Synthesis and Characterization of Silver-Titanium Nanocomposite via Horizontal Vapor Phase Growth (HVPG) Technique

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Abstract: Silver and Titanium Nanocomposite materials are well known as two materials that can effectively eradicate bacteria. Study on the synthesis and characterization of these materials using Horizontal Vapor Phase Growth (HVPG) technique was introduced to combine between Silver and Titanium in a single synthesis. Silver and Titanium powder was proportioned to 1:1 with the amount 17.5 mg.

The experiments were performed in high vacuum pressure ≈10^{-6} Torr. Quartz Tubes were sealed at both ends and were baked using horizontal furnace. The Experiments were conducted by varying temperature growth from 800°C, 1000°C, and 1200°C with a growth time of 4 hours, 6 hours, and 8 hours. The end products were characterized using Scanning Electron Microscopy (SEM) and Energy Dispersive X-ray (EDX) analysis to determine atomic proportion and Silver-Titanium nanocomposite shape and size. The study determined the optimum temperature and growth time in growing Silver-Titanium Nanocomposites material.

The optimum parameter in terms of the size of the nanomaterials produced is the temperature of 1000°C and growth time of 8 hours. Based in the experiments are studied also produced Silver-Titanium nanocomposite with different nano shape like Nanoparticles, Nanosphere, Nanotriangulars, and Nanorods.

Key Words: Silver-Titanium; Nanocomposite; HVPG

1. BACKGROUND

The synthesis of nanomaterials has been a very active area of research since materials in the nanoscale dimensions exhibit very interesting characteristics that would otherwise not be observed if the materials were in dimensions far greater than the nano-scale level. Among the many materials that are being studied for their nano-scale level behaviour and nano-scale level applications are silver and titanium dioxide.

Silver is a material commonly used in medical applications because of its anti-bacterial property. Several methods have been employed to to produce silver nanomaterials. In one study, Green synthesis was used to produce Nano-silver sol [1] with grain sizes of between 1 to 3 nm. Silver nano fiber with widths of around 180-190 nm were also produced in another study by Electrospinning method [2]. Other studies synthesized Silver nanorods using the Microwave heating method, Silver Nano-particles by facile synthesis methods, and Silver Nanotubes by Electrochemical method [3, 4, 5].

Titanium dioxide is also a material that has an anti-bacterial property. Several methods have been used successfully in the synthesis of titanium dioxide nanomaterials for medical application. Included in the list of methods as identified by Chen and Mao [6] are the Sol-gel method, the Micelle and Inverse Micelle method, the Sol Method, the Hydrothermal method, the Solvothermal method, the Direct Oxidation (DO) method, the Chemical Vapor Deposition (CVD) method, the Physical Vapor Deposition (PVD) method, the Electrodeposition method, the Sonochemical method, and the Microwave method.

Since both silver and titanium dioxide exhibit anti-bacterial properties, several researchers have attempted to produce Silver-titanium dioxide nanocomposite materials. In a study by Wang et. al. (2013) made use of both Hydrothermal and Irradiation method to produce silver-titanium nanobelts [7]. Other researchers used various methods to produce Silver-Titanium dioxide (Ag/TiO_2) nanocomposite materials including: Chemical reaction method [8], Reactive Magnetron Sputtering (RMS) method [9], and Photocatalytic method [10].

Another promising method of synthesizing nanomaterials but has not been used in synthesizing silver-titanium dioxide nanocomposite materials is the Horizontal Vapor Phase Growth (HVPG) Technique. In this study, the said method was employed in the attempt to produce novel Silver-Titanium dioxide nanocomposite materials.

2. METHODOLOGY

Horizontal Vapor Phase Growth (HVPG) technique was used in this study to synthesis Silver-Titanium dioxide Nanocomposite material. Scanning Electron Microscopy (SEM) and Energy Dispersive X-ray (EDX) Analysis were done to characterize the morphology and chemical composition of the resulting products.

A mixture of 17.5 mg of 99.99% purity Silver (Ag) powder form Aldrich and 17.5 mg of Titanium dioxide (TiO_2) P25 powder from Degussa were loaded in 9 closed end fused silica tubes. Each tube was then evacuated down to a pressure of around 10^{-6} Torr in a Thermionics High Vacuum System and then sealed using an LPG-Oxygen blowtorch.

Each sealed tube is then inserted halfway through one end of a Horizontal Vurnace. Such a configuration creates a temperature gradient needed for the nanomaterials to grow inside the tube.

The parameters varied in the study are the baking temperature (800°C, 1000°, and 1200°C) and the baking time (4 hours, 6 hours, 8 hours).

