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Time Dependent pH-Controlled Biosynthesis and Characterization of Nickel Nanoparticles using *Moringa oleifera* Leaf Extract

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Abstract

Biogenic synthesis of metal nanoparticles has recently emerged as a novel technology for the synthesis of metallic nanoparticles due to wide medical, catalytic and electronic applications as well as its eco-friendly nature. We herein report bio-inspired synthesis of nickel nanoparticles using *Moringa oleifera* aqueous leaf extract. The reduction of nickel chloride was carried out at pH3, pH7 and pH12. The synthesized nanoparticles were characterized using UV-Visible spectrophotometer and Atomic force microscope. The time of formation of the nanoparticles proceed faster at high pH and size of the nanoparticle also decreases with increasing pH implying that the time and size of the metal nanoparticle can be controlled.

Keywords: Nickel nanoparticles; Phytochemicals; pH–controlled synthesis; *Moringa oleifera*

Introduction

Nanotechnology is highly inter-disciplinary, involving physics, chemistry, biology, material science, and the full range of the engineering disciplines. The word nanotechnology is used as short hand to refer to both the science and the technology in this emerging field. Narrowly defined nanoscience concerns a basic understanding of physics, chemistry, and biological properties on atomic and near atomic scale, it also employs controlled manipulation of these properties to create materials and functional system with unique capabilities as reported by Hochbaum and Yang (2010). Metallic nanoparticles, those with primary dimension between 1 and 100 nanometres are of value owing to their unique size, shapes, composition, crystallinity and structure-dependent physiochemical and optoelectronic properties (Bagotsky, 2006).

At nanoscale dimensions the properties of materials no longer depend solely on composition and structure in the usual sense instead they display new phenomenon associated with quantized effect and with the preponderance of surfaces and interfaces. Quantized effects arise in the nanometre regime because the overall dimensions of objects are comparable to the characteristic wavelength for the fundamental excitation in materials .this is in accordance with Khan, Hussain and Hashmi (2012).

The physical and chemical properties of nanoparticles are often significantly different from the same material in bulk due to high surface to volume ratio at the nanometre scale (Adekunle and Ozoemena, 2010; Hochbaunm and Yang, 2010; Cheng and Yu, 2012).

The size, shape and controlled dispersion of nanoparticles plays a vital role in determining the physical, chemical, optical and electronic properties attributing its application in environment (Cheng, Zhang and Zhu, 1999); biotechnological (Duan and Lieber, 2000) and biomedical fields (Kasthuri, Kathiravan and Rajendiran, 2009).

Various physical and chemical processes have been exploited in the synthesis of several inorganic metal nanoparticles by wet and dry approaches namely ultraviolet irradiation, aerosol technologies, lithography, laser ablation, ultrasonic fields and photochemical radiation technologies as reported by Clemente and Glystein (2008) and Krpetic *et al* (2012). However these methodologies remain expensive and involve the use of hazardous chemicals as reported by Ali *et al* (2011) and Jin *et al* (2003). These methods are generally classified as top down methods. One of the promising alternatives to the afore mentioned top-down method is the bottom up methods as reported by

Gaware *et al*. (2012), this involves the employment of non-toxic plant extracts as reducing agent for the metal salt or oxide to form nanoparticles. The growing concern for the development of alternative, environment friendly and sustainable methods, has led to increasing awareness towards green chemistry and biological process which are cost effective and eco-friendly procedures (Clemente and Glystein, 2008; Ali *et al,* 2011). Phototropic eukaryotes such as plants, algae and diatoms and heterotrophic cell and some biocompatible agents have been reported to synthesize metal nanoparticles (Ali *et al*, 2011; Cheng, Zhang and Zhu, 1999; Farooqi, 2012; Gaware *et al*, 2012; Hochbaum and Yang, 2010; Hou *et al*, 2005; Jin *et al*, 2003; Kasthuri, Kathiravan and Rajendiran, 2009).

The use of phototropic and heterotrophic eukaryotes is yet to be fully exploited. In this study *Moringa oleifera* leaf extract has been used as the reducing and capping agent in the synthesis of nickel nanoparticles. The effect of varying the pH condition of the extract was investigated. Supposedly, the active component of the extract are the phytochemicals namely; flavonones, tannins, steroids carbohydrates and amino acid.

Materials and Method

Fresh leaves of *Moringa oleifera* were obtained locally at Adamawa State University Mubi, Nigeria. Nickel chloride hexahydrate assay 97% BDH Chemicals Ltd Poole England; Sodium hydroxide assay 99-100% Sigma-Aldrich Laborchemikalien Gmb; Nitric acid assay 68.5-69.5% BDH Chemicals Ltd Poole England.

