TEMPLATE FREE AND POLYMER FREE METAL NANOSPONGE AND A METHOD OF PREPARATION

International Patent Application.
PCT/IN2009/000266

IDENTIFYING OUT-LICENSING PARTNERS

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UNDERSTANDING THE NEED OF THE PRESENT TECHNOLOGY
-problems encountered in the known technologies

• Metal sponges are a class of materials identified for their unique properties such as low density, gas permeability and thermal conductivity. Metal nanosponges have widely shown their potential in applications such as catalysis, fuel cells, membranes, serve as substrate for Surface-Enhanced Raman Spectroscopy (SERS), exhibit antibacterial activity mainly for purification/filtration of water and also used as optical limiter in opticoelectronic devices.

• Significant progress has been made in making and manipulating high surface area metal oxide sponges, but the same is not true for their metallic counterparts.

•The most versatile template based approach, used for the synthesis of porous metal oxides did not give the desired results with the metals and in particular, the noble metals such as Ag, Au, Pt and Pd which are industrially more valuable.

UNDERSTANDING THE NEED OF THE PRESENT TECHNOLOGY
-problems encountered in the known technologies .....contd.

- The known methods suffer from many disadvantages,

->The template removal needs high temperature calcinations which sinter the metallic structure and thereby reduces the surface area drastically.

->The low temperature route, on the other hand uses colloidal crystals templates such as silica or latex spheres which involves multi-step process in addition to the dissolution of templates in organic solvents or HF.

->The Pattern-forming instabilities during selective dissolution of silver from Ag-Au alloys are reported to give nanoporous gold with controlled multi-modal pore size distribution.

THE PRESENT TECHNOLOGY AND THE ADVANTAGES

- An instantaneous formation of high surface area noble metal sponges through a template free, polymer free, one-step, inexpensive, method.

- The process generates a three dimensional porous structure made up of nanowire networks having porous low density, high surface area, high gas permeability and thermal conductivity.

- This process involves a simple, room temperature reduction of metal salts with sodium borohydride.

- It can be scalable to any amount (Industrially Scalable Process)

- The technology is developed by a prestigious Research Institute Jawaharlal Nehru Centre for Advanced Scientific Research (JNCASR)

AN OUTLINE OF THE PROCESS

- Mixing equimolar concentration of one part of metal precursor (such as Ag, Au, Pt or Pd) and five parts of reducing agent (such as Sodium Borohydride) solution;

- This results in spontaneous formation of effervescence and formation of nano sized ligament metallic networks which aggregate to form a black spongy solid floating on the reaction medium;

- Filtering and washing the spongy solid followed by drying at room temperature to obtain the metal nanosponge.

- The whole reaction process is completed within time duration of 5 minutes.

IMPORTANT USES OF THE PRESENT METAL NANOSPONGES IN INDUSTRY

- The template free and polymer free metal nanosponge can be used as substrates for **Surface-enhanced Raman Spectroscopy (SERS)**
- The template free and polymer free metal nanosponge can be used for **anti-bacterial activity**.

IP/ PATENT STATUS

- International PCT Application PCT/IN2009/000266
- United States Patent Publication US 20110014300 (A1)
- Japanese Patent Publication JP2011523677 (A)
- European Patent Publication EP2276691 (A2)
- Indian Patent Publication 1105/CHE/2008

STABILITY STUDIES

- The template free and polymer free metal nanosponges as developed by the present inventors are very stable at room temperature for months and also to temperature upto 200 °C, as tested for silver nanosponge.
  - Evidence provided in the form of FESEM images at 200, 300 and 500 degrees Celsius. (Figures 1 and 2)

- The said metal nanosponges can withstand pressure upto 10 kiloNewton
  - Evidence provided in the form of FESEM images and surface area data to conform the same. (Figures 3 and 4)

- The said metal nanosponges are inert to moisture and to common organic solvents
  - Tested in laboratory conditions

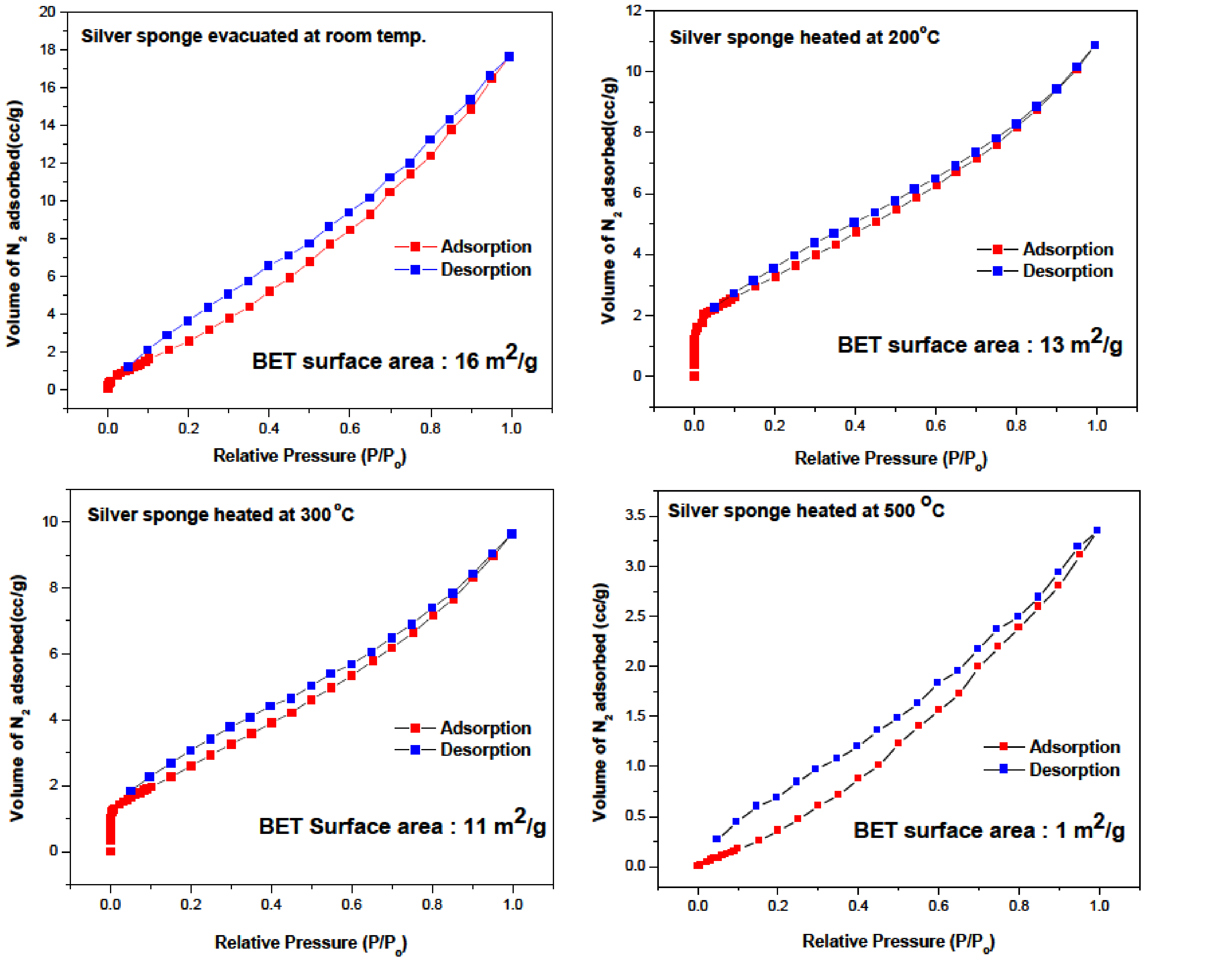

Figure 1: Nitrogen adsorption/desorption isotherms (at 77 K) for silver sponge treated at different temperatures

