Uninterrupted galvanic reaction for scalable & rapid Synthesis of metallic and bimetallic sponges/dendrites for efficient catalyst for 4-nitrophenol reduction

Barun Kumar Barman and Karuna Kar Nanda*

Materials Research Centre, Indian Institute of Science, Bangalore-560012, India

The yield calculation:

Cu:

Mol. Wt. of $CuSO_4$, $5H_2O$ = 249.70, Mol. Wt. of Cu = 63.546

1000 ml of 1M CuSO4, $5H_2O$ solution ≡ 63.546 gm. Cu

2.5 ml of 0.2M $CuSO_4$, $5H_2O$ solution ≡ 0.0317 gm. Cu

Experimentally, when we immersed ~26 mg (3cm) of Mg ribbon in 2.5 ml of 0.2M $CuSO_4$, $5H_2O$ solution + 0.5 ml of 5M H_2SO_4 solution, we get ~26.4 mg of Cu.

The yield of Cu ≡ 83 %

For synthesis of 1 gm. of Cu spongy structures, accordingly, we increased the corresponding precursor. For this case, we used 100 ml of 0.2 M of $CuSO_4$, $5H_2O$ solution + 20 ml of 5M H_2SO_4 solution along with 1.4 gm. of Mg ribbon. Then we get around 0.96 gm. of Cu sponge. So, we can really predict the synthesized product.

Ag:

Mol. Wt. of AgNO3 = 169.87 Mol. Wt. of Ag =107.86

1000 ml of 1 M $AgNO_3$ solution ≡ 107.86

2.5 ml of 0.05 M AgNO3 solution ≡ 0.0135 gm. of Ag

Experimentally, when we immersed ~26 mg (3cm) of Mg ribbon in 2.5 ml of 0.05 $AgNO_3$ solution + 0.5 ml of 5M H_2SO_4 solution, we get ~10.93 mg of Ag.

The yield of Ag ≡ 81 %

Sn:

Mol. Wt. of $SnCl_2$, $2H_2O$ = 225.64 Mol. Wt. of Sn=118.17

1000 ml of 1M $SnCl_2$, $2H_2O$ solution ≡ 118.71 gm. of Sn

2.5 ml of 0.2 M $SnCl_2$, $2H_2O$ solution ≡ 0.059 gm. of Sn

Experimentally, when we immersed ~26 mg (3cm) of Mg ribbon in 2.5 ml of 2.5 ml of 0.2 M $SnCl_2$, $2H_2O$ solution + 0.5 ml of 5M H2SO4 solution, we get ~ 47 mg of Sn.

The yield of Sn≡ 80%

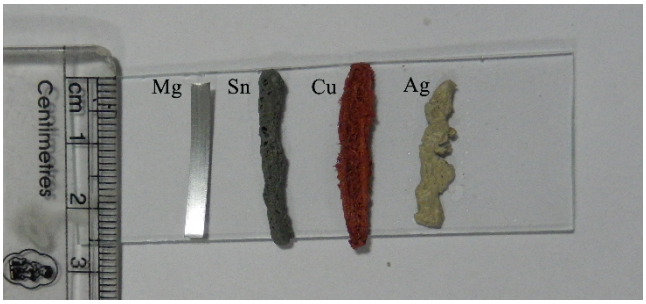

Fig1. Photograph of ~2.5 cm of Mg ribbon and Sn, Cu and Ag sponge structures after complete etching of the Mg ribbon.

