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Synthesis of nickel nanoparticles by pulsed laser ablation in different liquid media

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Abstract. Nickel nanoparticles exhibit superior ferromagnetic properties. This has attracted great interest due to its significant potential applications in various fields. The properties of nanomaterials are greatly affected by the environmental conditions during the synthetization process. In this work, nickel nanoparticles were synthesized by pulsed laser ablation in distilled water, ethylene glycol, chitosan, gum Arabic and walnut oil. For all samples, the ablated process was conducted for 5 minutes. The optical properties of colloidal nanoparticles were investigated by UV-Visible spectroscopy and Fourier Transform Infrared (FT-IR) spectrometer. Data from experimental works released that walnut oil has highest absorbance intensity which might be contributed to the existence of alkene functional group.

1. Introduction

Nickel nanoparticles have been studied extensively for their superior ferromagnetic properties, including chemical stability, magneto-crystalline anisotropy, and high coercive forces [1]. In recent years, Ni nanoparticles have proven significant potential in a wide range of applications such as battery manufacturing [2], textile industry [3], enhanced pseudo-capacitance [4] and catalyst [5]. There are several methods to synthesize nanoparticles such as electrochemical mechanical milling [6], solvothermal [7], electrolysis [8] and others [9-11]. Pulsed laser ablation in liquid (PLAL) is a promising technique due to its simplicity, require less sample preparation and free from contaminations [12-13]. Moreover, PLAL could control the size, shape and crystallinity of nanoparticles by varying the laser parameters such as laser energy, pulse duration, applied number of shots and wavelength [13][14]. Synthesis solvent is also influenced the nanoparticles properties [15] [16]. The laser parameters as well as solvent properties would lead to the changing of physical and chemical properties of the nanoparticles [17-18] due to the different liquid properties such as refractive index and viscosity [27]. Various nanoparticles have been synthesized in different liquid media such as ethanol [19], de-ionized or distilled water [20], hexane [21], ethylene glycol [22], and acetone [23]. As for nickel nanoparticles, researchers used deionized (DI) water, methanol, hexane, acetonitrile [24] [25] and acetone [26]. Therefore, this work was conducted to synthesize nickel nanoparticles in distilled water, ethylene glycol, chitosan, gum

Arabic, and walnut oil using PLAL technique to investigate the effect of solvent properties towards the properties of nanoparticles. Later, the colloidal NiO nanoparticles (NiO-NPs) were characterized using UV-Vis spectrophotometer and FTIR spectroscopy. The optical properties of the NiO-NPs on various solvents were evaluated. Results were analyzed, discussed, and compared with other works.

2. Methodology

2.1 Synthesis of nickel nanoparticles

A Nickel (Ni) plate with dimension of 10 mm X 5 mm x 1.5 mm with purity of 99.95% of Ni was used as a target. The Ni plate was polished using sandpapers with three different grit sizes; 800, 1200, and 2400. Then, the plate was washed with acetone and ethanol medium to remove the superficial oxide layers on the target surface in order to prevent the simultaneous oxidation.

Ethylene glycol (Merk Co.), chitosan, walnut oil, and gum Arabic (purchased from local stores) as well as distilled water were used as laser ablation media. The solutions will be prepared in-house. Chitosan and gum Arabic with mass of 0.2 g and 15 g respectively were dissolved in 100 ml distilled water at 60-degree Celsius and stirred for 60 minutes until completely dissolved.

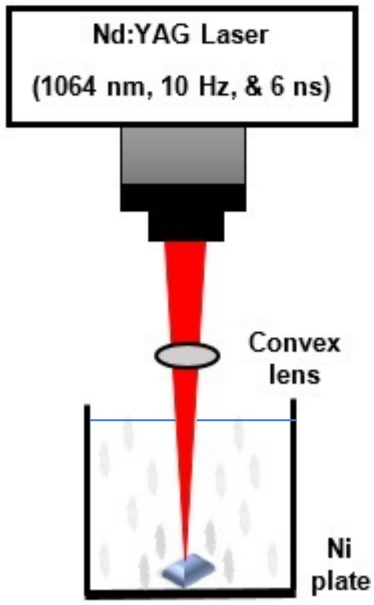

Figure 1 Synthesis of Nickel nanoparticles using Nd:YAG laser.

