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Green synthesis of silver nanoparticles using cellulose extracted from an aquatic weed; water hyacinth

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ABSTRACT

As part of the desire to save the environment through "green" chemistry practices, we herein report an environmentally benign synthesis of silver nanoparticles (Ag-NPs) using cellulose extracted from an environmentally problematic aquatic weed, water hyacinth (WH), as both reducing and capping agent in an aqueous medium. By varying the pH of the solution and reaction time, the temporal evolutions of the optical and morphological properties of the as-synthesised Ag-NPs were investigated. The as-synthesised cellulose capped silver nanoparticles (C–Ag-NPs) were characterised using Fourier transform infrared spectroscopy (FTIR), ultraviolet–visible spectroscopy (UV–vis), transmission electron microscopy (TEM) and high resolution transmission electron microscopy (HRTEM). The maximum surface plasmon resonance (SPR) peak decreased as the pH increased indicating that an increase in the pH of the solution favoured the formation of smaller particles. In addition, instantaneous change in the colour of the solution from colourless to brown within 5 min at pH 11 showed that the rate of reduction is faster at this pH compared to those at lower pH. The TEM micrographs showed that the materials are small, highly monodispersed and spherical in shape. The average particle mean diameters were calculated to be 5.69 ± 5.89 nm, 4.53 ± 1.36 nm and 2.68 ± 0.69 nm nm at pH 4, 8 and 11 respectively. The HRTEM confirmed the crystallinity of the material while the FTIR spectra confirmed the capping of the as-synthesised Ag-NPs by the cellulose. It has been shown therefore that based on this synthetic method, this aquatic plant can be used to the advantage of mankind.

1. Introduction

Metal nanoparticles have received a lot of attention in the recent years due to their unique optical, electrical, and biological properties which make them central to numerous applications such as in catalysis, bio-sensing, imaging, drug delivery and optical spectroscopy including surface-enhanced Raman scattering (SERS) (Abdel-Halim & Al-Deyab, 2011; Bankar, Joshi, Kumar, & Zinjarde, 2010; Hebeish, El-Rafie, Abdel-Mohdy, Abdel-Halim, & Emam, 2010). Amongst different types of metal nanoparticles such as copper, zinc, titanium, magnesium and gold, Ag-NPs are of particular interest because of their potent antimicrobial activity against different types of pathogens such as *Bacillus subtilis*, *Staphylococcus aureus*, *Klebsiella pneumonia* and so on (Li, Ma, Zhang, Liu, & Sun, 2011; Rai, Yadav, & Gade, 2009; Shameli et al., 2012). This extends their application to medicine, dental materials, coating of stainless steel materials, water treatment, sunscreen lotions, and elimination of microorganisms on textile fabrics (Oluwafemi et al., 2013; Rai et al., 2009). The smaller the particles, the greater the antimicrobial effect (Baker, Pradhan, Pakstis, Pochan, & Shah, 2005; Morones et al., 2005). However, these smaller particles are very reactive and consequently form aggregates thus losing their fundamental properties (Chen, Wang, Zhang, & Jin, 2008). To circumvent this challenge, passivating agents such as polyvinylpyrrolidone (PVP), polyvinyl alcohol (PVA), hyperbranched polyurethane (HP), and polyacrylonitrile (PAN) with reducing agents such as sodium borohydride, hydrazine, hydroxylamine, n-hexadecyltrimethylammonium bromide (HTAB) to mention a few, have been used successfully in the synthesis and stabilisation of silver nanoparticles (Abu Bakar, Ismail, & Abu Bakar, 2007; Hasell, Yang, Wang, Brown, & Howdle, 2007; Maria et al., 2009; Sharma, Yngard, & Lin, 2009). The utilisation of some of these passivating and reducing agents introduced yet another challenge of high cost and toxicity therefore threatening environmental sustainability and limiting the biological applications of these noble materials. These challenges have undoubtedly led to the current drive towards integrating the basic principles of 'green chemistry' – a concept that promotes the use of non-toxic chemicals, environmentally benign solvents, and renewable materials (Raveendran, Fu, & Wallen, 2003; Sharma et al., 2009; Vigneshwaran, Nachane, Balasubramanya, & Varadarajan, 2006; Wegner & Jones, 2009) into synthetic chemistry in general and in this case into the synthesis of