Preparation of Moringa oleifera aqueous leaf extract

The leaves of *Moringa oleifera* were washed with tap water and then with ultra-pure deionized water. 10 g of the fresh leaves were weighed and boiled in 50 mL ultra-pure deionized water for 10 minutes. The resulting mixture was filtered using Whatman filter paper No. l.

Procedure for adjustment of pH of the leaf extract

Prior to synthesis of the metal nanoparticles, extracts of the plant with different pH was prepared. For pH 3, 0.1 M HNO_3 was added drop wise while for pH 12, 0.1 M NaOH solution was added drop wise to the extract. The pH readings were taken using Jenway 6405 pH metre. Three portions of aqueous extract were obtained; neutral, pH3 and pH12.

Synthesis of nickel nanoparticles

10 mL of 0.001M aqueous solution of nickel chloride was taken in a beaker. 2 mL of plant extract was added drop wise for one hour with continuous stirring this was done for pH3, pH7 and pH12 of the plant extract.

Surface characterization

Topographical images of the nickel nanoparticles were obtained with an Agilent 5500 atomic force microscope employing the contact mode technique with a cantilever having resonant frequency of 17 kHz and spring constant of 0.8 N/m. Sheet of mica was used as a substrate for placing the nanoparticles.

Results and Discussion

Reaction Colour

Fig. 1 is the observed reaction colour when the faint light blue solution of nickel chloride was exposed to the extract of *Moringa oleifera*, obviously, there is a change in colour from the clear solution of nickel chloride to dark brown of the nickel nanoparticles. Such change in reaction colour has been observed by other workers. For pH3 a colour change was observed after one hour. For neutral pH colour change was observed after 48 minute and for pH12 colour change occurred after 36 minutes, implying that the reaction proceeds faster under alkaline condition.

Figure 1: Solution of nickel chloride (A), *Moringa oleifera* leaf extract (B) and nickel nanoparticles (C).

Atomic Force Microscopy (AFM)

Figure 2 shows the AFM topographical 3D image of nickel nanoparticles. The particles appear as aggregate "nano rings" having average size distribution of approximately 70 nm.

Ramesh *et al.*, (1998) had earlier reported the aggregation of nickel nanoparticles; this may be due to strongly intermolecular forces such as Van der Waals attraction and π–π interaction. For magnetic nanoparticles such as nickel, magnetic dipole–dipole interaction makes this attraction stronger (Hou *et al.*, 2005).