Figure 1 depicts a schematic diagram of the experimental setup. A pulsed Q-switched Nd:YAG laser (Quantel laser) with wavelength of 1064 nm, pulse duration of 6 ns and repetition rate of 10 Hz was used to generate pulses to ablate the Nickel target. The laser emits pulses with output energy around 15 mJ/ pulse. The Nickel plate was immersed in 10 ml of liquid media and ablated by focusing the incident laser using a 100 mm focal length lens. The ablation process happened at room temperature (27 ^{0}C) for 5 minutes. Nickel nanoparticles was synthetized in different liquid media which are distilled water, ethylene glycol, chitosan, walnut oil, and gum Arabic. Table 1 provides the samples code and their viscosity.

Table 1 The code and viscosity of samples.

Sample code	Liquid Medium	Viscosity of the sample (mPa)

A1	Distilled water	1
A2	Ethylene glycol	16
A3	Chitosan	22
A4	Gum Arabic	37
A5	Walnut oil	43

2.2 The mechanism of nanoparticles production

The nickel colloidal nanoparticles are produced due to the interaction between a high energy laser pulses with the nickel target. This interaction produced seed electrons through multiphoton ionization. Later, a plasma plume containing ablated material is produced and expanding into the surrounding environment. This followed by a shockwave emission. The hot plasma plume is then cooled down and energy is released into the solution environment. Next, a cavitation bubbles is generated, expanded, collapsed and later generated a second shockwave. During the plasma cooling stage, nanoparticles are produced and diffused into the surrounding liquid and forming a colloidal solution [28].

2.3 Characterization of nanoparticles

A UV-Vis spectrophotometer (UV-3600Plus Series) with wavelength range of 220 to 800 nm was used to record absorption spectra of the colloidal nanoparticles. The measurements employed a quartz cuvette with optical path length of 10 mm. FTIR spectra of all colloidal samples were recorded using a PerkinElmer Frontier FT-IR Spectroscopy with wave number range of 650 – 4000 cm^{-1}.

3. Results and discussions

Figure 2 shows samples which are distilled water, ethylene glycol, chitosan, gum Arabic and walnut oil after being ablated for 5 minutes using laser. It is obvious to observe light grey particles in all samples. During the ablation process, the samples gradually from transparent and became opaque and after five minutes, the colour changed to light grey and silver. This colour changes indicate the formation of Nickel nanoparticles in the liquid media.

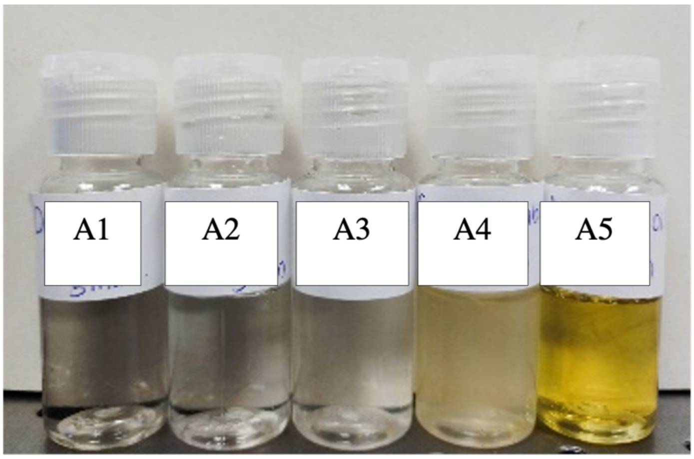

Figure 2 The sample after ablated for 5 minutes in (A1) distilled water, (A2) ethylene glycol, (A3) chitosan, (A4) gum Arabic, and (A5) walnut oil.

