**Facilely green synthesis of 3D nano-pyramids Cu/Carbon hybrid sensor electrode materials for simultaneous monitoring of phenolic compounds**

Excessive usage of phenolic compounds such as catechol (Cat-), cresol (Cre-) and p-nitrophenol (PNP) in food processing and pharmaceutical industry may lead to severe health issues including nerve cells disruption and even infertility. Thus, designing sensitive nanoscale electrochemical sensors to boost single screen signalling of these phenolic compounds from environmental samples is highly demanded. However, control monitoring and quantification of these phenolic compounds from real samples such as commercially available pesticides and insecticides is quite challenging because of their low concentration along with excess of co-existing interfering species. Herein, for the simultaneous electrochemical monitoring of these phenolic pollutants, we fabricate C-encapsulated three-dimensional Cu nano-pyramids ([email protected]) through a simple, scalable freeze-drying and carbonization process. The resulting hybrid contains 3D arrangement of controlled Cu nano-pyramids with atomic arrangement along all four lateral faces with sharp apex which result in exposition of greater fraction of Cu {111} catalytic active sites, large number of sp² carbon atoms, and maximum surface defects along with dipolar/quadrupolar radiative plasmons, thus offering highly sensitive and selective behaviour toward simultaneous monitoring of Cat-, cresol and PNP. The designed [email protected] electrode also shows fast diffusion and kinetics of ions compared to [email protected] (AL)-like Cu nanoparticles (NPs) sample, thus indicating shape dependent efficacy of the designed electrode. Because of high sensitivity, the developed novel [email protected] electrode was successfully applied to monitor PNP from commercially available insecticide. The results demonstrated the potential of the designed electrode to be used in preventing serious health issue associated with phenolic pollutants.