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nanomaterials. Reveendran et al., in 2003, reported the first completely green synthesis of Ag-NPs using starch as stabilising agent and glucose as reducing agent (Sharma et al., 2009). These materials are environmentally benign, non-toxic, abundant, renewable, and biodegradable. Several syntheses based on this modification have been reported (Darroudi, Ahmad, Abdullah, & Ibrahim, 2011; Darroudi, Ahmad, Zamiri, et al., 2011; García-Serrano, Pal, Herrera, Salas, & Ángeles-Chávez, 2008; Giuffrida, Ventimiglia, & Sortino, 2009; Hu, Wang, Wang, Zhang, & Yu, 2008; Oluwafemi et al., 2013). Li, Jia, et al. (2011) also prepared cellulose–silver nanocomposites using cellulose dispersed in N,N-dimethylacetamide (DMAc) as the stabilising agent and ascorbic acid as the reducing agent. The same group further reported the microwave synthesis of cellulose–silver nanocomposites using ethylene glycol (EG) as reducing agent (Li, Ma, et al., 2011). However, EG and DMAc are organic solvents and are not environmentally friendly. Therefore, preparation of Ag-NPs using water – a more environmentally benign solvent will be most preferable for a completely green chemistry approach (Raveendran et al., 2003).

One of the abundant aquatic weed species is the water hyacinth *(Eichhornia crassipes)*. This deadly aquatic weed (Fig. 1) found in tropical and subtropical regions such as Asia, South America, North America, Central America, Africa, and Oceania is a major problem to the environment (Wissel, Mayr, & Lücke, 2008). It is known to impede navigation through waters, blocking river waters and canals, clogging irrigation and hydropower systems. It also provides favourable breeding zones for disease-causing insects, quickens evapotranspiration and reduces biodiversity. Huge amounts of money are spent worldwide to remove this weed using chemical, biological, and mechanical methods which are aimed at its total eradication without making good use of it. In addition to this fact, some of these methods of removal also cause havoc like environmental pollution and destruction of other useful plants. Thus, its eradication and utilisation towards the advantage of mankind have become a major interest. Recent reports have shown that this weed has a high fibre content and contains a high percentage of cellulose in its shoot and roots (Sundari & Ramesh, 2012; Zhou et al., 2009). However, there are no studies indicating the use of this aquatic weed in the synthesis of silver nanoparticles. In this study, as part of the quest for a completely green synthesis of Ag-NPs and finding an economical and hazard-free method of disposing this weed, we report the first synthesis of silver nanoparticles using the cellulose extracted from the shoot of this plant (water hyacinth) as both the reducing and stabilising agent without any accelerator. By varying the pH of the solution and reaction time, control over monodispersity, size and shape of the as-synthesised C–Ag-NPs was investigated.

2. Materials and methods

2.1. Materials

Water hyacinth plant shoot collected from a local river in the Mthatha area (South Africa) was used as a source of cellulose. Toluene (Sigma–Aldrich), ethanol (Shalom Lab Supplies cc), sodium hydroxide pellets, hydrogen peroxide and silver nitrate (Sigma–Aldrich). All of these reagents were of analytical-grade and were used as received without further purification. The reaction was carried out in the dark to avoid any photochemical reactions

2.2. Extraction of cellulose from water hyacinth

The cellulose from the plant was extracted based on the method by Teixeira et al. (2011), with slight modification. Briefly, the water hyacinth shoot was washed, oven dried and cryocrushed in a mortar with a pestle followed by particle separation using 500 μm sieve. The resultant powder (10 g) was soxhlet extracted for 6 h with a 450 mL toluene/ethanol mixture in the ratio 2:1 (v/v) at 80 °C. The powder was then oven dried at 60 °C for 16 h after which it was dissolved in 400 mL distilled water. The resultant solution was stirred vigorously with a high energy homogeniser for about 30 min and filtered. The filtration was followed by bleaching the residue in a mixture of 100 mL 5% sodium hydroxide and 100 mL 11% hydrogen peroxide at 60 °C for 1 h and then re-filtered. The process of bleaching and filtration was repeated twice. The final residue was washed with distilled water to obtain a neutral pH and oven dried at 60 °C until constant weight.