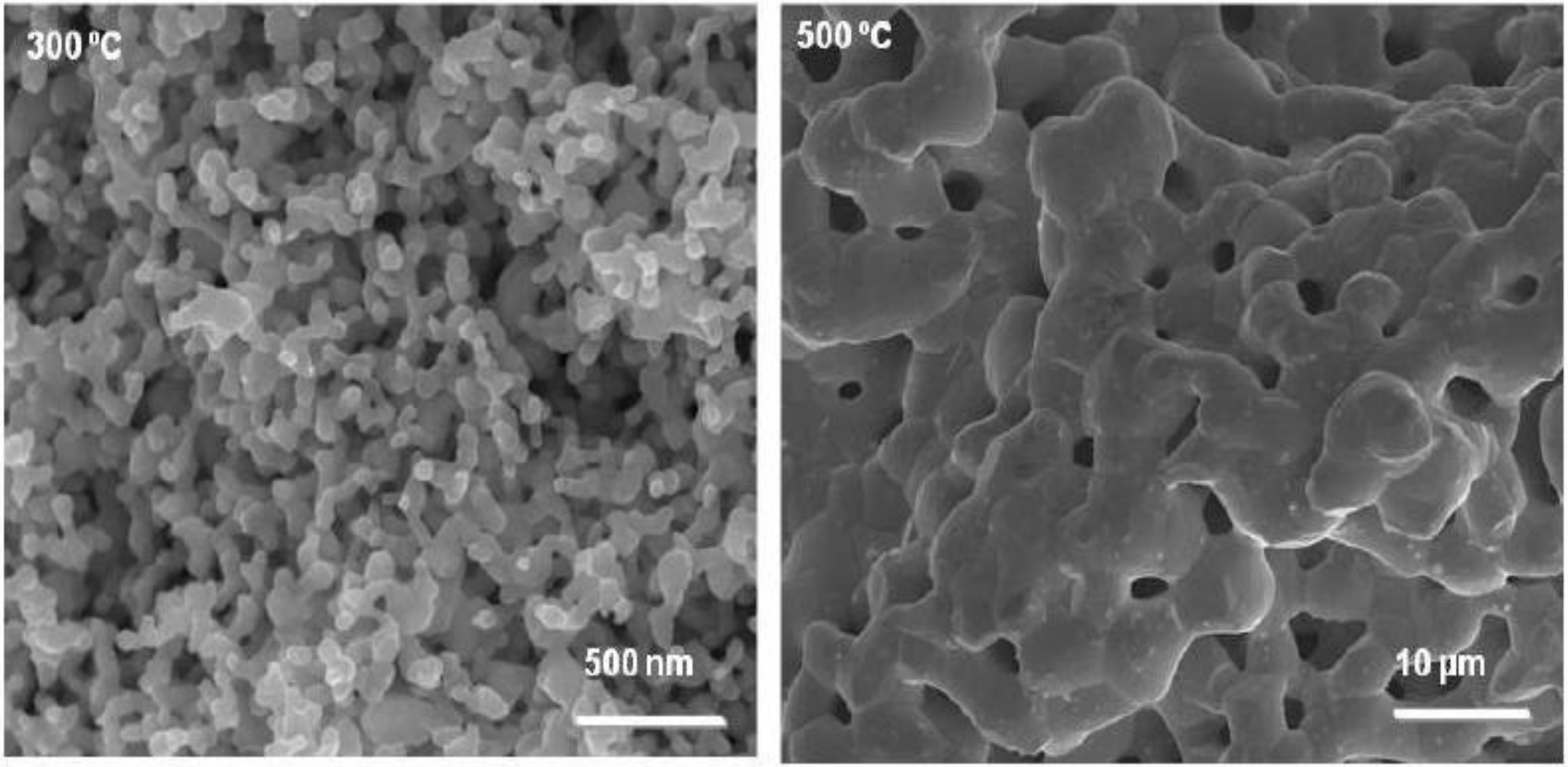

Figure 2: FESEM images of the silver sponge treated in vacuum at 300 °C and 500 °C for 3 h

