Chemical and thermal stability of different AgNP concentrations incorporated in 0.1% DMPT UPE resin

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Abstract

Purpose – In response to the growing demand for a polymer with improved chemical and thermal stability in the construction sector, this study aims to thoroughly explore the characteristics of silver nanoparticles (AgNP) and their various concentrations. The primary goal is to determine the effect of these nanoparticles on the chemical and thermal stability of unsaturated polyester (UPE) resin doped with dimethyl-para-toluidine (DMPT) when exposed to high temperatures.

Design/methodology/approach – Silver nanoparticles were first synthesized from the chemical reaction between silver nitrate and trisodium citrate before its addition to the resin. The nanocomposites were thoroughly examined using advanced analytical methods such as Fourier transform (FTIR), Raman spectroscopy and scanning electron microscope to determine chemical stability. Thermal stability tests were carried out using thermogravimetric analysis, differential thermal analysis and derivative thermogravimetry methods; viscosity and peak exotherm were also examined.

Findings – The data shows that increasing nanoparticle concentration improves resin chemical stability, reduces peak exotherm duration and increases viscosity. Clearly, only 1.5% AgNP concentration outperformed neat UPE resin, while 0.5% and 1% AgNP concentrations fall short in terms of thermal stability.

Originality/value – The enhanced resin highlights the subtle influence of nanoparticle addition, which has a greater impact on the chemical structure of the composite rather than its thermal properties.

Keywords Crosslinking, Concentration, Degradation, Silver nanoparticles

Paper type Research paper

Nomenclature

Abbreviation	= Meaning
Ag	= Silver
AgO	= Silver oxide
$AgNO_3$	= Silver nitrate
AgNP	= Silver nanoparticles
C	= Carbon
$C_6H_5O_7H_3$	= Citrate
DMPT	= Dimethyl-para-toluidine
DTA	= Differential thermal analysis
DTG	= Derivative thermogravimetry
EDX	= Energy dispersive X-ray analysis
FTIR	= Fourier transform
H	= Hydrogen
H_2O	= Water
O	= Oxygen
Na	= Sodium
Pristine	= Neat resin (no additions)
SEM	= Scanning electron microscope
UPE	= Unsaturated polyester
TGA	= Thermogravimetric analysis

The utilization of resin-based materials in structural applications, particularly in construction and civil engineering, has steadily increased due to their advantageous properties such as lightweight, high strength, hydrophobicity, heat resistance and corrosion resistance, to name a few (Chohan *et al.*, 2022; Oladele *et al.*, 2020; Shen *et al.*, 2020). There are various resins, including acrylic, polypropylene, polystyrene, polyethylene, silicone, epoxy, polycarbonate, phenolic and polyester, among others (Anandhan *et al.*, 2021). However, polyester resins are further

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divided into two main types, namely, saturated and unsaturated (Hofmann *et al.*, 2022).

Unsaturated polyester (UPE) resin has emerged as a significant player, finding wide-ranging applications in sectors such as marine, aerospace, thermal insulation, petrochemicals, construction and mining, to mention a few (Mehdipour-Ataei and Mohammadi, 2023). For instance, it is utilized in manufacturing anchor bolt capsules for mining roof support in the mining sector due to its flexibility and ability to create durable and robust composite structures (Chen *et al.*, 2022). Consequently, there has been an increase in mining roof support failures attributed to various natural occurrences (Zingano and Andrade, 2021). Hence, there is a need for reinforcement or alternative materials capable of mitigating or eliminating such occurrences. In this context, we opted to enhance the current material to reduce costs. One way to enhance the material is through the use of nanomaterials. Nanomaterials have found applications in diverse fields of science and technology due to their excellent and unique properties, including physical, chemical, electrical and magnetic properties (Asha and Narain, 2020). The introduction of nanomaterials, such as carbon nanotubes, titanium oxide, clay, graphene and silver nanoparticles (AgNPs), among others, has drawn attention because of its potential to enhance the performance characteristics of UPE resin (Meer *et al.*, 2016; Zhan *et al.*, 2020).