2.3. Synthesis of silver nanoparticles

45 mL of Cellulose solution (0.5 g/100 mL of distilled water, homogenised at 60 °C for 1 h) was put in three separate flasks. The pHs of two out of the three solutions were adjusted from its original value of 8.0 to the desired pH (4 and 11) by the dropwise addition of dilute solutions of hydrochloric acid (0.5 M) or sodium hydroxide (0.5 M). To each of the solutions above, at the different pH values of 4, 8 and 11, 2.5 mL of 1 M silver nitrate solution was added at 75 °C. Aliquots were taken at different reaction times to monitor the reactions. Each aliquot was diluted using distilled water in the ratio 1:15 for characterisation.

3. Characterisation

3.1. UV–vis spectroscopy

The absorption spectra of the synthesised samples were obtained using Perkin Elmer lambda 25 UV–vis spectrophotometer in the 200–700 nm wavelength ranges.

3.2. Transmission electron microscopy (TEM)

Transmission electron microscopy was used to observe the size, shape, and morphology of the synthesised nanoparticles. A drop of a diluted specimen was mounted on carbon coated copper grid and placed in a JOEL JEM 2100 (TEM) operating at the voltage of 200 KV.

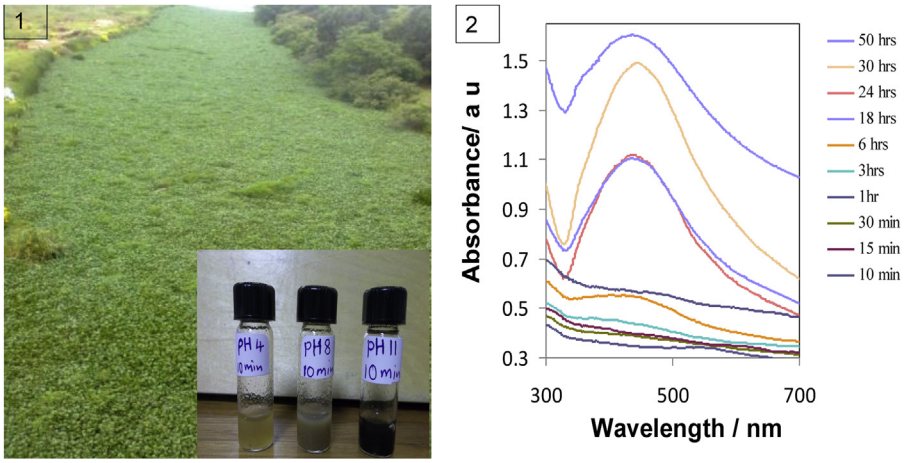
3.3. Fourier transform infrared spectroscopy (FTIR)

The FTIR spectra were measured using a Perkin Elmer spectrum, one FTIR spectrometer equipped with the universal ATR sampling accessory. The measurements were done in the transmission mode within a 4000–400 cm^{-1} range and 4 cm^{-1} resolution maintaining a total of 50 scans for each sample analysed.

4. Results and discussion

4.1. UV–vis spectroscopic analysis

In this reaction, cellulose extracted from water hyacinth shoot acted as both reducing and stabilising agent. The colour of the solution in the reaction flask changed from white to intense brown within 5 min for the reaction at pH 11 (Fig. 1a inset). However, for the reaction at pH 8 and 4, a gradual development of the brown colour was observed within 30 min and 6 h respectively. The instant change in the colour of the solution at pH 11 indicated that the rate of reduction was faster at this pH compared to those at lower pHs. The UV–vis spectra shown in Figs. 2–4 depict the surface plasmon resonance (SPR) peaks of the as-synthesised C–Ag-NPs at pH 4, 8, and 11 respectively. The spectra confirmed that the rate of the silver nanoparticles is slowest in the acidic medium. At pH 4 (Fig. 2)



Figs. 1 and 2. **(1)** Water hyacinth plant captured from a local river in Mthatha area (South Africa) (inset: colour of the C–Ag NPs at different pH after 5 min). **(2)** Absorption, spectra of C–AgNPs at pH 4 for different reaction times.

the plasmon resonance band appeared after 6 h while for pH 8 and pH 11 (Figs. 3 and 4), it appeared within 10 min although a broad spectrum was obtained for pH 8 at the beginning of the reaction. The smaller sized nanoparticles obtained in the basic medium as confirmed by the TEM analysis have been attributed to the high dissociation rate of the hemiacetal and hydroxyl groups present in the cellulose which are responsible for the reduction of silver ions to silver nanoparticles (Park, Hong, Weyers, Kim, & Linhardt, 2011). As the reaction time increased from 5 min to 50 h for pH 8 and 11, the peak intensity also increased indicating increase in the concentration of C–Ag-NPs. This trend is also observed at pH 4 except that the peak intensity was broad at 50 h indicating broad size distribution.