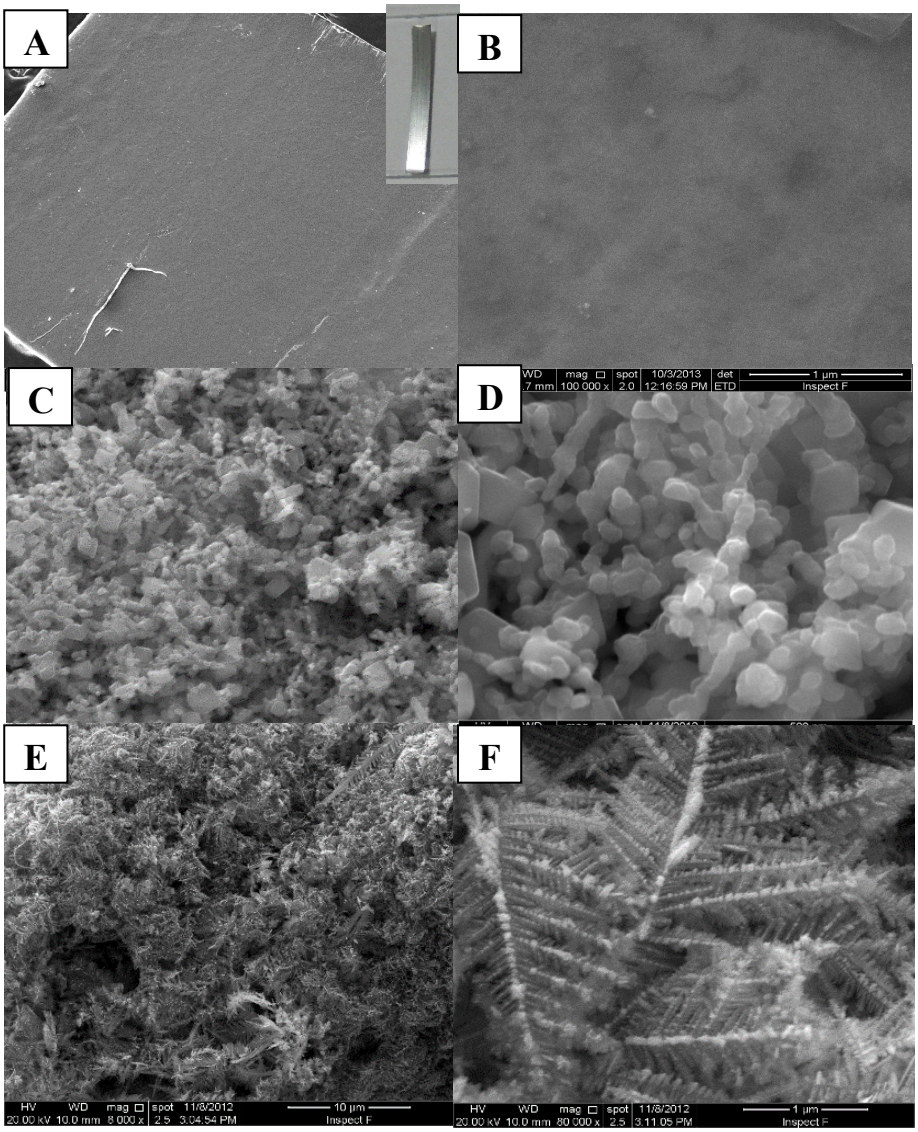

Fig.2 SEM images of Mg ribbon (A & B) and Cu (C &D) and Ag (E & F) nanostructures grown on the Mg ribbon (inset) in absence of acid.

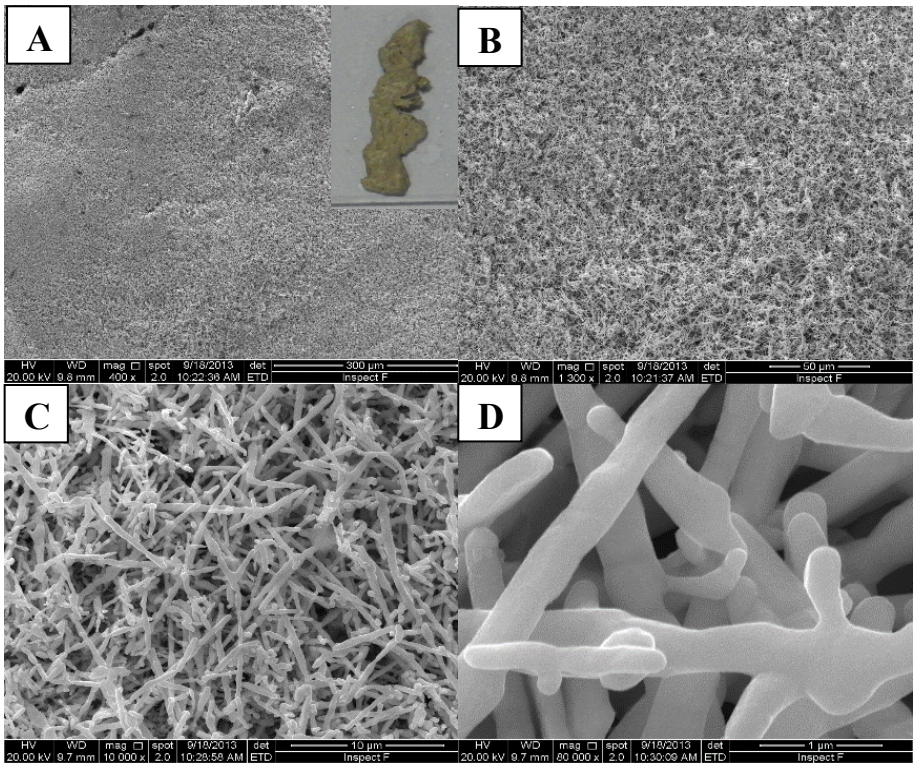

Fig.3 (A-D) SEM images of different magnification of spongy Ag nanostructures obtained with 2.5 ml of 0.05 M $AgNO_3$ and 0.6 ml of 5M H_2SO_4.

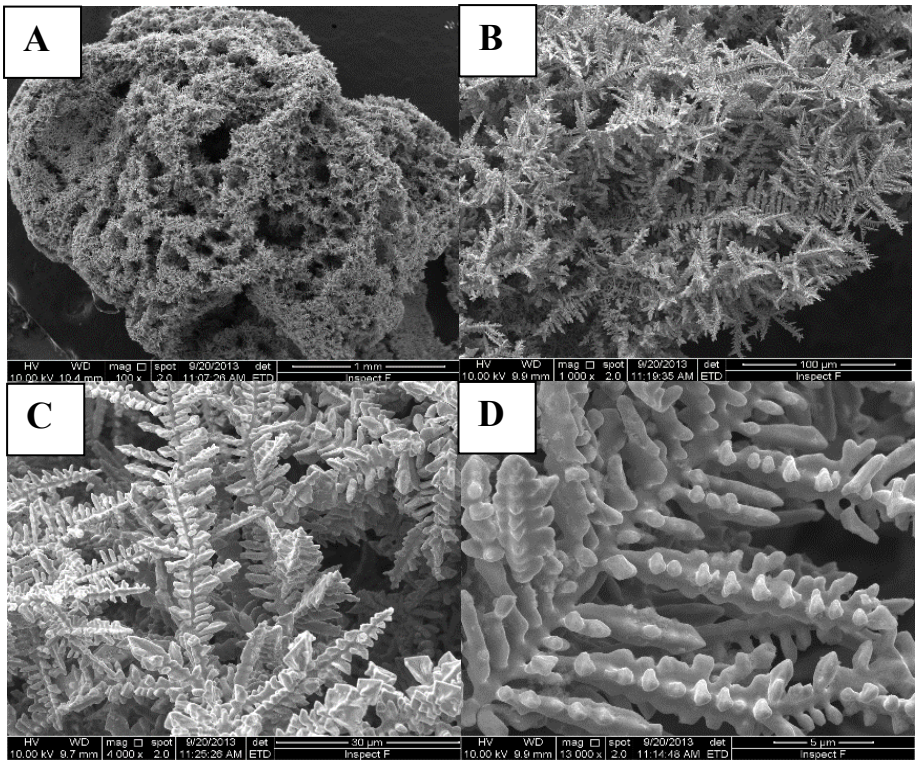

Fig.4 SEM images of spongy Sn obtained with 2.5 ml of 0.1M $SnCl_2$, $2H_2O$ and 0.6 ml of 5 M H_2SO_4.

