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Synthesis and characterization of Silver-Silica heterogeneous nanocomposite particles by Lithium Aluminum Hydroxide reducing method

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ABSTRACT

We have used Lithium Aluminum Hydroxide reducing method of silver nitrate to prepare heterogeneous silver-silica nanocomposite particles with thiol and amino groups serving to bind the Ag nanoparticles to the surfaces of the SiO_2 nanoparticles. We examined products of these reductions using FTIR, SEM, XRD and UV-vis. spectroscopy. The SiO_2 nanoparticles had diameters ranging from 70 to 90 nm; the Ag nanoparticles that formed on the surfaces of the SiO_2 nanoparticles had an average size of ca. 15 nm.

Keywords: *Silver-silica heterogeneous nanocomposite; Lithium Aluminum Hydroxide; Thiol; Amino; XRD; SEM.*

INTRODUCTION

Nanotechnology (sometimes shortened to "nanotech") is the study of manipulating matter on an atomic and molecular scale. Generally, nanotechnology deals with developing materials, devices, or other structures possessing at least one dimension sized from 1 to 100 nanometers. A nano composite is as a multiphase solid material where one of the phases has one, two or three dimensions of less than 100 nanometers (nm), or structures having nano-scale repeat distances between the different phases that make up the material. Silver is a metallic chemical element with the chemical symbol Ag and atomic number 47. A soft, white, lustrous transition metal, it has the highest electrical conductivity of any element and the highest thermal conductivity of any metal. The metal occurs naturally in its pure, free form (native silver), as an alloy with gold and other metals, and in minerals such as argentite and chlorargyrite. Silver is a very ductile, malleable (slightly harder than gold), monovalent coinage metal, with a brilliant white metallic luster that can take a high degree of polish.

It has the highest electrical conductivity of all metals, even higher than copper, but its greater cost has prevented it from being widely used in place of copper for electrical purposes.

Despite this, 13, 540 tons were used in the electromagnets used for enriching uranium during World War II (mainly because of the wartime shortage of copper). An exception to this is in radio-frequency engineering, particularly at VHF and higher frequencies, where silver plating to improve electrical conductivity of parts, including wires, is widely employed. Another notable exception is in high-end audio cables, where manufacturers claim that scaling copper conductors by 6% achieves slightly better results.

Heterogeneous nano composite materials, such as gold-platinum, gold-palladium, silver-silica, and gold-titania, have been studied widely in recent years [1-5] because their catalytic properties differ dramatically from those of their single components. For example, the absorbance of metals coated on photo catalysts can be tuned selectively to any wavelength across the visible and infrared regions of the spectrum simply by adjusting the ratio of the size of the dielectric core to the thickness of the metal over layer [6, 7]. In addition, Au/ Pt composite nano materials exhibit a more efficient catalytic activity than do mono disperse Pt nanoparticles for both the hydrogenation of olefins and the visible-light-induced generation of H_2 from water [8]. In general, these types of nano composite structures have been fabricated by depositing nano scale metal particles onto dielectric spheres. In particular, metal-coated SiO_2 surfaces have been fabricated using such approaches as electroless deposition [9], seed plating [10], surface functionalization [11], sputtering, layer-by-layer processing, and nonchemical deposition. These SiO_2-coating procedures are usually complex because they involve multi-step processes that make it difficult to obtain dense, uniform nanoscale metal layers of high purity. Several problems arise when immobilizing metal nanoparticles onto SiO_2 surfaces, including incomplete coverage, rough surfaces, and non uniformity of size and composition. Therefore, simple and controllable processes must be developed if these materials are to find industrial applications. We used FTIR, SEM, XRD and UV-Vis spectroscopy to characterize the resulting composite particles.

EXPERIMENTAL

All reagents were supplied by Merck and were used without further purification. The FT-IR spectra were recorded by KBr disk using a Bruker Tensor 27 M 420 FT-IR spectrophotometer. The UV–Vis spectra in DMF were recorded with a Perkin Elmer 25 Lambda spectrophotometer. XRD diffractions were studied with X-ray diffraction device Italy and USA MDP3000 model. The morphology and size of the nano composites were studied with a Leo-1430 VP scanning electron microscope (SEM).