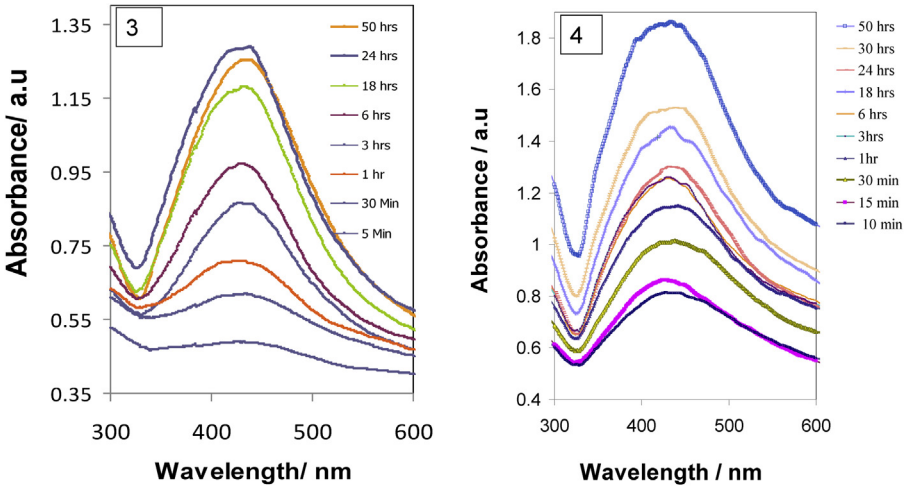
4.2. Transmission electron microscopy

Fig. 5 shows a typical representative of the TEM micrographs for the as-prepared Ag-NPs at different pHs. The TEM results indicate that the particles are small, well dispersed and spherical in shape.

The average diameter of the as-synthesised C–Ag-NPs taken after 6 h were determined to be 5.69 nm at pH 4, 4.53 ± 1.36 nm at pH 8, and 2.68 ± 0.69 nm at pH 11. Contrary to most of the reported bio-synthesis of Ag-NPs using plant extract, all the particles are isotropic (spherical) in shape at the different pHs. The smallest particle with high degree of monodispersity was obtained at pH 11. The HRTEM images (inset of Fig. 5) also provide further insight into the microstructure (shape) and crystallinity of the as-prepared Ag-NPs. The HRTEM images show that the as-synthesised materials are spherical and consist of well ordered single crystals with distinct lattice fringes. The clear and uniform lattice fringes confirmed that the spherical particles are highly crystalline. The measured lattice spacing (d) of 0.230 nm corresponds to the $d(1\,1\,1)$ spacing for face-centred cubic (fcc) Ag. The clear singular reciprocal points shown by the fast Fourier transform (FFT) patterns in Fig. 5 (inset) confirms that the obtained C–Ag-NPs are single crystals. The solutions of dispersed silver nanoparticles are highly stable and showed no signs of aggregation even after six months of storage indicating that cellulose extracted from this weed successfully reduced and passivated silver nanoparticles.

4.3. Fourier transform infrared spectroscopy (FTIR)

The surface chemistry of the as-synthesised C–Ag-NPs was investigated using FTIR spectroscopy. The FTIR spectra of the crude WH and extracted cellulose shown in Fig. 6A and B indicate the disappearance of the bands at 1737 cm^{-1} and 1514 cm^{-1} in the extracted cellulose. This has been attributed to the successful removal of either acetyl and uronic ester linkage of the carboxylic acid group of the ferulic and p-coumeric acids of lignin and/or hemicelluloses (Chen et al., 2011). A typical FTIR spectrum of the



Figs. 3 and 4. **(3)** Absorption spectra of C–AgNPs at pH of 8 and **(4)** pH 11 at different, reaction times.

