



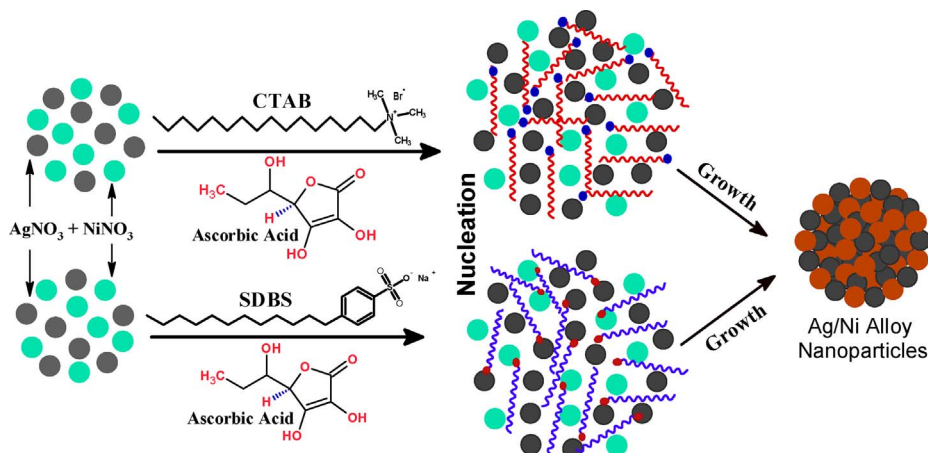
Role of surfactants: One step facile synthesis of hetero structured Ag-Ni alloy by seed less approach



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GRAPHICAL ABSTRACT



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ABSTRACT

Surfactant assisted seedless synthesis of Ag-Ni alloy nanoparticles were carried out in an one step process by co-reduction of mixed metal salts ($\text{AgNO}_3 + \text{Ni}(\text{NO}_3)_2$) solutions using ascorbic acid as reducing agent. To explore the role of surfactant, two types of surfactants were studied; cationic cetyltrimethylammonium bromide (CTAB) and anionic sodium dodecylbenzenesulphonate (SDBS). The surface plasmon resonance peak (SPR) of CTAB capped Ag-Ni and SDBS capped Ag-Ni located at 450 nm and 410 nm wavelengths, respectively. For SDBS, visual observations and time resolved spectra indicates that the reaction mixture containing different mixing orders of reactants (SDBS + $\text{Ag}^+ + \text{Ni}^{2+} + \text{ascorbic acid}$ and SDBS + $\text{Ni}^{2+} + \text{Ag}^+ + \text{ascorbic acid}$) have different colours as well as different SPR band positions with time. Presence of SDBS delayed the nucleation processes. The Ag-Ni alloy with both surfactants shows that the a red shift to ca. 410 nm and 450 nm with respect to the SPR of AgNPs. The absence of SPR bands at ca. 375 nm and 500–700 nm ruled out the possibility to the formation and /or deposition $\text{NiO} + \text{Ni}(\text{OH})_2$ on the surface of Ag-Ni alloy. The average number of Ag-Ni atoms per nanoparticle, molar concentration of Ag-Ni and free energies (surface, volume and excess energy due to mixing) were calculated and discussed. The Ag-Ni alloy used as a catalyst to the photo-assisted degradation methyl orange and showed a higher photo catalytic activity. The synthetic method reported here suggests a very promising use of the suitable stabilizer for the preparation of an alloy nanoparticles having different optical properties, which is a subject of immense interest.

