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Full Length Research Paper

Synthesis and characterization of Nickel hydroxide/ oxide nanoparticles by the complexation-precipitation method

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In the present research work, the synthesis of Ni $(OH)_2$ and NiO nanoparticles with narrow size distribution and uniform shape, by complexation-precipitation method using ammonia as complexing agent, was investigated. The reactions were carried out at the temperature about 70°C in the solution and the precipitated nickel hydroxide precursor, in the form of the nanosized soft agglomerates, was calcined at 300 and 400°C. The hydroxide and the oxide samples were then characterized by XRD, SEM, and IR spectroscopy. The NiO particles obtained after calcination at 300°C, could preserve the size and shape of the precursor material, whereas, the one obtained after calcination at 400°C showed a slight change in the size and morphology of the particles. In general, the results indicate that using this method, $Ni(OH)_2$ and NiO nanoparticles, with homogeneity in size and shape, suitable for various applications, can be prepared.

Key words: Complexation-precipitation, Ni hydroxide/oxide, nanoparticles, synthesis, characterization.

INTRODUCTION

Nanostructured materials have been extensively explored for the fundamental scientific and technological interests in accessing new classes of functional materials with unprecedented properties and applications (Wu et al., 2007; Neuberger et al., 2005; Schiffrin, 2001; Ramesh et al., 2006; The-Long et al., 2008). In recent years, there has been an increasing interest in the synthesis of nanosized crystalline metal oxides because of their large surface areas, unusual adsorptive properties, surface defects and fast diffusivities (Ying et al., 2007). Nickel oxide (NiO) is a very important material extensively used in catalysis, battery cathodes, gas sensors, electrochromic films, and magnetic materials (Yoshio et al., 1998; Moon et al., 1995; Alcantara et al., 1998; Wu et al., 2008; Miller and Rocheleau, 1997; Gabr et al., 1992) . Recently, several methods have been developed to prepare ultrafine nickel oxide powder, including low-pressure spray pyrolysis (Lenggoro et al., 2003), surfactant-mediated method (Wang et al., 2002), simple liquid phase process (Wang et al., 2005) and other techniques (Tao and Wei, 2004; Wang and Ke, 1996; Cherrey et al., 2002 Souza et al., 2007). In many of them, the main objective is to producue non-agglomerated nanoparticles with reduced costs and technological applications (Souza et al., 2007).

In the present research work therefore, synthesis of $Ni(OH)_2$ and NiO nanopowders by the complexation-precipitation method using ammonium hydroxide, as the complexing agent has been investigated. This method has been found to be simple, cheap, capable of being scaled up, which would result in production of nanosized $Ni(OH)_2$ powders as well as NiO nanoparticles with homogeneity in size and shape, making the products as suitable compounds for application in rechargeable batteries, heterogeneous catalysts and etc.

EXPERIMENTAL

Materials

All the chemical reagents used in this study were of analytical grade and are used without further purification. Distilled water was used

