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Topic: Synthesis of monometallic nanoparticles and their usagefor the removal of CR dye and heavy metals

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This chapter includes the synthesis of monometallic nanoparticles via various approaches such as simple reduction, polyol, and co-precipitation methods and their utilization for the removalof dyes and heavy metals. We have discussed the instability of monometallic particles and theirfailure to remove dyes and heavy metals.

Introduction

In recent decades, nanomaterials have attracted wide attention of the research fraternity due totheir diverse industrial applications in the fields of spintronics, microelectronics, sensors, magnetic storage materials, optoelectronics, catalysis, conducting paints and biomedical technology [1]. Transition-metal nanoparticles such as gold, silver, platinum and palladium have already stabilized in the most important areas such as catalysis and the medical industry [2]. But the high cost restricted their use on a large scale and forced the research community toexplore other cheaper metallic nanoparticles. From the 3d-transition series, Co and Ni have attracted wide attention due to their ferromagnetic nature and possibility to be potentialcandidates in the fields of high-performance magnetic storage materials and superconductivity.Recently, a superconducting phase was also found in antimony crystals [3]. The pure Co has special significance in technology due to its existance in both hexagonal (HCP and α-cobalt) as well as cubic (FCC or β-cobalt) structures [4-5].

The properties of nanomaterials are highly dependent on their size and shape which is ultimatelyrelated to the synthetic route. So, various synthesis procedures have been used for the synthesisof Co, Ni and Sb nanoparticles. Song et al. prepared Co nanoparticles via a microfluidic route [1]. Guo et al. synthesized Co nanoparticles in both fcc and hcp phase using ethanol hydrazinealkaline system (EHAS) at room temperature [4]. The Co nanoparticles were also synthesized by the reduction of Co $(NO_3)_2$ [5] and Bis (2-hydroxyacetophenato) cobalt (II) [6] precursors, by supercritical methanol and thermal decomposition method, respectively. The spherical Ni nanoparticles were prepared by using nickel chloride as metal precursor and hydrazine as reducing agent in presence of stabilizing agent CTAB (cetyltrimethylammoniumbromide) under

inert atmosphere [2, 7]. Self-assembled and uniform flower-like microstructure were synthesized by hydrazine based reduction of nickel chloride in the presence of polyvinylpyrrolidone (PVP) in ethylene glycol under microwave irradiation [8]. Also, Ni

Nanoparticles were synthesized by reduction of nickel acetylacetonate using sodiumborohydride in presence of hexadecylamine (HDA) and trioctylphosphine oxide (TOPO) as capping agents [9]. But the synthesis of antimony nanoparticles are not explored much. To best of our knowledge, these nanoparticles have never been used for the adsorptive removal of organic dyes and heavy metals.

Until now, metallic nanoparticles are synthesized using highly expensive techniques or expensive metal precursors and stabilization agents. Our idea is to synthesize metal nanoparticles via simple routes and with relatively cheap metal precursors and use them to remove hazardous substances such as organic dyes and heavy metals from aqueous solutions.

Result and discussion

XRD analysis: The cobalt nanoparticles were synthesized by two different routes polyoland simple reduction approach. The XRD pattern of the as synthesized particles is displayed in

Fig. 1. The results clearly indicates that crystallization of Co nanoparticles is dependent on