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1. Introduction

Synthesis and characterization of mono-, and bi-metallic nanomaterials (BNMs) have been the subject of various investigators around the different continents from two decades due to the tremendous applications in chemistry, physics, biology, technology, and in all areas of industry [1–12]. Bimetallic NMs exhibits superior catalytic and sensing properties compared to their mono-metallic counterpart due to new bifunctional or synergetic effects [13–15]. El-Sayed et al. in his pioneering review suggested that enhanced applications depend on the morphology (shape, size, composition, structure, and surface chemistry) of the NMs [16]. Among the various BNMs, Ag@Ni, Ni@Ag and Ag-Ni alloy had attracted great attraction due to their very valuable properties, pronounced lattice mismatch between the Ag and Ni metals, lower surface energy of Ag, complete immiscibility, and large difference between their reduction potentials ($\text{Ag}^+(\text{aq}) + \text{e}^- \rightarrow \text{Ag}(\text{s})$ and $\text{Ni}^{2+}(\text{aq}) + 2 \text{e}^- \rightarrow \text{Ni}(\text{s})$ are + 0.7994 V and -0.25 V in an acidic solution) [17–20]. For example, Prieto et al. [21] used derived seed-mediated growth method for the synthesis of bimetallic core-shell Ag@Ni by utilizing indium tin oxide modified Ag NPs as seeds. Wu et al. synthesized Ag-Ni alloys NPs by reducing AgNO_3 and $\text{NiSO}_4 \cdot (\text{H}_2\text{O})_6$ in the presence of poly(N-vinylpyrrolidone) and NaBH_4 as a stabilizing and reducing agent, respectively [22]. Nouneh et al. reported the synthesis of AgNPs and NiNPs from seed growth solution reduction process by using NaBH_4 as reducing agent in presence CTAB and poly(N-vinylpyrrolidone) [23,24]. Wu and Chen also reported the synthesis of pure NiNPs by the hydrazine reduction of Ni^{2+} ions in ethylene glycol at 60 °C. They also suggested that the presence of NaOH was necessary for the formation of NiNPs [25]. In the seed-growth method, first step includes preparation of a seed solution, in which metal salts are reduced with a strong reducing agent in water. The growth solution was prepared by the subsequent reduction of other metal salt with a weak reducing agent in the presence of a suitable stabilizer [11]. Generally, NaBH_4 was used as a reducing agent to the preparation of seed solution. An aqueous NaBH_4 solution is unstable and alkaline in nature due to the formation of NaOH after hydrolysis [26].

It has been established that the seedless co-reduction method is a simpler process than seeded growth and the most seedless methods can proceed in an one reaction vessel [27–29]. Recently we have prepared Ag-Cu core-shell NPs by reducing Ag^+ and Cu^{2+} ions with ascorbic acid in presence of CTAB solution at 25 °C, and found that Ag-Cu exhibited an SPR band at 425 nm and no SPR was observed for CuO or Cu_2O at 800 nm [30]. Chen et al. pointed out that the synthesis of alloy NPs was relatively easy than the core-shell NPs synthesis and their properties can be tailored by altering the metal ions composition [31]. To avoid the formation of metal hydroxide due to the use of NaBH_4 and NaOH in the seeds growth solution, the metal NPs must be synthesized in acidic media. In this study, we aimed to use seedless method to control the optical properties and morphology of Ag-Ni BMNPs by the ascorbic acid co-reduction of Ag^+ and Ni^{2+} in presence of cationic CTAB and anionic SDBS. We did not observe any SPR band at ca. 300–400 nm related to the oxidized Ni NPs as well as a broad absorption band at 500–700 nm related to the absorption of nanostructures containing Ni^{2+} ions for SDBS capped Ag-Ni NPs.

2. Experimental

2.1. Materials

Nickel nitrate hexahydrate ($\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, molar mass = 288.90 g mol⁻¹, purity = 99.9%), silver nitrate (AgNO_3 , molar mass = 169.87 g mol⁻¹), ascorbic acid ($\text{C}_6\text{H}_8\text{O}_6$, molar mass = 176.12 g mol⁻¹), cetyltrimethylammonium bromide ($\text{C}_{19}\text{H}_{42}\text{BrN}$, molar mass = 364.45 g mol⁻¹), sodium dodecylbenzenesulfonate ($\text{C}_{18}\text{H}_{29}\text{NaO}_3\text{S}$, molar mass = 348.48 g mol⁻¹), sodium hydroxide (NaOH, molar mass = 40 g mol⁻¹), and inorganic electrolytes

(NaCl, NaBr, Na_2SO_4 and NaNO_3) were purchased from BDH and used as received without further purification. All solutions were prepared with deionized double distilled water on molarity basis.

