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Synthesis of Ni Nanoparticles by Pulsed Laser Ablation Method in Liquid Phase

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Abstract

Laser ablation in liquids has been intensively studied in recent years, due to present numerous potentials in laser material micro-processing, including nanomaterials and nanostructures synthesize. Compared to others, typically chemical methods, pulsed laser ablation (PLA) in liquid is a simple and "green" technical method that normally operates in water or organic liquids under ambient conditions. Here, pure Ni nanostrutures were synthesized using PLA method in 30 mL of acetone. A simple fiber pulsed laser setup has been not only employed to reduce the micron Ni particles to nano-sized by the PLA method, but also with no-induced oxidation at room temperature and free additive in liquid. The particle size, spectral analysis and morphology of the products were characterized by UV-vis spectrometry, energy-dispersive X-ray spectroscopy (EDX) and transmission electron microscopy (TEM). The TEM image indicates spherical shapes with a narrow size distribution, compared with other methods, with ~10 nm in diameter respectively.

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Keywords: Pulsed laser ablation; liquid phase; green; Ni nanostructures; oxidation; free additive.

1. Introduction

The synthesis method is vastly applied within the recent decade and has been improved. Nickel (Ni) nanostructured materials, such as Nanospheres (NSs), nanorods (NRs), nanowires, and nanotubes, have attracted applications in fields

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such as pharmaceutical synthesis, Khurana and Yadav (2012), magnetic biocatalysis, Bussamara et al. (2013), biomolecular separation, Lee et al. (2004), and biosensor, Kalita et al. (2012).

Numerous methods such as sputtering, Akamaru et al. (2012), solution glow discharge, Saito et al. (2010), PLA, Kalita et al. (2012), reversed micelles, Calandra (2009), thermal decomposition, Gonzalez et al. (2012), and wet chemical reduction, Ma et al. (2009) have been developed for the preparation of Ni nanoparticles (NPs). Among other methods, the novel PLA method is favorable. This safety approach is able to optimize and control the NPs in term of structures, morphologies, sizes and size-distribution. The single-step process is facile, environment benign, reproductive and yielding high-quality Ni powder and colloidal forms, Colombier et al. (2005). High-yield, trustable, controllable, simple and fast is the main characteristics of this method, which makes it favorable over other methods. Additionally, the proper method can even affect the surrounding conditions of the nanoparticle generation. Therefore, this method was chosen to prepare nickel nanoparticles, with using absolutely no additives in liquid and without the oxidation event. Here we reported the synthesis of nickel NPs using PLA method in acetone, using powder source of micron size. UV-vis spectrometry was employed to check and measure particle size. The synthesized nickel NPs were characterized by energy-dispersive X-ray spectroscopy (EDX) for spectral analysis, and transmission electron microscopy (TEM) to access the size and morphology of the nanoparticles.

Nomenclature

C_{ext}	Extinction cross-section of the spheres
Q_{ext}	Extinction efficiency factor
R	Radius of sphere
ε_m	Dielectric constant of the surrounding medium
λ	Wavelength of the plasmon peak

2. Experimental Procedure

To synthesize pure nickel NPs via the laser ablation method, 10g of the nickel powder (Nilaco, 5μm powder size, 98% purity) was poured into a beaker containing 30 mL of acetone. The solution was then uniformly mixed by electric agitator during laser irradiation. During the ablation process, a 1070 nm of the fibre pulsed laser was focused on the suspension mixed Ni and acetone, inside the container. The used laser power was 0.4 W, leading to fluence of 4.8 J/cm^2, regarding the 72 μm spot size of the beam diameter on the Ni samples inside the container. To avoid scattering the laser beam and disturbing the ablation process, the laser parameters pulse were maintained and the laser irradiation did not exceed one hour. A schematic diagram of the experimental setup for the Ni nanosphere generation is shown in fig. 1. Before starting the ablation processing, the experimental setup and the conditions were optimized as shown in the authors' recent work, Ganjali et al. (2011).

The synthesized Ni NPs (8.4 g) were collected by a suction filter on a Büchner funnel, and well transferred to a filter paper. To ensure the stability of the synthesized Ni NPs; as one of the most active metals; it is vital to dry them safely to avoid an induced oxidation effect. Therefore, it is preferred to store them only under argon gas, as a shield during the ablating process, in a glove box.

