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FULL PAPER

Synthesis and characterization of TiO_2, Ag_2O, and graphene oxide nanoparticles with polystyrene as a nonocomposites and some of their applications

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In this study, metal oxides nanoparticles TiO_2 and Ag_2O NPs were synthesized by green method using sider leaves extract. The nano-composites were prepared from condensation reaction of polystyrene (PS), TiO_2, Ag_2O, and (GO) by a simple mixing method. These structures as (PS/GO/Ag_2O) and (PS/TiO_2/Ag_2O) were characterized by X-rays diffraction, FE-SEM, TEM, and thermal analysis. The measurements proved that all the components, of polystyrene (PS), graphene oxide (GO), TiO_2, and Ag_2O were present and that the size of the nanoparticles within nano-scale. Nano-composites were tested in biological activity applications as anti-bacterial and anti-fungal, the results proved that (PS/GO/Ag_2O) nano-composites gave a higher inhibition value than (PS/TiO_2/Ag_2O) nano-composites with bacteria, but it gave the result when applied to fungi.

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KEYWORDS

Nano-composite; plystyrene; graphene oxide; Ag_2O; TiO_2; metal oxide nanoparticles.

Introduction

The main parameters of nanoparticles are their shape, their size, and morphological substructure of the substance (including the aspect ratio where appropriate) [1].

Depending on their size, shape, and chemical and physical properties, nanoparticles can be categorized into different groups, some of which are considered semiconductor nanoparticles, ceramic nanoparticles, polymeric nanoparticles, carbon-based nanoparticles, lipid-based nanoparticles, and metal nanoparticles [2].

Nano-sized metal oxides have many outstanding properties, including a high removal capacity and heavy metals selectivity. As promising adsorbents to heavy metals, they have great potential. Metal oxide-based nano-materials include manganese oxides, nano-sized iron oxides, titanium oxides, cerium oxides, ZnOs, magnesium oxides, aluminum oxides, and zirconium oxides [3].

Titanium oxide (TiO_2) is further used in a variety of applications such as disinfection agents and white pigment, food color flavor enhancer additives, and decomposition of organic compounds [4], [5].

Titanium oxides (TiO_2) nano-metal is relatively less expensive than any other nanomaterial and exhibits good thermal and chemical stability as well as low human toxicity [6].

Silver oxide (Ag_2O) nanoparticles are spherical or faceted high surface area oxide magnetic nanostructured particles. Silver oxide nanoparticles are similarly available in coated and dispersed high purity, ultra-high purity, and transparent forms [7].

Graphene and its oxide have been widely used due to many industrial applications and in scientific research which keeps pace with the continuous demand for materials around the world, since graphene oxide has different properties including the electrical, mechanical, etc. It is can be prepared by graphite oxidation [8].

Polymers are extensively used in applications because they are lightweight, easily processed, and have design flexibility. Polymer composites, consisting of the polymer phase and an additional component(s), generally balance performance, mechanical properties, cost, and processing. The additional component, or filler, may be of reinforcing or non-reinforcing type. Reinforcing fillers aid in improving mechanical properties and abrasion resistance whereas non-reinforcing fillers may decrease the cost, modify density, improve barrier properties, or change color [9].

Experimental part

Materials

The materials used in the preparation of nano-composites were supplied by several companies:

- Silver nitrate ($AgNO_3$), Sodium nitrite ($NaNO_2$), and Potassium permanganate ($KMnO_4$) from CDH.
- Titanium isopropoxideTi[$OCH(CH_3)_2]_4$, Ammonium Hydroxide(NH_4OH), Sulfuric acid (con.) (H_2SO_4), and Hydrogen peroxide H_2O_2 fromSigma-Aldrich.
- Carbon tetra chloride(CCl_4) and Graphite from Fasco Expoxies.

Preparation of sider leaves aqueous extract

Typically, 150 ml of distilled water was added to 4 g of Sidr leaf extract with heating and stirring for 15 minutes, then, filtered the solution for utilizing in the synthesis [10].

Synthesis of metal oxides nanoparticles

A- Synthesis of Ag_2O nanoparticles by green method

A quantity of (250) ml of ionic water was added to (2,2) of silver nitrate, the solution was heated to 100 °C with stirring for 30 minutes, after that, the aqueous extract of Sidr leaves prepared in the previous way was added slowly to the silver nitrate solution by a dropper until the color changed. The resulting mixture was heated to 400 °C for two hours and the solution was allowed to cool at room temperature. Finally, the mixture was dried and the precipitate formed [11].