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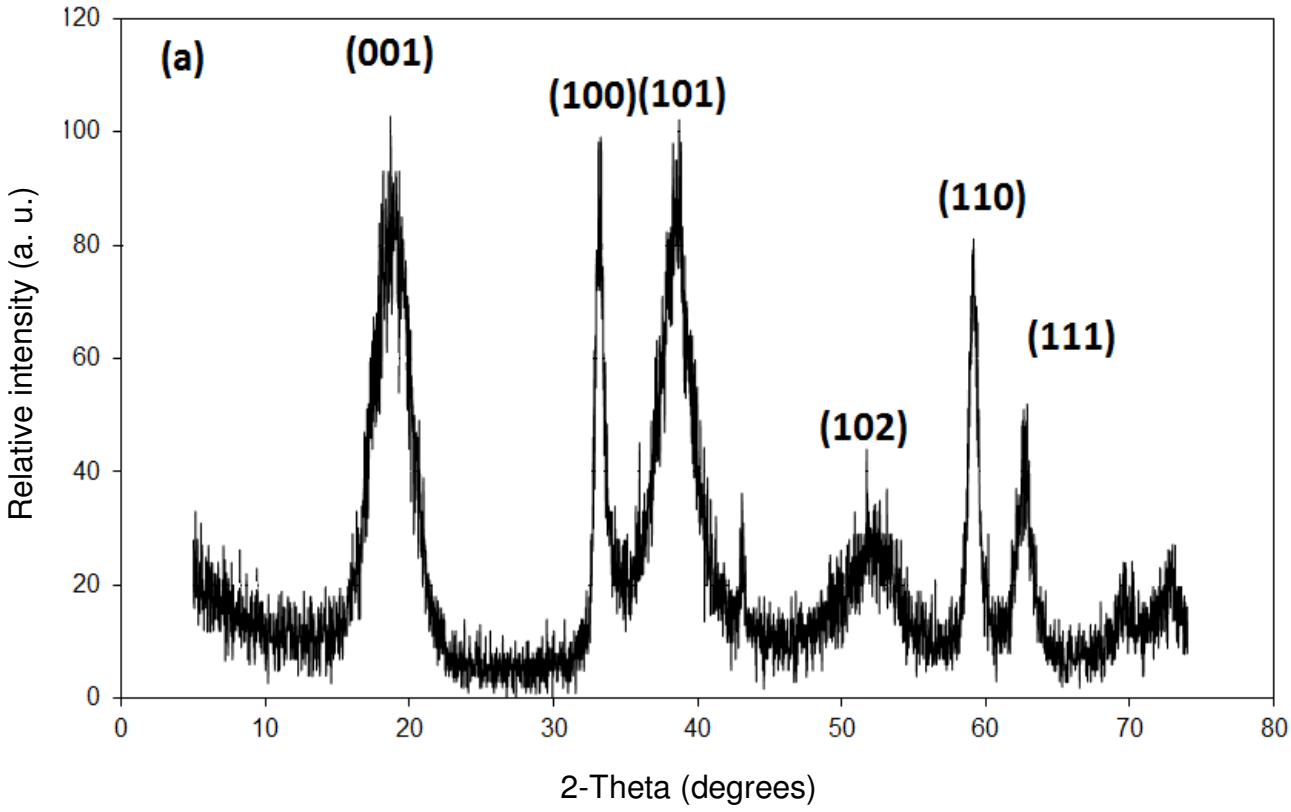
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