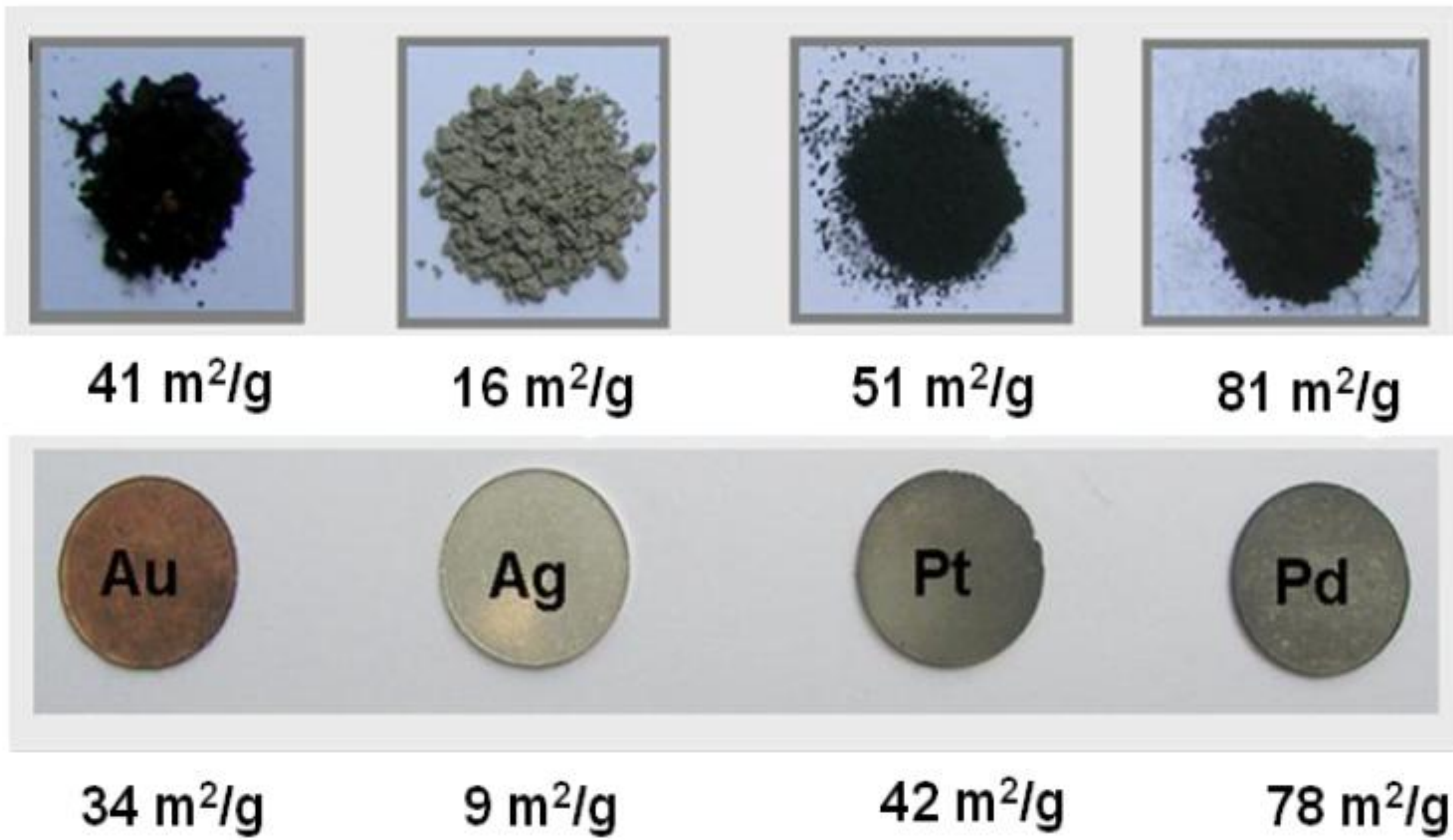

Figure 3: Photographs of the sponges and sponge pellets with their respective surface areas (Pellets are made by applying a pressure of 10 kN)

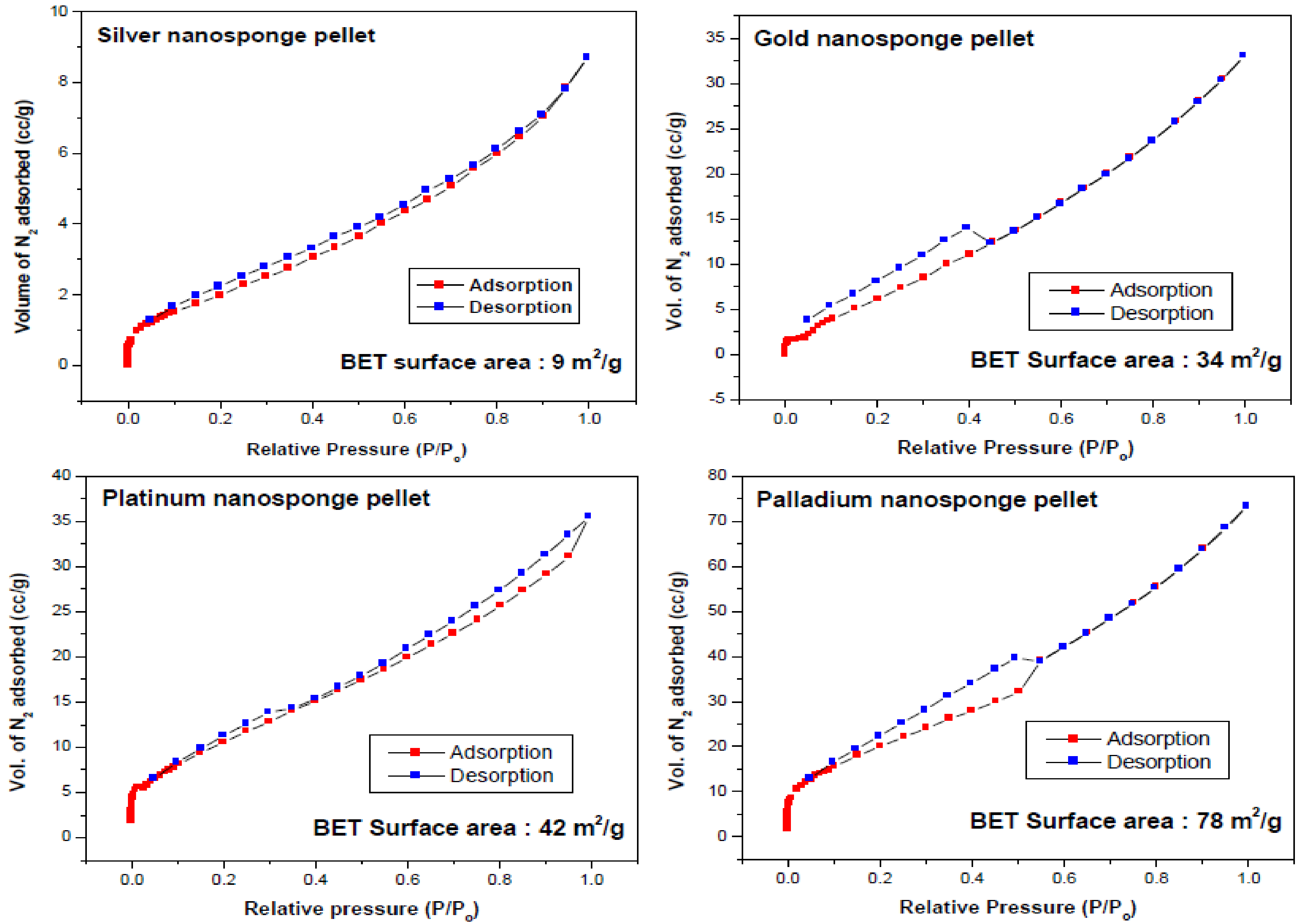

Figure 4: Nitrogen adsorption/desorption isotherms (at 77 K) for the sponge pellets pressed by applying a pressure of 10 kN

PARTICLE SIZE AND SURFACE AREA

- The experimental evidence for particle size and surface area is provided in TEM, FESEM and Adsorption data (Figures 5 to 9)