B- Synthesis of TiO_2 nanoparticles by sol-gel method

Fifteen mL of acetic acid was mixed with 150 mL of distilled water and stirred for 30 min, and then 5 mL of titanium tetra isopropoxide was added slowly, and stirred for 2 hours to get clear solution.

The above solution was (ultrasonicated) for 30 min, and then kept in the dark for 24 hours, after that the mixture retain in a hot oven at 80 ◦C for 3 hours to get a gel, then the gel left in an oven at 100 °C until dry.

The last mixture was dried and calcinated at 400 °C for 3 hours to get the white color of TiO_2 nanoparticles [12].

C- Synthesis of graphene oxide nanoparticles (Hummers' method)

Graphene oxide (GO) was prepared according to Hummers' method, 1 g of graphite was added into cool (50) ml concentrated H_2SO_4 and stirred in an ice bath for 15 minutes. A quantity of (4) g from ($NaNO_2$) and (6) g of

($KMnO_4$) were added to the above-mentioned solution with stirred in an ice bath for 6 hours. The ice-bath removed and the temperature of the mixture was kept at 35 °C in water path for 30 minutes to became the mixture pasty (deep red- brown in color). 50 ml of DI water was then added into above mixture. The temperature then raised to 90-98 °C. The above mixture was diluted by addition (250) ml warm DI water. After that, (30) ml of H_2O_2 was added until solution turned bright [13].

Synthesis of nano-composites

A- Synthesis of (Polystyrene/TiO_2/Ag_2O) nano-composites

Typically, 25 ml of CCl_4 was added into (1) g of polystyrene and the mixture was refluxed for 1 hour at 50 °C (**Solution A**), 0.1 gm of TiO_2 NPs was mixed with (0.1) gm of Ag_2O NPs into (10) ml of CCl_4. Then, the mixture was exposed to ultrasound for 5 minutes to ensure mixing (**Solution B**).

Mixture solution B was added to solution A (its above) and refluxed at 50 °C for 5 hours, after that, the mixture was left to cool at room temperature and finally collected [14].

B- Synthesis of (polystyrene/GO/Ag_2O) nanocomposites

Typically, 25 ml of CCl4 was added into (1) g of polystyrene and the mixture was refluxed for 1 hour at 50 °C (**Solution A**), (0.1) gm of GO NPs was mixed with (0.1) gm of Ag_2O NPs into (10) ml of CCl_4. Then, the mixture was exposed to ultrasound for 5 minutes to ensure mixing (**Solution B**).

Mixture solution B was added to solution A (its above) and refluxed at 50 °C for 5 hours, after that, the mixture was left to cool at room temperature and finally collected [15].

Results and discussion

X-Ray diffraction Of PS/TiO_2/Ag_2O nanocomposites

X-Ray diffraction pattern in Figure (1) of Polystyrene/TiO_2/Ag_2O nano-composites indicated the polymer diffraction peaks at [°2Th.] 20.5040° and 27.6154° corresponding to (244.28°) and (185.27°) planes were observed, while XRD revealed TiO_2 nanoparticles (NPs) diffraction peaks at 26.0445°, 48.20°, 63.40°, and 76.3232° corresponding to 267.41, 224.54°, 189.33°, and 52.07°, respectively as compared with the standard reference for Tio2 NPs [16].

Moreover, XRD indicated in Figure 1 Ag_2O NPs diffraction peaks at [°2Th.] 32.1599°, 38.5212°, 46.0770°, and 54.7755 corresponding to 406.37, 87.34, 168.48, and 76.71, respectively as compared with the standard reference for Ag_2O NPs [17].

The presence of PS/TiO_2/Ag_2O nano-composites in X-Ray diffraction is solid evidence for the success of the reaction, as presented in Table 1.

