Report of the minor UGC project entitled

"Synthesis and Characterization of polymeric Nanocomposites"

from Sept 2009 to Sept 2010

By

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Introduction:

Nanotechnology is an intensive branch of science that interesting in the materials among the size of 1-100 nm with different shapes of spherical nanoparticles, nanorods, nanoribbons, nanobelts and nanoplatelets [1,2]. The unique physical and chemical properties are due to its high surface-to-volume ratio comparing with micro or bulk-sized [1,2]. The nanomaterials can be obtained with different methods such as solid−liquid discharge process [3], novel quick-precipitation [4] and direct thermal decomposition [5] methods. Nanomaterials are now become available and useful in all the man daily life applications such as in: medicine, solar cells, water purification, pharmaceutical and catalysts [6].

Experimental:

I. Copper oxides were prepared as shown in a table 1

Table 1

Experimental procedures and products

Experiment C (series 1)	Preparation procedure	Result
C1	10.0mL 1M $Cu(NO_3)_2$ + 10.0mL1M Urea Heated on low flame	CuO spherical particles width = 430nm
C2	10.0mL 1M $Cu(NO_3)_2$ + 10.0mL 1M Urea + 10 mL of Castor oil and Heated on low flame	CuO Honey comb like assembly width = 437nm
C3	10.0mL 1M $Cu(NO_3)_2$ + 1M Urea + 5 mL gum acacia and Heated on low flame.	CuO nanopetals length less than 0.85-2.0 µm width = 280 nm
Experiment C (Series 2)		
C4	10.0mL1M $Cu(NO_3)_2$ + 10.0mL1M Thiourea Heated on low flame	CuO flower like assembly with number of nanopetals of length less than 1.1-2.7 µm width = 680nm

C5	10.0mL 1M $Cu(NO_3)_2$ + 10.0mL 1M Thiourea + 10 mL of Castor oil and Heated on low flame	CuO flower like assembly with number of nanopetals of length less than 0.941-3.5 µm width = 352nm
C6	10.0mL 1M $Cu(NO_3)_2$ + 1M Thiourea + mL gum acacia and Heated on low flame	CuO flower like assembly with number of nanopetals of length less than 0.57-4.28µm width = 300nm.

Copper oxide (CuO) was prepared by combustion method, using fuels like urea & thiourea. Effect of additives like castor oil & gum acacia was studied. CuO obtained was characterised by IR, XRD, SEM/EDAX.

II. Copper oxide prepared in above manner was doped with PANI. By mixing with 10, 20 mass percent copper oxide during in situ polymerization of aniline in the presence of potassium dichromate as an oxidant. PNC's thus prepared were characterized by IR, XRD etc. Electrical conductivity was measured by two probe method.

Result & Discussion:

As shown in Fig. 1 Infra red spectra revealed absorption band for metal oxygen, three characteristic peaks of CuO positioned at 606, 500 (a Cu–O stretching along [−202] direction) and 422 cm^{-1} (a Cu–O stretching along [202] direction) were observed. The XRD pattern of CuO samples prepared by combustion method are presented in Fig. 2, all diffraction peaks can be indexed to the monoclinic phase of CuO crystals (JCPDS 48-1548). No peaks of impurity are detected in the XRD patterns, indicating that pure crystalline CuO was fabricated by the presented procedure. By using the Debye-Scherer formula. the average crystallite size of the C1-C6 samples was calculated to be in the range 30-78 nm from CuO (111), as shown in Table 2. From SEM micrographs it was observed that microflowers were obtained in presence of thiourea and this microflowers were found to be shattered after addition of additives like castor oil & gum acacia Fig. 3 & 4 shows the SEM micrographs of C1-C6. EDAX

measurements help to give the metal percentage present in copper oxide as shown in Fig. 5. The properties such as electrical conductivity were measured by two probe method. Dielectric constant & resistivity was measured w. r. t. to varying frequency the graphs are presented in figures from 6 & 7. Later nanocomposites were obtained by doping Copper oxide prepared as above in polyaniline. Composites obtained were characterised by IR, XRD etc. Again electrical properties were measured.IR and XRD are presented in Fig. 8 & 9. Fig. 10 shows the conductivity plots.