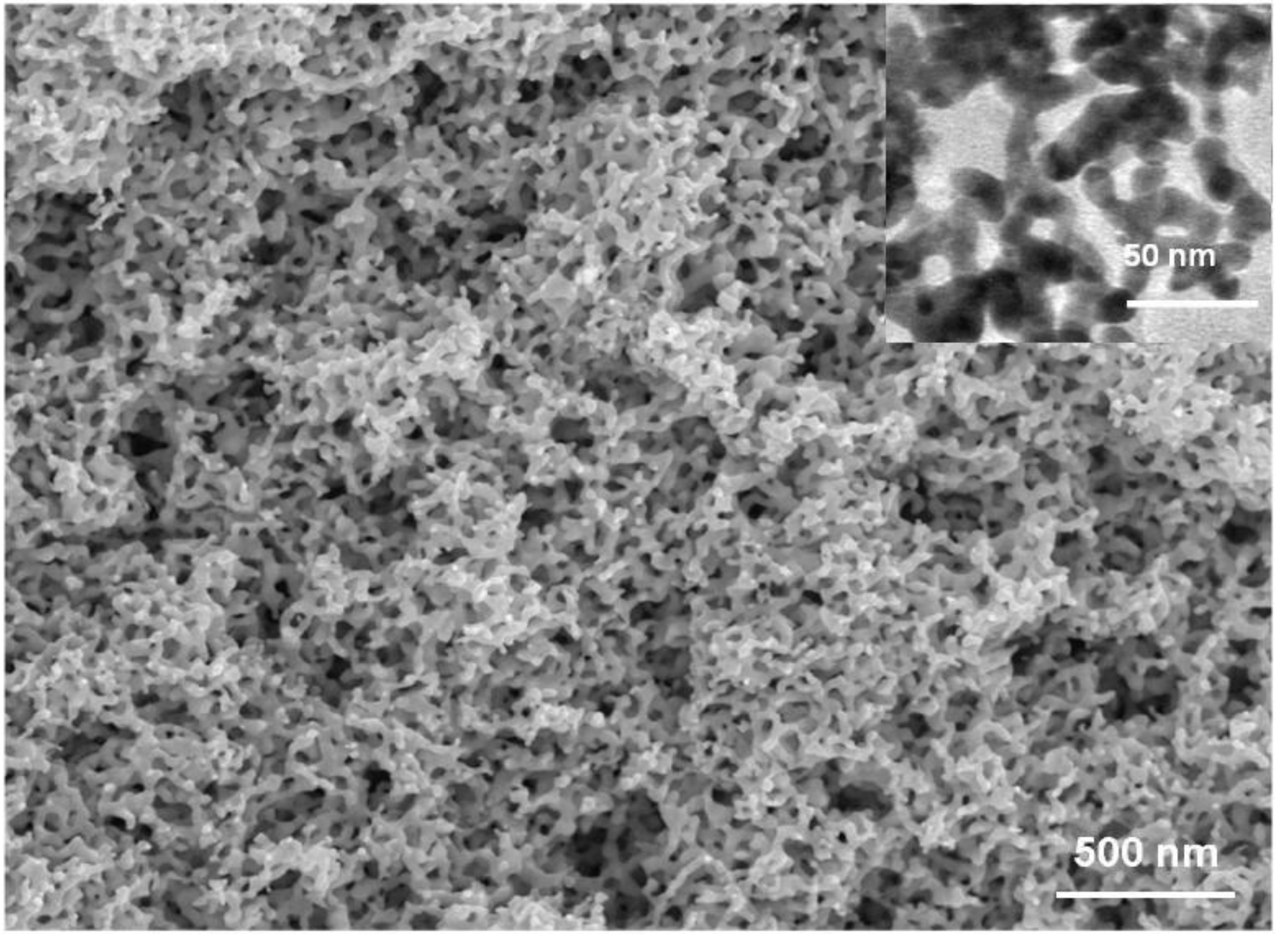

Figure 5: Higher magnification FESEM image of the gold nanosponge showing porous interconnected networks with ligaments of size 20 – 50 nm. Inset shows the TEM image of the gold nanosponge

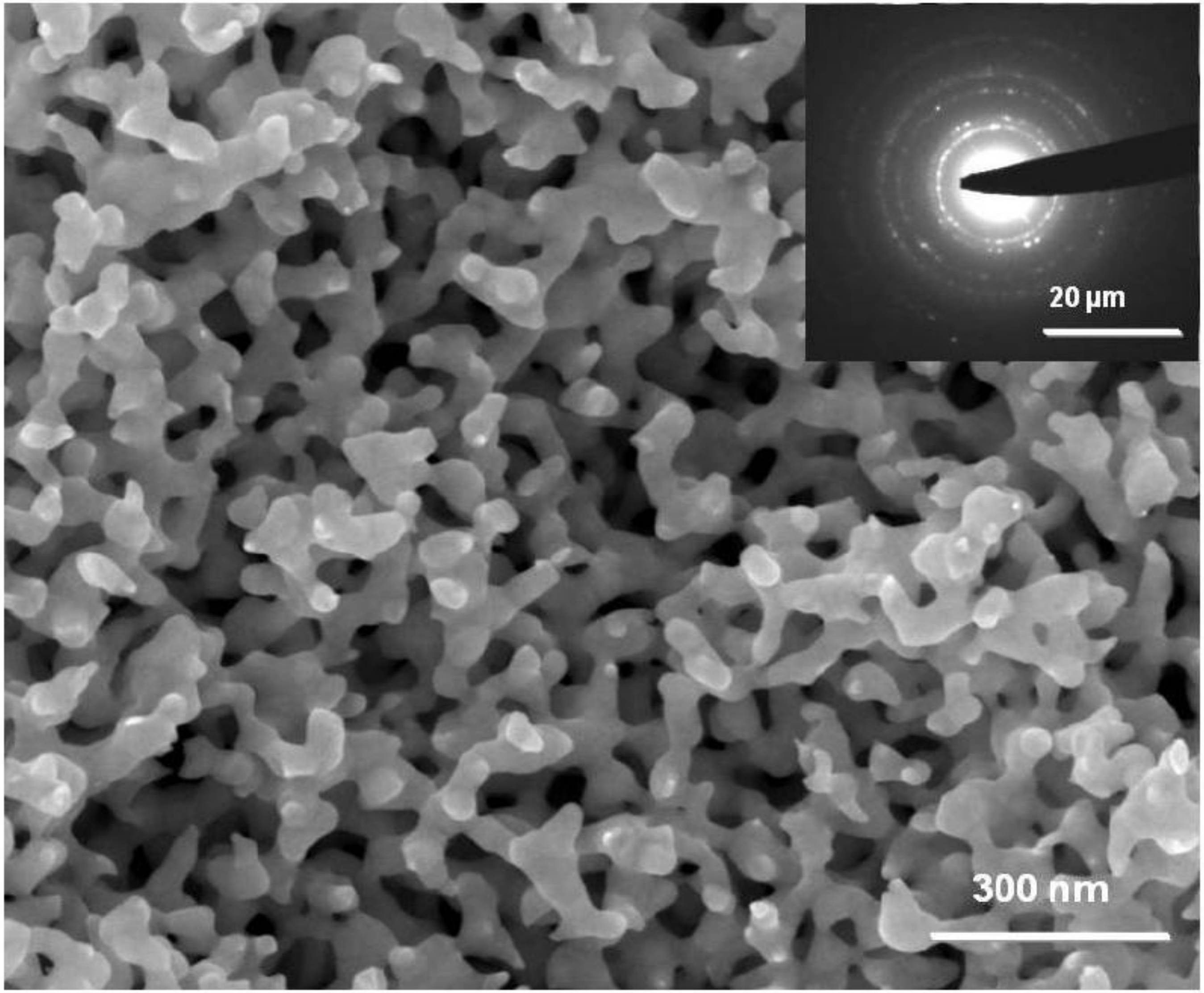

Figure 6: FESEM image showing gold nanostructure with highly interconnected ligaments of size 20 – 50 nm. Inset shows the electron diffraction (ED) pattern with polycrystalline nature