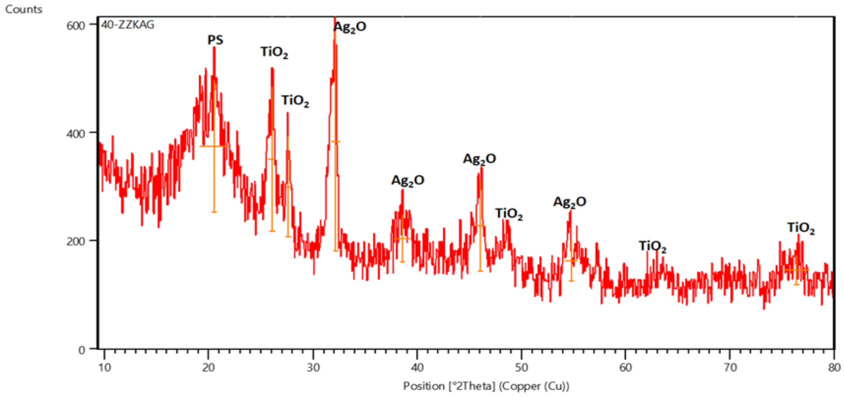

FIGURE 1 XRD pattern of ($PS/TiO_2/Ag_2O$) Nano-composites

TABLE 1 XRD record of ($PS/TiO_2/Ag_2O$) nano-composites

Pos. [°2Th.]	FWHM Left [°2Th.]	Matched by	Particle size	Average particle size (nm)
20.5040	2.7552	Polymer	3.06 (nm)	
26.0445	0.6888	TiO_2 NPs	12.37 (nm)	
27.6154	0.3936	Polymer	21.72 (nm)	
32.1599	0.8856	Ag_2O NPs	9.75 (nm)	
38.5212	1.5744	Ag_2O NPs	5.59 (nm)	13.92 (nm)
46.0770	0.7872	Ag_2O NPs	11.46 (nm)	
48.2000	0.3936	TiO_2 NPs	23.10 (nm)	
54.7755	1.1808	Ag_2O NPs	7.92 (nm)	
63.4000	0.2236	TiO_2 NPs	43.64 (nm)	
76.3232	2.3616	TiO_2 NPs	4.47 (nm)	

X-Ray diffraction Of $PS/GO/Ag_2O$ nano-composites

Figure 2 displays the XRD pattern for $PS/GO/Ag_2O$ nano-composites which described the graphene oxide diffraction peaks at [°2Th.] 13.3°, and 42.84° corresponding to 94.31 and 42.27, while its revealed polymer diffraction peaks at [°2Th.] 23.91° corresponding to 78.84, as compared with the standard reference for GO NPs and polystyrene [18], [19].

Moreover, XRD in Figure (2), depicts Ag_2O NPs diffraction peaks at [°2Th.] 32.20°, 34.8866°, 46.1155°, 53.4483°, 67.2397°, and 76.6936°, respectively corresponding to 227.26, 556.55, 271.30, 134.53, 71.11 and 131.26, as indicated in Table 2.

