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Nanotechnology: Principles and Practices

Third Edition

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Chapter 14
Practicals

14.1 Introduction

In this chapter we shall briefly discuss some easy ways to perform experiments which will enable the beginners to get some experience in the synthesis of nanomaterials and characterization. We shall mention the equipment required and outline the procedure along with some results. These experiments were actually performed in the author's laboratory and are possible to perform in any moderately equipped laboratory without heavy investment.

For chemical synthesis discussed here, simple and inexpensive set of equipment is required. As shown in Fig. 14.1, a multiple neck, round bottom, glass flask can be used. Chemical synthesis reaction is generally carried out under some inert gas (N_2 or Ar) atmosphere to avoid uncontrolled or undesirable oxidation of the nanoparticles by constant flow of the gas in the round bottom flask. Dropwise addition of the reacting solution is carried out. A centrally inserted refluxer in the flask also helps in some cases. One can use other necks for introducing temperature measuring devices (e.g. a thermometer), pH electrode, gas insertion device etc.

After preparing the appropriate precursor solutions by proper weighing/measuring and using appropriate volumes of the chemicals, reactions can be carried out following the steps given in the flow charts.

The nanoparticles are usually separated out from the liquids as a precipitate by using a centrifuge (approximately 2,000–3,000 r.p.m. speed). Precipitate should be washed using appropriate solvents like water, acetone, ethanol or methanol to remove unreacted chemicals or byproducts. Precipitate can be then dried to get sample in the form of powder.

Characterization of samples can be done using some of the techniques given below. The basic principles of characterization techniques were discussed in Chap. 7. All these techniques may not be available/required for high quality research but sometimes observation of colour change and techniques like optical absorption spectroscopy and X-ray diffraction are good enough to indicate the formation

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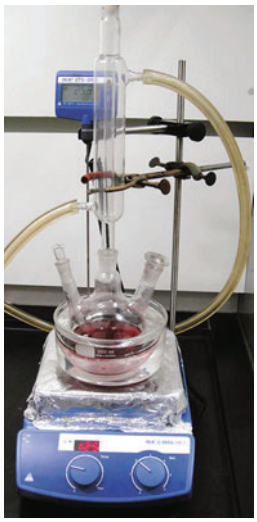


Fig. 14.1 A typical set-up for synthesizing nanoparticles by chemical method

of nanomaterials. Transmission Electron Microscope (TEM), Scanning Electron Microscope (SEM), and photoluminescence are further useful techniques available at many places.

Usually for optical absorption investigations, nanoparticle sample dispersed in a liquid is used. For transmission or scanning electron microscopy (TEM or SEM) or other microscopies a drop of liquid is placed on suitable grid or substrate and liquid is allowed to evaporate before the measurements are carried out. In some other analysis like photoluminescence, X-ray diffraction (XRD) powder samples are preferred.

It may be noted that the following procedures are only illustrative and only to give one a flavour of nanotechnology experiments and are not necessarily the best possible procedures for obtaining particular nanoparticles. One can follow the literature and obtain better materials following some more stringent conditions or procedures.

14.2 Synthesis of Gold/Silver Nanoparticles

Aim: To synthesize metal nanoparticles of gold and silver.

