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Preparation of Mn Nanoparticles, Mn as a Catalyst and its Properties

Dr. Pramod Kumar ,Dept. of Chemistry, GDC Jaithra Etah

Dharmendra Dwivedi, Dept. of Physics, Gdc Bangarmau

Dr Rajat Srivastava, Dept. of Engineering, University Researcher,Uni. Salento, Italy

Abstract

In this paper, we report on the prepration of Mn nanoparticles through a single-precursor route by controlling the growth temperatureThe understanding of this self- assembling process will help us develop reliable and reproducible methods to synthesis other three dimensional nanostructuredarchitectures and increase the knowledge of self-assembly. As catalyst its nanoparticles of these nanoparticles of different sizes have been tested on the yield of production of biphenyl from the iodobenzene in reaction. The as obtained products were specified by powder X-Ray diffraction ,Scanning electron microscopy , Energy-dispersive X-ray analysis Spectra etc.

Keywords- Nanoparticles, Diffraction, Scanning Etc.

Introduction-

Therefore, the development of uniformation of particles has been intensively pursued. But the methods usually offer materials with poor optical and physicalproperties, as the nanocrystals can continue to grow slowly at now temperature via coarsening or epitaxial attachment because ofFor the preparation of nanoparticles with desired properties, it remains a challenge to explore robust pathway and purify principles towards the systematic manipulation of their size and shape. Nowadays the method of thermal decomposition of organometallic precursors in coordinating solvents is used more frequently, which results in relatively stable nanoparticles for organic monolayer protection. Synthesis of Mn anostructures has been activelyresearchedfor manydecades because Mn is an important industrial material due to its novel physicalandchemicalproperties.Amongallmetals used in modem electronic circuits, Mn is the most common oneTo date, several synthetic approaches- the microwave-induced method, the electrochemical synthetic method, the vapor deposition method , the sonochemical method, the solution-phase synthesis method, and so on, have been developed for the preparation of nanosized Mn. Mn is one of the most normal conductors in modern technologies and could be used widely in nano device.Many methods have been devotedtothe shape controlled synthesis of Mn nano crystals, nanoparticles, nanowires and nanorods .In this paper, we have reported thesynthesis of copper nanoparticles through a single- precursor route by controlling the growth temperature. The catalytic activities of these nanoparticles have been tested on synthesis of biphenyl from iodobenzene.

Material&Experimental-

Copper (II) acetylacetonate (Mn (acac)2, 99.99%),oleylamine(OA,80–90%),DMSO, CH_2Cl_2 were purchased from Aldrich and iodobenzen.

Synthesis of Mn nanoparticles

In a typical synthesis, about 0.001 mol Mn (acac)2 was added to the three necked flask, which contained 8ml oleylamine (OA) under Ar. The solution was heated to certain temperatures for the synthesis of Mn nanoparticles, the temperature is 220°C in a few minutes, and keptatthosetemperaturesfor6hours.Afterthe solutions were cooled to room temperature, nanoparticleswere obtainedbycentrifugation and washing. The as-prepared nanoparticles were dried in vacuum at 60°C for 12 hours for following characterization.

Synthesisofbiphenyl-

The Mn nanoparticles prepared through the above-said method were used for catalytic testing without furtheractivation.In a typical reaction, 50 mg of Mn nanoparticles were added to 1 ml of DMSOsolution containing 100 mg of iodobenzene and the mixture was refluxed at 200°C for5 h. After the required time the reaction system was cooled rapidly to room temperature and the product was separated by thoroughly mixing with 2 ml of saturated ammonium chloride followed by 2.5 mlof dichloromethane which separated two layerswith the upper layer containing the product dissolved in dichloromethane.

Result&Descussion-

The XRD pattern of copper nanoparticles for the freshly prepared sample at 300˚C. The peak positions coincide with those of metallic copper with a specific structure andconacience without regard for his own life : I shall never cease from the practice and teaching of philosophy. "So Socrates the man and philosopher amounts to a cardinal philosophical error. broadeningof the peaks due to crystallites with sizes in the nanometer scale is also observed. The Peaks are very sharp due to the high nano crystalline nature of Mn. valuesof 43.3˚, 50.4˚, and 74.1˚, corresponding to (111), (200), and (220) planes of copper, respectivelyθThree peaks at 2Peaks corresponding to Manganese Oxide were absent from this spectra.Shows SEM images of Mn nanoparticles. Copper nanoparticles by this method show the monodispersed distribution of particle sizes. The average particle sizes of the Mn nanoparticles were obtained around 50 nm with spherical shape. The composition of copper nanoparticles was further probed by energy-dispersive X-ray (EDX) analysis. In this spectra there are number of sharp peaks that indicate that copper nanoparticles were prepared. Shows the absorption spectrum of the copper nanoparticlesA modest blue shift of the absorption edgerelative to that of the bulk copper powder (600 nm) is observed. This observation alludes to the size effect of the copper nanocrystallites. On the other hand, the thermally prepared copper nanoparticles display a broad absorption from 550 to 700 nm.The broadness of the absorption peak probably stems from the wide size distribution The condensation of iodobenzene to biphenyl was chosen as the model reaction for catalytic activity due to its significant role in organic synthesis.When iodobenzene is heated at 200°C in presence of Manganese powder powder it forms diphenyl. Aryl halides couple among themselves to form diaryl derivatives, an important drug intermediate, in presence of Mn catalyst powder showed a 43% conversion of iodobenzene to biphenyl in 5 h underour experimental conditions.Sameamountof copper nanoparticles (size, ~ 50 nm diameter) showed 88% conversion of iodobenzene to biphenyl,whichincreasedtoabout95%when8nm diameter capped copper nanoparticles are used. Surprisingly, 5 nm size Mn nanoparticles showed no change in the yield of about 95% . The higher catalytic activity towards the condensation of iodobenzene could be due to the higher catalytic surface area of small nanoparticles. It is presumed that the electrophilic nature of the catalyst surface renders a weak bond between theπ-system of benzene ring and vacant d system of copper atoms, which gives slightly positive charge over iodine atom. The electrophilic nature of copper surface obviates that when the particles are extremely small in size, the electrons are pumped into copper by the benzene π-system, whichusuallyreducesthebandgapbetweenFermi Any aggregation of the particles in aqueous dispersion leads to lower efficiency. The freshly prepared and capped C, Mn nanoparticles also showed good activity for this reaction type of condensation indicating that any oxide layer formation on the particle surface perhaps inhibits the catalytic activity Table.Dependenceofyieldofiodobenzene(%)onsizeof the Mn nanoparticles

Particlessize(nm)	Approximateyield(%)
92	82
69	90
37	94
10	97
7	97
Uncapped Mn Particles of Undefined large Size	47

Conclusion-

In summary, Mn nanoparticles have been selectively synthesized by using Mn(acac)2 as precursor by controlling the growth temperature. Selective adsorption of OA on various crystal planes may play an important role in the growth process. Furthermore, the understanding of thisself-assembling process will help us develop reliable and reproducible methods to synthesisother 3D nanostructured architectures and increase the knowledge of selfassembly. Moreover, the synthesis route is mild and environmentally friendly. It is believed that this method will be re- considered toward the research in the nanometer regime. Also their chemical reactivity in the reaction of synthesis of biphenyl is proportional to their surface area, and their reactivity.

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