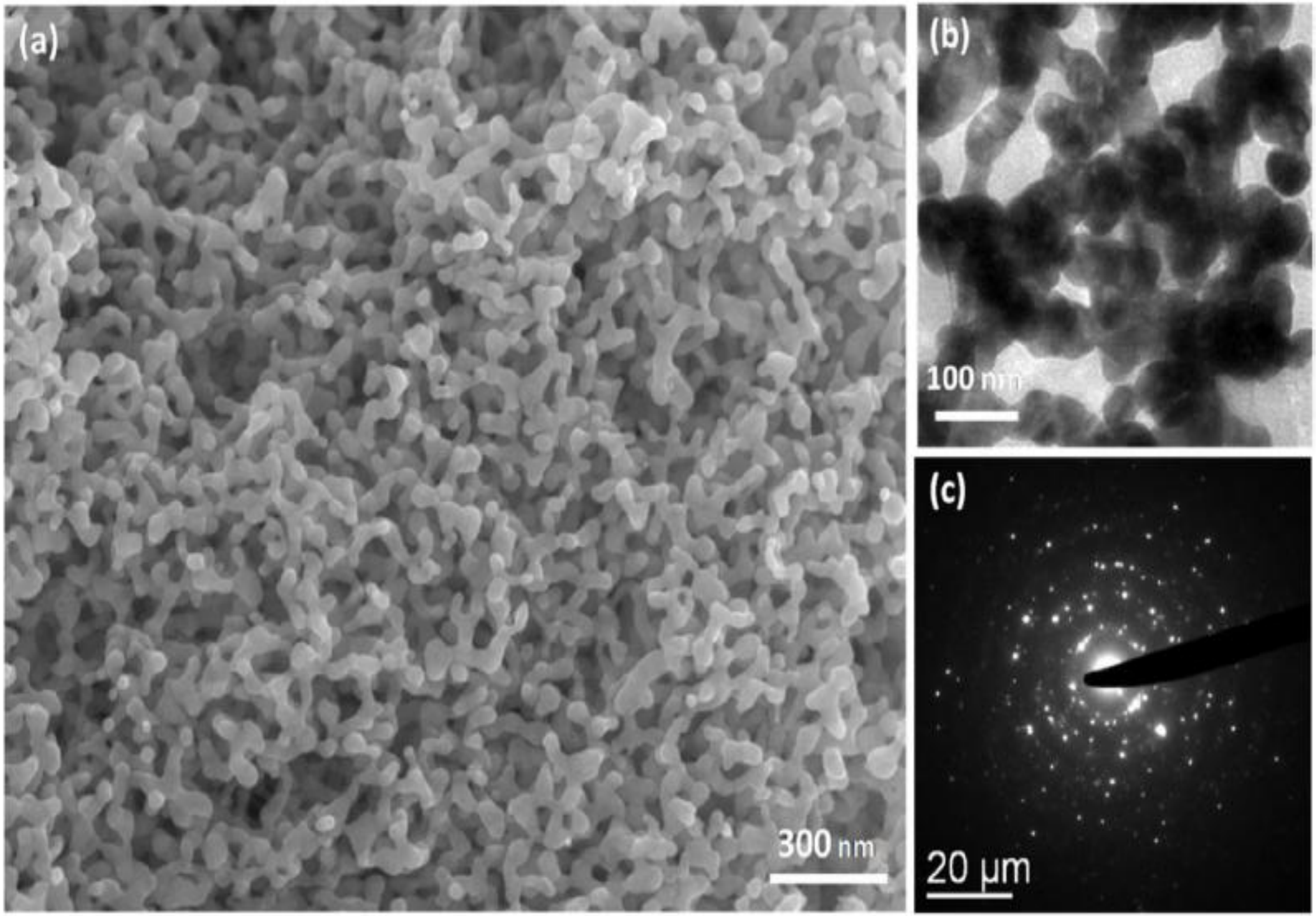

Figure 7: (a) FESEM image of silver nanosponge showing the highly interconnected ligaments. (b) TEM image of the silver sponge showing network of ligaments of size 50 – 60 nm (c) Electron diffraction pattern showing the polycrystallinity of the silver sponge