Carbon nanotube was added to epoxy to achieve improved thermal stability (Lorero *et al.*, 2024). Similarly, silica nanoparticles were incorporated into epoxy and vinyl ester resin to enhance mechanical behaviour at elevated temperatures. Halim *et al.* (2020) studied the effect of silica aerogel–Aluminium trihydroxide nanoparticles hybrid on the thermal stability of unsaturated polyester resin and discovered that the thermal stability was improved by 30°C. In the work of Abdulridha *et al.* (2022), titanium dioxide (TiO_2) nanoparticles were dispersed in acrylic resin to achieve enhanced mechanical strength.

Focusing on its application as anchor bolt capsules for roof construction in the mining sector, this study investigates the intricate relationship between the concentration of AgNP and the chemical and thermal stability of UPE resin treated with 0.1% dimethyl-para-toluidine (DMPT). Since anchor bolt capsules secure structures to underground roofs and endure various environmental factors, it's crucial that the encapsulating materials remain reliable and durable (Alhaidary and Al-Tamimi, 2021).

AgNP has demonstrated robust chemical stability and enhanced thermal characteristics (Pryshchepa *et al.*, 2020). Synthesized AgNPs, depending on the method, exhibit a degree of thermal stability and find applications in diverse areas such as biomedical, food packaging, the textile industry, energy storage, sensor technology and construction, among others (Gupta *et al.*, 2023; Panhwar *et al.*, 2022). Chemical stability in the UPE resin matrix is expected to be enhanced by the inclusion of AgNPs (Shenashen *et al.*, 2014), which are known for their resistance to chemical degradation, enhanced optical absorption, high catalytic activity, high melting point, good thermal and electrical conductivity, robust mechanical properties and strong sterical properties, among others (Shenashen *et al.*, 2014; Singh *et al.*, 2021; Zaman *et al.*, 2023).

Since anchor bolt capsules are exposed to harsh environmental factors such as moisture, chemicals and temperature changes, chemical stability is essential (Abdulridha *et al.*, 2022). The resin curing rate relies on the type and amount of promoter employed, such as dimethyl-para-toluidine (DMPT). DMPT has proven to be an effective curing promoter that has been applied at ranges (Mounika *et al.*, 2023). DMPT, at a concentration of 0.1%, was chosen as the UPE resin promoter due to its shown ability to increase mechanical properties, thermal stability and resistance to water absorption and create a slow-setting (Aoki and Nishio, 2010; Subramani *et al.*, 2003). The synergistic combination of DMPT and AgNPs is projected to result in a composite material with improved chemical and thermal stability, making it an ideal candidate for anchor bolt capsule applications. This study aims to elucidate the crucial relationship between AgNP and its concentration on chemical and thermal stabilities to enhance a material's behaviour under high-temperature circumstances relevant to anchor bolt capsules.

In summary, since not much attention has been given to the chemical stability of UPE resin laced with nanoparticles, the current study aims to enhance the chemical stability for resin grout applications by establishing value-adding activities of Ag nanomaterial on UPE resin for mine bolt application. Additionally, the study aims to investigate the possibility of creating sophisticated polymer composites, which would improve structural dependability and safety in construction and civil engineering procedures.

2. Experimental procedures

2.1 Material

Unsaturated polyester resin, aminedimethyl-para-toluidine, (DMPT), Calcium Carbonate fillers, $AgNO_3$ and trisodium citrate were purchased from Merck, South Africa.

2.2 Synthesis

To synthesize the silver nanoparticles, 50 ml of 0.001 M $AgNO_3$ was heated to boiling at 100°C. Subsequently, 5 ml of 1% trisodium citrate was added dropwise to the solution, which was vigorously stirred with a magnetic stirrer on a hot plate until a pale-yellow colour change was observed at 60°C. The solution was then removed from the heating device and stirred until it cooled to room temperature.

