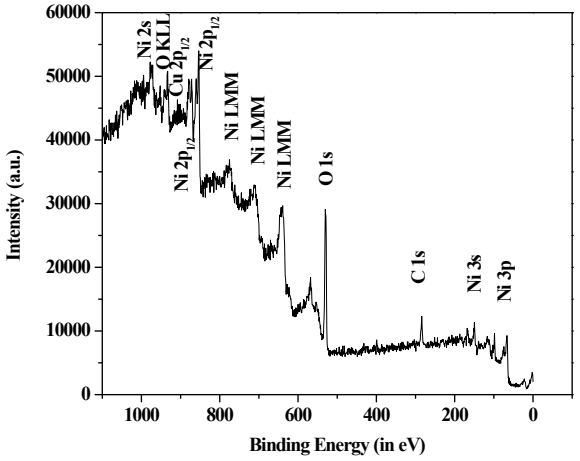
Electronic Supplementary Material (ESI) for Environmental Science: Nano.

Synthesis of $Cu_xNi_{(1-x)}O$ coral like nanostructures and its application in design of re-usable toxic heavy metal ion sensor based on adsorption mediated electrochemical technique

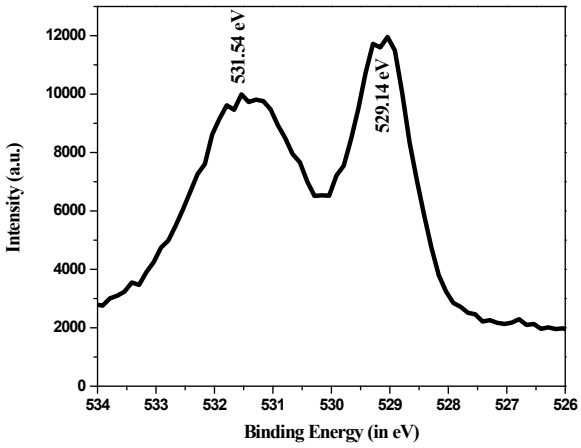
Electronic Supplementary Information

Synthesis of NiO, Ni_2O_3 and $Cu_xNi_{(1-x)}O$ hierarchical nanostructures

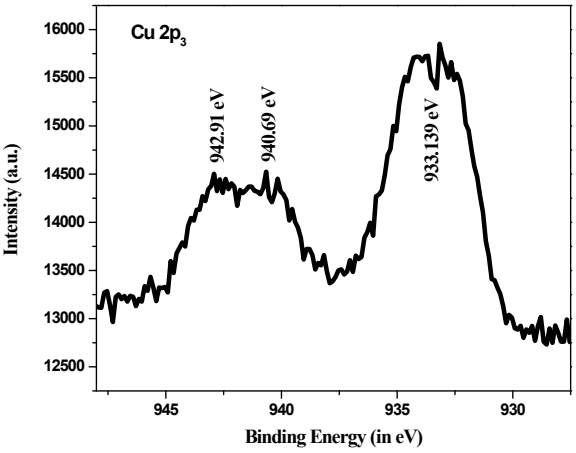
In a typical experiment, 1.57 g of $Ni(NO_3)_2.6H_2O$, 1.57 g or NH_2-CO- NH_2 (Urea) and 0.3 g polyethylene glycol (PEG) were dissolved in 65 ml DI water. For doping, the a mixture of Cu and Ni salts are added maintaining desired ratio of Cu:Ni (by weight). The nucleation is started by adjusting the pH at around 11 when the precipitation of $Ni(OH)_2$ just starts. Then the solution is transferred to a Teflon lined steel autoclave and was treated hydrothermally at 100^{o}C for 5 hours. The resultant precipitate so obtained was washed with DI water for several times and dried to obtain power of pure/doped nickel hydroxide. For NiO and Cu doped NiO, the pure/doped hydroxide was calcined at 450^{o}C for 3 hours. While for Ni_2O_3, the hydroxide is treated with sodium hypochlorite till the effervescence ceases. The black precipitate is centrifuged, washed and dried to obtain fine powder of Ni_2O_3 hierarchical nanostructures.