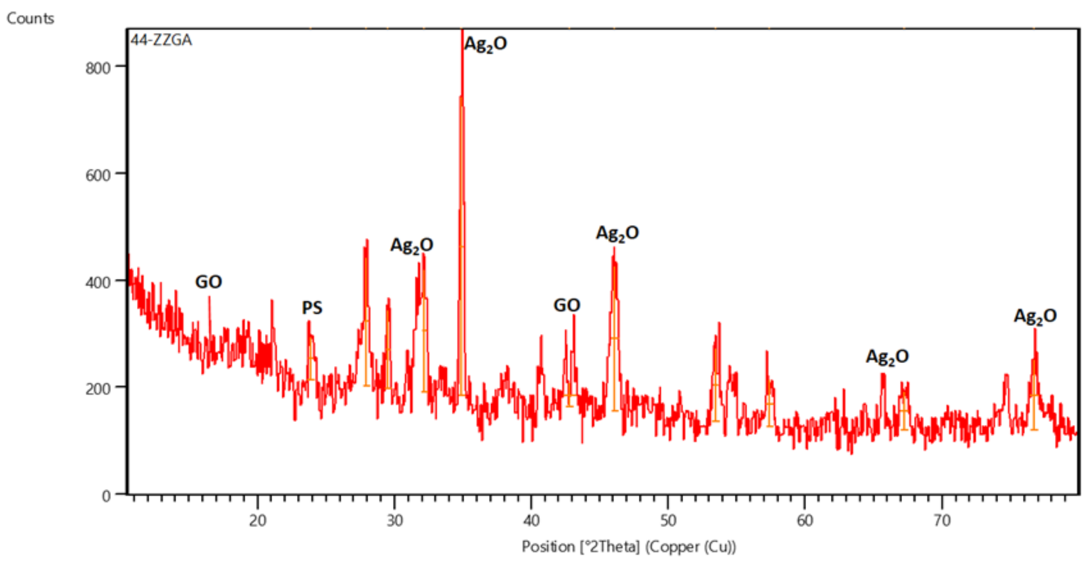

FIGURE 2 XRD pattern of (PS/GO/Ag_2O) Nano-composites

TABLE 2 XRD record of (PS/GO/Ag_2O) nano-composites

Pos. [°2Th.]	FWHM Left [°2Th.]	Matched by	Particle size	Average particle size
13.3532	7.6883	GO	1.09 (nm)	19.18 (nm)
23.9114	0.3936	Polymer	21.56 (nm)	
29.5265	0.2952	Ag_2O NPs	29.08 (nm)	
32.2072	0.3936	Ag_2O NPs	21.95 (nm)	
34.8866	0.2460	Ag_2O NPs	35.37 (nm)	
42.8410	0.9840	GO	9.06 (nm)	
46.1155	0.5904	Ag_2O NPs	15.28 (nm)	
53.4483	0.3936	Ag_2O NPs	23.61 (nm)	
67.2397	0.5904	Ag_2O NPs	16.89 (nm)	
76.6936	0.5904	Ag_2O NPs	17.93 (nm)	

Thermogravimetric analysis (TG) of PS/TiO_2/Ag_2O and of PS/GO/ Ag_2O nano-composites

Thermal decomposition of prepared nano-composites under nitrogen atmosphere, the heating range (35-800) °C and their atmospheres are displayed in Figure 3; the following results were explained according to the analytical suggestions mentioned in literature [20]:

Thermogravimetric analysis of PS/TiO_2/Ag_2O nanocomposites

1- The first stage: At 35-120 °C with a weight loss percentage of 3.32% attributed to the loss of water (absorbed from the atmosphere) which adsorbed physically of nano-composite due to the fact that nano-material have very high surface area.

2- The second stage: At 120-440 °C and third stage at 440-800 °C with a total weight (percentage) loss equal to 85.77% (found) corresponding 83.3% (calculated), these stages include the starting of styrene unit loss and there is decomposition of carbon skeleton of the polymer.

3- The third stage: At temperature more than 800 °C with weight (percentage) of 14.229% (found) corresponding 16.6% (calculated). This stage could be attributing to the amount of TiO_2 and Ag_2O NPs.

It is worthy notes that the obtained weight percentage (Wt%) of the polymer and metal oxide nanoparticles (TiO_2, Ag_2O) are in agreement with the calculated weight percentage (Wt%) ones, therefore, this was considered as a good evidence for the reaction success, as indicated in Table 4.

Thermogravimetric analysis of PS/GO/Ag_2O nano-composites

Thermogravimetric analysis of prepared nano-composites in Figure 4 depicts four stages of weight loss, and the following results were explained according to analytical suggestions mentioned in literature [21]:

1- The first stage: At 35-120 °C with a weight loss percentage of 7.30% attributed to the loss of water (absorbed from the atmosphere) which adsorbed physically of nano-composite due to the fact that nano-material have very high surface area.

2- The second stage: At 120-440 °C and third stage at 440-800 °C with a total weight (percentage) loss equal to 88.23% (found) corresponding 83.3% (calculated), these stages include the starting of styrene unit loss and there are decomposition of carbon skeleton of the polymer.

3- The third stage: At temperature more than 800 °C with weight (percentage) of 11.72% (obtained) corresponding 16.6% (calculated). This stage could be attributing to the residue amount of GO and Ag_2O NPs.