The mechanism of reaction could be expressed in equation (1):

$$4AgNO_3 + C6H_5O_7Na_3 + 2H_2O \rightarrow 4AgO + C_6H_5O_7H_3 + 3Na + + H + + O_2 \uparrow \quad (1)$$

2.3 Nanocomposite

For composite preparation, prior to nanoparticle introduction, the resin was mixed with 0.1% DMPT to enhance resin reactivity for subsequent experiments. The blend was achieved by mixing the resin with DMPT at medium speed with a mechanical mixer. Calcium carbonate fillers were incorporated at 82% in the nanocomposites. The resin was weighed into a stainless-steel bowl, followed by the addition of fillers and blending for 900 s at medium speed with a mechanical mixer. To initiate resin curing, a catalyst paste containing 2.5% active oxygen from benzoyl peroxide was utilized. Resin setting time

was determined by mixing a known quantity of 75% resin and 25% catalyst paste in a plastic cup, conditioning it in water for 900 s at 20°C. The graphical representation of the preparation is detailed in Figure 1.

2.4 Characterization

Various analytical techniques were employed to study resin nanocomposites. Chemical bonding and molecular structure were investigated using a Perkin-Elmer Spectrum 100 FT-IR spectroscope with ATR fitted with a diamond crystal. Raman measurements were conducted using Thermo-Fisher's FT-Raman Module integrated with a Nicolet-6700 FT-IR spectrometer, employing a laser with a wavelength of 1064 nm. X-ray diffraction (XRD) analysis utilized a PANalytical X'Pert Pro-powder diffractometer with CuKα radiation. Morphology and structure were examined with a Tescan SEM equipped with EDX. The time-to-peak exotherm test involved a calibrated water bath, digital timer and APO K-Type thermocouple. Thermal degradation behaviour was studied using a Hitachi STA7200RV. This comprehensive experimental approach provided insights into the resin nanocomposites' composition, structure and thermal properties.

3. Results and discussion

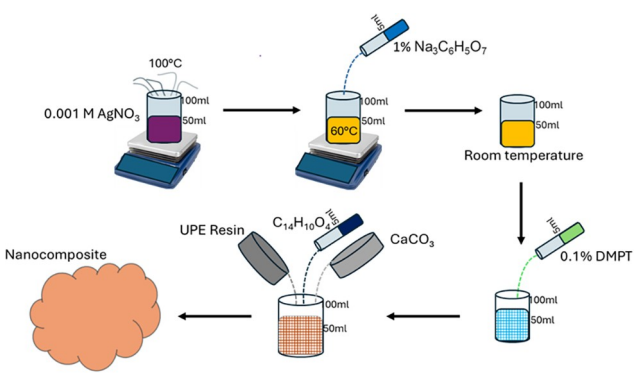
3.1 Chemical structure

Figure 2a illustrates the chemical structures of neat UPE and various concentrations of UPE/Ag nanocomposites. Some disparities are noticeable among the spectra, particularly in the fingerprint regions.

In general, the spectra of all the samples exhibit the -CH_3 and -CH_2 methylene groups stretching vibration of peak C–H between usually observed in the region 2850–3000 cm ^{1} (Rhouma *et al.*, 2023). In this case, no shift was observed in all the samples, and it occurred at 2963 cm ^{1}. The stretching peak of the C = O ester can be seen at 1717 cm ^{1} followed by the disubstituted (cis) C = C stretching peak at 1645 cm ^{1} (Volkov *et al.*, 2021). It is worth noting that the intensity of the peak reduces as the concentration of the nanoparticle increases. The C-O-C vibration peak was registered at 1260 cm ^{1} in all the samples. Also, at 1152 cm ^{1}, the C-O stretching peak (ester group) is evident and succeeded by the C-H stretching vibration peak of CHR=CHR at 972 cm ^{1}and the C = C peak in the vinyl group for styrene monomer at 865 cm ^{1} (Huynh *et al.*, 2022). A sharp and moderate band observed at 755 cm ^{1} was assigned to the skeletal deformation γ(CH), vibration. An emerging peak at 701 cm ^{1} indicates the presence of metal oxide vibration (Ag-O). This observation indicates that the Ag nanoparticle is packed inside the UPE resin matrix and is not chemically bound with any functional groups of the resin components. The major difference in the spectra is found at peak 701 cm ^{1} for 1.0 and 1.5% composites, whereas the peak is absent in the 0.5% and the control UPE. The absence of a peak for 0.5% could be attributed to the nanoparticle's low quantity and sparing distribution. Also, the peak within 755 and 765 cm ^{1} range of both neat UPE and 0.5% AgNP/UPE is 1,2-disubstituted C-H bending, while that of 1.0 and 1.5% are monosubstituted C-H bending (Ravutsov *et al.*, 2021). The dispersion of nanoparticles within the resin matrix was facilitated primarily by cohesive, adhesive and potentially van der Waals forces (Qiu *et al.*, 2022). As a result, the molecular structures of the nanocomposite closely resembled those of the neat resin. This similarity is likely attributed to the low quantities of nanomaterials integrated into the resin matrix. At 1.5% nanoparticle contents, the metal vibration is more visible (Anancharoenwong *et al.*, 2021).