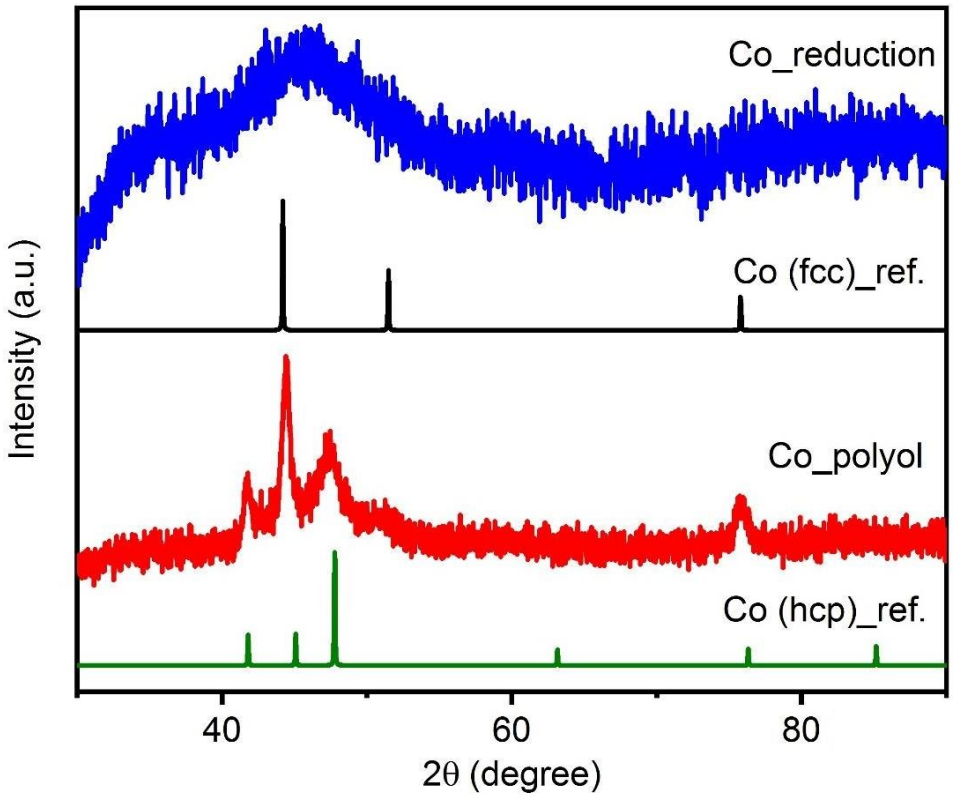

Fig. 1. XRD pattern of Co nanoparticles synthesized by polyol &simple reduction approach.

Synthetic procedure as we got α-cobalt (HCP) and β-cobalt (FCC) nanoparticles on the synthesisvia polyol and simple reduction approach, respectively. The broad hump of peak in case of Conanoparticles of redcution method is either due to small particles size or amorphous nature of particles. But, the broadness of peaks in polyol cobalt is due to small size particles as intensityof peaks indicates the crystalline nature. Both reduction and polyol cobalt are well consistent with FCC (ICDD # 04-015-0419) and HCP (ICDD # 04-007-7985) structures, respectively. Butthe variation in the relative intensity of peaks in polyol cobalt compared to reference data, indicates the preferred orientation along the (002) plane.

Similarly, Ni and Sb nanoparticles were synthesized by reduction and co-precipitation approach, respectively and resulted pattern are given in Fig. 2. The broad hump in case of Ninanoparticles is probably due to small size particle formation in reduction approach like Co particles above. But the position of hump is in good agreement with the reference peaks (ICDD #04-015-2543) which suggests the formation of pure nickel nanoparticles. Also, it suggests thecrystallization of particles in cubic (CCP) structure in space group 225 (Fm-3m). The XRD pattern generated from Sb particles, clearly indicates the high cystalline nature of the particleswhich can be justified based on the high temperature treatment in co-precipitation synthesis method. The insignificant broadning or less FWHM (full width at half maximum) of peaks suggests the larger size of particles as compared to Co and Ni. The relative intensity and peakspositions of peaks are well consistent with reported data in rhombohedral structure with sapce