Preparation of Ag-SiO_2 Nano composites

For synthesis of Ag nano composites, 0.5 (S1) and 1 (S2) g $AgNO_3$ was taken to the beaker (50 ml) and added $LiAlH_4$ (4×10^{-4}). Then this component of the beaker was added to another beaker that included 100 g of SiO_2. The whole solution was mixed 24 h; finally the compound was precipitated and separated. The product dried at 50^0C for 24 h. Then the nano composite was taken to the electrical furnace at 185 ^{0}C for 5 h until to calcination. In S_1: Mp 380º C Yield: 72%. FTIR (KBr pellet, cm^{-1}): 3454 (m, O-H), 1635 (w, NH), 1078(m, Al-O), 806(s, Si-O) and 473 (s, Ag-O). UV–Vis, λmax (nm)/ε ($M^{-1}cm^{-1}$); 240, 310 and 380. In S_2: Mp 380º C Yield: 65%. FTIR (KBr pellet, cm^{-1}): 3462 (m, O-H), 1635 (w, NH), 1098(m, Al-O), 807(s, Si-O) and 479 (s, Ag-O). UV–Vis, λmax (nm)/ε ($M^{-1}cm^{-1}$); 240, 320 and 380. In S_3: Mp 380º C Yield: 77%. FTIR (KBr pellet, cm^{-1}): 3454 (m, O-H), 1635 (w, NH), 1104(m, Al-O), 805(s, Si-O) and 472 (s, Ag-O). UV–Vis, λmax (nm)/ε ($M^{-1}cm^{-1}$); 240, 310 and 380 (Figure 1, 2, 3, 4, 5, 6) (Table 1, 2, 3).

RESULTS AND DISCUSSION

Vibrational spectra of (FT-IR)

FTIR absorption was used in order to check the characteristic bands of the synthesized nano composites. The bonds of these compounds were at 470-800 cm^{-1}. The bands at 3454, 3462 cm^{-1} were related to OH bonds in S_1, S_2 and S_3. The spectrum shows strong IR absorption bands at 806, 473, 807, 479, 805 and 472cm^{-1} which are characteristic of Si-O and Ag-O. The spectrum shows weak IR absorption band at 1635 cm^{-1} which is characteristic of NH in these nano composites.