The structure characteristics of AgNP/UPE nanocomposite and neat UPE resin were also investigated using Raman spectroscopy. As shown in Figure 2b, the spectra of all AgNP/UPE nanocomposites exhibit the D and G bands, including neat UPE and Ag. D and G bands are characteristic peaks of C atom crystals, which were at 1458 cm ^{1} and 1522 cm ^{1}, respectively. In the UPE spectra, the D band relates to disordered carbon structure, and the G band is associated with the C—C vibration mode in organized carbon structure (Dai *et al.*, 2020). The bands at 480 cm ^{1} and 639 cm-c can be assigned to C C C ring in-plane vibration mode, C H out-of-plane bending, and C H in-plane bending mode, respectively (Sui *et al.*, 2021). After the addition of Ag nanoparticles, the band at 143 cm ^{1} shifts to 99.9 cm ^{1} with 0.5% Ag, 94.4 cm ^{1}cm ^{1} with 1.0% Ag, and 89.56 cm ^{1} with 1.5% Ag at the radial breathing mode. This means that the band at 480 cm ^{1} disappears with the addition of Ag nanoparticles, indicating an attachment of Ag nanoparticles to UPE resin molecules. The bands at 1871 cm ^{1} at 0.5% Ag, 1605 cm ^{1} and 1733 cm ^{1} at 1.0% Ag indicate the nanosilver vibrations. At 1.0% Ag nanoparticles, the Raman spectrum exhibits a band attributed to C C stretching at 3069 cm ^{1} (Ouyang *et al.*, 2016). In all the samples, a 2G band mode is also observed. In the pristine material, a broad peak is observed at 2737 cm ^{1}, while a shift and narrower peaks are observed in AgNP/UPE at 2728, 2726 and 2693 cm ^{1}. The higher the concentration, the lower the Raman wavelength of the material. This project shows that the composites tend towards the G-band and, therefore, have the tendency to improve the materials' chemical stability.

Figure 1 Experimental set-up image for the nanocomposite preparation



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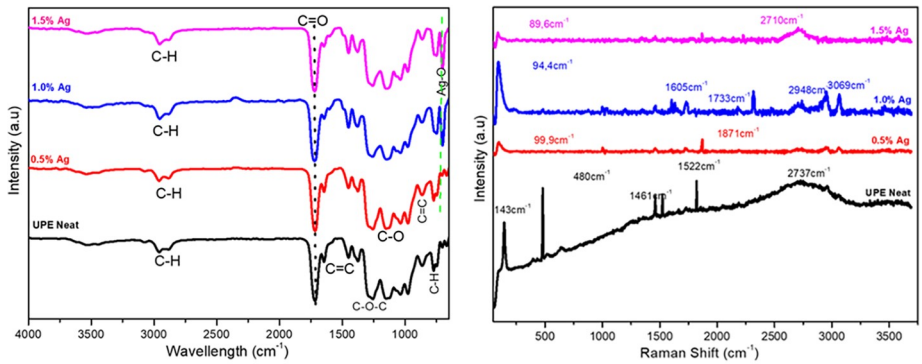
3.2 Morphology

The morphology of the pristine and AgNP/UPE composites was obtained using the scanning electron microscope, as shown in Figure 3.