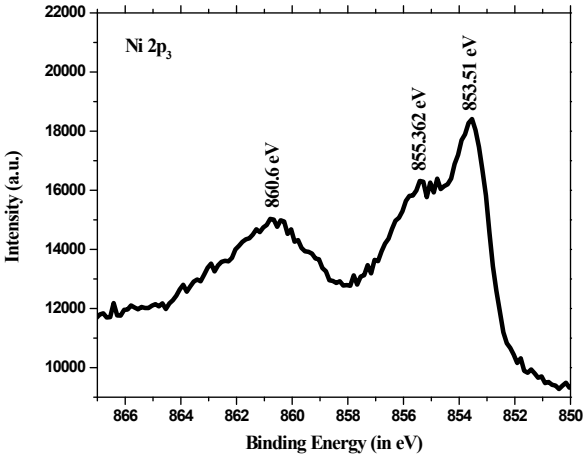
(a)



(b)



(c)



(d)

Figure S1: XPS Spectra of $Cu_xNi_{(1-x)}O$ nanostructures for chemical composition analysis (a) Overall XPS spectrum of the sample (b) XPS peaks of O 1s (c) XPS peaks of Cu $2p_3$ (d) XPS peaks of Ni $2p_3$

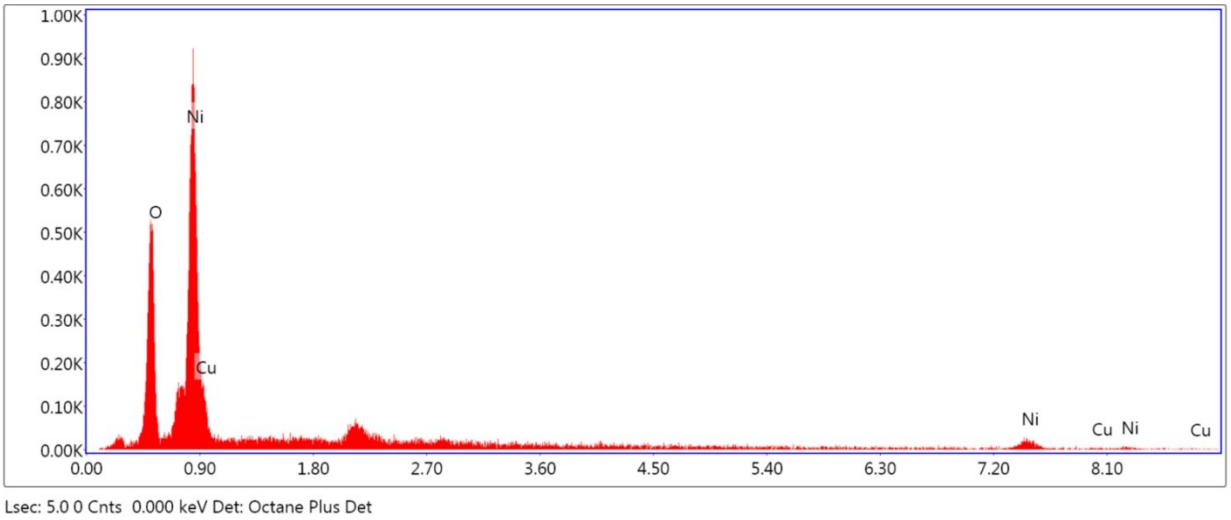


Figure S2: EDAX of Cu doped NiO hierarchical nanostructure

Element	Weight %	Atomic %	Net Int.	Error %	K ratio	Z	R	A	F
O K	24.73	54.92	1,293.04	6.52	0.21	1.23	0.89	0.67	1
Ni L	64.95	39.31	1,670.02	4.14	0.54	0.93	1.04	0.89	1
Cu L	10.32	5.77	166.36	19.07	0.05	0.89	1.05	0.5	1