The structural analysis of Ni NPs was carried out by the means of UV-Vis spectrometer (Perkin-Elmer 550 ES). The nanostructure of the synthesized samples were observed and analyzed by Transmission Electron Microscopy (TEM) equipped with an Energy-Dispersive Spectroscope (EDX) to assessing the formation of Ni NPs and determine the elemental analysis or chemical characterization of the Ni suspension respectively.

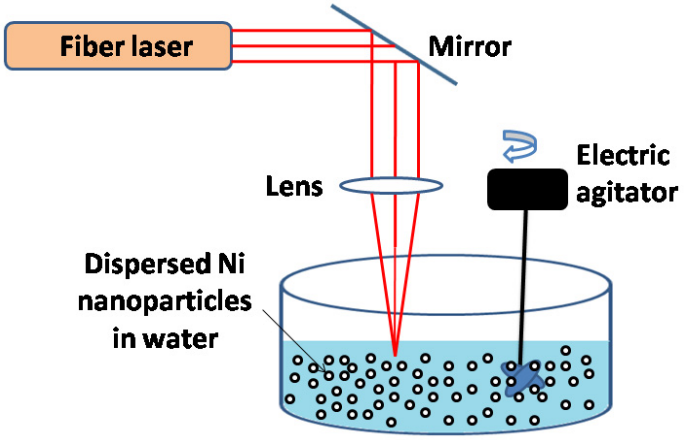

Fig. 1. Schematic diagram of the experimental setup.

3. Results and Discussion

The optical properties of Ni NPs in acetone medium as indicated in fig. 2 (dot line) were studied via UV-Vis spectrometry. The conduction band electrons of the metal NPs are coherently oscillated during the electromagnetic field's interactions with the sample. The oscillation of electromagnetic field can result in the changing of colour in the metallic NPs colloid, named Surface Plasmon Resonance (SPR), Zeng et al. (2014). Agreeing with Mie's theory, and previous work, Creighton and Eadon (1991); the SPR absorption of the colloidal Ni NPs was exhibited 350 nm in the UV–Vis spectrum. A blue shift of the absorption occurs with decreasing size of the Ni NPs while the increase of the kinetic curve is sensitively detected for a stable product. Conversely, as the Ni nanosize increases, the slope of the obtained straight line decreases due to linearization of UV-vis spectroscopy. Moreover, the Peak width significantly depends on the width of distribution of Ni nano sizes, i.e. a wider peak causes wider size distribution of NPs. Light interaction with NPs strongly depends on the size, shape, composition, and medium container. Haiss and colleagues could determine size and concentration of gold NPs throughout UV-vis spectra using Mie's theory, Haiss et al. (2007). Mie theory is the exact solution to Maxwell's electromagnetic field equations for a plane wave interacting with a homogenous sphere of radius R with the same dielectric constant as bulk metal. The extinction cross-section (C_{ext}) of the spheres can be obtained as a series of multipole oscillations if the boundary conditions are specified. The extinction efficiency factor Q_{ext} is defined as the ratio of the extinction cross-section to the physical cross-sectional area (πR^2). It is a sum of both scattering and absorption. In a more specific case, when the diameter of the spherical particle is much smaller than the wavelength of the radiation ($2R << l$) and only dipole oscillation contributes to the extinction cross-section, the electrodynamic calculation could be simplified via ignoring high order terms. This gives the most popular form of Mie theory for spherical particles:

$$C_{ext} = \frac{24\pi^2 R^3 \varepsilon_m^{3/2}}{\lambda} \frac{\varepsilon_2}{(\varepsilon_1 + 2\varepsilon_m)^2 + \varepsilon_2^2} \quad (1)$$

The dielectric constant of the surrounding medium, (ε_m) and $\varepsilon = \varepsilon_1 + i\varepsilon_2$- the complex dielectric constant of the metallic particle are respected. From equation (1), a resonance peak occurs while the condition of $\varepsilon_1 = -2\,\varepsilon_m$ is satisfied. This is the SPR peak which accounts for the brilliant colors of various metallic nanoparticles.

Figure 2 (square line) shows the extinction spectra calculated based on Mie's theory for Ni nanospheres synthesized by the PLA method. The complex refractive index n (i) for bulk nickel was taken from the experiment by Johnson and Christy (1974). This spectrum also shows a maximum at 300 nm to 400 nm, which is quite close to the present result for the Ni NPs in acetone medium. By replacing the values for parameters of the Ni NPs and the acetone as a medium,

the plasmon peak value of 350 nm with the size radius average of 10 nm were approximately defined (shown in fig. 2).