Figure 2 FTIR and RAMAN spectra of neat UPE resin and AgNP/UPE composites



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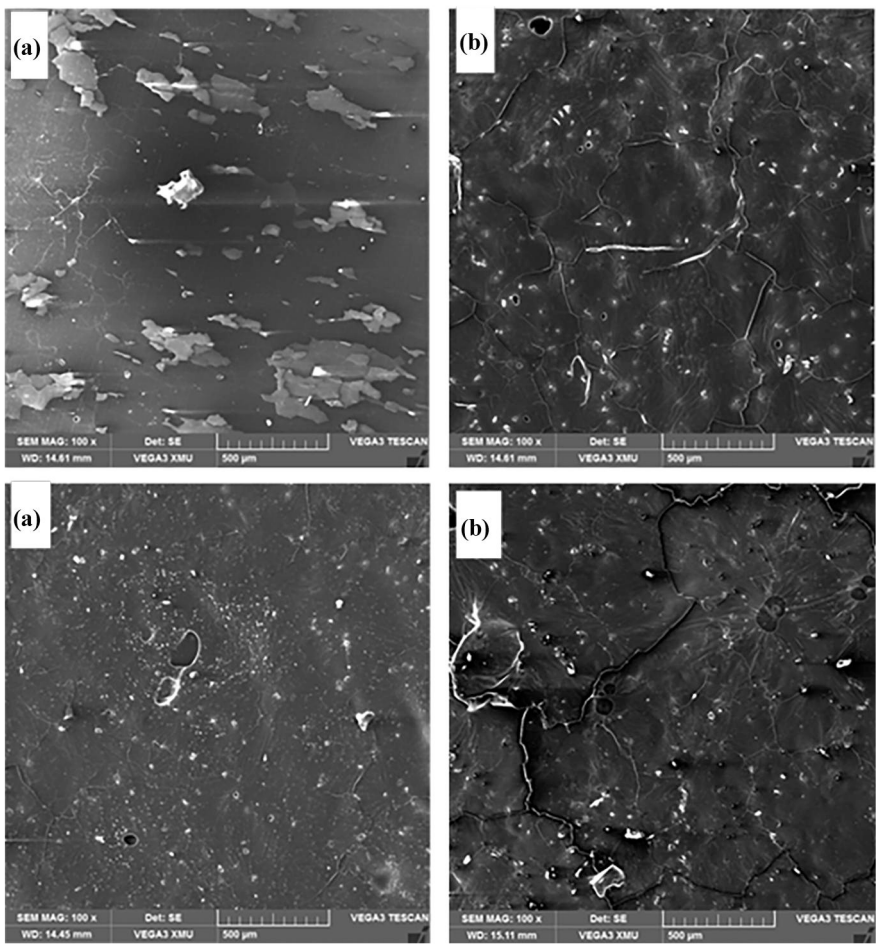
In Figure 3a, the neat UPE resin appears smooth, flat and compact, indicating a fully and perfectly cured resin. However, the introduction of AgNP starts to change the morphology as the concentration increases due to functionalization. The distribution of the particles heightens the roughness of the surface. The higher the concentration, the greater the surface roughness, and shape irregularity of the composites (Sallal *et al.*, 2020). The impact is also evident in the spatial

Figure 3 The surface morphology of (a) UPE resin, (b) 0.5% AgNP, (c) 1.0% AgNP and (d) 1.5% AgNP



