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Title of the Thesis: Synthesis, Characterization and Properties of Metal (Au and Ag)

Nanoparticles and Their Nanosized Oxides.

Abstract

Nanotechnology has experienced a rapid growth recently because nanostructures exhibit physical and chemical properties that are distinctly different from those of the bulk solid. In particular, metallic nanostructured materials have been the subject of intense scientific research by virtue of their fundamental importance and potential applications. To achieve these desired target properties, synthesis of nanoparticles with controlled size and morphology is critical. Here in this thesis we report the various important methods like microemulsion, sonochemical and solvothermal methods for the synthesis of silver and gold nanostructures and their oxides of desired morphologies and controlled size distributions.

Chapter 1 deals with the introduction about nanotechnology, nanomaterials, quantum size effect of nanomaterials arising due to their dimenstions. It also gives detailed knowledge about various aspects of synthesis of nanomaterials and different characterization techniques used for the identification of the synthesized nanomaterials.

Chapter 2 deals with the study of effect of surfactant type on the particle size, morphology and properties of the silver nanoparticle formed inside the microemulsions. TEM studies show the formation of cubes, spheres and disc shaped silver nanostructures with the size in the range from 8 nm to 40 nm, showing surface plasmon resonance (SPR) peaks between 400 nm and 500 nm. Surface area increases from 3.45 m^2/g to 15.06 m^2/g with decreasing the size of silver nanoparticles (40 nm to 8 nm).

Chapter 3 deals with large scale synthesis of silver nanoparticles using modified solvothermal method using ethanol as the refluxing solvent. TEM images show the formation of spherical and monodisperse silver nanoparticles of the average grain size of 5 nm with the surface area of 34.5 m^2/g. The percentage yield of silver nanoparticles was found to be as high as 98.5 %.

Chapter 4 deals with the study of effect of the reducing agents on the particle size, morphology and properties of the silver nanoparticles synthesized under sonochemical conditions using sodium borohydride and sodium citrate as the reductants. Strong reducing agent ($NaBH_4$) produced spherical silver nanoparticles of the average size of 10 nm where as weak reducing agent (Sodium citrate) produces silver nanoparticles of the average size of 3 nm. UV-Visible studies show the presence of surface plasmon resonance peak at 405 nm. Reflectance spectra give the broad band spreading between 340 and 360 nm which is characteristics surface plasmon resonance peak for the quasi

spherical silver nanoparticles. Citrate stabilized silver nanoparticles also give small peaks at 600 nm and 640 nm due to the higher order plasmon modes. The specific surface area was found to increase from 2.6 m^2/g and 13.1 m^2/g and pore radius decrease from 15.2 Å and 12.3 Å as the size of the silver nanoparticles decreases from 10 nm to 3.5 nm.

Chapter 5 deals with the effect of the gold salt ($HAuCl_4$) concentration on the particle size, morphology and surface area of the gold nanoparticles synthesized inside Tergitol and Triton X-100 as the surfactants. Particle size first increase from 20 nm to 25 nm and then decreases to 15 nm as the concentration of gold salt decreases from 0.05 M to 0.03 M inside Triton-X 100 microemulsions. UV-Visible spectroscopy shows the peaks of variable intensity around the main surface plasmon resonance band arising due to various types of plasmon resonance modes in gold nanocrystals. The surface area of the gold nanoparticles synthesized by the Triton X-100 based microemulsions decreases from 107 m^2/g to 3.45 m^2/g and then increases up to 16.70 m^2/g with decrease in the concentration of the gold salt in the aqueous phase.

Chapter 6 deals with the study of the effect of strength of the reducing agents on the particle size, morphology and properties of the gold nanoparticles synthesized by the solvothermal approach using tinchloride ($SnCl_2$) and sodium borohydride ($NaBH_4$) as the reductants. Tin chloride produced gold nanocrystals of the average grain size of 15 nm (s. s. area 269 m^2/g), while borohydride produced spherical gold nanoparticle of the average grain size of 7 nm having the highest surface area of 329 m^2/g.

Chapter 7 deals with the sonochemical synthesis of the gold nanoparticles using tinchloride and sodium borohydride as the reductants. TEM images show the formation of gold nanodiscs of average disc diameter of 30 nm (s. s. area 179.5 m^2/g) using tinchloride and where as sodium borohydride produced gold polyhedral nanocrystals in the size range of 20 to 40 nm having the surface area of 150.6 m^2/g.

Chapter 8 deals with the synthesis of silver oxide (Ag_2O) and gold oxide nanoparticles using sonochemical, solvothermal and microemulsion routes. TEM studies show spherical silver oxide nanoparticles with average size of 5 nm (s. s. area 19.68 m^2/g) and 8 nm (s. s. area 12.6 m^2/g) prepared by the sonochemical and solvothermal methods respectively. However, microemulsion method results in the formation of non spherical silver oxide nanoparticles of the size of 10 nm and 40 nm by using Tergitol and Triton X-100 surfactants respectively. UV-Visible spectroscopy shows the bands at 400 nm and 420 nm corresponding to the surface plasmon resonance of silver in silver oxide nanoparticles. Various chemical methods were also used for the preparation of gold oxide nanoparticles but none of the methods used were successful in synthesizing the target material.

In Annexure gold and silver nanoparticle were synthesized by the biosynthetic route using cell free extract of the fungus, Candida albicans. The nanoparticle interactions with proteins were confirmed by FT-IR spectroscopy and thermal gravimetric analysis (TGA). TEM micrographs show the formation of gold and silver nanocrystals of average size of 5 nm and 30 nm respectively. The formation of gold and silver nanoparticles was confirmed by the appearance of a surface plasmon band at 540 nm and 450 nm respectively. The specific surface area was found to be 18.87 m^2/g for gold nanoparticles and 187.35 m^2/g for silver nanoparticles.