts the heat flow (mW) of the silver conductive polymer in relation to temperature (°C). Initially, the samples are heated from a baseline temperature, with an endothermic process beginning at 28.06°C and a heat flow rate of 20 mW for all samples. As the temperature rises to 34.06°C, the heat flow reaches 21.79 mW for the 40% silver sample, 21.39 mW for the 60% silver sample, and 20.69 mW for the 80% silver sample. During this initial phase, the heat flow remains relatively stable, indicating no significant thermal events.

Upon further heating, the materials undergo a glass transition, marked by a sharp rise in the heat flow curve. For the 40% and 60% silver conductive polymers, the DSC curves exhibit glass transition temperatures (T_g) at 188.29°C and 178.13°C, respectively. These glass transitions indicate the temperatures at which the polymer matrices transition from a rigid, glassy state to a more flexible, rubbery state. The change in specific heat capacity (ΔCp) for these transitions is 0.108 J/g°C for the 40% silver sample and 0.130 J/g°C for the 60% silver sample. The higher ΔCp value for the

60% silver sample shows a more pronounced change in heat capacity and a significant transition from a glassy to a rubbery state. According to Kouediatouka *et al.* [23] the composite is unlikely to have a sharp melting temperature because PDMS-OH itself does not have a well-defined melting point due to its amorphous nature [23].

In contrast, the 80% silver content sample does not show a distinct Tg but instead exhibits a crystallization peak at 97.49°C with an area of -29.300 mJ and ΔH of -2.3629 J/g, indicating the formation of crystalline regions and increased brittleness. This peak represents an exothermic crystallization event, where the polymer matrix releases energy as it transitions from an amorphous to a crystalline state. This exothermic behavior could be influenced by the higher silver content that agglomerates due to high van der Waals attractions, as described by Ibrahim *et al.* [11] and Bachok *et al.* [18]. Furthermore, crystallization increases the polymer's density and enhances mechanical properties such as stiffness and tensile strength, but it can also make the material more brittle.

These findings demonstrate that increasing the silver content in conductive polymers significantly affects their thermal behavior. This highlights the complex interplay between the polymer matrix and the silver particles, impacting the thermal stability and transitions of the conductive polymers. The heat flow trends reveal that the 60% and 40% silver samples have consistent thermal transitions, with the 60% sample showing a higher heat flow, indicating a better thermal response. In contrast, the heat flow for 80% of the silver increases at a different rate and shows a distinct peak, indicating a different thermal behavior due to the crystallization event. The 80% silver sample's distinct crystallization event increased rigidity and brittleness, which might not be desirable for applications requiring flexibility.

Overall, the 60% silver content sample provides the best balance between thermal stability and flexibility, making it the most advantageous for high-performance applications, while the 40% sample remains more rigid, and the 80% sample demonstrates increased brittleness due to crystallization. It is evident that although adding silver can increase thermal stability, having too much of it could have negative consequences.

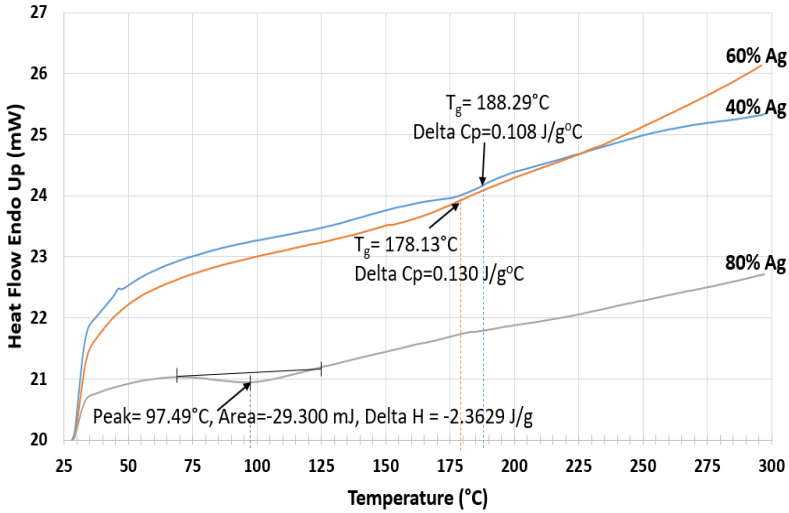

Figure 3: DSC analysis of conductive polymer with 40%, 60% and 80% of silver

Thermogravimetric Analysis (TGA)

The thermal degradation, decomposition, and stability of silver conductive polymers were examined using Thermogravimetric Analysis (TGA) over a temperature range of 30°C to 900°C, with a heating rate of 10°C/min. The analysis was conducted in a nitrogen atmosphere with a purge rate of 70 ml/min.