Table S1: EDAX data of $Cu_xNi_{(1-x)}O$ nanostructures

Batch adsorption technique for Cr(VI) ion adsorption

For batch adsorption technique, 0.4 g of $K_2Cr_2O_7$ is dissolved in 200 mL DI water to prepare the stock solution. The different concentration is prepared by performing serial dilution to the stock solution. In a typical experiment of a particular concentration, 5 mL of stock solution of required dilution is taken in a 100 mL beaker and 0.02 g of adsorbent is added to it under constant stirring. The stirring is continued for 5 minutes. Then the solution is filtered and the supernatant is tested under UV-Visible spectrophotometer to observe any decrease in the peak for Cr(VI) ion in the spectra with respect to control solution (solution not exposed to the adsorbent). Any decrease in absorbance can be related to the amount of Cr(VI) ions adsorbed and hence adsorption can be tested.

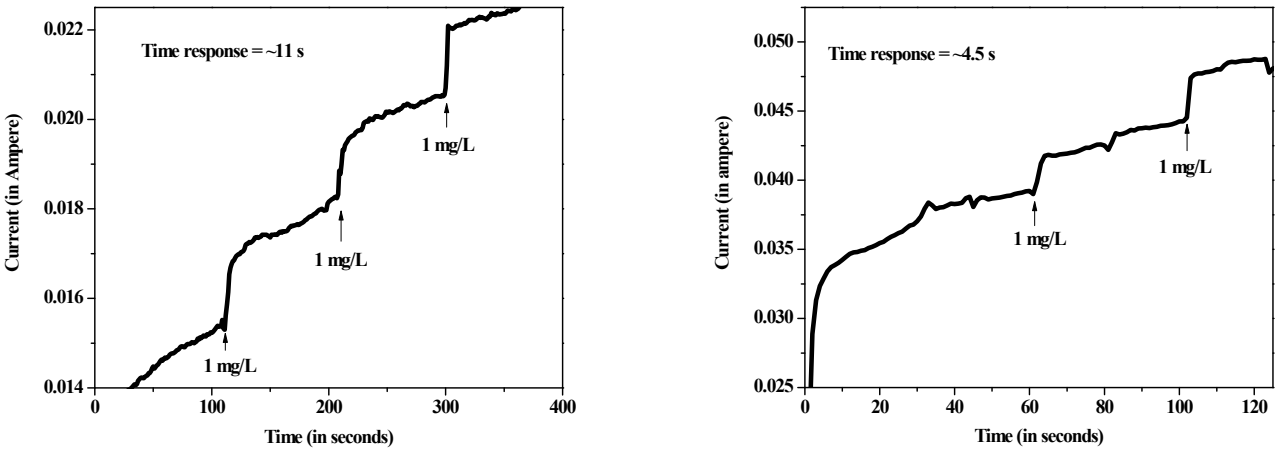
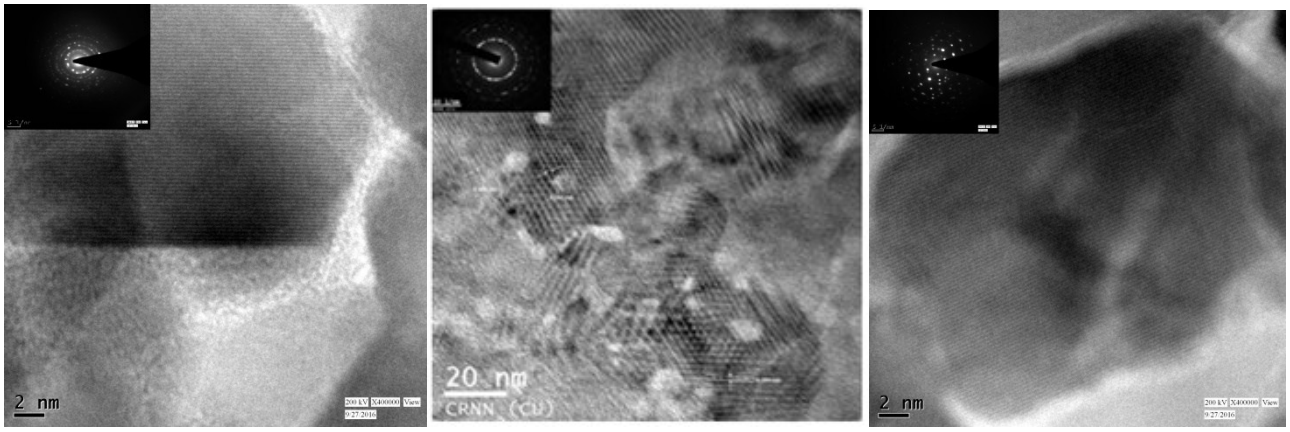