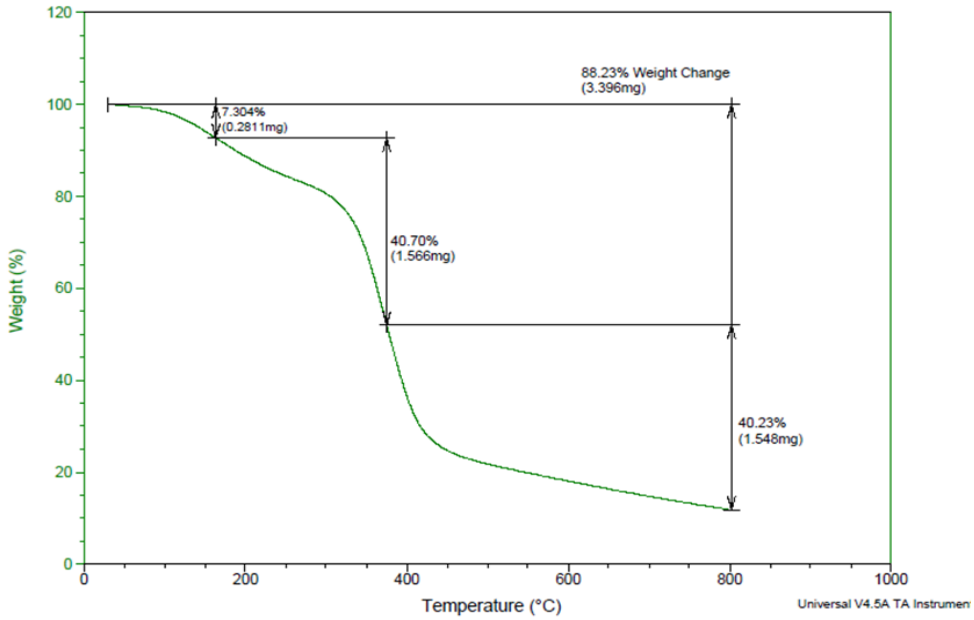

FIGURE 3 TG analysis of (PS/GO/Ag_2O)

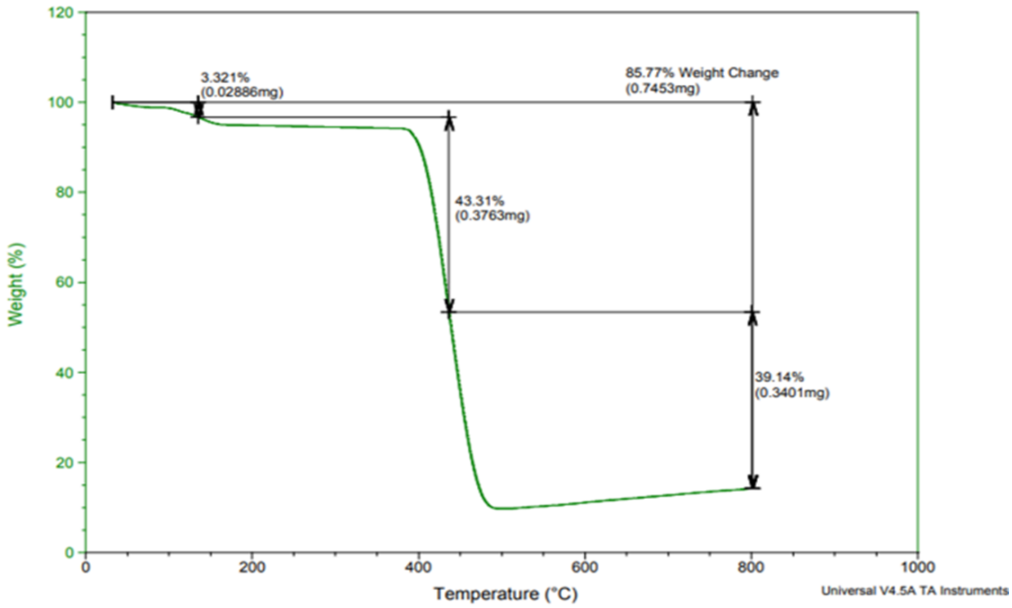

FIGURE 4 TG analysis of (PS/TiO_2/Ag_2O)

Differential scanning calorimetry (DSC) of PS/TiO_2/Ag_2O Nano-composites

Differential scanning calorimetry (DSC) analysis of the prepared PS/TiO_2/Ag_2O nano-composites included two endothermic stages: First, at 177.09 °C equal ΔH to 568 J/g for PS/TiO_2/Ag_2O, at 353.82 °C equal ΔH to 429.3 J/g for PS/GO/Ag_2O nano-composites, the glass transition of polystyrene in nano-composites was considered as compared with the standard reference of polystyrene which is 100 °C [22]. Second, 400.56°C, the conceded melting point of polystyrene in nano-composites was compared with the standard reference of polystyrene which was 270°C. This was another indication of the reaction success, as displayed in Figure 5.