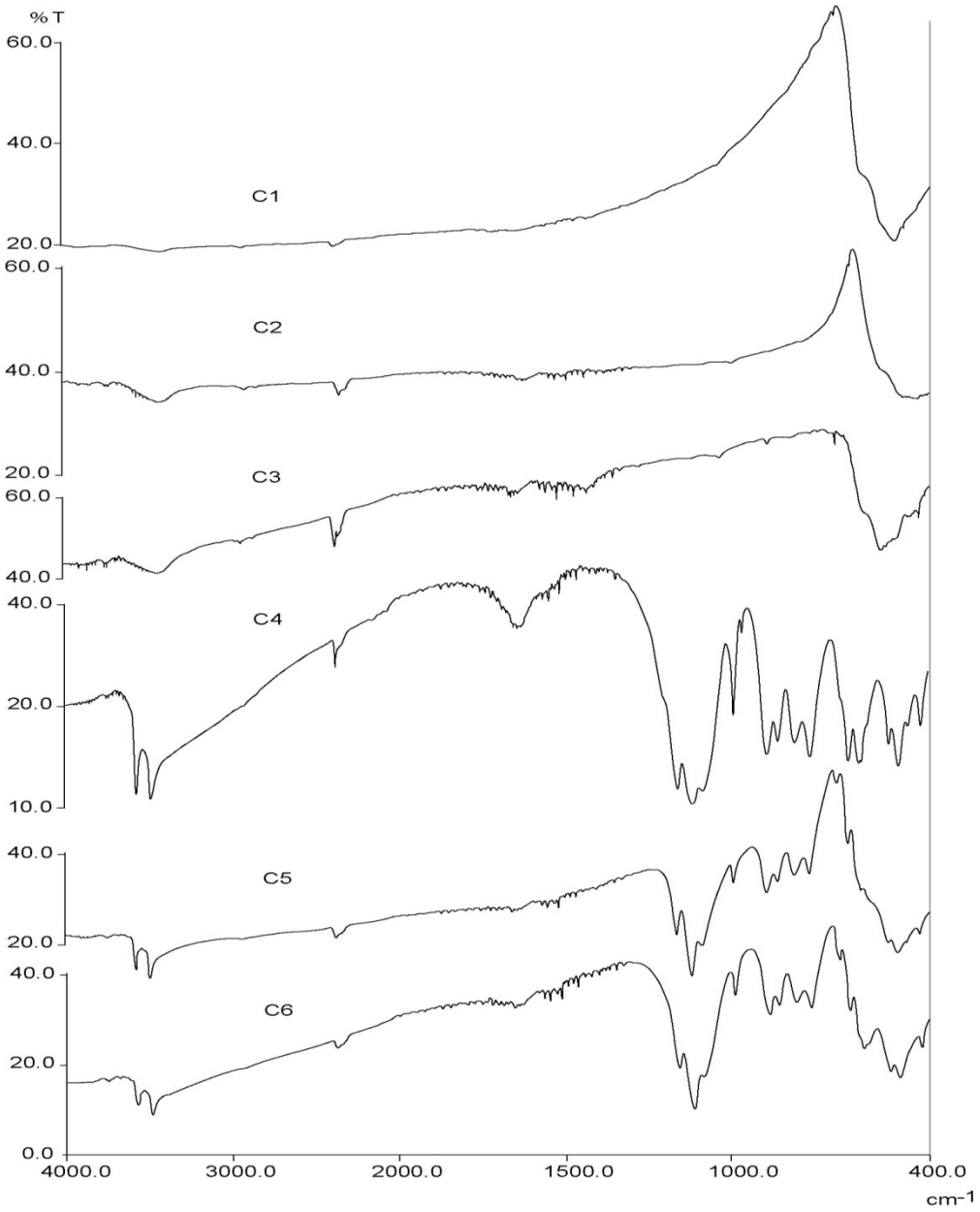

Fig.1 IR spectrum of CuO samples

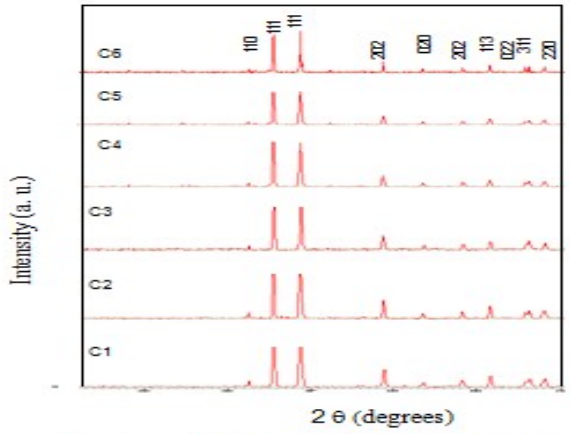

Fig. 2 X- Ray Diffraction Patterns of C1- C6 Samples

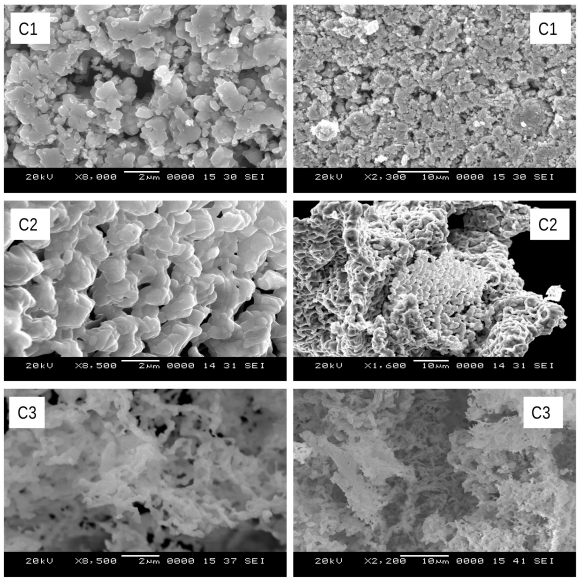

Fig. 3 SEM images of samples C1 to C3

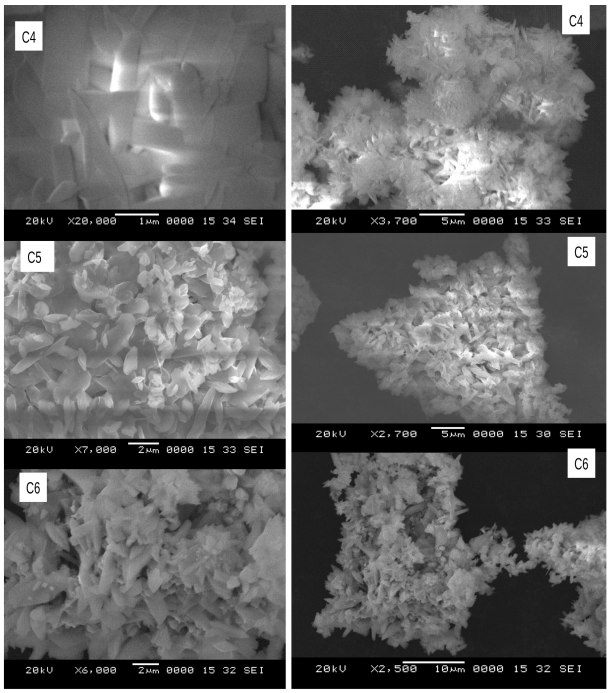

Fig.4 SEM images of flower like assemblies corresponding to C4-C6