Figure S3: Time response of 5% and 20% Cu doped NiO hierarchical nanostructure respectively (amperometric measurement)



(a) **(b)** **(c)**

Figure S4: Lattice images and SAED patterns of (a) NiO (b) Ni_2O_3 (c) $Cu_xNi_{(1-x)}O$ coral like nanostructures

Sample Name:	CuNiO 1		
SOP Name:	mansettings.nano		
File Name:	rishi-go.dts	**Dispersant Name:**	Water
Record Number:	95	**Dispersant RI:**	1.330
Date and Time:	Thursday, September 22, 2016 3:00:19 ...	**Viscosity (cP):**	0.8872
		Dispersant Dielectric Constant:	78.5

Temperature (°C):	25.0	**Zeta Runs:**	12
Count Rate (kcps):	75.9	**Measurement Position (mm):**	2.00
Cell Description:	Clear disposable zeta cell	**Attenuator:**	6

			Mean (mV)	**Area (%)**	**St Dev (mV)**
Zeta Potential (mV):	5.51	**Peak 1:**	5.51	100.0	4.94
Zeta Deviation (mV):	4.94	**Peak 2:**	0.00	0.0	0.00
Conductivity (mS/cm):	0.248	**Peak 3:**	0.00	0.0	0.00

Result quality : Good

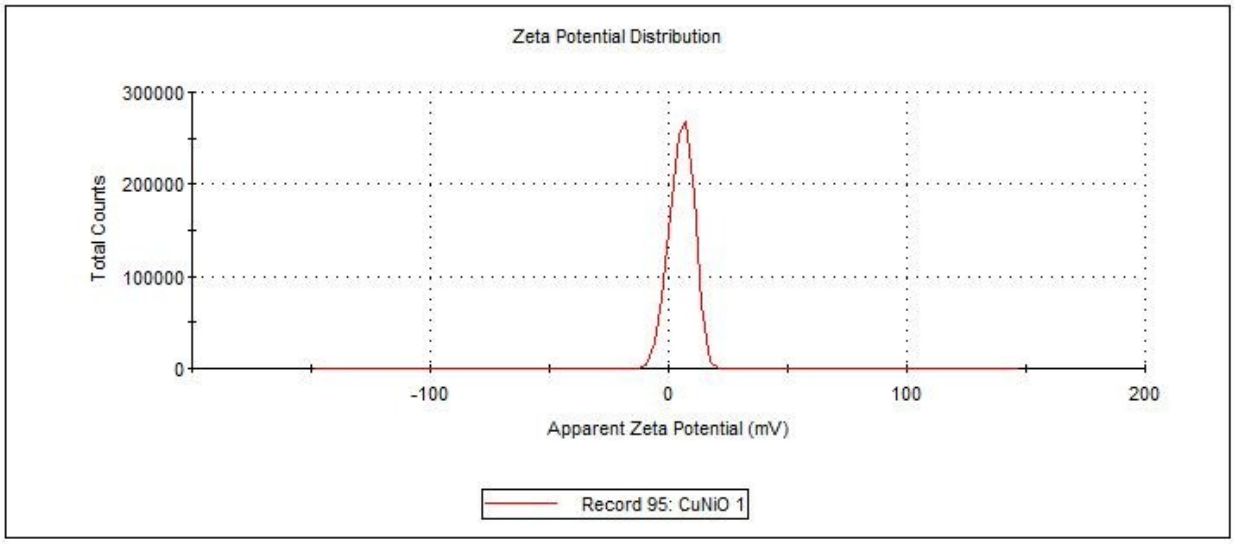


Figure S5: Zeta potential analysis (ZetaSizer nano NS) for $Cu_xNi_{(1-x)}O$ coral like nanostructures