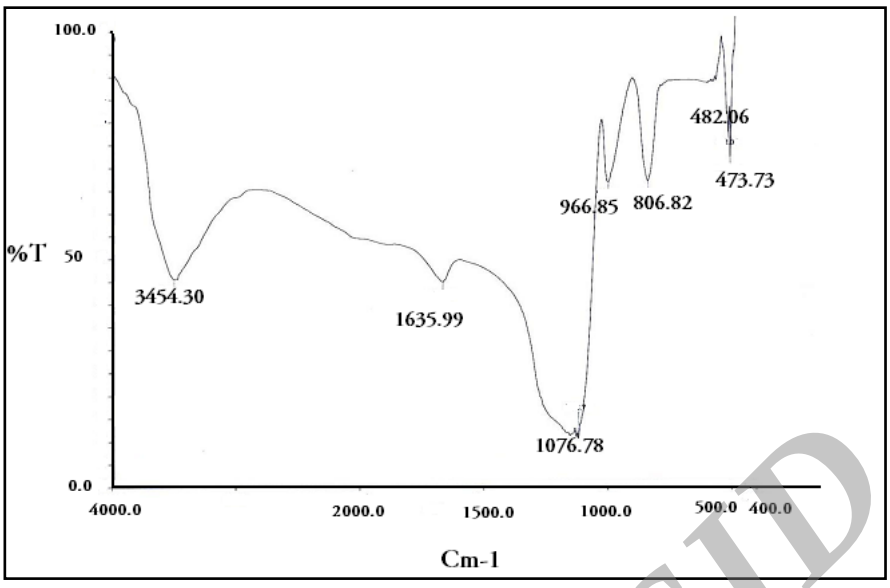

Fig. 1. FT-IR spectra of nano-composite S1 in KBr disk

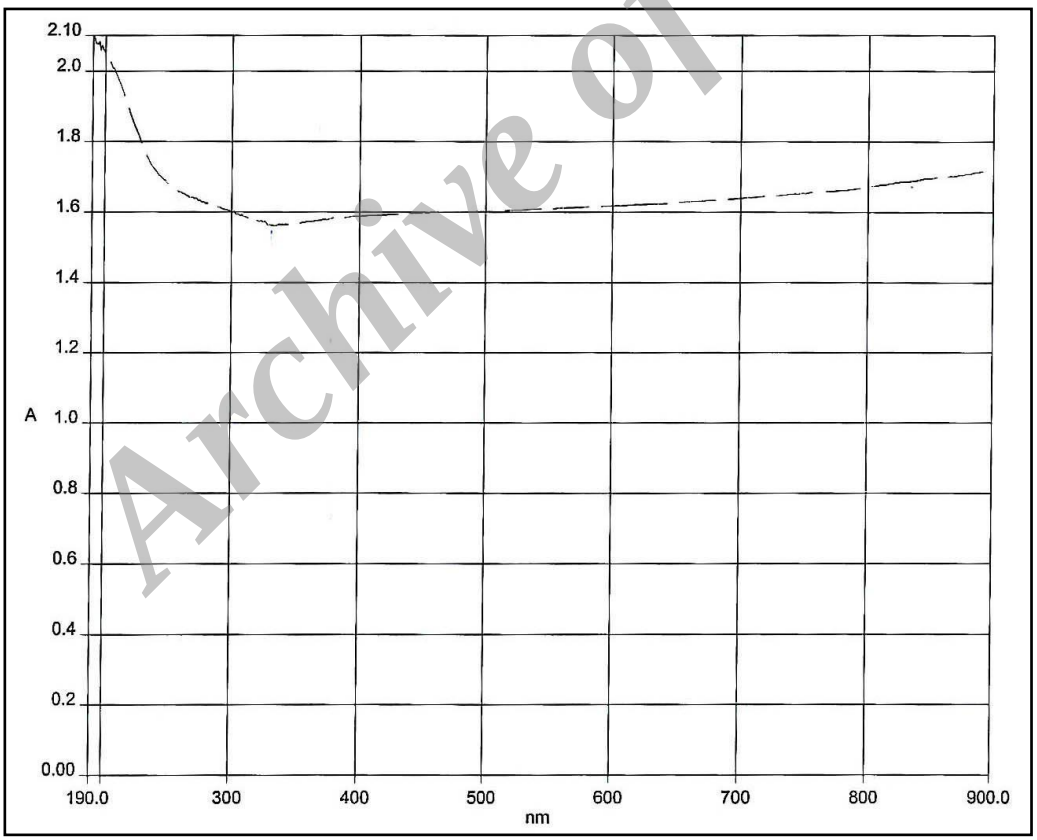

Fig. 2. UV/Vis spectrum of S1 (in DMF, C= 10^{-3}M)