Known spherical NPs with only one oscillation, a transverse oscillation, Avasthi et al. (2010) is detected in the UV-visible absorbance curve with one peak for nanosphere shape particles. A high field intensity absorbance is illustrated during SPR, Zijlstra et al. (2012). Therefore, the difference between the theoretical and experimental curves in the Fig 2, is an evidence to prove the created spherical nanonickel particles.

The typical Ni NPs is demonstrated by the TEM image, as shown in fig. 3a. It is clearly indicated that the Ni particles synthesized by the PLA method, in nano scale, narrow size distribution and spherical shapes. Also, the diameter of Ni NPs was measured from digital images using "ImageJ" software. Thus, a size average of the spherical Ni NP was ~ 10 nm. The histograms of this data were calculated as shown in the fig.3b.

The EDX spectrum result in the fig. 4 confirms formation of the pure Ni NPs synthesized with no oxidized material. According to previous study, Zijlstra et al. (2012), the EDX and the UV-visible results of synthesized Ni NPs, the authors claim that the PLA with a versatility setup is proper approach to fast prepare a metallic NPs with no concentration of material oxides.

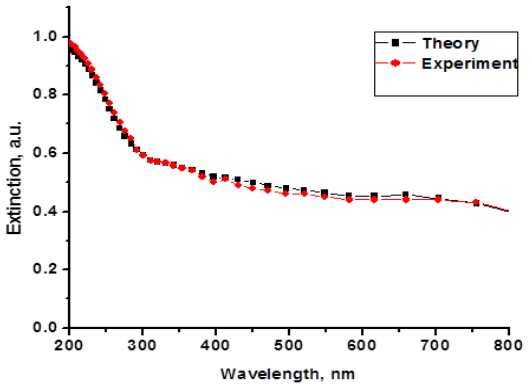

Fig. 2. UV-vis spectrum of the Ni NPs.

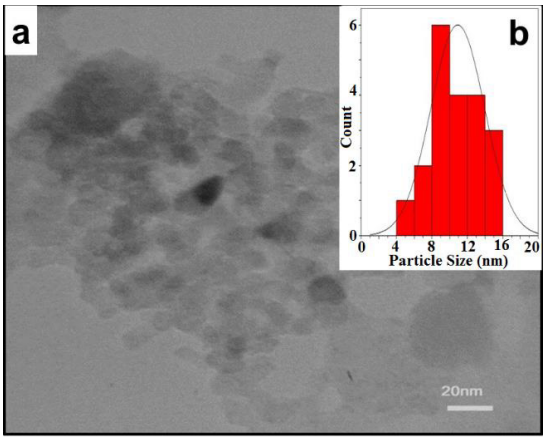

Fig. 3. (a) TEM image; (b) histograms of the Ni NPs.

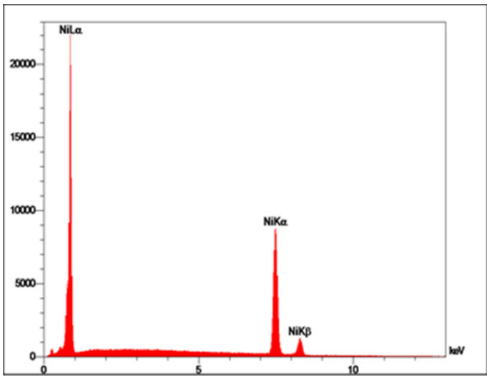

Fig. 4. EDX spectrum of the Ni NPs.

4. Conclusion

The stabilized and purified nickel NPs were synthesized into a nanospherical shape and a narrow size distribution via the PLA method while dispersing the microsized Ni powder in the liquid phase, acetone. The described method can accurately provide alternative versatility, controllable, predictable, repeatable, sensitive, precise and simple technique, compared with conventional ones. These characterization techniques (UV-Vis absorbance, TEM images and EDX) suggest that synthesized particles, nanosphere, are pure nickel without any oxidation. The colloidal or powder prepared Ni NPs are reliable materials and convenient to use. Moreover, the PLA method is successfully able to synthesis the Ni NPs without the use of surfactants or other additives.

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