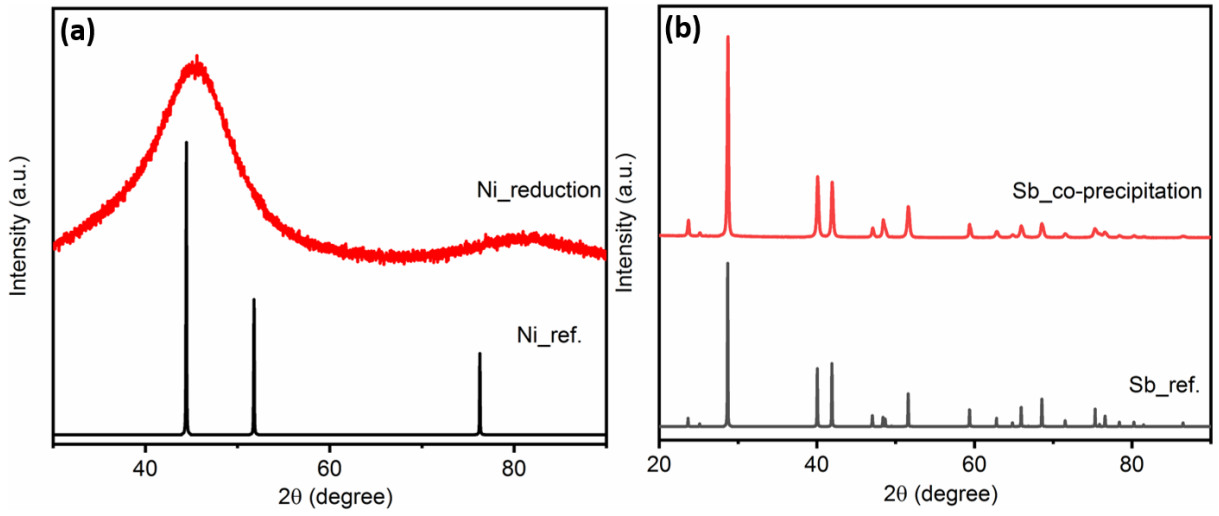

Fig. 2. XRD pattern of as synthesized Ni nanoparticles synthesized by reduction method (a)& Sb nanoparticles synthesized by co-precipitation approach (b).

group 166 (ICDD #04-014-2871). The absence of any external peak clearly suggest the pure phase synthesis of antimony particles.

Congo red dye removal: From preliminary investigation of the particles using XRD, wehave confirmed the synthesis of Co, Ni, and Sb nanoparticles. As we found broad peak in somecases indicates smaller particle size and ultimately larger surface area, therefore, we have usedCongo red dye as the synthesized particles for removal. Adsorption experiments were carried out separately with equal amounts (1 g L^{-1}) of Co, Ni and Sb nanoparticles as adsorbents alongwith CR dye of 5 mg L^{-1} concentration. The experiment was continued for a contact time of 5 hours at room temperature and the residual concentration of CR dye was determined by UV- visible spectroscopy and the resulting data are depicted in Fig. 3. The spectra clearly indicate that the particles synthesized by reduction approach are comparatively good for CR dye removal as Ni and Co nanoparticles adsorbed 40 and 36 % dye, respectively (Fig. 3.3b). The lowest removal efficiency of 12.5% was achieved with Sb particles, which may be due to the low surface area due to the large particle size. Overall, monometallic particles (Co, Ni, and Sb)were not found to be more effective for CR dye removal.