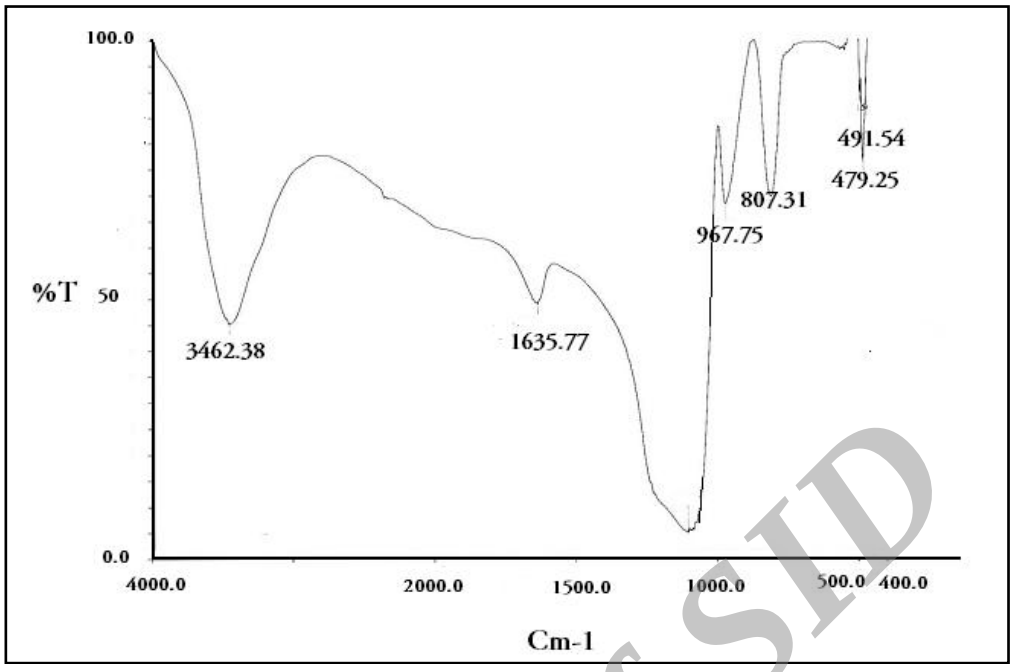

Fig. 3. FT-IR spectra of nano-composite S2 in KBr disk

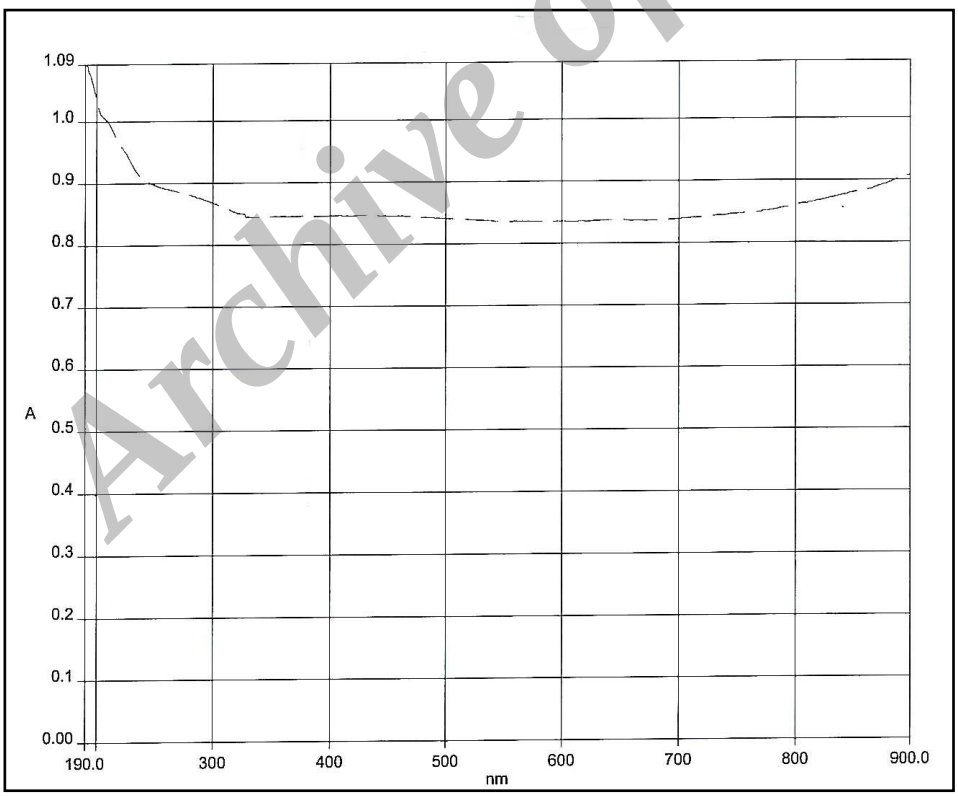

Fig. 4. UV/Vis spectrum of S2 (in DMF, C= 10^{-3}M)