3.1 UV-Visible spectroscopy (UV-VIS)

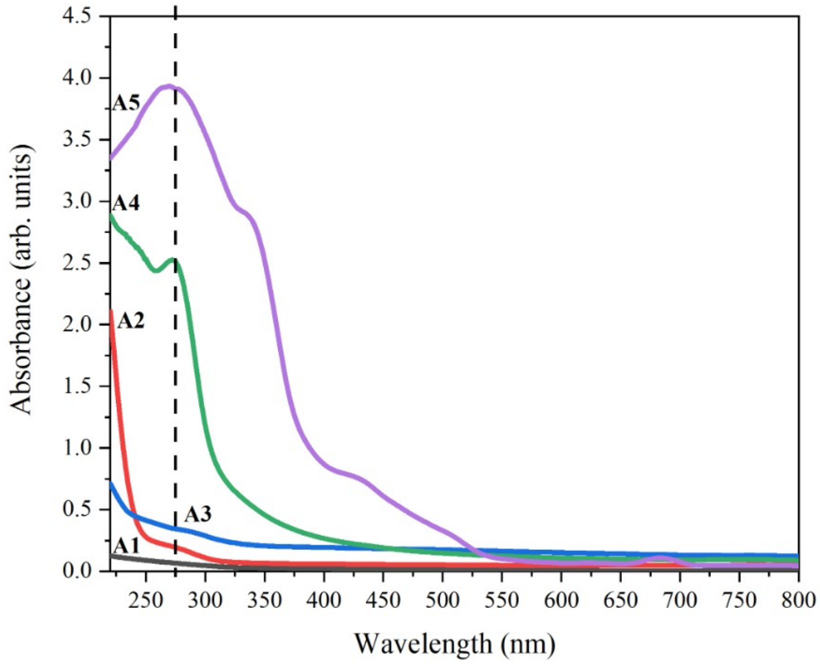

Figure 3 The absorption spectra of nickel nanoparticles in (A1) distilled water, (A2) ethylene glycol, (A3) chitosan, (A4) gum Arabic, and (A5) walnut oil.

Figure 3 indicates the optical properties of colloidal nickel nanoparticles in distilled water, ethylene glycol, chitosan, gum Arabic, and walnut oil which were obtained using UV-Vis spectrometry. The surface plasmon resonance (SPR) absorption of the colloidal Ni NPs in gum Arabic and walnut oil are exhibited at around 275 nm. Previous work shows similar absorption peak of Ni NPs in ionized water [29]. Absorption peak of walnut oil is more intense and broader than others. While the absorbance peak of gum Arabic is sharper and narrower. According to Ali *et al*, the variation of absorption peak intensities and positions indicates the various morphology of the nanoparticles [15]. However, very small absorbance peak is observed in distilled water, ethylene glycol, and chitosan spectra. Table 2 below tabulates the viscosity and absorption peak intensity of all sample. Walnut oil which has higher viscosity produced a highest absorption peak intensity than other solvents. Thus, this indicates the sample's viscosity plays role in formation and expansion of plasma.

Table 2 The absorption peak and viscosity of samples

Sample	Viscosity of the sample (mPa)	Absorption Peak (arb. units)
Distilled Water, A1	1	0.068
Ethylene Glycol, A2	16	0.198
Chitosan, A3	22	0.347
Gum Arabic, A4	37	2.519
Walnut Oil, A5	43	3.922

3.2 Fourier-Transform Infrared spectroscopy (FTIR)

The colloidal Ni NPS was characterized using FTIR to investigate the functional group of the samples. The FTIR spectra in Figure 4 are spectra of Ni nanoparticles synthesized by pulsed laser ablation in distilled water, ethylene glycol, chitosan, gum Arabic, and walnut oil respectively. The spectra were recorded in the range of 500-4000 cm^{-1}. Several significant absorption peaks can be observed. The IR spectra A1-A4 show broad absorption band at range of 3298 cm^{-1} to 3325 cm^{-1} which corresponds to O-H stretching bands that attributed to absorbed water or hydroxyl groups of nanoparticles [12]. Colloidal Ni NPs in Ethylene glycol, A3 and walnut oil, A5 spectrum show another significant absorption band at 2854 and 2939 cm^{-1} which indicates the complex vibrations of CH + CH2 + CH3 groups [32][33]. Medium absorption band is recorded at 1737 to 1743 cm^{-1} that related to C=O stretching indicates the ester carbonyl function group of triglycerides [33]. As can be seen, in walnut oil, A5 spectrum the stretching vibration shift is sharp and appear at 1743.48. The shifted upwards mainly due to carbonyl, cyclic ketones and carboxyl groups [34]. Another strong O-H bending mode is observed in A1, A3, and A4 spectra at absorption band between 1635 -1638.18 cm^{-1} which corresponds to the secondary group of OH. A low absorption peak at 1459 cm^{-1} is recorded in walnut oil spectrum that indicates the bending vibration of CH_3, corresponds to alkene functional group [35]. Absorption region between 900 cm^{-1} to 1400 cm^{-1} is the fingerprint region. Another OH bending is observed in spectra A1-A4 at region 1365-1366 cm^{-1}. Spectra A1 and A3 show absorption peak at 1217 cm^{-1} which indicates the C-O stretching vibration which corresponds to ether functional group. Similar functional group can be identified at 1205 cm^{-1} and 1083 cm^{-1} for sample A2 and 1160 cm^{-1} for sample A5. In sample A4, small absorbance peak can be observed at 1074 cm^{-1} which indicates the presence of galactan [36]. This is the characteristics peak of gum Arabic and fingerprint of carbohydrates [37]. In walnut oil, the stretching vibrations of CH=CH is observed at 966 cm^{-1} and 722 cm^{-1} which corresponds to alkene functional groups.