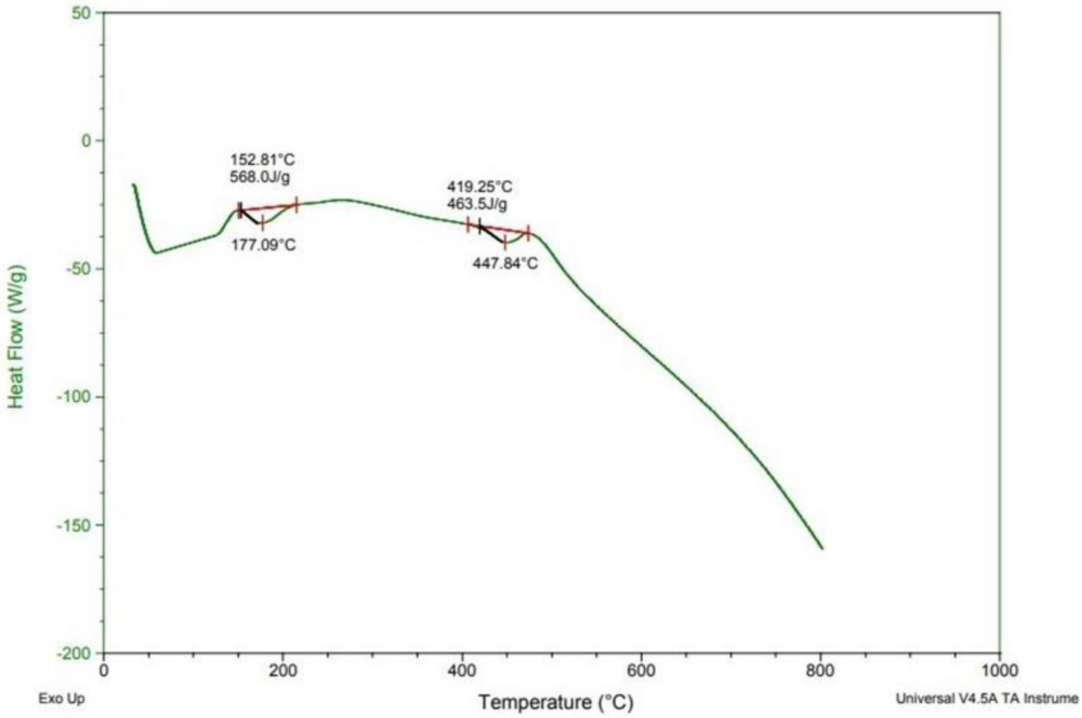

FIGURE 5 DSC/TGA of (PS/TiO_2/Ag_2O) nano-composite

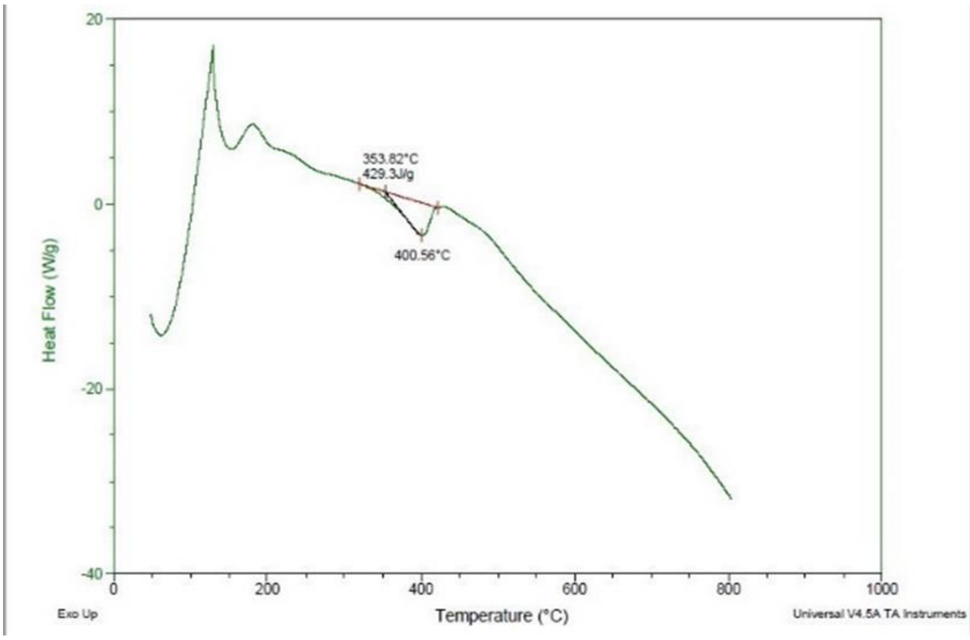

FIGURE 6 DSC/TGA ($PS/TiO_2/Ag_2O$) nano-composites

The field emission scanning electron microscopy (FE-SEM) of $PS/TiO_2/Ag_2O$ and $PS/GO/Ag_2O$ nanocomposites

The Field Emission Scanning Electron Microscopy (FE-SEM) measurements were conductive to $PS/TiO_2/Ag_2O$ and $PS/GO/Ag_2O$, as displayed in Figures 6 and 7. The FE-SEM reveals that two different nano-structures, which are irregular sphere like those ones with nano-scale range, sheet like nanoparticles with a thickness of approximately 30-78 nm in $PS/TiO_2/Ag_2O$, these nano-structures means that the prepared nano-composite is within the nano-scale [23].