The morphology and chemical composition of the synthesized nanomaterials deposited on the inner walls of the tubes were then characterized by Scanning Electron Microscopy (SEM) and Elemental Dispersive X-ray (EDX) analysis using a JEOL JSM-5310 Scanning Electron Microscope.

Table 1 summarizes the parameters used for each tube used in the study.

Table 1. Variable baking

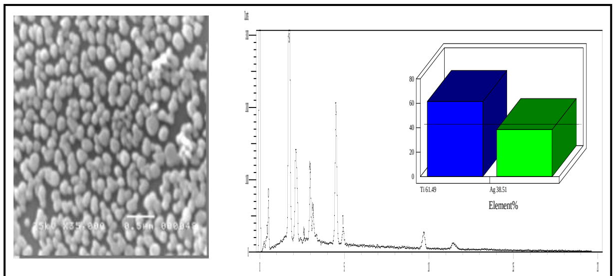
No. Tubes	Temp. (°C)	Baking Time (h)
1	800	4
2	800	6
3	800	8
4	1000	4
5	1000	6
6	1000	8
7	1200	4
8	1200	6
9	1200	8

3. RESULTS AND DISCUSSION

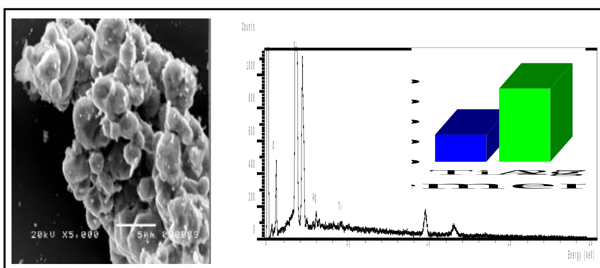
Table 2 summarizes the range of sizes of the micro- and nano- composites synthesized in this study. Table 2 shows that the sizes of the micro- and nano- materials formed in the tubes vary. The smallest range of sizes of composite material formed was found in tube 6, the tube baked at 1000°C for 8 hours.

Table 2. Size of Nanocomposite from quartz cubes

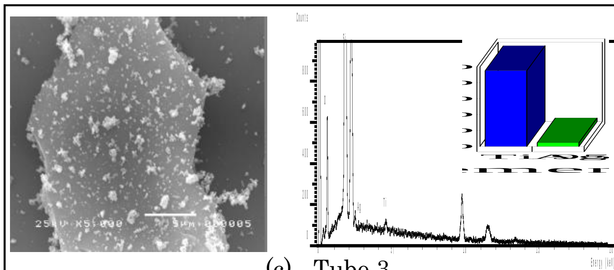
Tubes	Result	Size range (μm)
1	Micro-Nano Composite	0.16 - 7.37
2	Micro-Nano Composite	0.3 - 3.57
3	Micro-Nano Composite	0.82 - 3.32
4	Micro-Nano Composite	0.34 - 1.43
5	Nano-composite	0.45 - 0.69
6	Nano-composite	0.38 - 0.67
7	Micro-Nano Composite	0.84 - 7.69
8	Micro-Nano Composite	0.52 - 4.79
9	Micro-Nano Composite	0.6 - 8.43