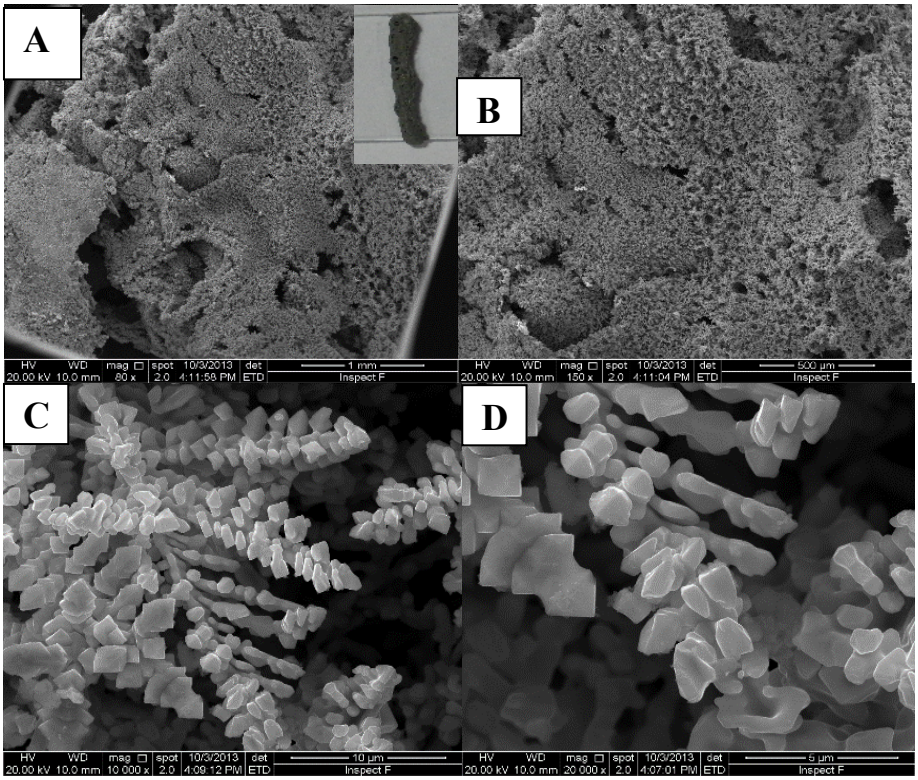

Fig.5 SEM images of spongy Sn obtained with 2.5 ml of 0.2 M $SnCl_2$, $2H_2O$ and 0.6 ml of 5M H_2SO_4.

Cu sponge:

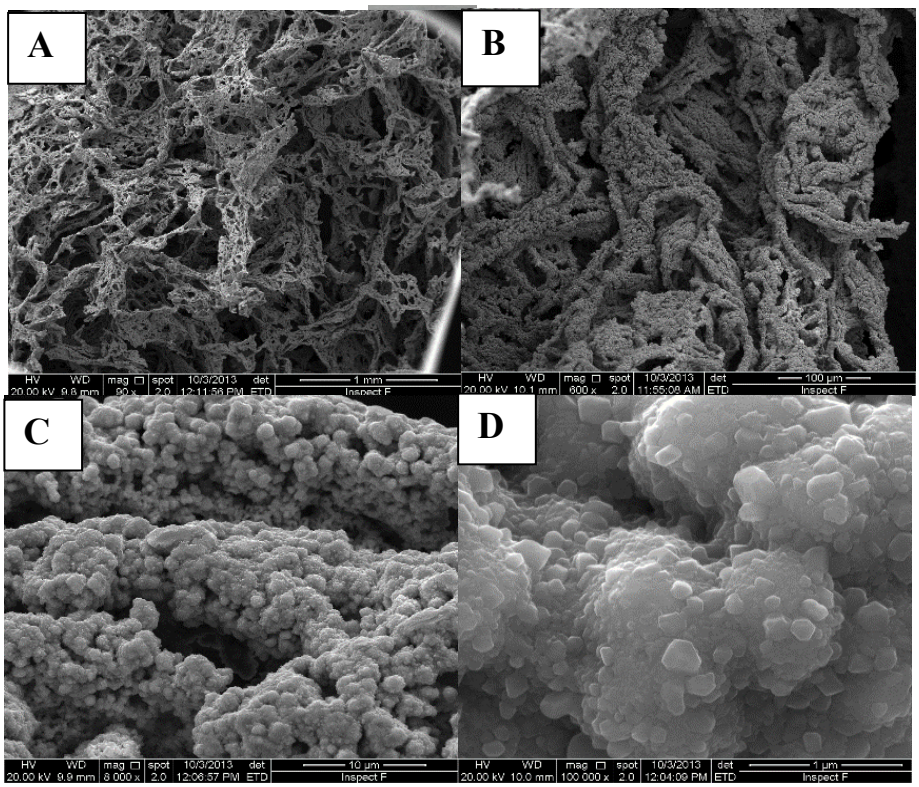

Fig.6 SEM images of spongy Cu obtained with 2.5 ml of 0.2 M $CuSO_4$, $5H_2O$ solution and 0.5 ml of 5M H_2SO_4. No change in morphology was observed when 0.1 M CuSO4, $5H_2O$ solution was used. However, the amount was half in quantity.