Figures 4-6 illustrate the TGA-DTG curves for the 40%, 60%, and 80% silver conductive polymer samples, respectively, highlighting their thermal stability and decomposition characteristics. The TGA curve, shown in black, depicts the weight loss of the samples as the temperature increases, while the derivative (DTG) curve, shown in red, illustrates the rate of weight loss as a function of temperature or time and are derived from TGA (Thermogravimetric Analysis) data. The peaks in the DTG curve correspond to significant changes in the TGA curve, indicating rapid decomposition. These curves reveal that the material undergoes multiple thermal events that show multistage decomposition. Additionally, the incorporation of silver nanoparticles significantly enhances the thermal conductivity of the composite, potentially affecting its heat distribution and thermal stability.

40% Silver Conductive Polymer

Figure 4 shows the 40% silver conductive polymer sample remains stable up to approximately 308.68°C. The first stage of decomposition begins at an onset temperature of 308.68°C and continues up to 357.62°C, with a weight loss of 9.477%. This initial weight loss is attributed to the loss of moisture content, or any solvents at high temperatures. The second stage of decomposition occurs between 357.62°C and 521.08°C, resulting in an additional weight loss of 5.757% as the composite material further breaks down. The delta Y value of 34.43% signifies a substantial change in the sample's weight, confirming significant decomposition.

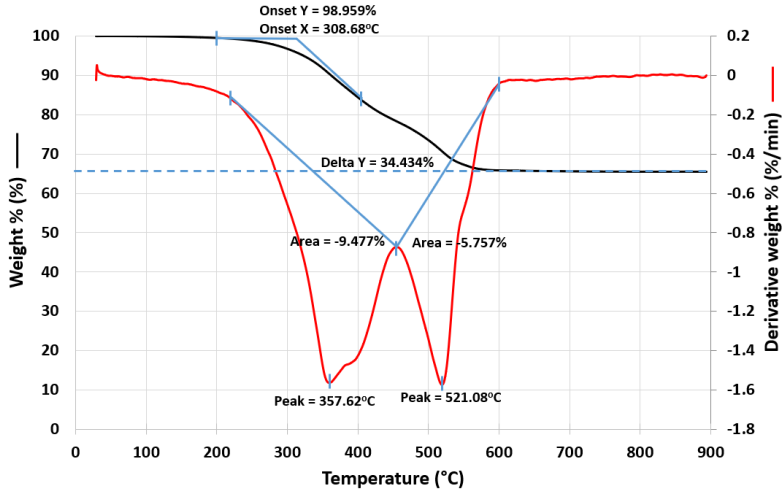

Figure 4: TGA-DTG curve of the 40% silver conductive polymer sample

60% Silver Conductive Polymer

The thermal behavior and decomposition properties of the 60% silver conductive polymer sample are illustrated in Figure 5.

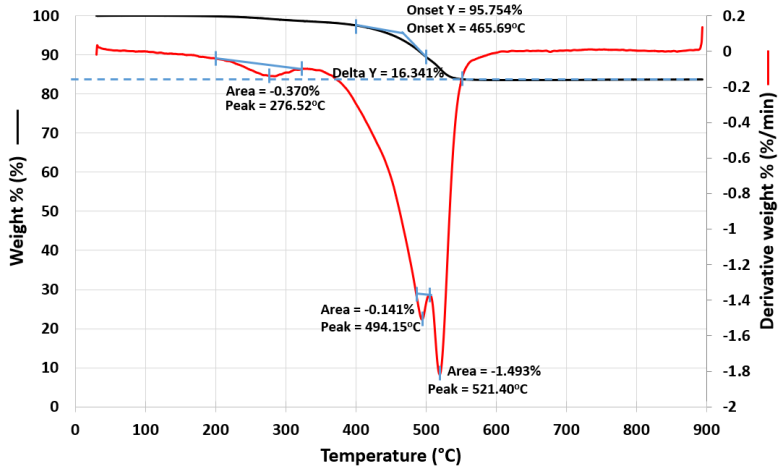

Figure 5: TGA-DTG curve of the 60% silver conductive polymer sample

This sample remains stable up to approximately 276.52°C. The first decomposition stage begins at 276.52°C and continues until 465.69°C, resulting in a minor weight loss of 0.370%. This initial decomposition event is evident only in the DTG curve, indicating a minor decomposition that the TGA curve does not capture due to its insignificant contribution to the total weight loss. The onset temperature in the TGA curve is 465.69°C, marking the beginning of more substantial decomposition. The second decomposition stage occurs between 465.69°C and 494.15°C, with an additional weight loss of 0.141%, indicating further degradation of the polymer matrix. The third and most significant stage of decomposition occurs between 494.15°C and 521.40°C, with a weight loss of 1.493%. This stage shows continued breakdown of the composite material, as reflected by the major peaks in the DTG curve. Overall, the delta Y value of 16.34% confirms substantial decomposition, indicating a significant change in the sample's weight throughout thermal analysis.