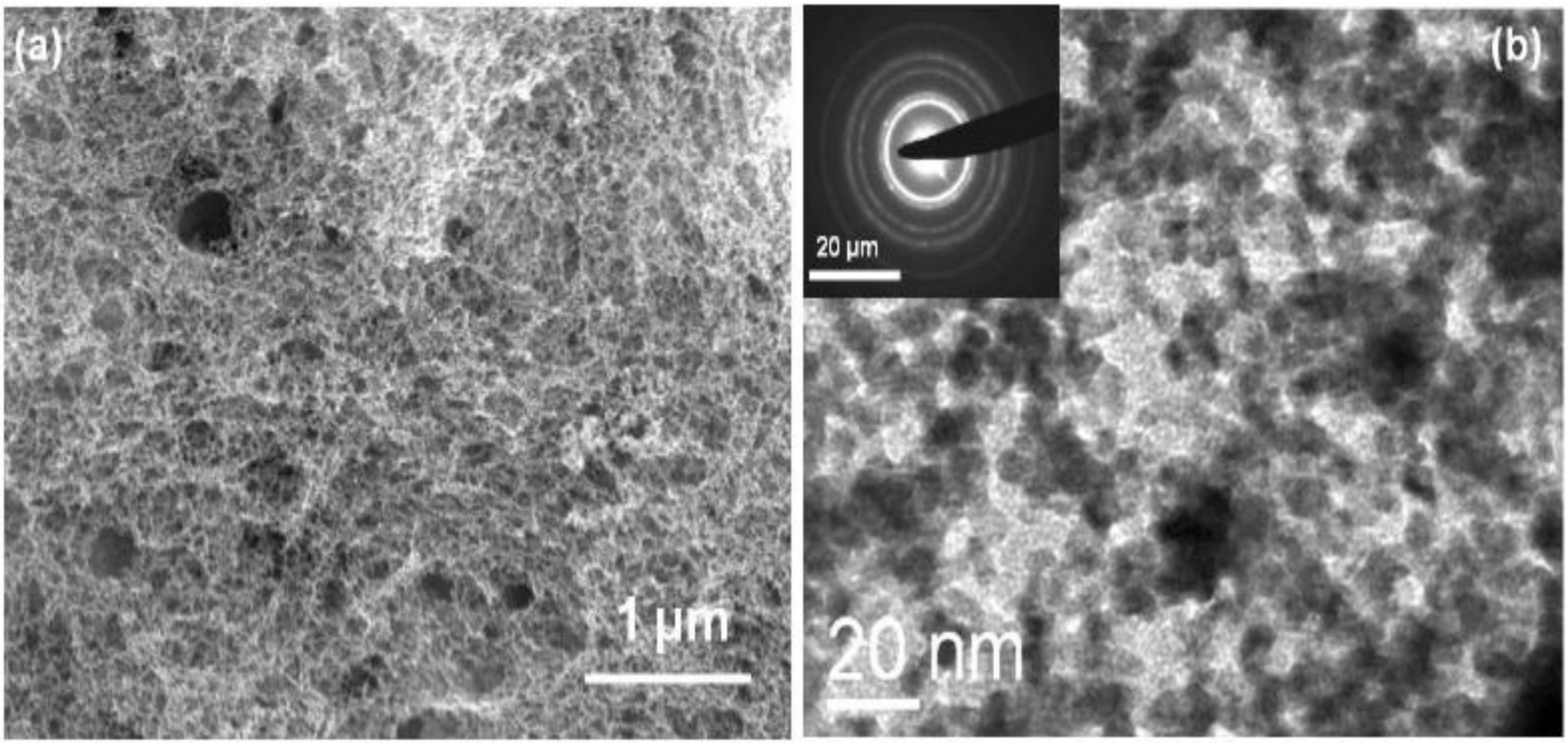

Figure 8: (a) Low-magnification FESEM image of palladium nanosponge (b) TEM image showing palladium nanoparticles of 5 – 10 nm size fused to form a network structure. Inset shows the ED pattern

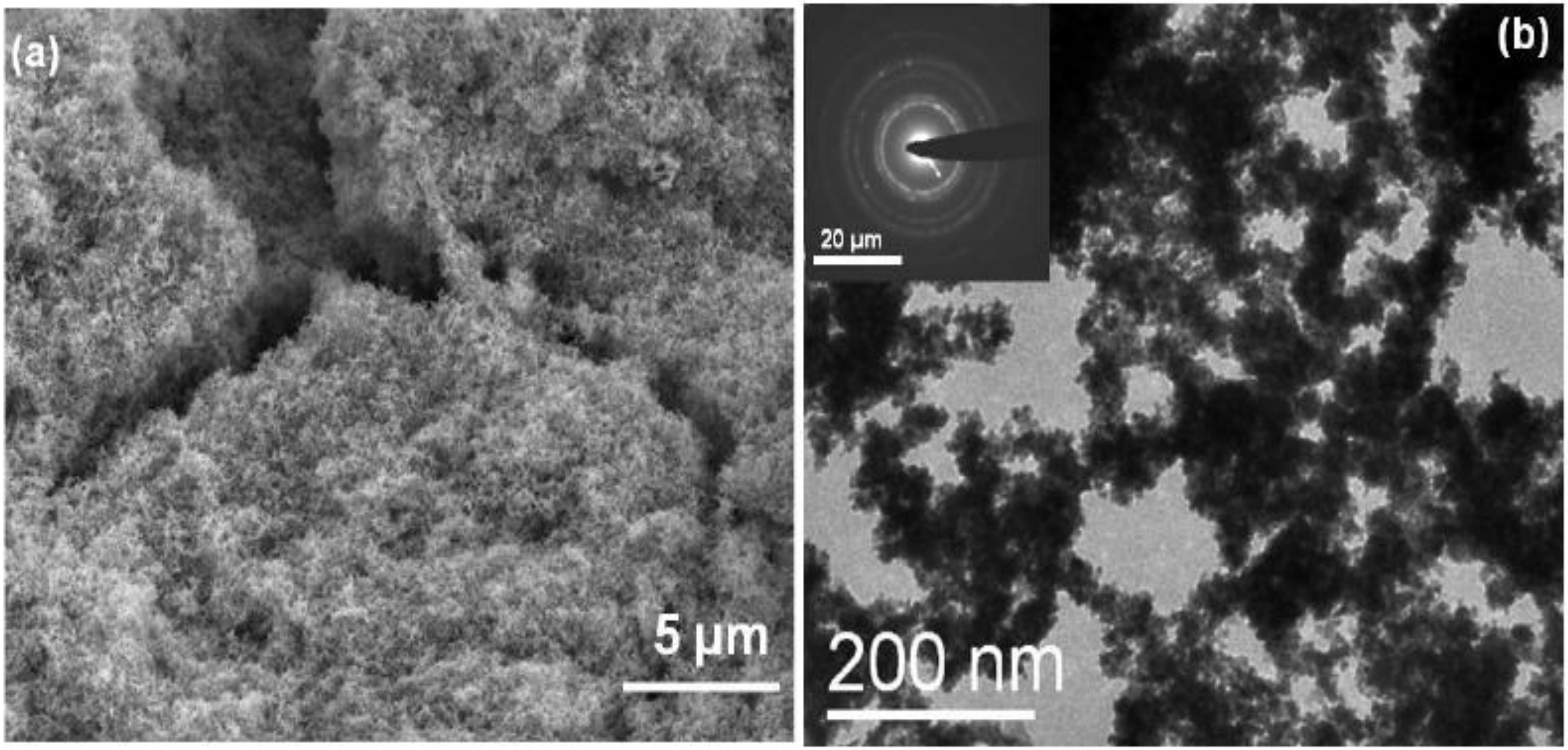

Figure 9: (a) Low-magnification FESEM image showing the platinum nanosponge (b) TEM image showing the platinum networks made up of nanoparticles of size 5 – 10 nm. Inset shows its ED pattern

APPLICATIONS IN THE INDUSTRY

• The template free and polymer free metal nanosponge are useful as substrates for **Surface-enhanced Raman Spectroscopy (SERS)** and for **Antibacterial Activity.** (Figures 10 to 13)