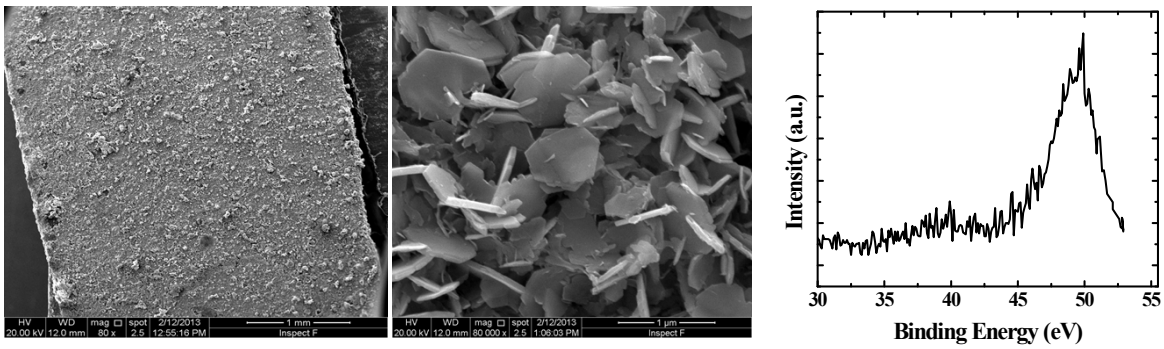

Fig.7 (A & B) SEM images of $Mg(OH)_2$ nanostructures formation on Mg foil surfaces when we performed the experiment in absence of acid medium. (C) XPS spectrum of Mg 2p in $Mg(OH)_2$ (corresponding of to the binding energy 49.6 eV).[1]

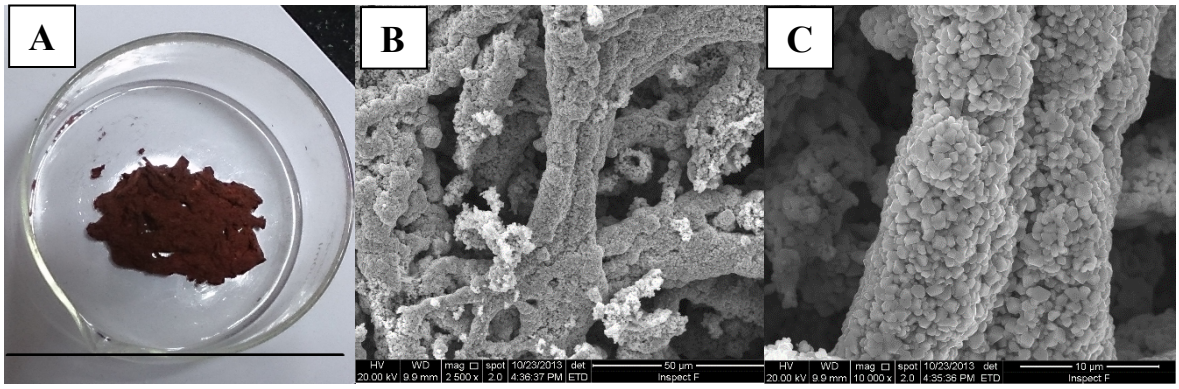

Fig.8 Photograph and SEM images of spongy structures of gram scale synthesis of Cu. The scale bar corresponds to 9 cm.