80% Silver Conductive Polymer

Figure 6 displays the thermal behavior and decomposition characteristics of the 80% silver conductive polymer sample. The curve starts with minimal weight loss up to approximately 246.92°C, indicating that the conductive polymer is stable at lower temperatures. The first

significant weight loss occurs around 289.43°C, where a peak is observed. The area under this peak is 0.820%, suggesting the decomposition of a component within the conductive polymer. The second weight loss observed at 497.48°C, with a peak indicating substantial weight loss. The area under this peak is 2.479%, highlighting a notable decomposition process. This substantial decomposition is confirmed by the delta Y value of 19.34%, showing a significant change in weight.

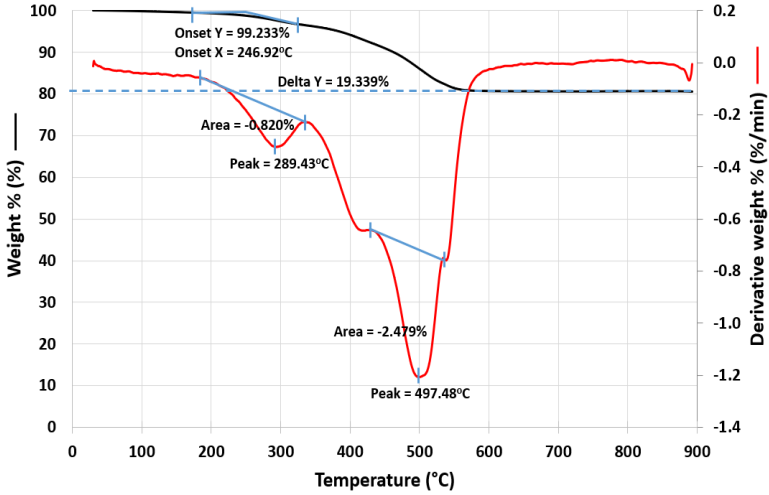

Figure 6: TGA-DTG curve of the 80% silver conductive polymer sample

Silver Conductive Polymers Effectiveness

Table 2 presents the results of a thermogravimetric analysis (TGA) conducted on conductive polymers with varying silver (Ag) content: 40%, 60%, and 80%. The table details the onset temperatures of weight loss, total weight loss percentages, and the decomposition temperature ranges for each sample.

Table 2: Thermogravimetric analysis of conductive polymers

Sample	T_{onset} (°C)	Total weight loss (%)	Decomposition range temperature (°C)
Sample 1 - 40% Ag	308.68	34.43	308.68-599.29
Sample 2 - 60% Ag	465.69	16.34	276.52-559.93
Sample 3 - 80% Ag	246.92	19.34	246.92-579.98

This analysis helps to understand the thermal stability and decomposition behavior of the polymers based on their silver content. Figure 7 complements this data by providing the TGA curves for the same samples, illustrating the relationship between temperature and weight loss for the conductive polymers.

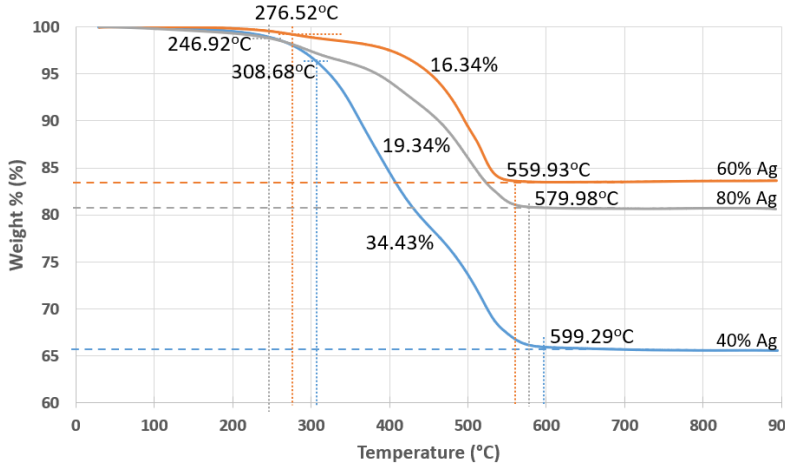

Figure 7: TGA curve for 40%, 60% and 80% of silver conductive polymer samples

The curves visually demonstrate the decomposition process, highlighting the differences in thermal stability and weight loss behavior among the 40%, 60%, and 80% silver content samples.