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distribution of nanoparticles within the matrix. Nevertheless, the roughness will contribute to the material's mechanical properties, such as mechanical interlock as a result of increased surface area (ASTM C581 20, 2013). It is evident from Figures 3b–d that the higher the percentage of the nanoparticles in the mix, the greater the agglomeration of particles and uneven distribution. The random shape of the synthesized nanoparticles could also have contributed to the clustering observed within the matrix (Sallal *et al.*, 2020). There are no microgaps visible in the structure of all the samples. This indicates a perfect bonding between the UPE resin and AgNP and a high probability of reduced failure (Mourad *et al.*, 2020). This supports the information from the infra-red spectroscopy that a cohesive and adhesive bonding occurs in the matrix. The cohesive bonding is established because of the covalently attached Ag of Ag-O, which enhances the interaction between the Ag-O nanoparticles and UPE resin due to the multifunctional groups within the mix. The mixability and traces of silver nanoparticles were also investigated in the matrix using the EDX analysis presented in Figure S1. It establishes that the electron beam penetrates the embedded nanoparticles and detects a perfect crosslinking between the nanoparticles and the resin either at the surface or within the matrix (Aksoy, 2023). Furthermore, the nanocomposites reflect Ag element, indicating nanoparticles in the matrix. The data obtained from the EDX elemental mapping (not shown) indicate that the higher the concentration of the nanoparticles, the higher the Ag and the lower the oxygen composition in the matrix. It demonstrates a strong intra-particulate of carbon, calcium, silicon and silver in the Ag-O-loaded resins. The EDX analysis also reveals the absence of impurities in the nanocomposites, which indicates the purity of the resultant materials (Sallal *et al.*, 2020).

3.3 Peak exotherm

In supplementary file, Figure S2 and Table S1 show results obtained from resin nanocomposite exotherm and test results. An addition of 0.5%, 1.0% and 1.5% Ag nanoparticles into the resin matrix increases exotherm temperature at reduced peak time, as deduced from Table S1. Ag nanoparticles react or bond with the resin during crosslinking with styrene, producing microgels that emit good heat transfer (Spasojevic *et al.*, 2021). The heat transfer increases exotherm temperature and reduces the peak time by reducing the gel time. Figure S2 highlights the extracted peak exotherm from Table S1. A reduction in Peak Exotherm is an indication of a quicker resin nanocomposite cure, significantly affecting nanocomposite thermal stability and mechanical strength (Athawale and Pandit, 2019). There is a 50-second reduction in peak time and a 46°C higher exotherm in a sample containing 1.5% AgNP compared to neat UPE. Similarly, an addition of the nanoparticles results in a reduction in the time it takes to peak at the highest temperature by 50 s. These results indicate that Ag improves the catalysis process for curing the resin nanocomposites. This was also confirmed by (Athawale and Pandit, 2019) in their review on UPE resin, vegetable oil, clay and Ag nanoparticles. The more effective the catalysis, the quicker the cure will be, with a higher exothermic reaction.

Figure S3 shows thermograms of an unsaturated polyester resin cured isothermally at 20°C utilizing the same amounts of peroxide initiator and amine accelerator at different nanomaterials quantities. The reaction induction period shortens as the quantity of nanomaterial indicates an increase in peroxide decomposition or an increase in catalytic activities from AgNP. This catalytic effect results in the rapid production of alkoxy and peroxyl free radicals that initiate polymerization reactions (Liu *et al.*, 2021) and thermo-chemical reactions (Ouyang *et al.*, 2016). Similar results were obtained by (Farsane *et al.*, 2023) in their evaluation of UPE resin curing at different quantities of initiator and accelerator. The free radicals initiated the exothermic copolymerization, resulting in increased reaction heat in exotherm temperature. The increase in temperature increases the resin cure due to an increase in the reaction speed and the molecular weight of the polymer (Aziz *et al.*, 2023; Brondi *et al.*, 2023). Therefore, the addition of AgNP will improve both the thermal and chemical stability of the polymer. It is also evident that the higher the concentration of the nanoparticles, the greater the stability of the material in application.