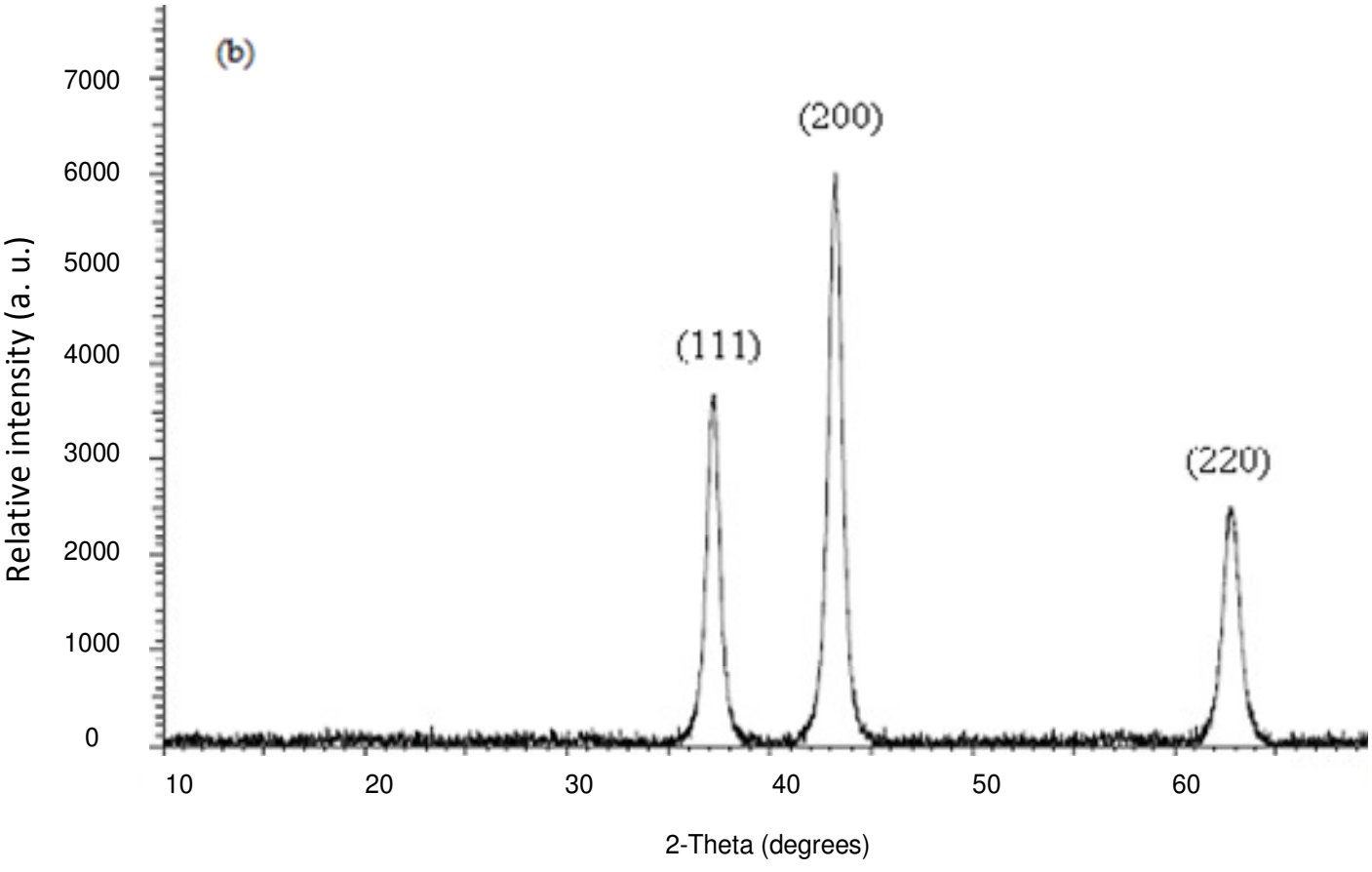
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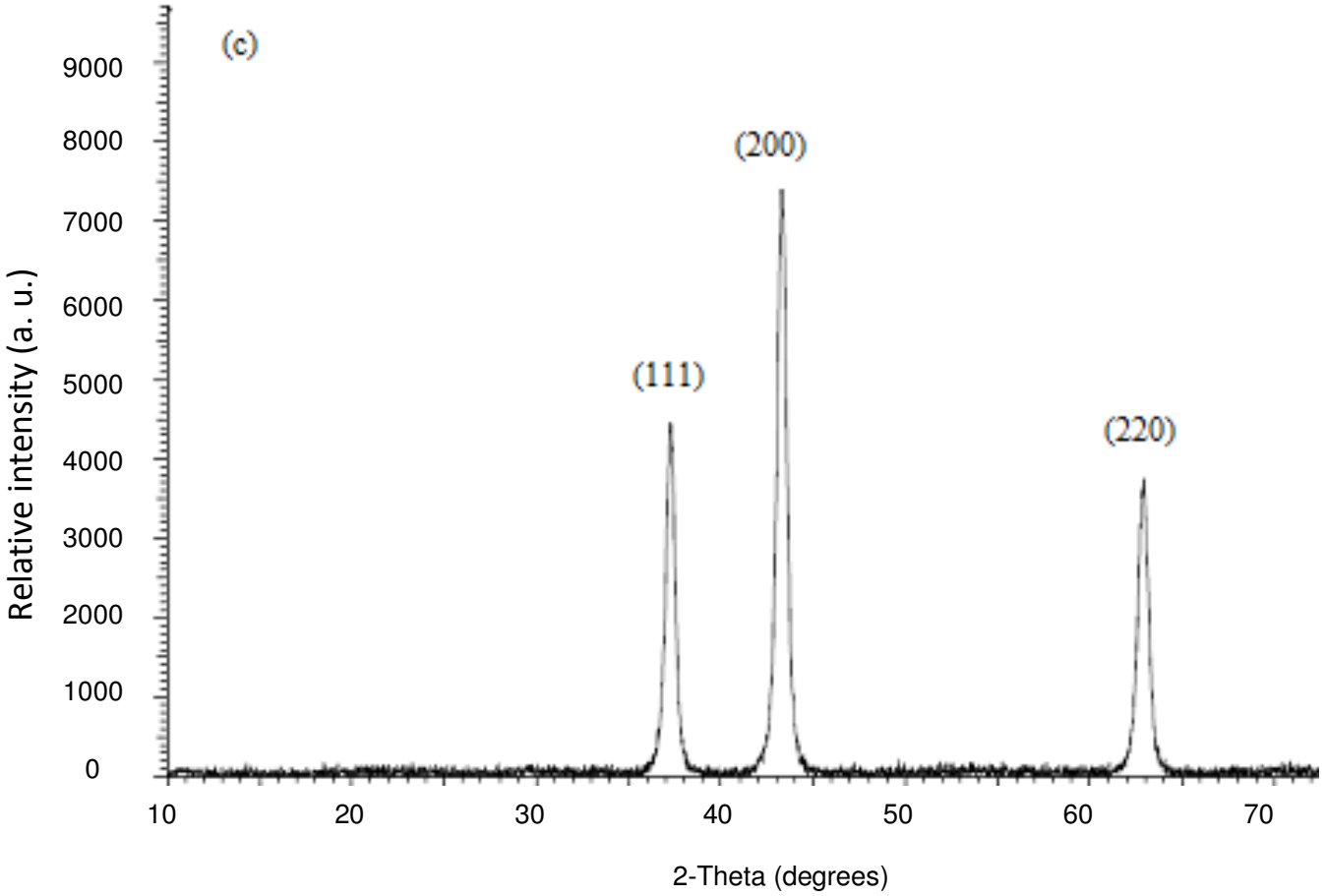