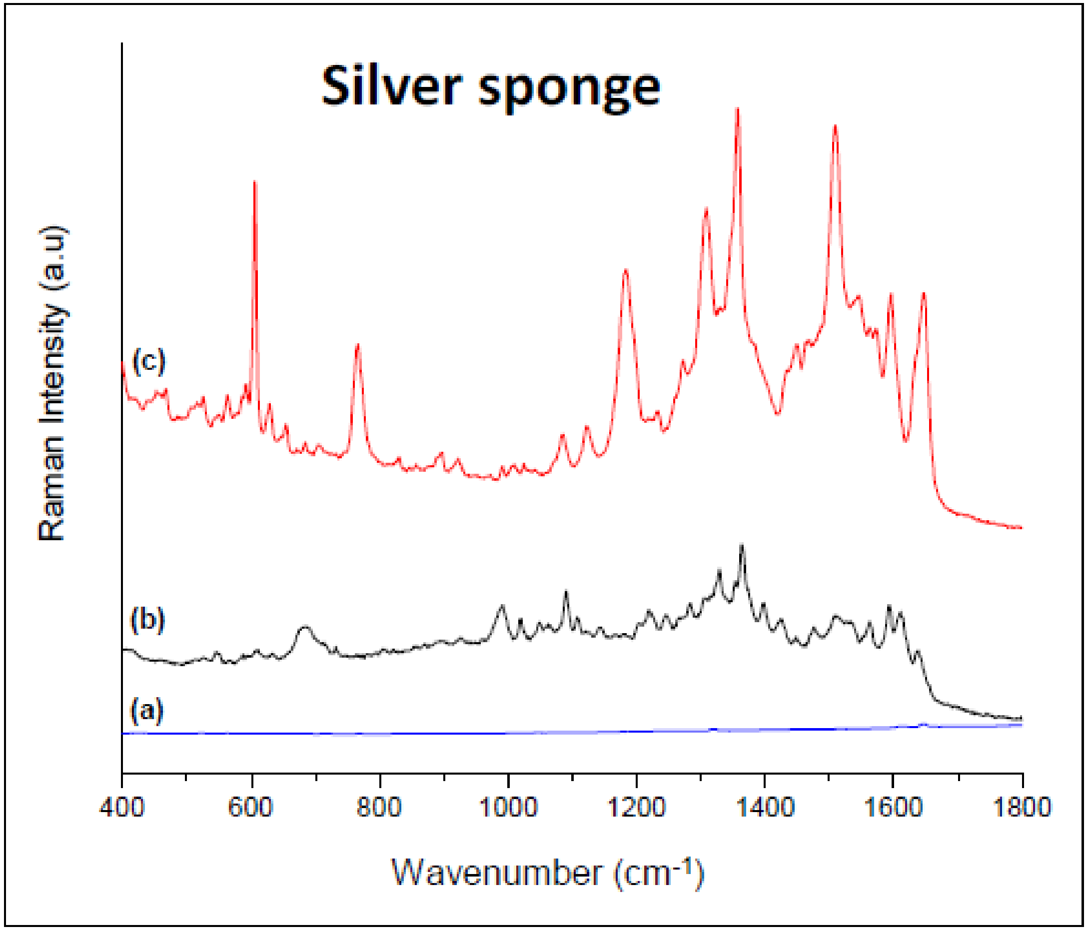

Figure 10: Surface-enhanced Raman spectra (SERS) of (a) Rhodamine 6G (10-4M), (b) Rhodamine 6G (10-6M) with Silver nanosponge and (c) Rhodamine 6G (10-4M) on silver nanosponge

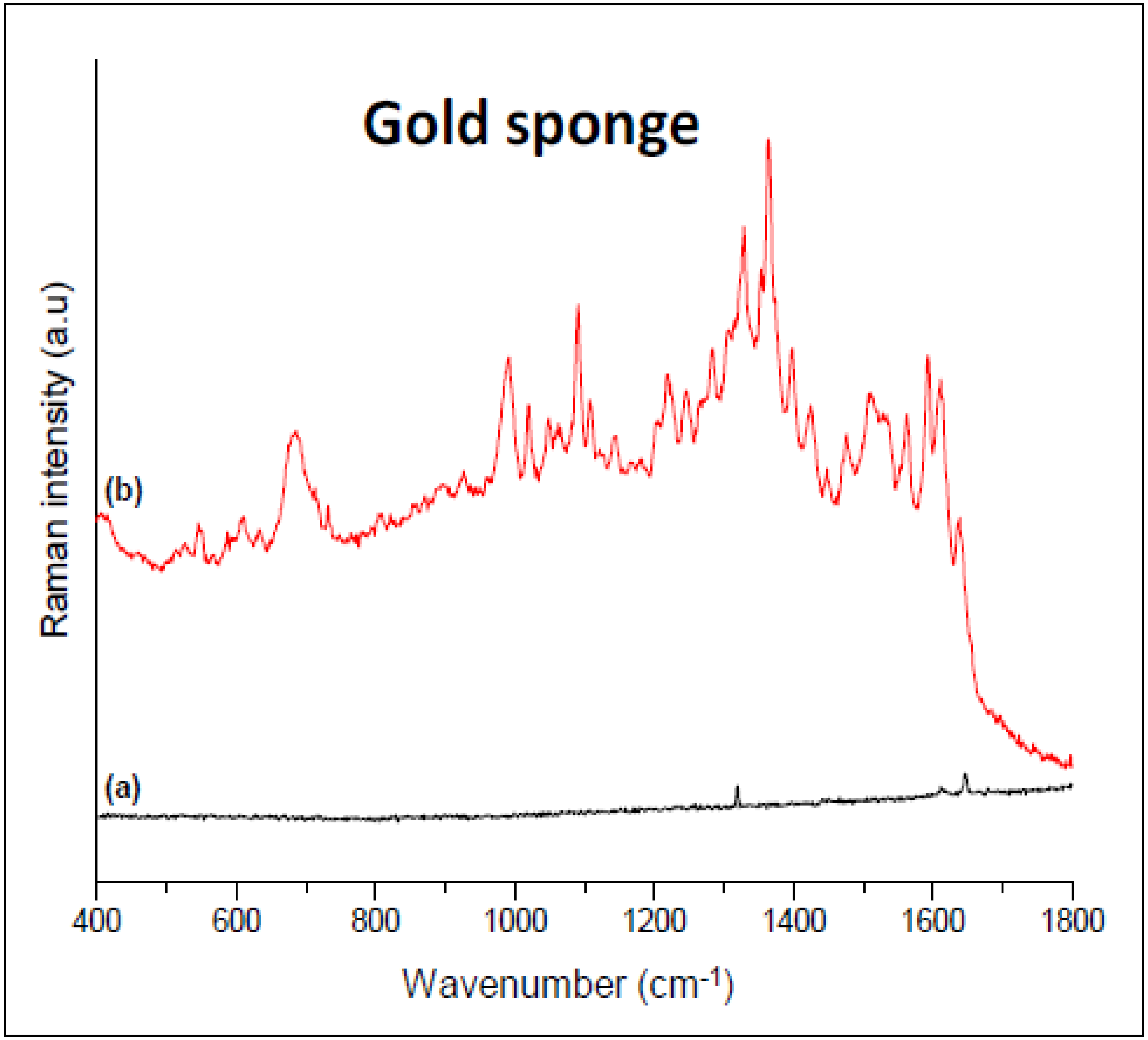

Figure 11: Surface-enhanced Raman spectra (SERS) of (a) Rhodamine 6G(10-4 M) and (b) Rhodamine10-6 M) on gold nanosponge