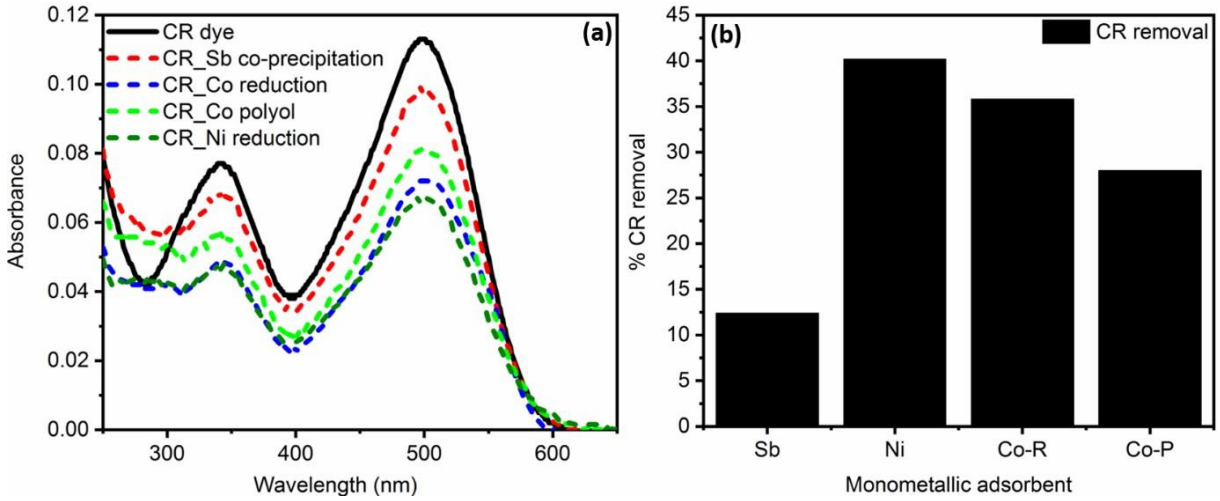

Fig. 3. Uv-Visible spectra of congo red dye (black) along with treated ones with differentmonometallic adsorbents (a) & coresponding removal efficiency of CR dye (b).

Cr (VI) and Pb (II) removal: The as-synthesized particles were used for the sorbent removal of heavy metals Cr (VI) and Pb (II) from aqueous solutions. Adsorption experiments were conducted separately for all metal nanoparticles of the same volume of 1 g L^{-1} for a contacttime of 24 h with Cr (VI) and Pb (II) solutions of 5 mg L^{-1} concentration. The obtained results were displayed in Fig. 3.4, which indicates that Co and Ni nanoparticles are better than Sb particles for heavy metal removal. A maximum removal efficiency of 30 % was achieved for

Ni nanoparticles after 24 h, which indicates the inefficiency of monometallic nanoparticlestowards heavy metal removal.

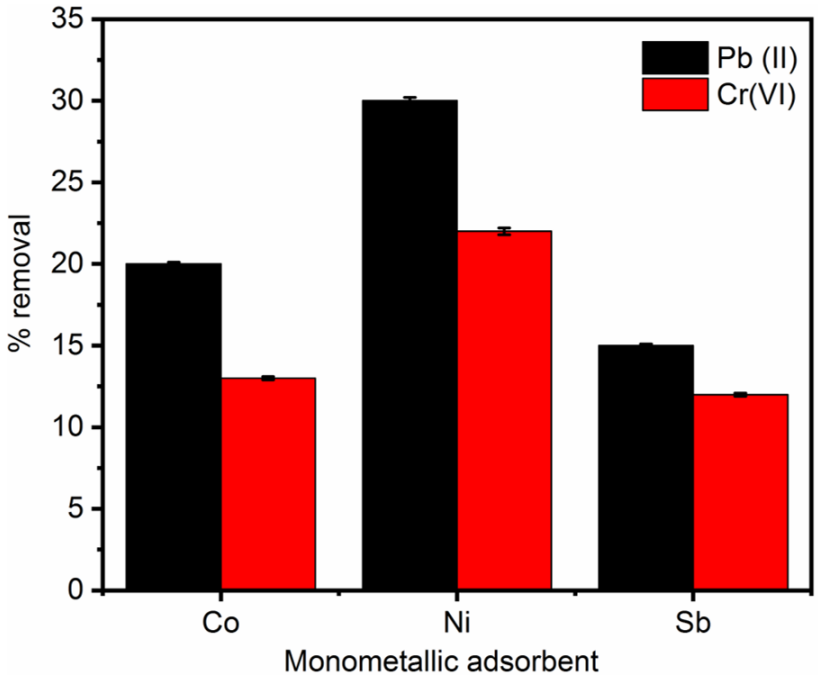

Fig. 4. Percentage removal of Cr (VI) and Pb (II) after the treatment with monometallic nanoparticles.

Conclusion

To summarize, we have successfully synthesized monometallic nanoparticles (Ni, Co, and Sb)via cost-effective reduction, polyol, and co-precipitation approaches without using expensive stabilization and coating agents. The obtained results confirmed the inefficiency of Ni, Co, andSb nanoparticles for the removal of organic Congo red dye and the adsorption of heavy metalsCr (VI) and Pb (II). Overall, this study suggests that monometallic nanoparticles can be used toadsorb organic dyes and heavy metals, but much more potentization is required for better results.

Therefore, after various tests we found that monometallic nanoparticles are not more effectivefor adsorbent removal of dyes and heavy metals. Therefore, we have decided to focus our further research work on the synthesis, characterization of biometallic (antimony based intermetallics) particles and their employment for the removal of hazardous materials from simulated wastewater.

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