Figure 1. XRD patterns of the precursor (a), and calcined powders at 300 (b), and 400°C (c).

throughout the experiments.

Preparation method

100 ml of 2 M NH_4OH solution was added drop wise into 50 ml of 0.5 M $Ni(NO_3)_2.6H_2O$ solution which was stirred by magnetic stirring apparatus (about 1000 rpm) at 70°C temperature. The resultant light-green suspension was filtered, and then washed with deionized water and ethanol in molar ratio of 1:1 for 5 to 10 times. It was then dried at 70°C for 24 h, and finally calcined at 300 and 400°C for 2 h.

Characterization techniques

X-ray diffraction (XRD) measurements were performed using a JEOL-8630 diffractometer with Cu $K\alpha_1$ radiation at 35 kV and 20 mA with a scanning speed in 2θ of 4°min^{-1}.

The infrared spectra were obtained by a SHIMADZU spectrum FT-IR-8400S spectrometer in the range of 400 to 4000 cm^{-1}. One milligram of each powder sample was diluted with 100 mg of vacuum-dried IR-grade KBr.

The surface morphology of the precursor and nickel oxide nanoparticles was observed by means of a scanning electron microscope (Philips XL30) operated at 30 kV.

RESULTS AND DISCUSSION

The XRD patterns of the precursor and the oxide products (after calcination) are shown in Figures 1a to c. The peaks positions and relative intensities obtained for the hydroxide precursor (Figure 1a match with the JCPDS card No: 1-1047 file, identifying it as β-$Ni(OH)_2$ with a hexagonal structure. The peaks seem to be appreciably broad which indicates the crystallites of the hydroxide can be in the nanosized range. Both the XRD patterns of the calcined samples at 300 and 400°C, shown in Figures 1b and c, match the JCPDS card No: 71-1179 file which indicates the formation of pure NiO product. However, it can be observed that, by increasing the calcination temperature up to 400°C , the peaks have been appreciably sharpened which indicates that a growth in the crystallite sizes of NiO has occurred.

The IR spectrum of the precursor shown in Figure 2a exhibits absorption peaks as follows. The broad absorption band centered at around 3500 cm^{-1} could be due to the O-H stretching vibrations, which is the characteristic of β-Ni(OH)2. The band at 1631 cm^{-1} can be attributed to the bending vibration of water molecules.