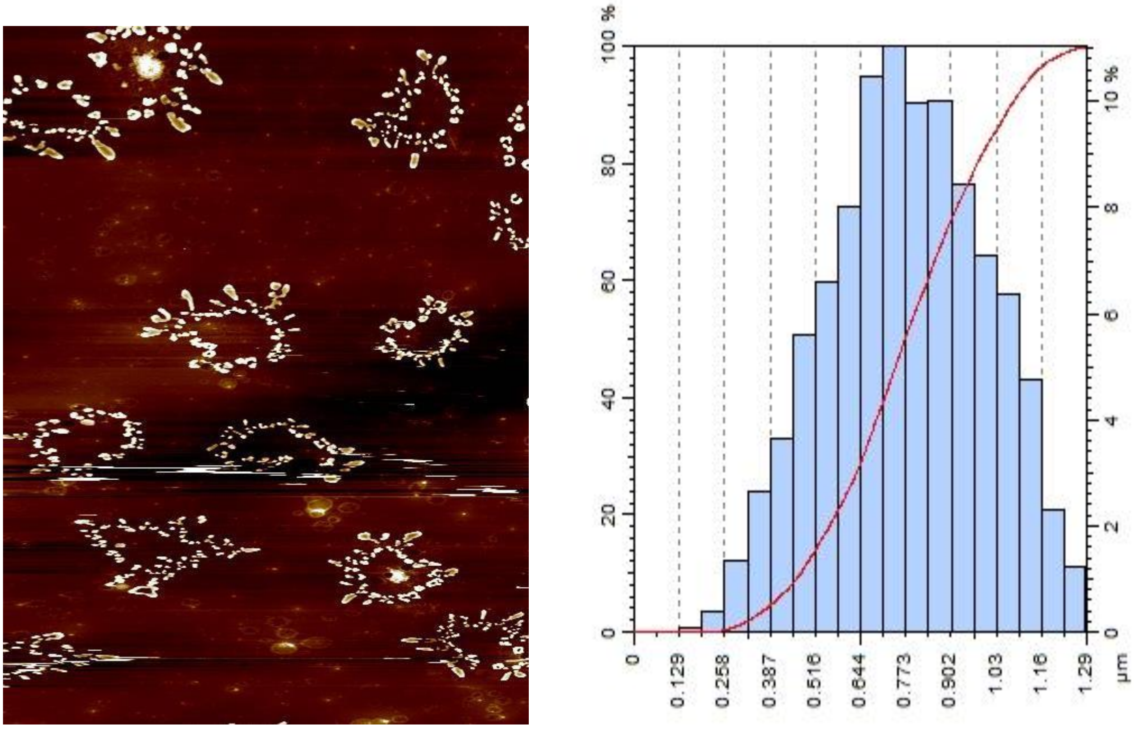

Figure 2: 3D image of nickel nanoparticles and size distribution of the nanoparticles

UV Visible Spectrophotometry of Nickel Nanoparticles

Using Jenway 6405 UV-visible spectrophotometer, surface Plasmon resonance of nickel nanoparticles was obtained for pH3, neutral pH and pH12; the results are presented in Figure 3. There was a shift of the band from 295 nm of the acid extract synthesized nanoparticle to 290 nm for the alkaline extract synthesized nanoparticle implying that under alkaline conditions formation of nanoparticles of small size is favoured.

Conclusion

Aqueous *Moringa oleifera* leave extract is effective at reducing aqueous nickel chloride to nickel nanoparticles (Ni^0). The phytochemicals act as reducing as well as capping agents. By varying the pH of the extract, the time required in the formation of the nanoparticles reduces with increasing pH also size of the nanoparticles followed similar path, inferring that synthesis of metal nanoparticles using plant extract may be favoured at high pH conditions.

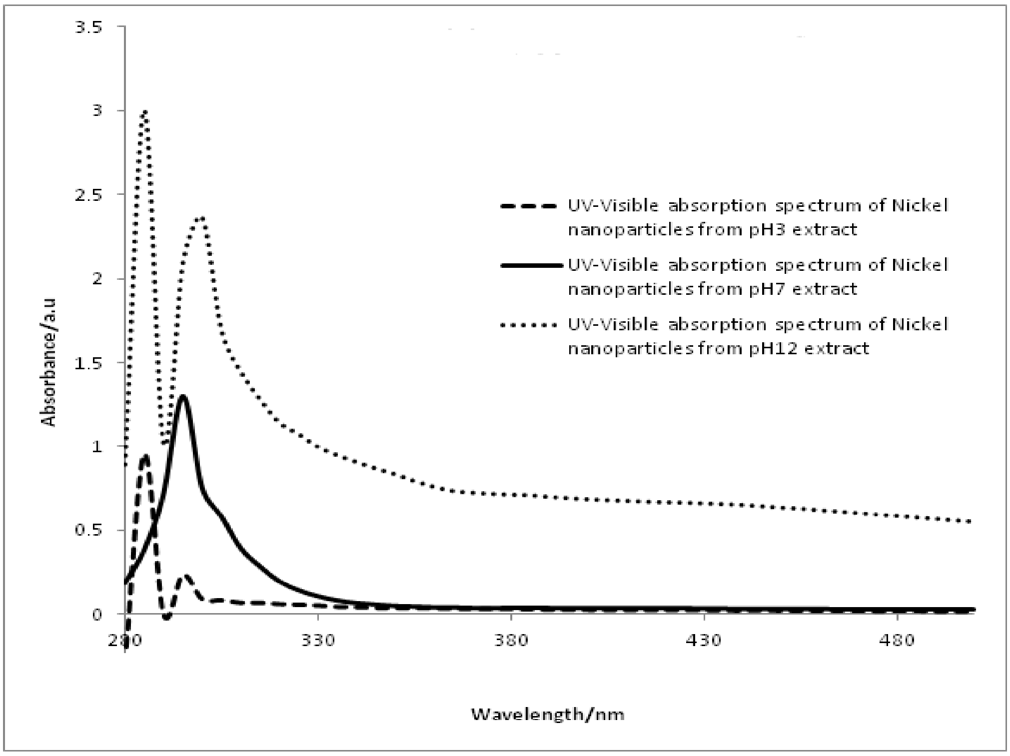

Figure 3: Comparative UV-Visible spectra of nickel nanoparticles at pH 3, pH 7 and pH 12.

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