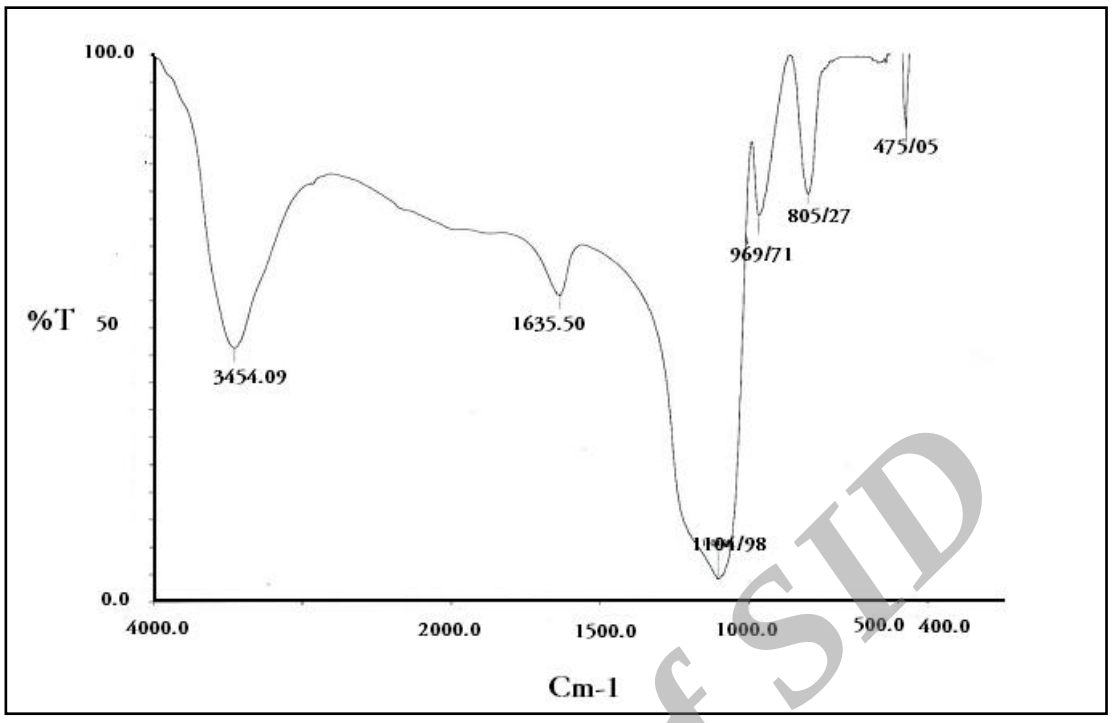

Fig. 5. FT-IR spectra of nano-composite S3 in KBr disk

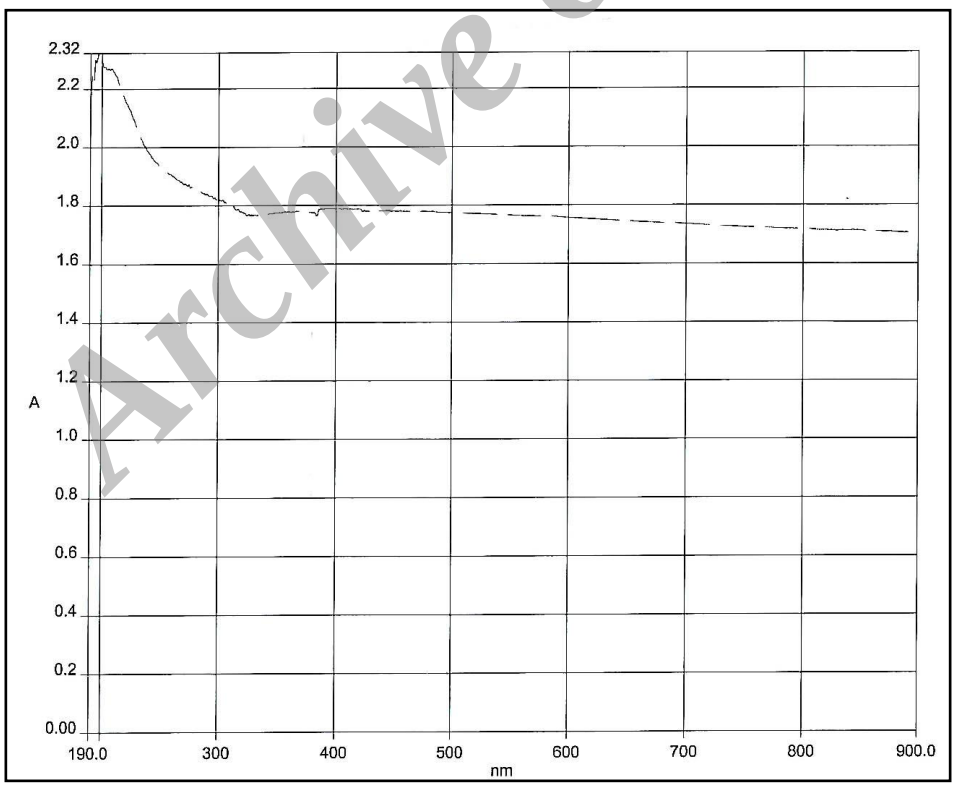

Fig. 6. UV/Vis spectrum of S3 (in DMF, C= 10^{-3}M)

Table 1. Solubility Test of S1

Solubility	Solvent
Insoluble	Methanol
Insoluble	Ethanol
Insoluble	Acetonitrile
Insoluble	Water
Insoluble	n-Hexane
Less soluble	1-Butanol
Insoluble	DMSO

Table 2. Solubility Test of S2

Solubility	Solvent
Insoluble	Methanol
Insoluble	Ethanol
Insoluble	Acetonitrile
Insoluble	Water
Insoluble	n-Hexane
Less soluble	1-Butanol
Insoluble	DMSO

Table 3. Solubility Test of S3

Solubility	Solvent
Less soluble	Methanol
Less soluble	Ethanol
Less soluble	Acetonitrile
Insoluble	Water
Insoluble	n-Hexane
Insoluble	1-Butanol
Insoluble	DMSO

X-ray diffraction analysis (XRD)

The over all shape of the pattern and angular peaks shows the nano properties of a nano composite. The characteristic pattern of components could be found in XRD pattern. XRD spectra using the approximate size of the particles in the nano-composite can be calculated. When particles are smaller than 100 nm XRD peaks are much wider than this factor can be used to estimate the size of the nanoparticles. Scherrer equation used for this purpose:

$$PS(nm) = K.\lambda/\beta.\ Cos\theta$$

In the above equation PS in terms of particle size, K=0.9, B corresponds to the width of the strongest peak at half height in radians, λ=0.154 nm and θ is the angle at which the peak appears. XRD analysis shows the synthesized nano composites.

SEM Pictures