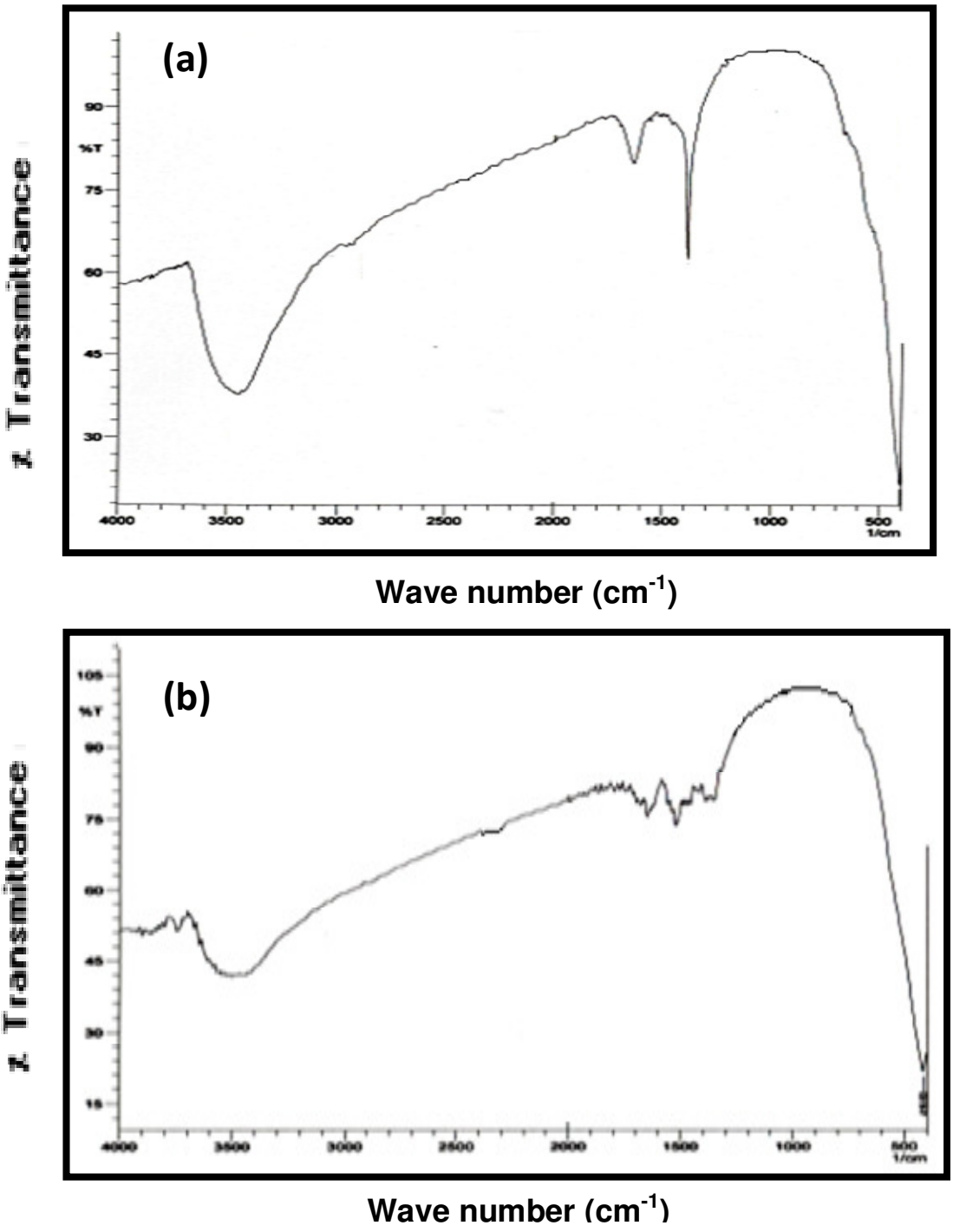

Figure 2. The IR spectra. (a) Precursor and (b) calcined powder.

The band at 1384 cm^{-1} is due to the presence of CO_3^{2-} anions. After calcination (Figure 2b), the strong band at 412 cm^{-1} corresponds to the vibration of Ni-O bond. The bands at 3445 and 1384 cm^{-1} in (Figure 2b) are due to the fact that the calcined powders tend to physically absorb water and carbonate ions, respectively.

The results of SEM study for the precursor and the calcined samples are presented in (Figure 3). The SEM image of the precursor $Ni(OH)_2$ product, clearly shows the uniform particles in the form of nanoflakes having a diameter about 500 nm and thickness less than 50 nm. The SEM micrographs of the calcined samples are shown in Figure 3b and c. The interesting to note is the similar morphology and size for the NiO particles