The figure clearly shows that the sample with 60% silver content (sample 2) has the highest onset temperature (465.69°C) and the lowest total weight loss (16.34%), indicating its superior thermal stability compared to the other samples. The onset temperatures for the 40% silver conductive polymer (sample 1) and the 80% silver conductive polymer (sample 3) are 308.68°C and 246.92°C, respectively. These results clearly demonstrate that the thermal stability and decomposition properties vary significantly with silver content. Higher silver content generally leads to increased onset temperatures, indicating enhanced thermal stability. This observation aligns with Nazarenko's findings, which suggest that the addition of fillers can improve thermal stability [3]. However, when the silver content increased

to 80%, the onset temperature dropped to 246.92°C. This anomaly could be due to the agglomeration of silver particles at higher contents [11, 18], which can create stress points and reduce the overall stability of the composite. This suggests that while the addition of silver can improve thermal stability, excessive silver content may lead to adverse effects.

Sample 2 (60% silver) shows the least extensive decomposition, with smaller weight loss areas of 0.370%, 0.141%, and 1.493%. In contrast, sample 1 (40% silver) exhibits the most extensive decomposition, with areas of 9.477% and 5.757% for its primary events. Sample 3 (80% silver) experiences moderate decomposition, with areas of 0.820% and 2.479%. The delta Y values further emphasize these differences, with sample 1 (40% silver) having the highest delta Y value of 34.434%, indicating the most significant weight loss, while sample 2 (60% silver) has the lowest delta Y value of 16.341%, indicating the least significant weight loss. Sample 3 (80% silver) falls in between with a delta Y value of 19.339%.

The decomposition range also provides insight into the temperature span over which significant weight loss occurs. Sample 3 (80% silver) has the broadest decomposition range, from 246.92°C to 579.98°C. Sample 2 (60% silver) has a narrower range from 276.52°C to 559.93°C, and sample 1 (40% silver) decomposes between 308.68°C and 599.29°C. The decomposition temperature range increases with higher silver content due to enhanced thermal conductivity. Increased thermal conductivity allows heat to be distributed more evenly and quickly throughout the material, leading to a broader temperature range for decomposition.

Overall, sample 2 with 60% silver content is the optimal conductive polymer sample. It has the highest onset temperature of 465.69°C, indicating superior thermal stability, and the lowest total weight loss of 16.34%, suggesting minimal degradation. These properties make it the most suitable for applications requiring high thermal resistance and stability.

CONCLUSION

In conclusion, this study examined the effect of varying silver content on the thermal behavior of PDMS-OH/silver conductive polymers using DSC and TGA analyses. Both DSC and TGA results demonstrated that adding more silver enhances the thermal stability of the material, but excessive silver can have negative consequences. The DSC analysis reveals that lower silver contents (40% and 60%) primarily influence the glass transition temperatures of the polymers. This heat flow analysis reveals that while both the 60% and 40% silver samples exhibit consistent thermal transitions, the 60% sample shows a higher heat flow, indicating a better thermal response. In contrast, a higher silver content (80%) leads to a distinct crystallization event, increasing the polymer's density and brittleness. This highlights the complex interplay between the polymer matrix and silver particles, impacting the thermal stability and transitions of the conductive polymers. The DSC results indicate that a 60% silver content provides a desirable balance between flexibility and processability, with a glass transition temperature around 178.13°C and a delta Cp of 0.130 J/g°C. Additionally, TGA results reveal that the 60% silver conductive polymer balances thermal stability and minimal degradation, making it the most advantageous choice for high-performance applications. Therefore, it can be concluded that the PDMS-OH conductive polymer with 60% silver content offers an optimal balance between flexibility, processability, thermal stability, and minimal weight loss, outperforming both the lower and higher silver content formulations.

ACKNOWLEDGEMENTS

The authors would like to acknowledge the support of the Ministry of Higher Education Malaysia for the Fundamental Research Grant Scheme (project code: FRGS/1/2022/TK10/USM/02/17) and Universiti Sains Malaysia (USM) for funding this project under Short Term Grant 304. PMEKANIK.6315494, both provided to the School of Mechanical Engineering, Universiti Sains Malaysia (USM).

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