Moreover, FE-SEM measurement indicated the surface roughness of nano-composite with a high number of nano-shrinkage about 20 nm. The presence of Ag_2O, TiO_2 and GO NPs causes surface roughness of polymer.

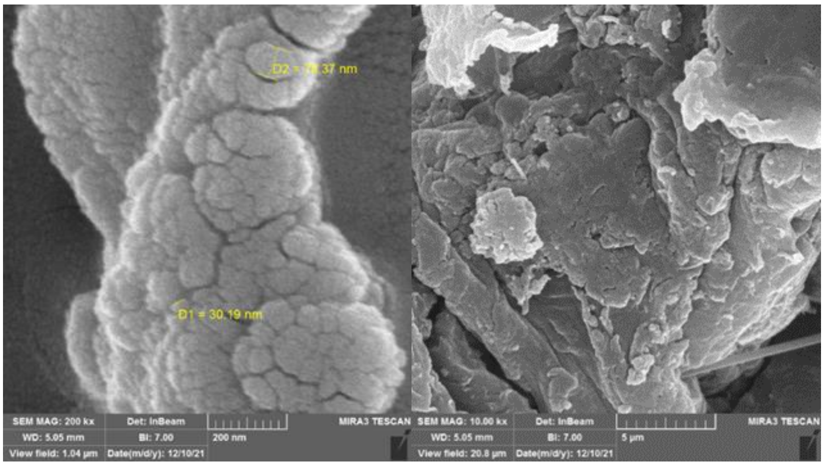

FIGURE 7 FE-SEM of ($PS/TiO_2/Ag_2O$) nano-composites

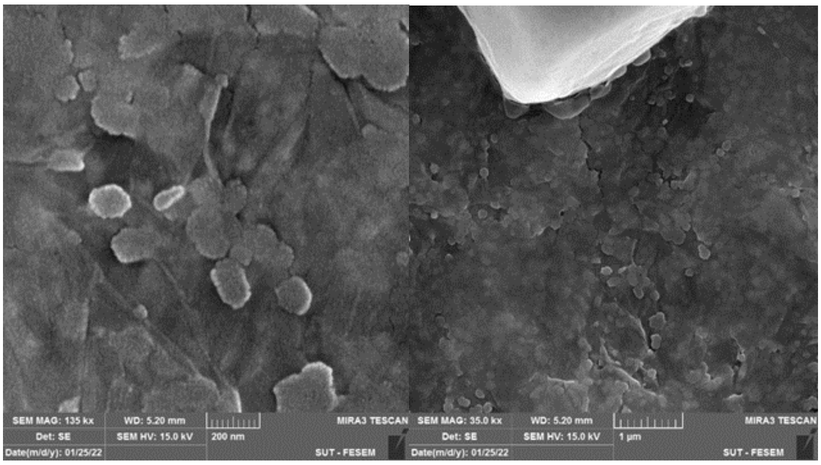

FIGURE 8 FE-SEM of (PS/GO/Ag_2O) nano-composites

Application

The biological activities of prepared nano-composites were tested against two types of bacteria (*Klebsiella pneumoniae*) and (*Staphylococcus aureus*) and demonstrated that nano-composites have a different effects on inhibiting the growth of the studied bacteria. This is due to the ability of these nano-composites to the production of free radicals leading to the oxidative stress, and thus damage to proteins, DNA, and cell membranes as well as binding to cytosolic proteins, DNA, and enzymes. This interaction cause decreased inhibiting respiratory chain, ATP production, and metabolic pathways [24, 25].

The results of the bacterial tests proved that PS/GO/Ag_2O nano-composite was more inhibiting bacteria (*Staphylococcus aureus*) with an effect size of 29 mm than PS/TiO_2/Ag_2O nano-composite, which had an effect of 16 mm, while the inhibition effect on bacteria (*Klebsiella pneumoniae*) was PS/GO/Ag_2O nano-composite with a size of 16 mm less than PS/TiO_2/Ag_2O nano-composite with a size of 18 mm, as displayed in Figures 8 and 9.