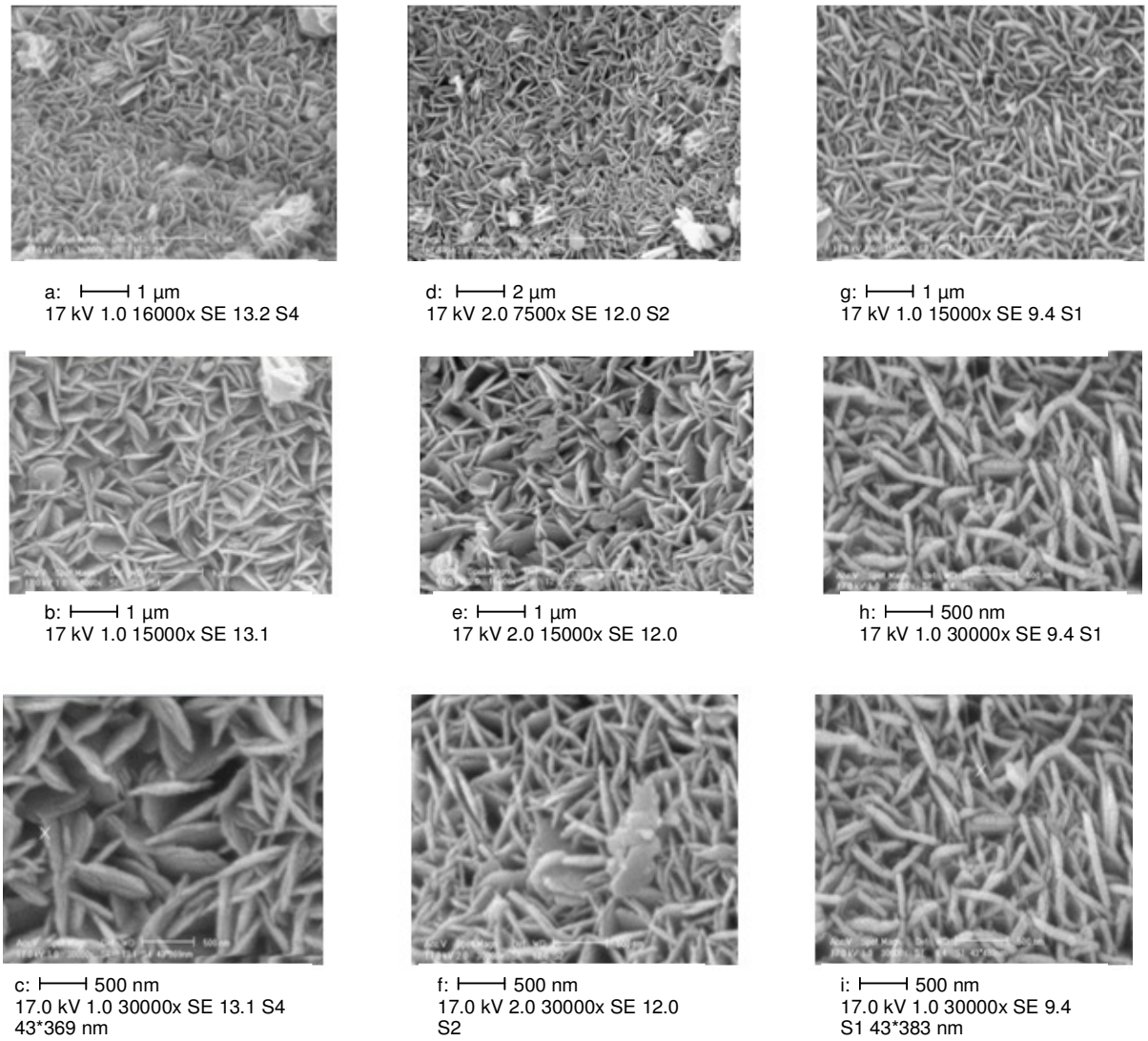

Figure 3. The SEM image of (a, b, c) precursor and calcined powder at (d, e, f) 300, and (g, h, i) 400°C.

obtained after calcinations at 300°C. However, calcination at 400°C, has led to a change in the particles shape and size, such that, almost a sort of nanoworms, can be observed.

Conclusion

$Ni(OH)_2$ and NiO nanoparticles could be successfully prepared by complexation-precipitation method using ammonia as the complexing agent. The XRD along with the IR spectroscopy results indicated the formation of β-$Ni(OH)_2$ product. The SEM image of the precursor $Ni(OH)_2$ product, clearly shows the uniform particles in the form of nanoflakes having a diameter about 500 nm and thickness less than 50 nm. These nanoflakes, in fact, may consist of nanoparticles of much smaller sizes, namely crystallites, where could be observed by TEM study. The interesting to note is the similar morphology and size for the NiO particles obtained after calcination at 300°C however, calcination at 400°C, has led to a change in the particles shape and size. The results clearly indicate that by using the present method with choosing the appropriate process conditions, nickel hydroxide and oxide nanoparticles, having various homogeneities in size and shape for different applications can be effectively prepared through a low cost and high yield process.

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