Sample Name:	NiO 1		
SOP Name:	mansettings.nano		
File Name:	rishi-go.dts	**Dispersant Name:**	Water
Record Number:	93	**Dispersant RI:**	1.330
Date and Time:	Thursday, September 22, 2016 2:44:54 ...	**Viscosity (cP):**	0.8872
		Dispersant Dielectric Constant:	78.5

Temperature (°C):	25.0	**Zeta Runs:**	13
Count Rate (kcps):	232.0	**Measurement Position (mm):**	2.00
Cell Description:	Clear disposable zeta cell	**Attenuator:**	10

			Mean (mV)	**Area (%)**	**St Dev (mV)**
Zeta Potential (mV):	36.3	**Peak 1:**	39.2	84.6	8.34
Zeta Deviation (mV):	12.5	**Peak 2:**	12.3	13.4	5.83
Conductivity (mS/cm):	0.0960	**Peak 3:**	62.4	2.0	2.48

Result quality : See result quality report

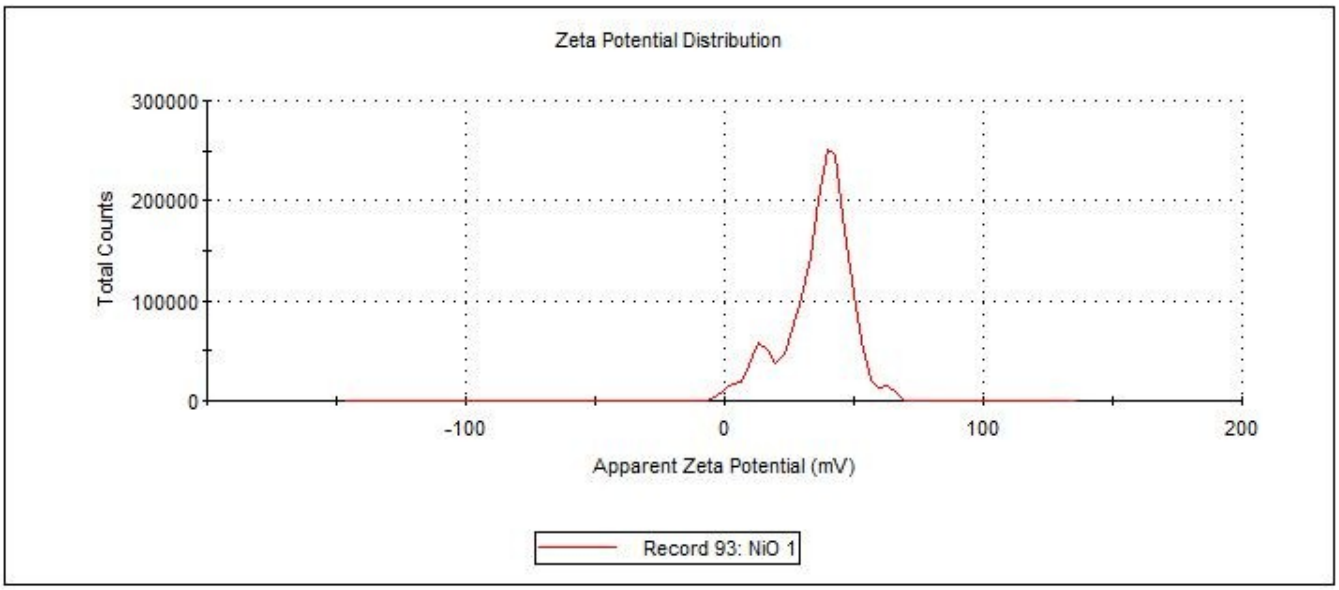


Figure S6: Zeta potential analysis (ZetaSizer nano NS) for NiO coral like nanostructures