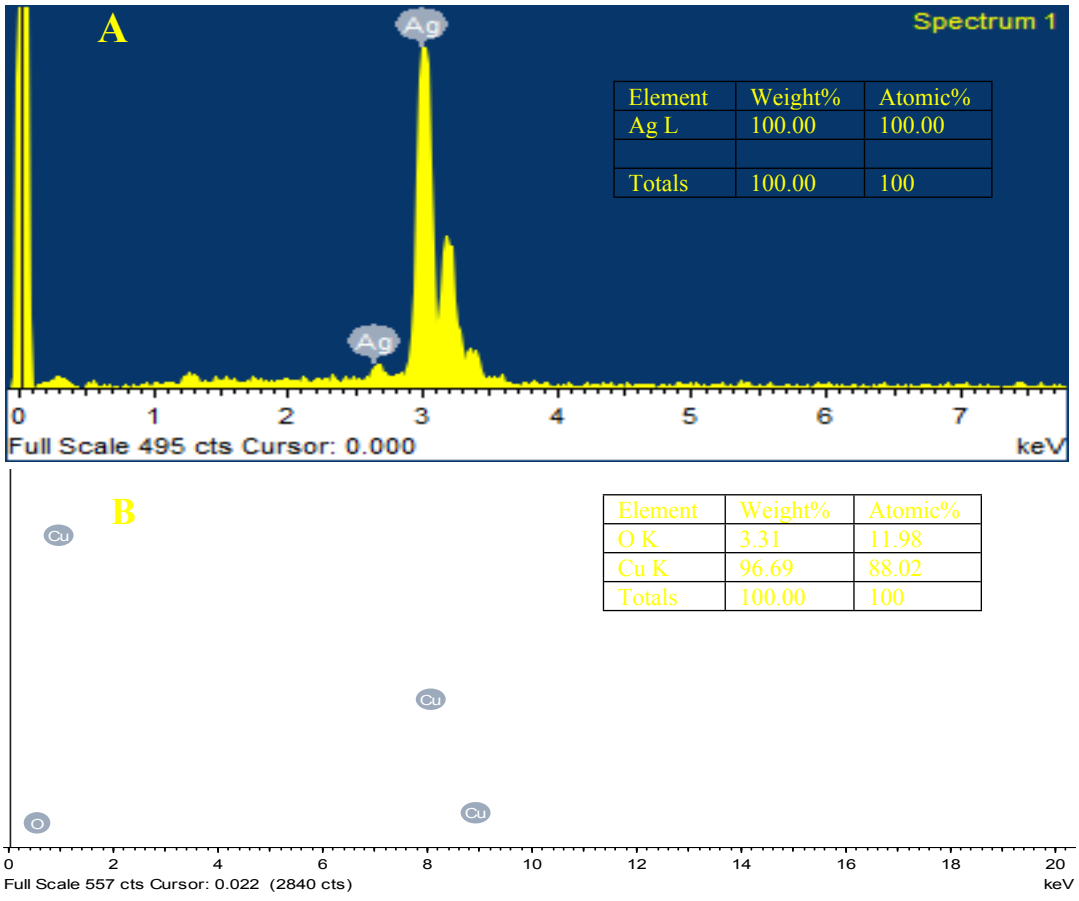

Fig. 8. (A and B) EDS spectra of spongy Ag and Cu nanostructures .

Cu-Ag bimetallic dendritic nanostructures:

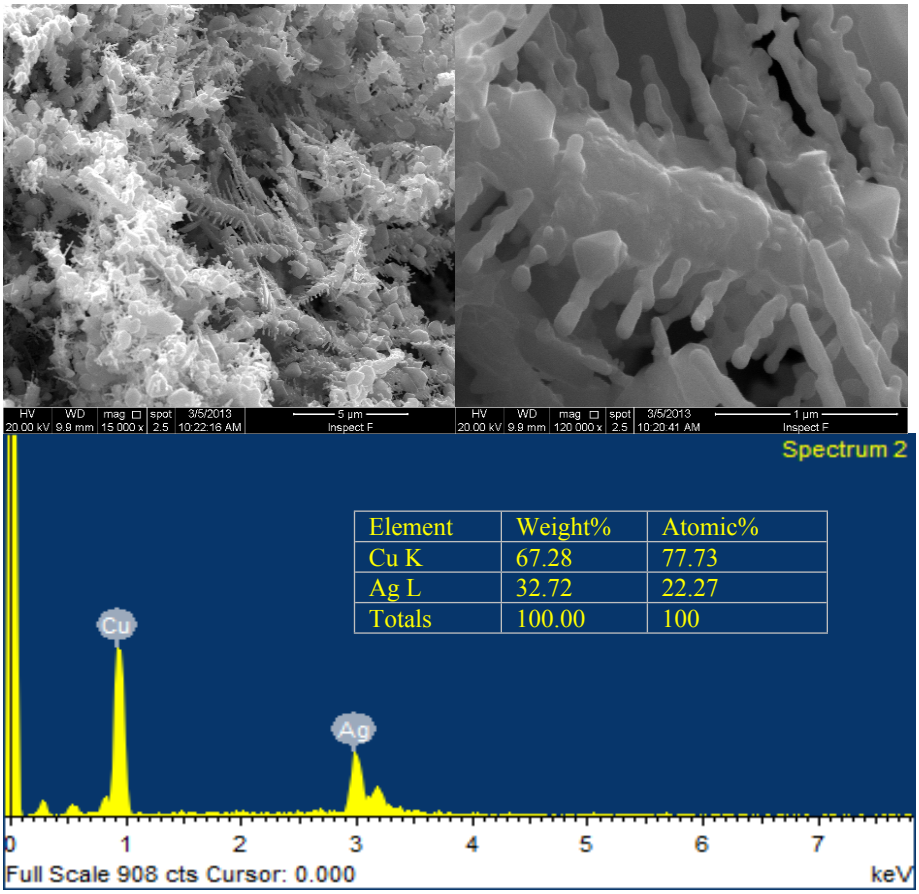

Fig. 9 SEM images with different magnifications and EDS spectra of $Ag_{33}Cu_{67}$.

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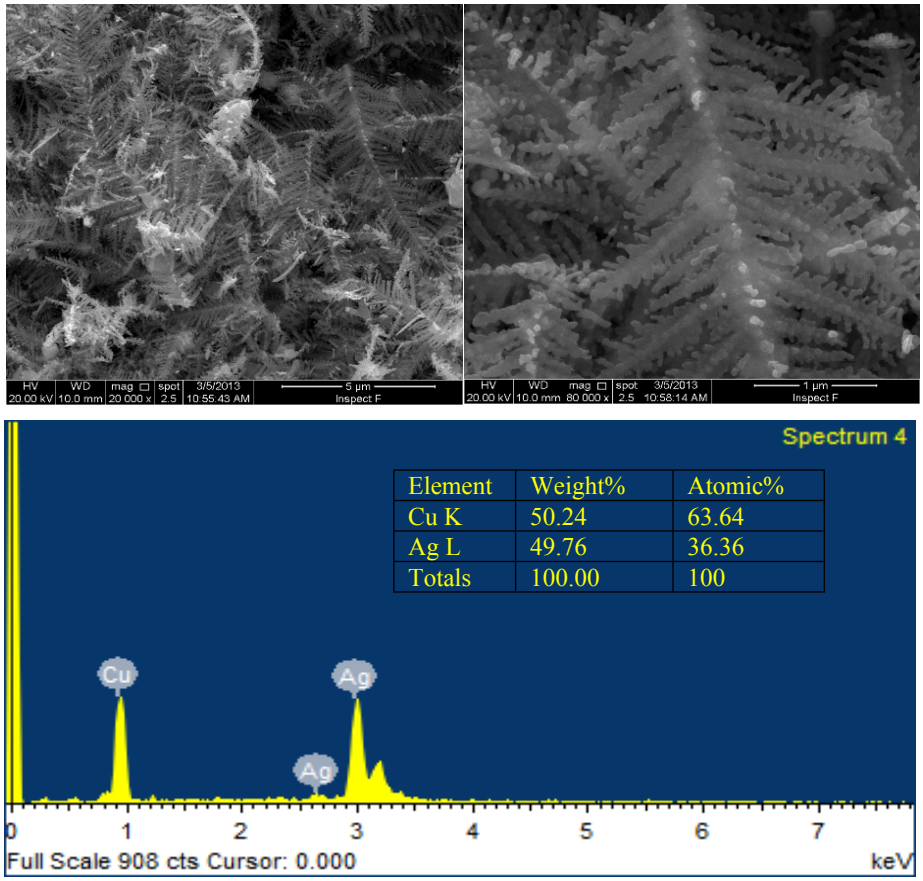