The anti-fungal activity of all nano-composites synthesized by the green and sol-gel method was studied on *Candida Albicans*, the results indicated that all nano-composites had strong and medium fungal activity. The results showed that the PS/ TiO_2/ Ag_2O nano-composites gave the strongest against *Candida Albicans* and the size of hole (affecting the bacteria) was approximately 18 nm, as compared with 15 nm for PS/GO/Ag_2O nano-composites.

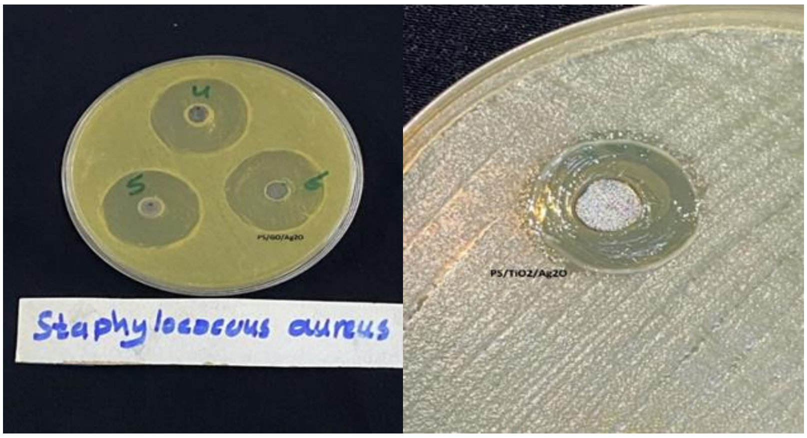

FIGURE 9 Biological test (Staphylococcus aureus) of (PS/GO/Ag_2O) and PS/TiO_2/Ag_2O nano-composites

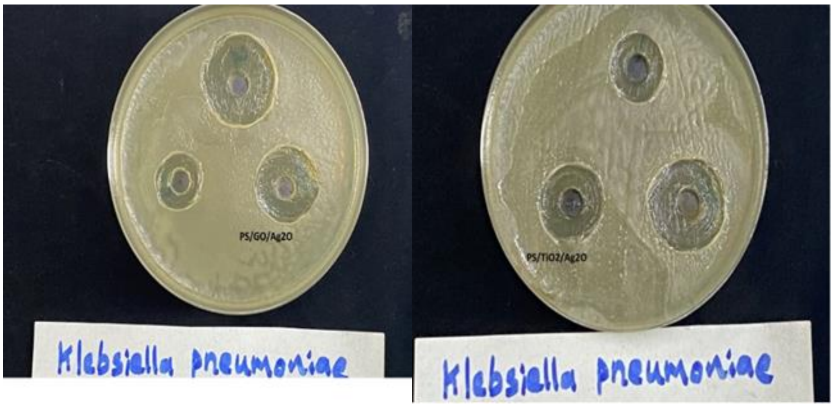

FIGURE 10 Biological test (Klebsiella) of (PS/GO/Ag_2O) and PS/TiO_2/Ag_2O nano-composites

Conclusion

The results proved the validity of the proposed method for nano-composites, as it gave the size of nanoparticles based on FE-SEM approximately 20 nm in PS/GO/Ag_2O nano-composite and 30 to 78 nm in PS/TiO_2/Ag_2O nano-composite and these two sizes are within the nano-scale. These nano-composites were used in the biological applications of bacteria and fungi, Gram-positive (*Staphylococcus aureus*) and Gram-negative (*Klebsiella*) bacteria were selected with *(Candida parapsilosis)*.

The results indicated that PS/GO/Ag_2O nano-composite gave a size of inhibition against bacteria (*Staphylococcus aureus*) 29 mm and against bacteria (*Klebsiella*) 16 mm, while the results showed less inhibition with PS/TiO_2/Ag_2O nano-composite, in which the inhibition size against bacteria (*Staphylococcus aureus*) was 16 mm and against bacteria (*Klebsiella*) 18 mm. As for the effect of nano-composites on *Candida parapsilosis*, the PS/GO/Ag_2O nano-composite was less than the inhibition of PS/TiO_2/Ag_2O nano-composite, which had the inhibition size of 15 mm and 18 nm, respectively.

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