14.2.1 Chemicals

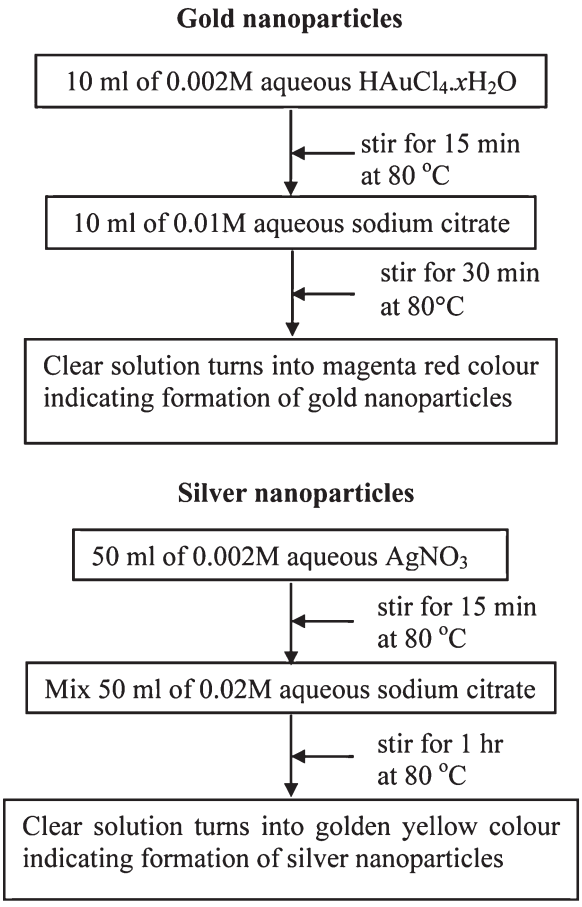
1. Chloro auric acid ($HAuCl_4$) for gold particles
2. Silver nitrate ($AgNO_3$) for silver particles
3. Trisodium citrate ($C_6H_5O_7Na_3$)
4. Double distilled water

14.2.2 Equipments

1. Round bottom flask
2. Magnetic stirrer cum heater
3. Optical absorption spectrometer (~250–700 nm)

14.2.3 Synthesis Procedure

Procedures of gold and silver nanoparticles synthesis are given in the flow chart form. Synthesis can be carried out using the glass apparatus or set up as shown in Fig. 14.1.



14.2.4 Results

The magenta red and yellow colours for gold and silver solutions respectively indicate the formation of nanoparticles. Changing the concentrations, reaction time, temperature etc. one can obtain different shapes/sizes of the particles. This changes the solution colour or shades. There is large literature on these aspects. Typical photograph of gold and silver particles obtained using above procedure are shown in Fig. 14.2a.

Optical absorption spectra can be recorded using a simple absorption spectrometer discussed in Chap. 7. Figure 14.2b illustrates typical spectra obtained for the synthesis described here. It can be seen that peak for silver appears at approximately 396 nm and that for gold at about 530 nm. These are Surface Plasmon Resonance (SPR) peaks discussed earlier.

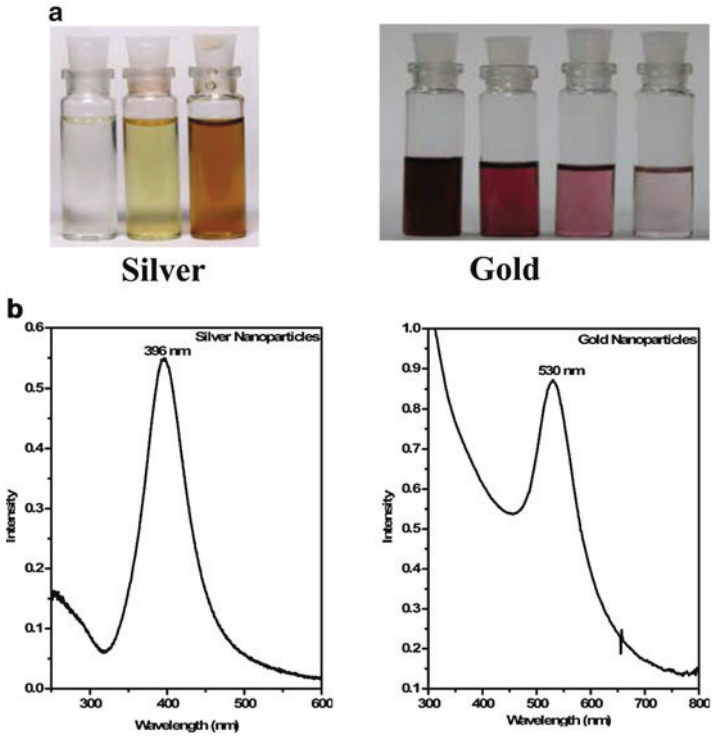


Fig. 14.2 (**a**) Gold and silver nanoparticles in solution. (**b**) Typical absorption spectra of silver and gold nanoparticles