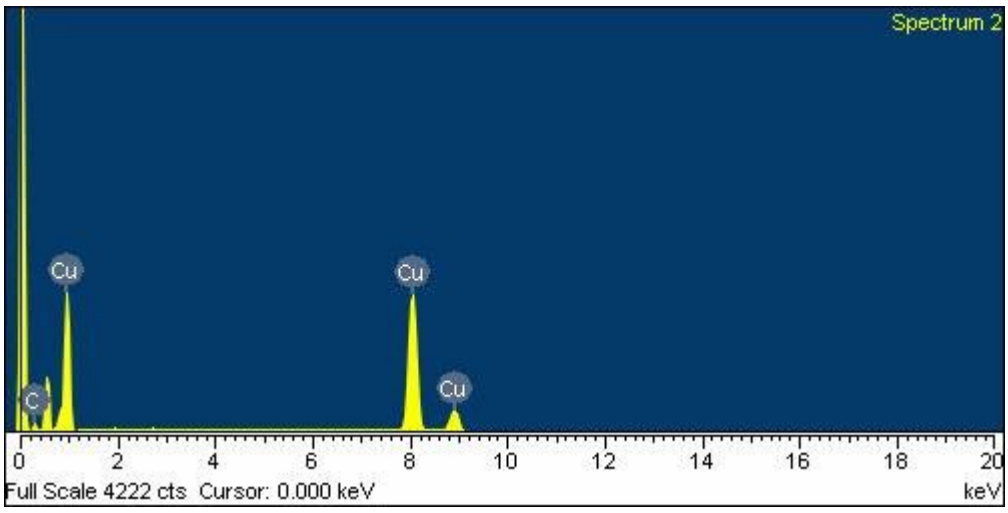

Fig. 5 Showing EDS of microflowers

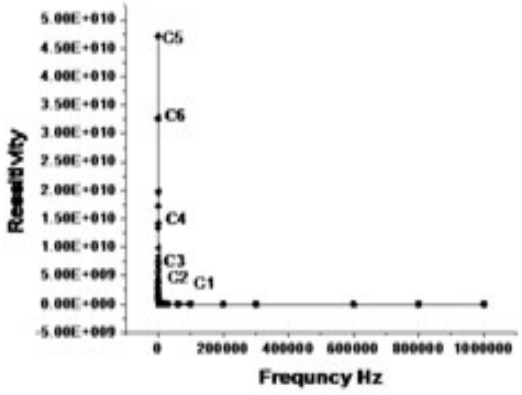

Fig. 6 plot of ressistivity vs frequency of C1-C6 samples

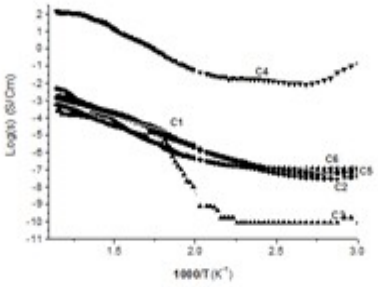

Fig. 7 Logarithm of electrical conductivity vs. receiprocal of temperature of C1-C6 samples

Table 2 showing some characteristics of CuO samples

Samples	Electrical Conductivity	Capacitance gap eV	€”	Band	Crystallite Size nm (Scherrer’s)	Dielectric constant
C1	1.2×10^{-3}	97.3	1.47	0.72	32	22.73
C2	6.0×10^{-3}	210.8	3.0	1.01	40	45.30
C3	2.6×10^{-3}	413.1 91.91	2.67	2.46	30	
C4	6.9×10^{-3}	248.39 63.91	0.71	0.75	34	
C5	1.7×10^{-3}	150.13 46.80	1.91	0.54	43	
C6	5.8×10^{-3}	118.9 26.51	0.8	0.73	78	