3.4 Viscosity

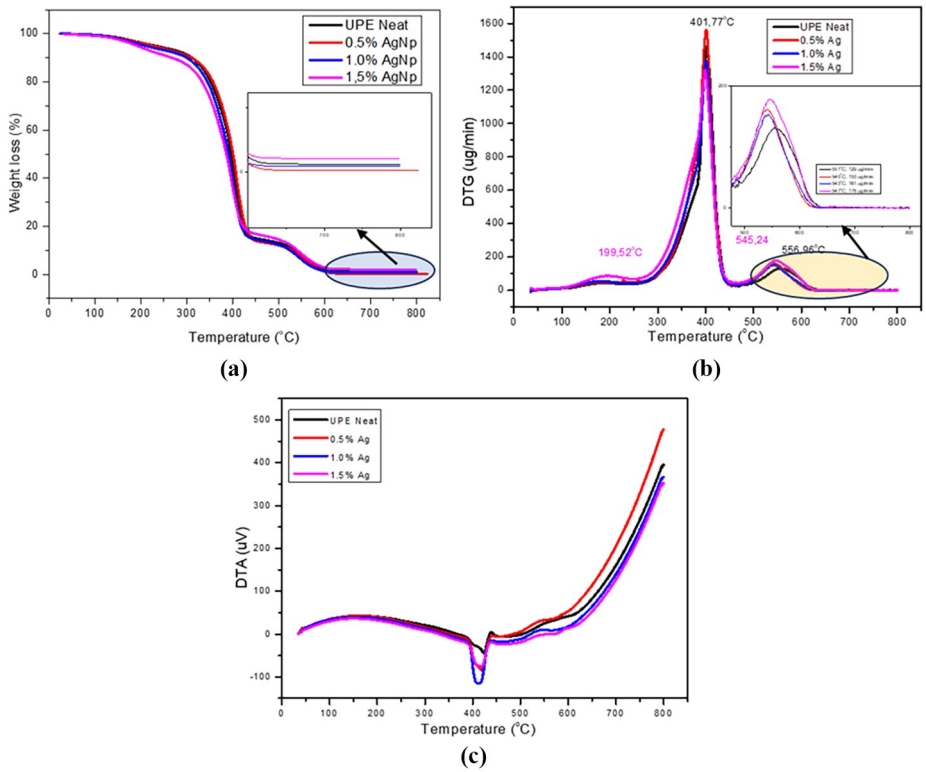
The viscosity is measured as a force required to push the resin composites through an orifice at a constant rate. The behaviour of the materials was studied for 90 days, as depicted in Figure S4. Surprisingly, the incorporation of nanomaterials shows an increase in viscosity over time. Increased viscosity indicates an increase in the filler wettability of the resin and a reduction in the mobility of resin molecules. This assertion differs from what other researchers observed, in which a reduction in viscosity of the matrix with the addition and increase in nanoparticles was observed (Apmann *et al.*, 2021; Tang *et al.*, 2013). In other studies, the reduced mobility in resin molecules was attributed to the ability of nanoparticles to close the gaps between the filler particles. For example, it is claimed that TiO_2 nanoparticles played a significant role in reducing the resin segments' mobility at the nanoparticles-matrix interfaces through the presence of physically attractive interactions (Shafaamri *et al.*, 2020). This is required to ensure that the resin does not drip during application and that it is not runny when mixed inside the holes or an area of application, especially vertical holes in buildings. Figure S4 shows that after 90 days, the viscosity of all nanocomposite samples increased considerably. The notable increase seen in 60 and 90 days owes to nanoparticles' higher surface area, resulting in improved physical interactions.

3.5 Thermal stability

Figure 4 describes the details of the thermal stability investigation on the unblended UPE resin and AgNp/UPE composites. Thermogravimetric analysis (TGA) was performed on the UPE/Ag nanocomposites. The hypothesis is that weight loss can help define the effect of nanoparticles on UPE resin thermal stability, which can also be influenced by its chemical properties. In Figure 4a, the TGA result shows that the pristine have better weight loss than 0.5 and 1.0% nanoparticles over the considered temperature range. The inset in Figure 4a projects that only 1.5% Ag have lower degradation mass compared to neat UPE and other nanocomposites. This indicates that at 1.0% AgNP, the concentration is not sufficient to improve the thermal degradation of the DMPT-promoted UPE resin.