2.2. Instruments

UV-vis spectra (from 200 to 800 nm range) were recorded on a Shimadzu UV-vis multi Spec-1501, spectrophotometer with 1cm quartz cuvettes. Fourier Transform infra-red, FT-IR spectra (from 40,000 to 500 cm⁻¹) were measured with a Bruker Tensor II FT-IR spectrometer, USA using the KBr pellet technique (few drops of as prepared sols were placed on KBr pellet and allowed to dry). Transmission electron microscopy images of BMNPs were obtained with a JEOL, JEM-1011, Japan, transmission electron microscope operating at 120 kV equipped with energy dispersion X-ray spectrometer (EDS). The surface characteristics (XRD) of Ag@Ni were detected with a Fison (VG) ESCA 210 spectrometer using a monochromic $\text{MgK}\alpha$ radiation. The pH of the aqueous solutions was adjusted by standard HCl and NaOH solutions. To diminish the formation of $\text{Ni}(\text{OH})_2$, all experiments were performed in a neutral-acidic media at pH ca. 5–6, unless otherwise specified.

2.3. Synthesis of silver-nickel alloy nanoparticles

The seedless chemical reduction method was used for the synthesis of Ag@Ni nanoparticles. In a typical experiment, An equimolar aqueous (0.01 mol dm⁻³) solutions of $\text{Ag}(\text{NO}_3)$ and $\text{Ni}(\text{NO}_3)$ in CTAB and/or SDBS were used as precursors, and were added to ascorbic acid solution (10 cm³ of 0.01 mol dm⁻³). The progress of the reaction, formation of perfect transparent sols under different conditions (SDBS + Ag^+ + Ni^{2+} + ascorbic acid, SDBS + Ni^{2+} + Ag^+ + ascorbic acid and CTAB + Ag^+ + Ni^{2+} + ascorbic acid) was monitored by recording the UV-vis spectra at different time intervals. As the reaction proceeds, the appearance of pale yellow, light brown to chocolate color, indicating the formation of Ag-Ni sols. The resulting sols has the SRP band at ca. 400 nm, and 450 nm with SDBS and CTAB surfactant stable for at least one months, and no precipitation and/or agglomeration was observed. For the characterization, the particles were separated from the solutions by centrifugation at 20, 000 rpm for 30 min. The resultant product was washed with deionized water.

They were then re-suspended in double distilled water and centrifugation was repeated twice so as to remove the impurities and stored in a dark glass bottle.

3. Results and discussion

3.1. Role of surfactant in the preparation of Ag-Ni alloy

The stability implies individually dispersed NPs and long term stability with out agglomeration or precipitation in aqueous media. Due to large surface to volume ratio, NPs tend to aggregate in solution in order to reduce their surface energy. The presence of suitable stabilizer is another morphology-controlling factor in the synthesis of NPs. The stabilizer does not only control the size and shape of NPs but also stabilized the colloids in solvents and prevents the metal surface from further oxidation [32]. To examine the influence of surfactant, reaction solution containing AgNO_3 , $\text{Ni}(\text{NO}_3)_2$, ascorbic acid (all 5.0 cm³ of 0.01 mol dm⁻³ + required amount of water for dilution), and different [CTAB] and [SDBS] surfactants were prepared. Figs. 1–3 present the optical absorption characteristics of the Ag-Ni sols in the UV-vis region as a function of time. It is evident that the resulting sols has quite a different extinction profile under our experimental conditions. The appearance of peak depends on the reaction time. If the ascorbic acid solution was added into reaction mixture (SDBS + Ni^{2+} + Ag^+) at room temperature, the color of the reaction solution did not changed for ca. 2h, which implies that the reduction of Ag^+ ions and/or Ni^{2+} ions did not occur under this mixing order (Fig. 1; UV-vis spectra). At

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