Fig. 10 SEM images with different magnifications and EDS spectra of $Ag_{50}Cu_{50}$.

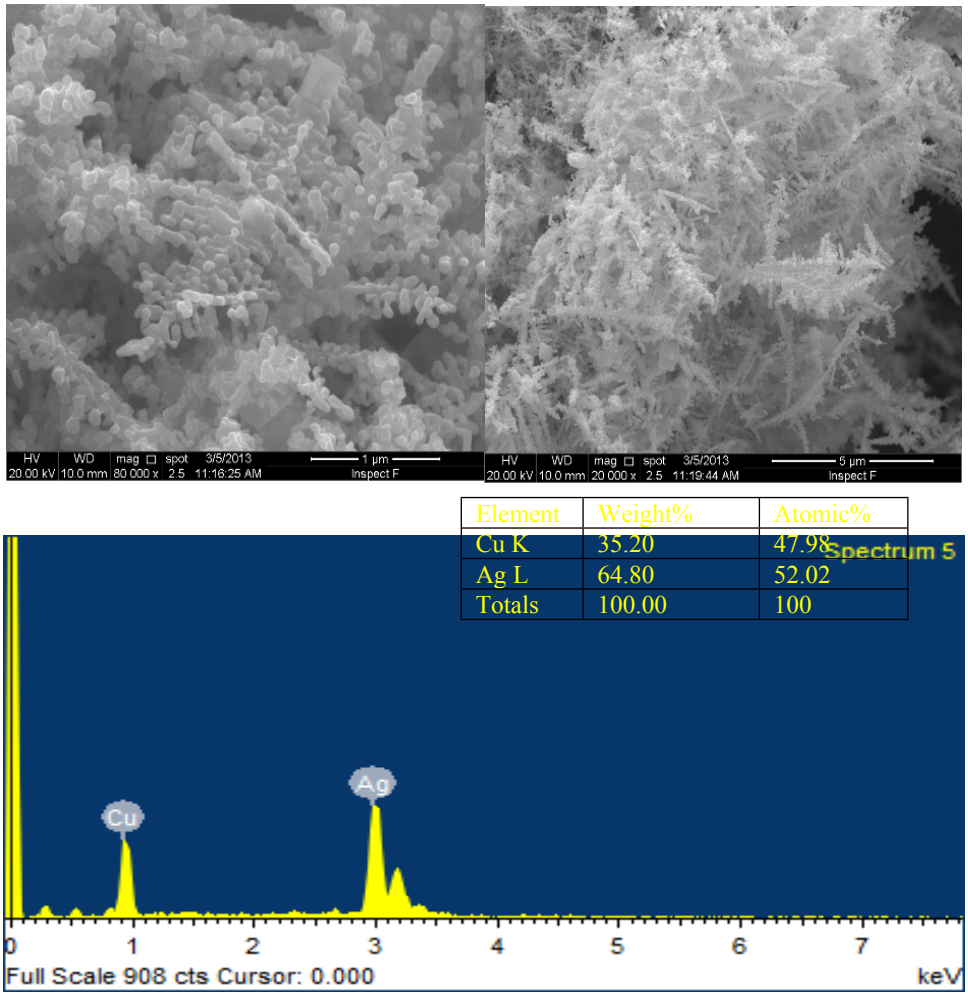

Fig. SEM images with different magnifications and EDS spectra of $Ag_{65}Cu_{35}$.