Scanning Electron Microscope image of Ag-SiO_2 nano-composites with different magnification for 5 hours at 185° C has been shown calcination (Figure 7, 8, 9). Scanning electron microscopic observations of Ag-SiO_2 nano-composites shows that morphology of this nano composite as seen in SEM pictures is mixed particles forms such as sand and ceramic. The size of these nano composites is 32-48 nm. As know the morphologies sometimes arise spontaneously as an effect of a templating or directing agent present in the synthesis such as micellar emulsions or anodized alumina pores, or from the innate crystallographic growth patterns of the materials themselves. Amorphous particles usually adopt a spherical shape (due to their microstructural isotropy) – whereas the shape of anisotropic microcrystalline whiskers corresponds to their particular crystal habit. At the small end of the size range, nanoparticles are often referred to as clusters. Spheres, rods, fibers, and cups are just a few of the shapes that have been grown.

Silver is a widely used metal for the deposition of nanoparticles onto SiO_2 substrates. In this study, a novel and simple method was developed for the synthesis of highly nanostructured, Ag-SiO_2 with $LiAlH_4$ reducing method. The results of characterization of the SiO_2 nano composite by FTIR, UV-vis absorption

spectroscopy, XRD, SEM demonstrated that fairly uniform sized nano composite of 32-47 nm diameter with spherical-shaped Anatase form were successfully obtained. In surveys taken from the IR spectra of Si-O bonds were in the range of 470 cm^{-} ^{1}-800 cm^{-1}. Research results indicate that with decreasing particle size to nano-meter amplifier and nano composite construction phase to ensure proper mixing amplifier Moreover, the strength, stiffness and increases flexibility.