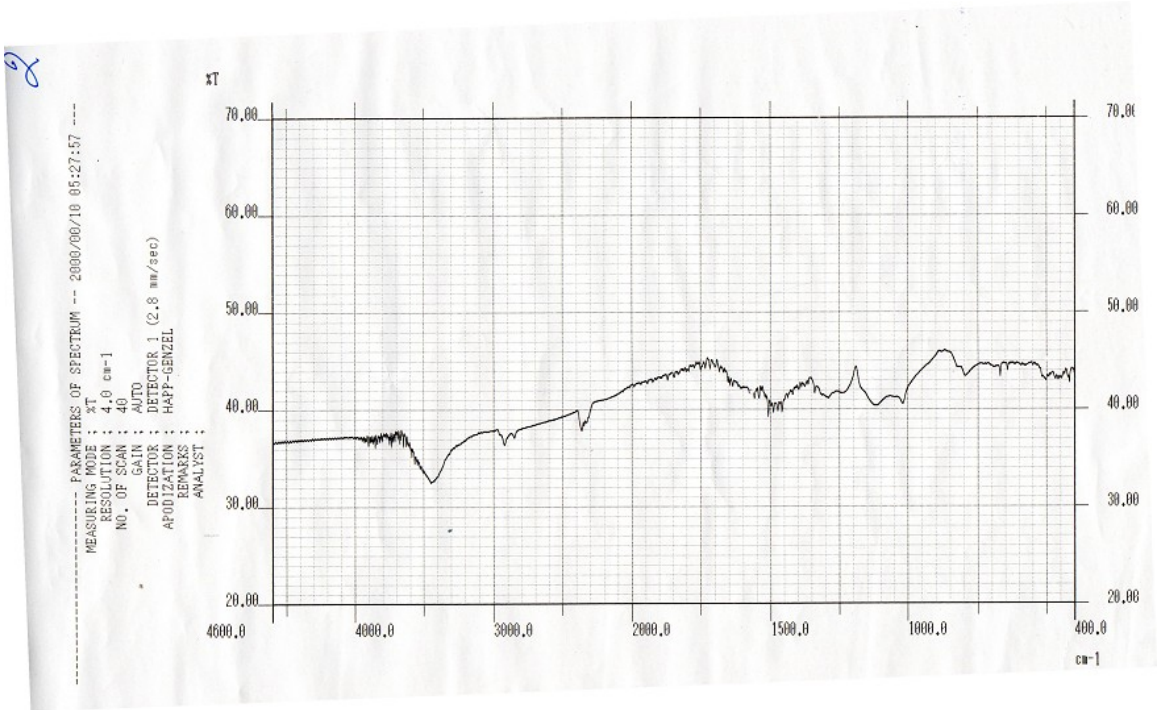

Fig. 8 IR spectrum of Nanocomposite

In addition to the absoption peaks of PANI, Peaks at 420cm^{-1},500cm^{-1} correspond to CuO absorption.

Fig. 9 XRD pattern of pani –CuO nanocomposite

With the increase in percentage of CuO in PANI the 2Θ value shifts slightly towards the lower side . And the intensity of the peak also decreases. The broad peaks observed in all hybrids XRD pattern indicates short crystallite diameter of the oxide, which was estimated by the Scherer's equation as ..... nm. Results obtained by Scherer's equation suggest that the CuO crystallite size is not affected by the polymer presence, and is approximately the same in the pure oxide and in all the hybrids materials.

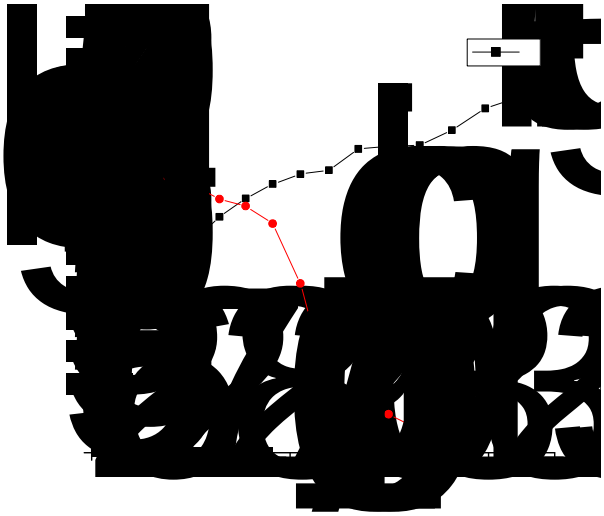

Fig. 10 log σ v/s 1000/T of Pani-CuO nanocomposites

Conclusion:

The nanostructured CuO microflowers were fabricated through a Combustion method. These microflowers were built up with petals of 0.941-3.5 nm in length and 352nm in width. Presence of thiourea was found to favour the formation of microflowers. But were found to show scattered petals morphology with addition of additives like castor oil and gum acacia as seen from SEM observations. It can be concluded that the presence of sulphur group from thiourea without additives leads to the formation of microflowers with fine arrangements. C3 was also found to show the lowest particle size and lowest average crystallite diameter. The highest dielectric constant value and the highest band gap were observed for C3. The effect of additive such as gum acacia was found to show decrease in particle size. The electrical conductivity for C1 to C6 was found to increase with increase in temperature showing semiconducting behaviour. This synthetic route was very simple and well reproducible, which could be applied in the preparation of other transition metal oxide hierarchical nanostructures.

References:

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