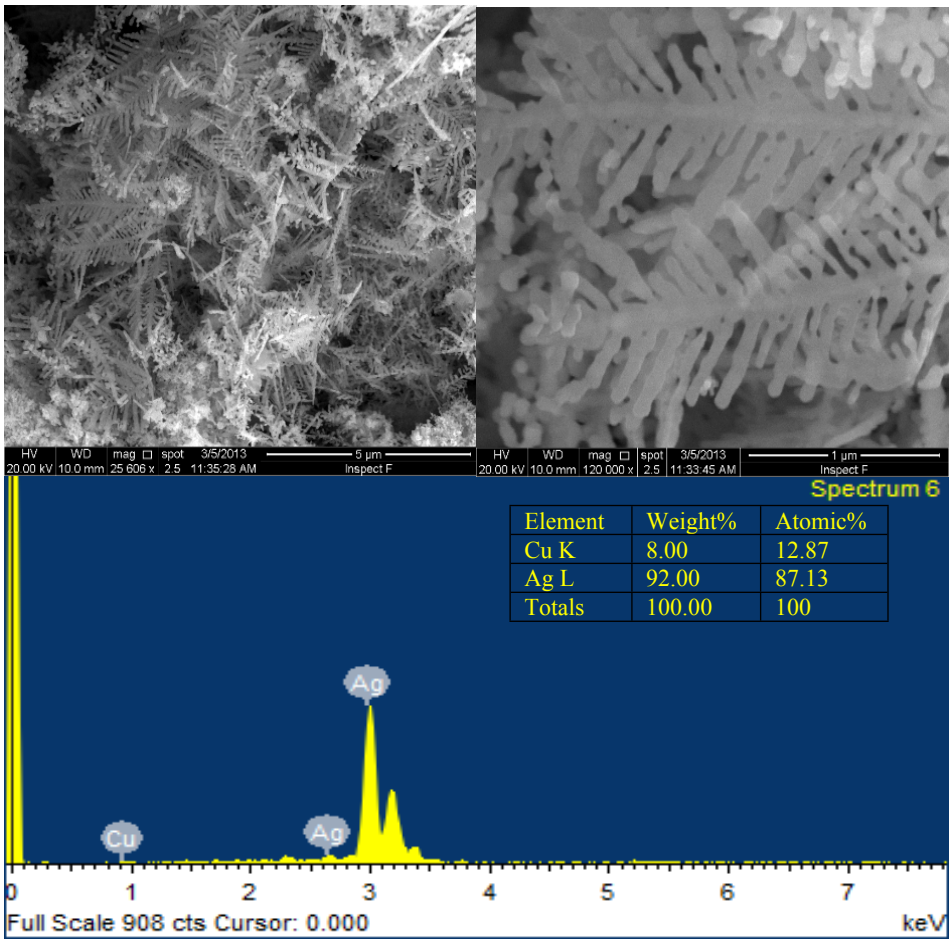

Fig. 12 SEM images with different magnifications and EDS spectra of $Ag_{92}Cu_8$.

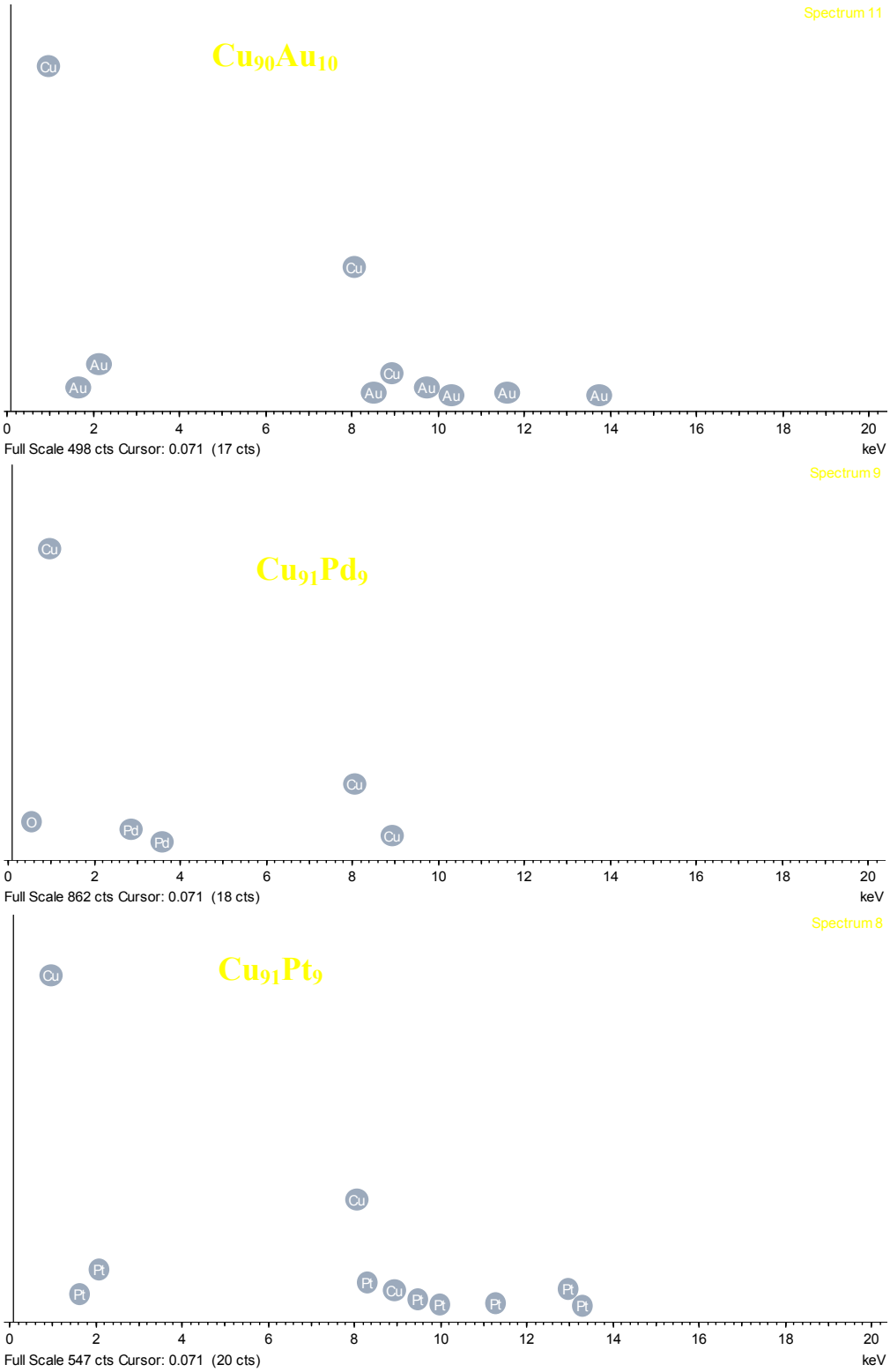

Fig. 13 EDS spectra of various kind of bimetallic nanostructures with the Au, Pt and Pd.