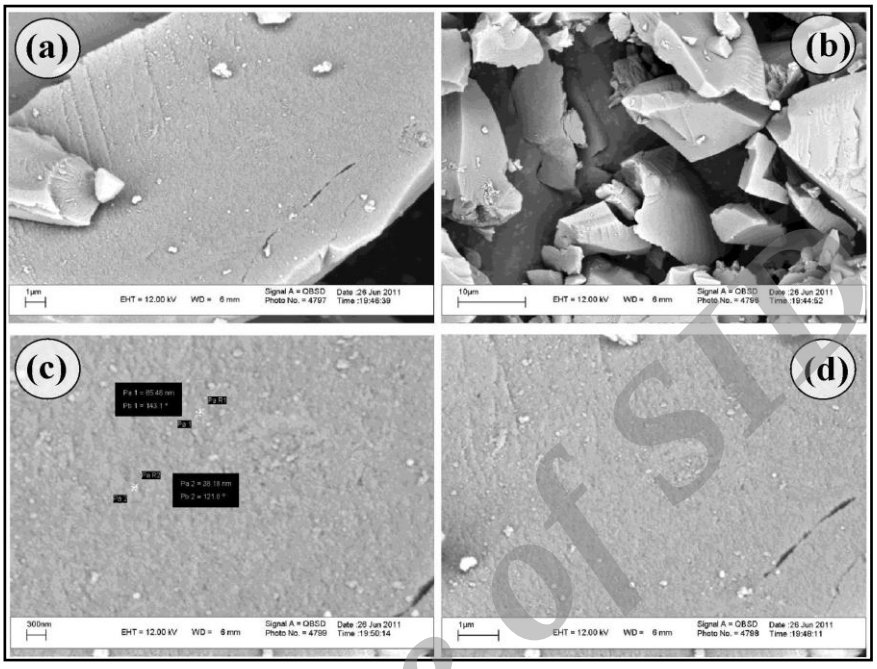

Fig 7. Nano-composite S1 morphology with Magnifictin a) 5000 times, b) 15000 times, c) 30,000 times, d) 50,000times

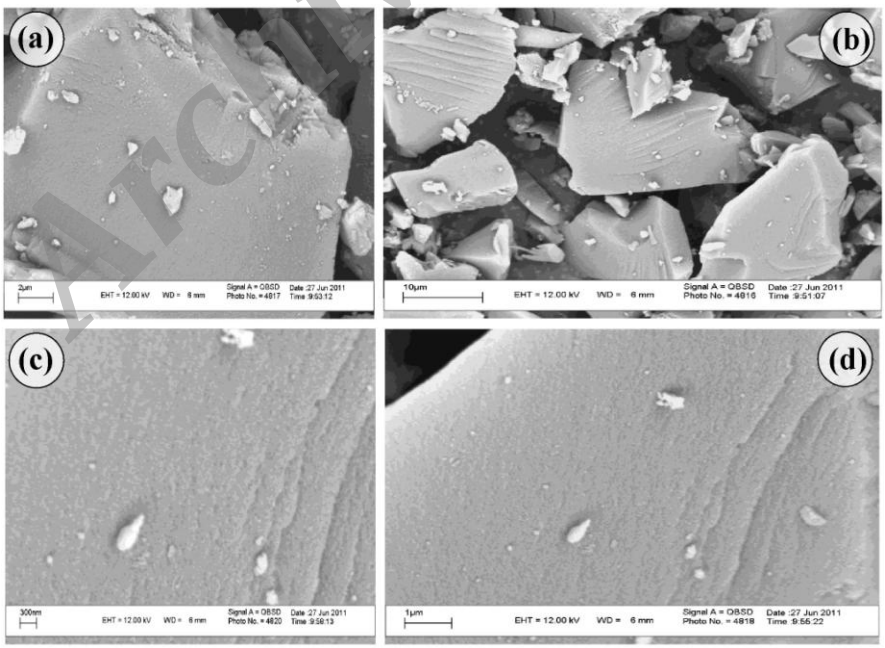

Fig. 8. Nano-composite S2 morphology with Magnification a)5000 times, b) 15,000 times, c) 30,000 times, d) 50,000times, f) 50,000times