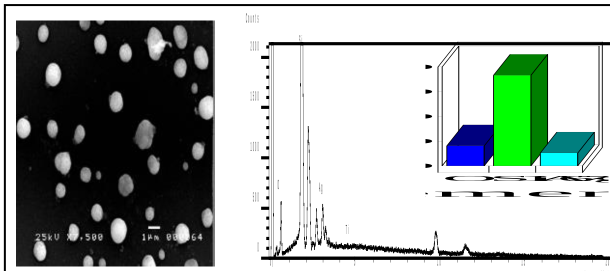
(a) Tube 1



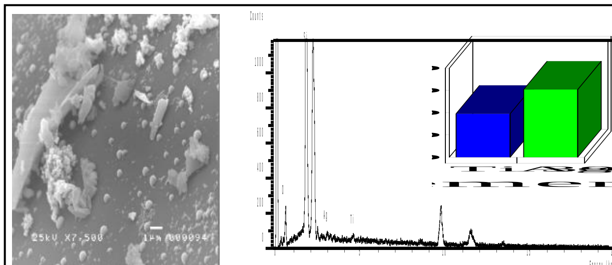
(b) Tube 2



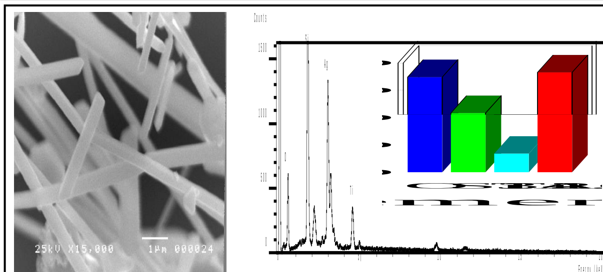
(c) Tube 3



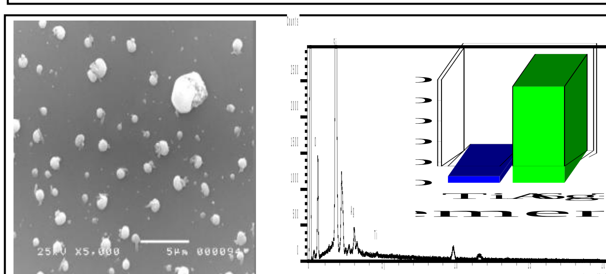
(d) Tube 4



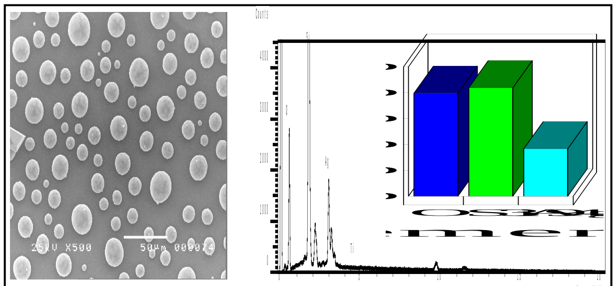
(e) Tube 5



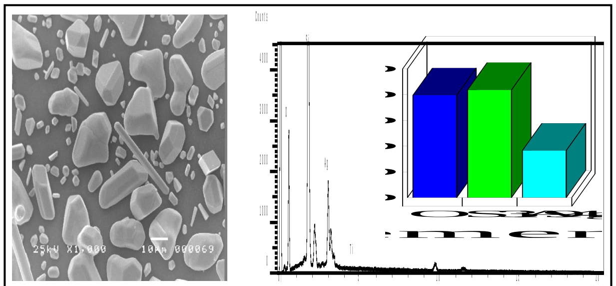
(f) Tube 6



(g) Tube 7



(h) Tube 8



(i) Tube 9

Figure. 2. Shape and EDX graph of micro- and nano-composite material typically in each tubes

Figure 2. are SEM micrographs of the majority of micro-nano-materials found in each tube and EDX graph of samples each tube.

Figure 2a reveals that there are a lot of agglomerated microparticles. These microparticles have dimensions that are smaller than the dimensions of the source material (silver and titanium dioxide). These microparticles could be the source powders that did not completely evaporate. This might be because of the fact that the baking time of 4 hours is too short and the fact that the temperature of 800°C is too low for the powders to evaporate and grow into the desired micro- and nano-materials.

Figure 2b reveals that there are a lot of silver-titanium composite materials formed tube 2, the tube baked at 800°C for 6 hours. These materials, however, have sizes in the micron range.

Figure 2c reveals that there are a lot of nanoparticles formed in tube 3, the tube baked at 800°C for 8 hours.

Figure 2d shows that there are a lot of spherical nanoparticles produced in tube 4, the tube baked at 1000°C for 4 hours.

Figure 2e shows that there are also a lot of spherical micro- and nano-particles formed in tube 5, the tube baked at 1000°C for 6 hours.

Figure 2f reveals that there are plenty of nanorods with sharp edges in tube 6, the tube baked at 1000°C for 8 hours.

Figure 2g reveals that for tube 7, the tube baked at 1200°C for 4 hours, there are silver-titanium nanocomposites formed. The spherical part of the nanocomposites were made up of silver while the middle portion, that looked like a sliver of icing on top of the spheres, were found to be made up of titanium dioxide.

Figure 2h shows that there are a lot of spherical particles in tube 8, the tube baked at 1200°C for 6 hours. These spherical particles are bigger in size than those in tube 4.

Figure 2i reveals that in tube 9, the tube baked at 1200°C for 8 hours, there is a presence of the largest particles found in the study. The said particles have sizes that ranged from 0.6 - 8.4 micrometers. The said particles also have flat faces and sharp edges. Some microcubes and microrods were also found.

4. CONCLUSIONS

This research presents the successful synthesis of Silver-titanium dioxide micro- and nano- composite nanomaterials at different growth temperatures and growth times. The method employed was the Horizontal Vapor Phase Growth (HVPG) technique. The result of materials form are nanoparticles, nanosphere, nanotriangulars, and nanorods, also reveal micro- materials such as microtriangulars, microparticles and microrods.

5. ACKNOWLEDGMENTS

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