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Non-Hydrothermal Synthesis of Cu(I)-Microleaves from Cu (II)-Nanorods

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Simultaneous transformation of structural morphology, material dimension and oxidation state of Cu (II)-nanorod was achieved with 3-(Triethoxysilyl)propylamine, (APTS) and 4-nitrobenzaldehyde (4-NB) under non-hydrothermal condition. Morphology of the modified Cu(I) material was found to resemble with the leaf of colocasea. Conversion of a Cu(II) based nono-material to a Cu(I)-based micro-material was confirmed from EPR, MAS- NMR and cyclic voltametric (CV) study. BET-surface area, Raman signal intensity and thermal stability of the Cu(I)-microleaf was greatly enhanced due to presence of Cu-O–Si linkage. Both the Cu(II) and Cu(I) material were found to be effective catalyst systems for nitro-aldol reaction as well as catalytic oxidation of methylene blue (MB) dye in presence of H_2O_2. High %yield (> 90%) of nitro-aldol product was obtained with both the catalyst. Catalytic reaction under microwave irradiation was found to bring substantial decrease in reaction time. Cu (II)-material was however not recyclable because of its soft nature. While the Cu(I)-microleaf was recycled upto four consecutive cycles. These materials were found to degrade methylene blue dye within 20 min in presence of H_2O_2 but in absence of light. Dark phase catalytic oxidation of MB was monitored both *via* UV-vis and cyclic voltametric study.

Introduction

Development of nano-based materials and nanotechnology has recently brought a new renaissance in designing of newer materials for various applications.[1] Starting from its applications in house utensils to the drug delivery, nanomaterials have captured a wide range of research area like energy conversion and storage, chemical manufacturing, biological applications, and environmental technology.[2–13] Catalysis by nanoparticles or nanomaterials is one of the important aspects where researchers from both industries and academics are paying much interest.[14,15] So far various transition metal like gold (Au),[16] platinum (Pt),[17] palladium (Pd),[18] silver (Ag)[19] etc have been well explored as nanocatalyst for transforming various organic reactions. In this context, Cu-containing nanomaterials or nanoparticles are gaining much interest in catalysis due to the high abundances of copper and low cost synthetic method. Cu-nanoparticles either in its oxide form CuO or as hydroxide, $Cu(OH)_2$ or as sulphides, CuS has recently been employed as catalyst in various type of catalytic reactions.[20]

Cu-nanomaterials or nanoparticles are mostly synthesized by hydrothermal or solvothermal methods using some suitable reducing and stabilizing agents.[20] In many cases Cu-salts with different counterions are taken as source of copper.[20] These methods however have some limitations i.e. control over the growth of nanoparticles. Temperature, pressure, molar ratios, nature of counterions and stabilizers plays vital role in architecting the surface morphology of Cu-nanomaterials.[21] So in recent years researchers are trying to synthesize Cu-based nanomaterials using some metal complex as precursors. Liu et al.[22] has synthesized CuS from Cu-thiourea complex, Yao et al.[23] has obtained CuS from Cu(I) complex of thioacetamide.

Researchers seemed to be more focused towards the hydrothermally synthesized Cu-nanomaterials in low oxidation state. But less emphasis has been given on synthesis of Cu(I) and Cu(II) material without following the conventional hydrothermal process. A very few reports are available for synthesis of Cu(II) complex in its nanodimension.[24,25] The objective of the present work is therefore to synthesize a Cu(II)- complex in nanoscale and its conversion to a Cu(I)-based material *via* a non-hydrothermal process. Such method will reduce the number of steps involved in hydrothermal process. It will also avoid the use of specific stabilizing agent and also the toxic reducing agents resulting in a development of one green method. Further, as one dimensional nanomaterials also finds high applications in optics, magnetism, and microsystems

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