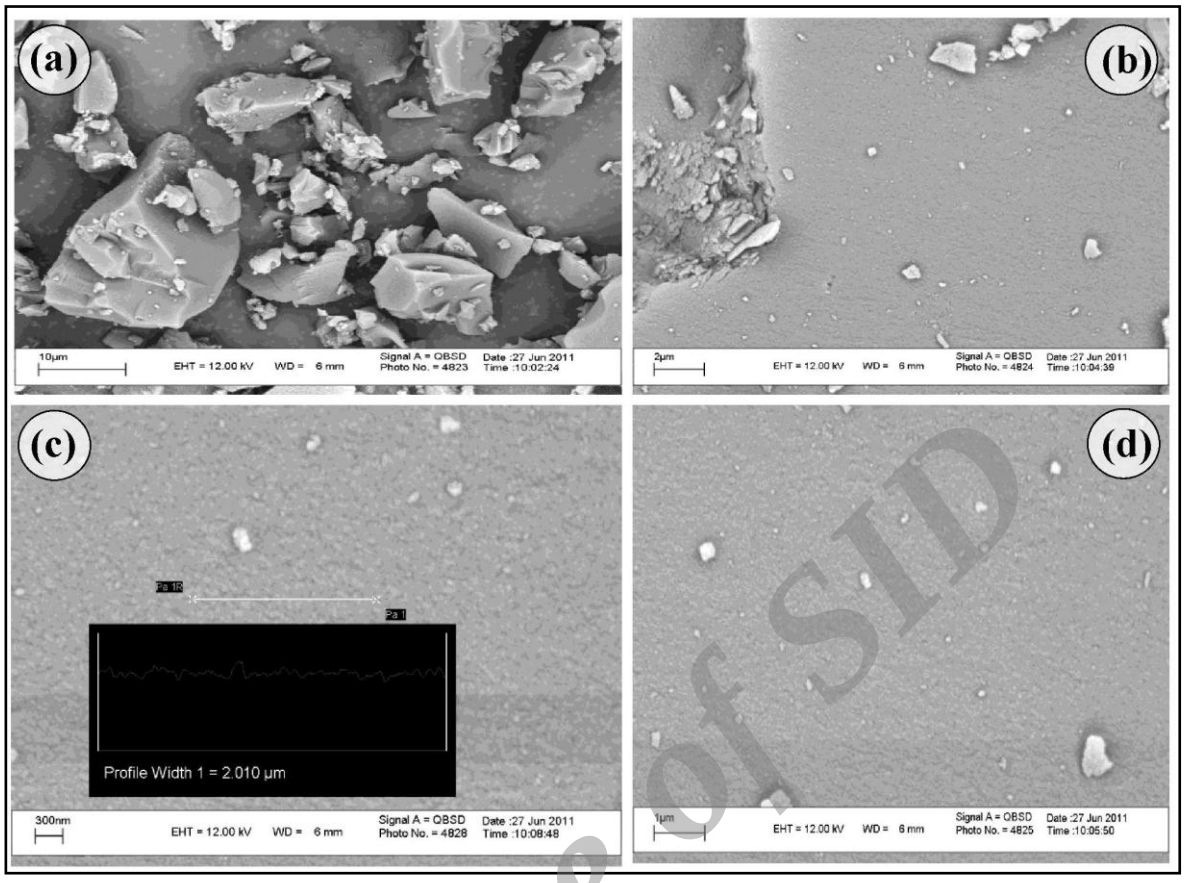

Fig. 9. Nano-composite S2 morphology with Magnification a)5000 times, b) 15,000 times, c) 30,000 times, d) 50,000times

CONCLUSIONS

In summary, the synthesis and characterization of nano composites have been described. We have used $LiAlH_4$ reducing method of $AgNO_3$ to prepare heterogeneous Ag-SiO_2 nano composites from SiO_2 nanoparticles. These compounds was Characterized by FTIR, Uv-Vis, SEM and XRD.

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