The results show the degradation temperature of UPE/AG nanocomposites between 291.23°C and 369.64°C. Nanocomposite with 1.5% Ag nanoparticles shows the first

Figure 4 Thermal degradation investigation using (a) TGA, (b) DTG and (c) DTA



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degradation temperature from 290.84°C to 368.01°C. Conflicting reports regarding the incorporation of nanoparticles exist in the literature. Some studies suggest that nanoparticles may accelerate the resin's degradation by enhancing thermal transport processes, while others indicate a reduction in the resin's typical degradation temperature (Ishii *et al.*, 2021; Li *et al.*, 2021). In this study, the observation suggests that modified nanoparticles provide better interfacial adhesion with the resin matrix, leading to delayed thermal degradation of the composites for the nanocomposite above 1.0%. The percentage residual increases with the addition of nanoparticles, remaining stable within the examined temperature range. The cause could be attributed to a lower surface interaction of the nanoparticles with the resin and limited crosslinking between the resin and the nanoparticles.

The temperature relating to degradation peaks can be seen on the derivative thermogravimetry (DTG) curve of the UPE/Ag as indicated in Figure 4b. Three levels of degradation were observed on UPE/Ag nanocomposites. The first degradation of UPE/Ag nanocomposite ranged from 150°C to 250°C, with 1.5% displaying the most improved thermal stability (199.52°C) compared to neat UPE resin and other nanocomposites. This stage is usually associated with the removal of any water molecules entrapped. The second degradation peak at 401.77°C is consistent with the degradation of UPE resin. This shows that Ag nanoparticles have little influence on the thermal stability of the UPE resin as result of limited crosslink between the resin and AgNP. A significant influence is observed in the third stage (inset), where the mass loss was due to the loss of carbon monoxide and methane formed by the degradation of the methylene bridge (Karami *et al.*, 2022). All the nanocomposites showed to have more stability at this stage. Although an increase in concentration improves thermal stability and reduces the rate of degradation, the char residue at the end of 800°C remains the same. These findings align with previous research, which identifies the third peak around 550°C as associated with the degradation of the polyester nanocomposite (Chu *et al.*, 2022).

Thermal stability is a crucial factor influencing material behaviour and holds significant importance in the production of nanocomposites. In the differential thermal analysis (DTA) curve depicted in Figure 4c, a singular endothermic peak is evident at 98 μV 410°C, suggesting a decomposition reaction within the UPE/Ag nanocomposites. This peak intensifies with increasing nanoparticle content, particularly noticeable with 0.5% Ag. Conversely, in neat UPE, this peak is attributed to the presence of low molecular weight polymeric/organic compounds. Thermal analysis demonstrates that the inclusion of inorganic/metallic Ag nanoparticles lowers the decomposition temperature compared to neat UPE, indicating successful nanoparticle integration into the UPE resin matrix and its influence on thermal stability. The data from this analysis suggest that nanoparticle presence accelerates the nanocomposite's decomposition, possibly due to nanoparticle agglomerations forming weak bonds at the interface.

4. Conclusions

The investigation into the influence of AgNP concentration promoted by DMPT was carried out successfully, and the study yielded several significant conclusions:

- The spectral analysis revealed distinct Ag-O vibrations within the UPE resin matrix at all concentrations of Ag nanoparticles. The disappearance of peaks at lower concentrations indicated limited dispersion of the nanoparticles, rendering them less visible.
- Non-covalent interactions facilitated the dispersion of nanoparticles within the resin matrix, and higher nanoparticle percentages improved the visibility of metal vibrations, signifying enhanced dispersion.
- The addition of AgNPs altered the surface morphology; higher concentrations resulted in rougher and more uneven forms, with increased nanoparticle percentages leading to particle aggregation and irregular dispersion.
- Despite these morphological changes, the absence of pores, voids or microgaps demonstrated perfect bonding between the AgNPs and UPE resin.
- The introduction of AgNPs to UPE influenced its degradation behaviour based on concentration; nanocomposites with 0.5% and 1.0% AgNPs experienced less weight loss than UPE without AgNPs, while 1.5% AgNPs exhibited superior thermal stability, suggesting an optimal concentration range.
- While higher nanoparticle concentrations may create weak links at the nanocomposite interface, the correlation between increased nanoparticle concentration and enhanced thermal stability was evident through constant char residue at 800°C and higher peak intensity, indicating effective integration and stability enhancement.

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Supplementary material

The supplementary material for this article can be found online.

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