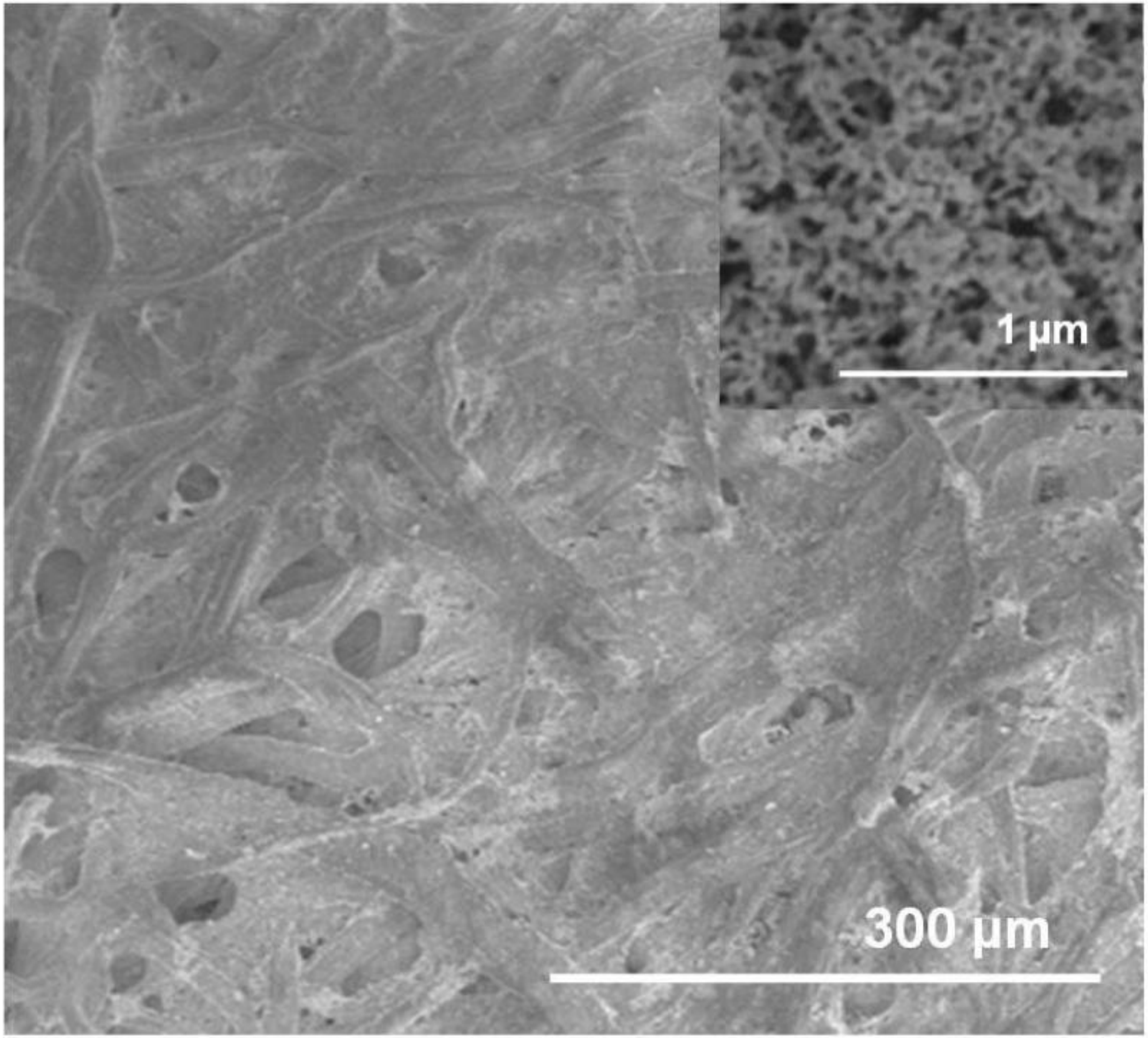

Figure 12: FESEM image of the silver nanosponge- Whatman filter paper composite. Inset shows the high magnification image of the silver nanosponge deposited on the paper

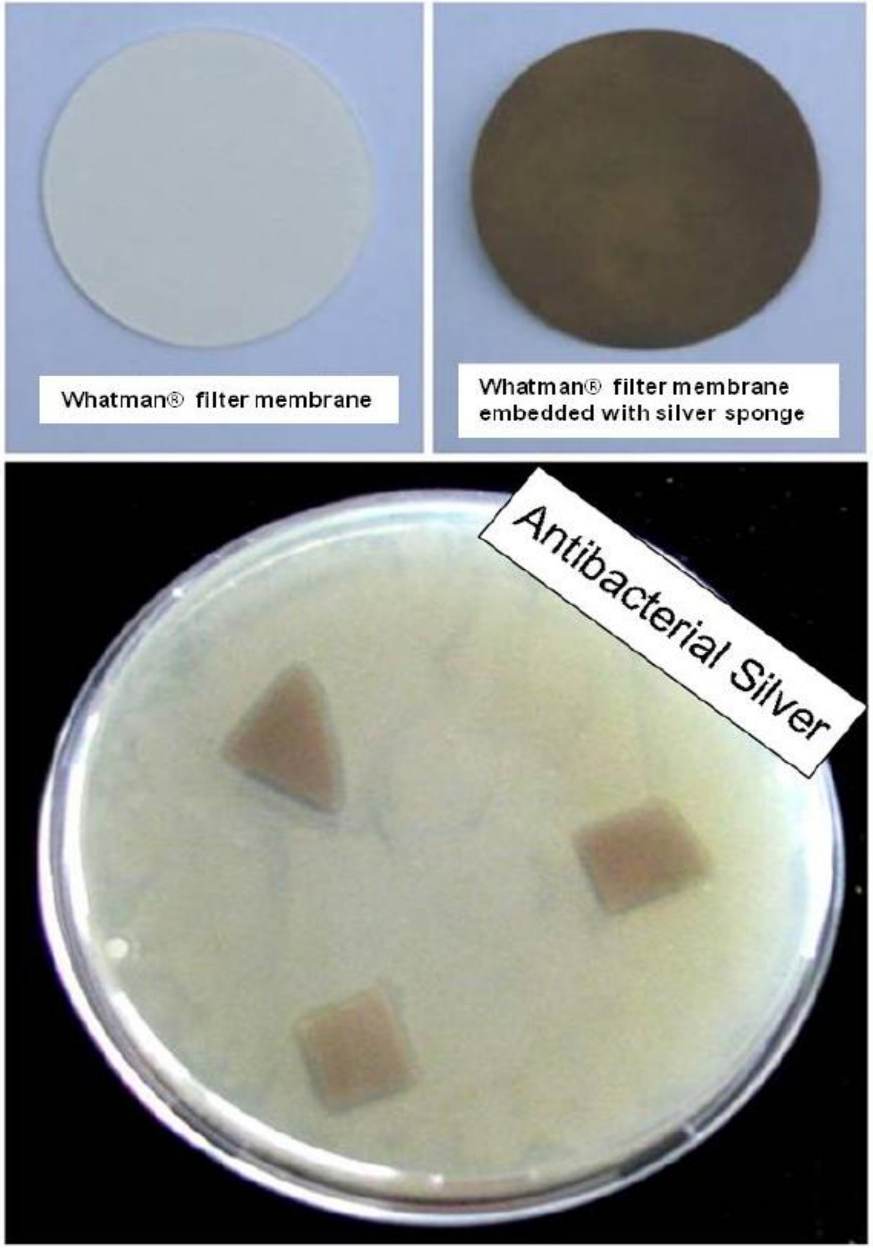

Figure 13: Photographs showing (a) Whatman filter membrane and (b) composite membrane embedded with silver nanosponge (c) Antibacterial activity of the silver nanosponge- Whatman filter membrane composite against E. coli bacteria. The inhibition zone against the bacterial growth is visible around the paper strips

EXPECTATIONS:

- Applicant seeks to Out-License the Patent Rights on Exclusive or Non-Exclusive Terms.

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