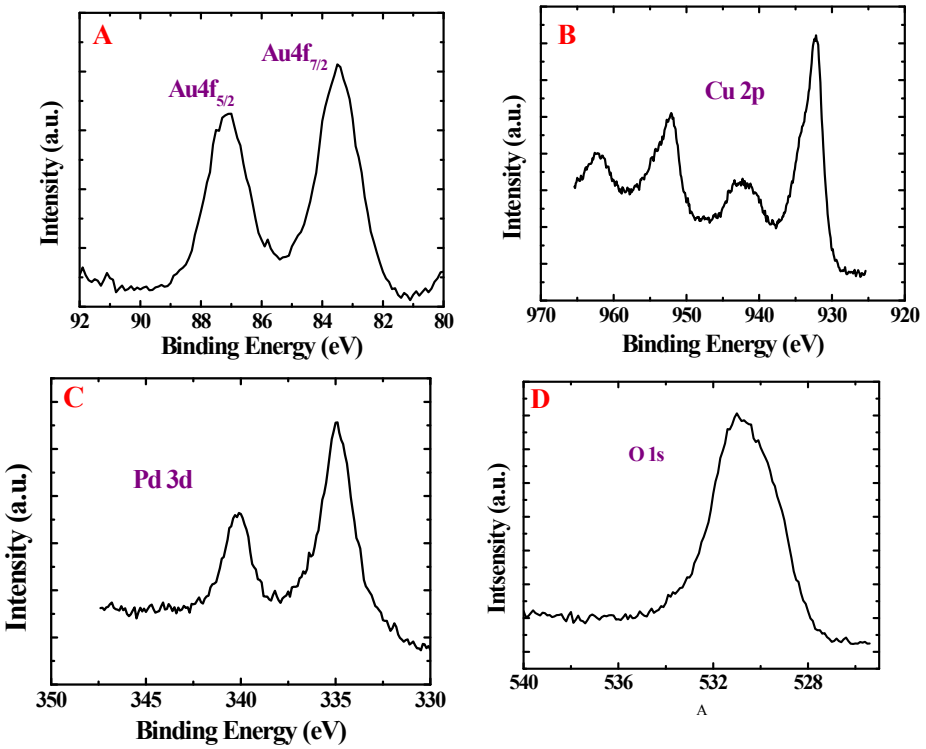

Fig. 14 (A-D) High resolution XPS spectra of individual Cu, Pd, Au and O in CuPdAu dendritic nanostructures.

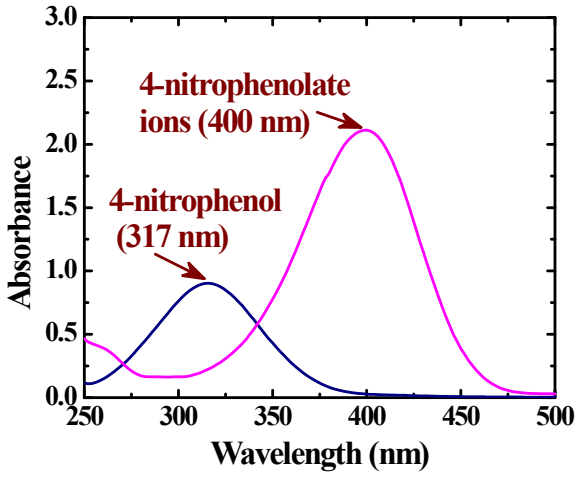

Fig.15 UV-vis spectra of 4-nitrophenol before and after adding $NaBH_4$ solution.

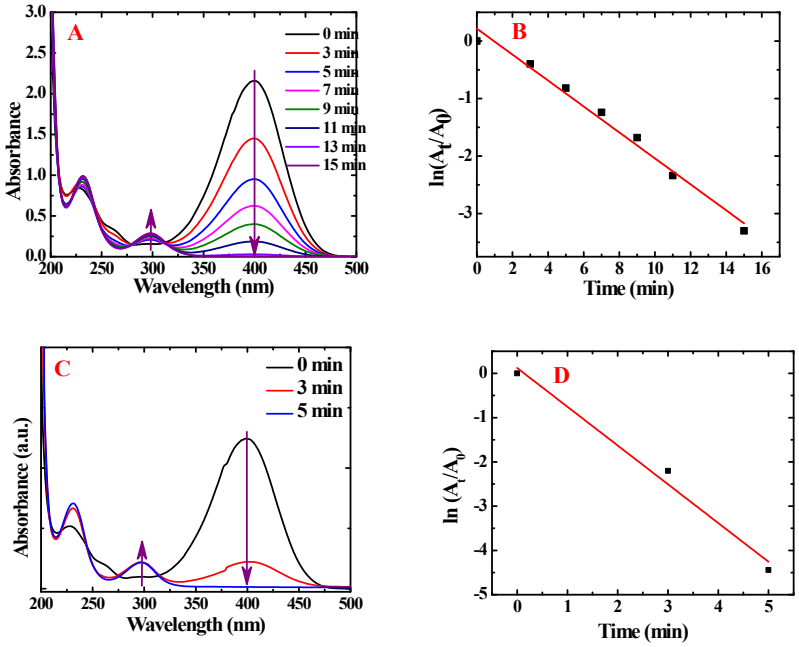

Fig. 16 (A and B) 4-NP reduction by commercially available bulk Cu powder (10 ml of 1 mM 4-NP and 10 ml of 50 mM $NaBH_4$ solution and 5 mg of catalyst) and its pseudo first order rate kinetics plot. (C and D) 4-NP reduction by $Cu_{35}Ag_{65}$ and its pseudo first order rate kinetics plot.

Reference

1. Y. Zhua, G. Wub, Y. H. Zhanga, Q. Zhaoa, *Applied Surface Science*, **2011,** 257, 6129–6137.