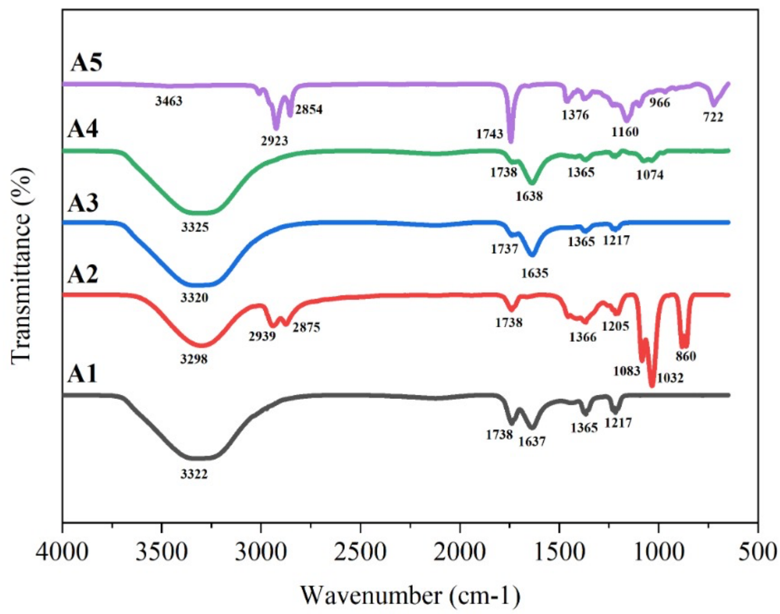

Figure 4 FTIR spectra of Ni-NPs in (A1) distilled water, (A2) ethylene glycol, (A3) chitosan, (A4) gum Arabic, and (A5) walnut oil.

4. Conclusion

Pulsed laser ablation in liquid technique successfully synthesized nickel in distilled water, ethylene glycol, chitosan, gum Arabic and walnut oil. The colour changes of the solvents after ablation indicates the presences of the nickel nanoparticles. Next, the effect of different solvents on the production of nickel nanoparticles by laser ablation has been investigated using UV-visible spectrophotometer and FTIR spectroscopy. Based on the UV-visible spectra, distilled water, ethylene glycol and chitosan demonstrated poor production of nanoparticles, while walnut oil indicates the best solution. The SPR peaks are observed at 275 nm which indicates the presence of nickel particles in the solutions. Ni NPs in walnut oil demonstrates the highest absorbance intensity followed by Ni NPs in gum Arabic. This indicates the solvent properties affecting the Ni NPS production. This is confirmed by the FTIR analysis where Ni NPS in walnut oil and gum Arabic demonstrates unique absorbance peak at 966 cm^{-1} and 722 as well as 1074 cm^{-1} which indicates the presence of alkene functional group and galactan respectively. Thus, the high absorbance intensity in UV-visible spectrum of Ni NPs in walnut oil might be contributed by the existence of the alkene group. Thus, it is concluded that the solvent properties have a significant influence in growth of nickel nanoparticles.

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