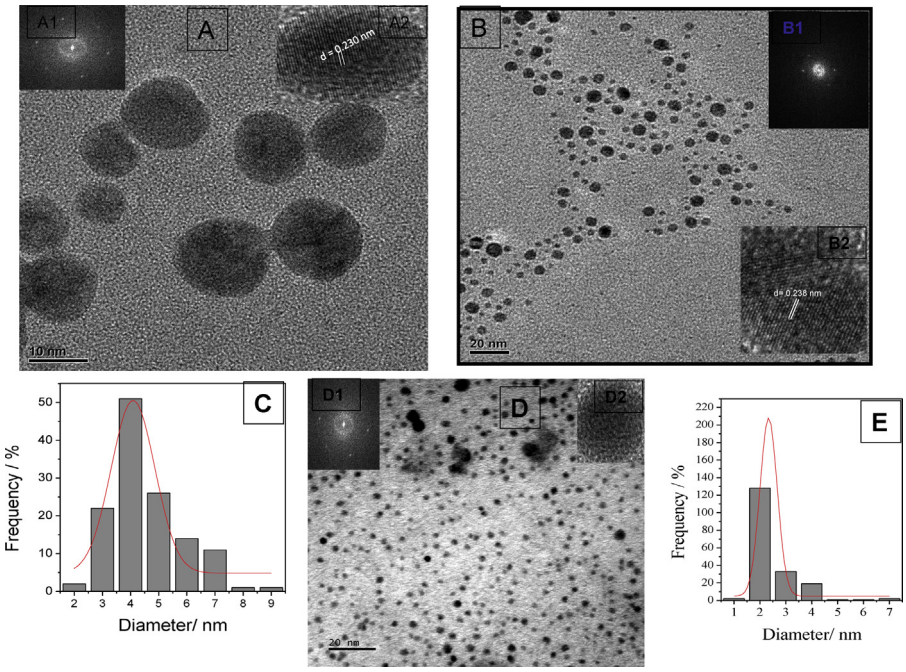


Fig. 5. TEM images at 6 h for pH 4 (A) with corresponding FFT (A1) and HRTEM (A2), pH 8 (B) with corresponding with corresponding FFT (B1), HRTEM (B2) and size distribution (C), pH 11 with corresponding FFT (D1), HRTEM (D2) and size distribution (E).

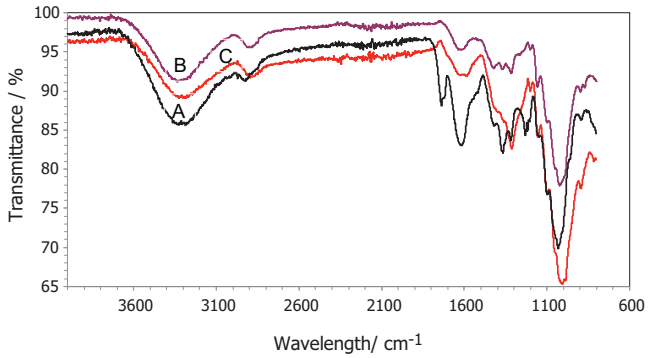


Fig. 6. FTIR spectra of crude WH (A), extracted cellulose (B) and C–Ag-NPs (C).

as-synthesised C–Ag-NPs in Fig. 6C shows the fundamental peaks present in the extracted cellulose as shown in Table 1. In addition, the disappearance of the band at 1366 cm^{-1} and the appearance of the band at 1588 cm^{-1} were observed in the C–Ag-NPs spectra. The band at 1588 cm^{-1} in the as-synthesised silver nanoparticles suggests possible coordination of the as-synthesised Ag NPs with the carboxylate ions formed during the reduction reaction. Similar spectra were obtained for the C–Ag-NPs at pH 8 and 4. All these measurements confirmed successful capping of the silver nanoparticles with cellulose extracted from the water hyacinth shoot.

5. Conclusion

We have demonstrated the synthesis of C–Ag-NPs using cellulose extracted from water hyacinth-known as a deadly aquatic weed for the first time. The synthesis is simple and completely green with the extracted cellulose acting as both reducing and stabilising agent. The temporal evolution of the particle size and shape was monitored by varying the pH of the solution and reaction time. Small, stable, spherical and highly monodispersed particles with an average diameter of 2.68 ± 0.69 nm were obtained at pH 11. This method offers an economical and hazard-free method of disposing water hyacinth from contaminated or invaded rivers.

Table 1
Summary of FTIR spectral bands for the crude water hyacinth (WH), extracted cellulose and C–Ag-NPs.

Bands (cm^{-1})			Assignments	References
Crude WH	Cellulose	C–Ag-NPs		
3311	3335	3311	O—H stretching vibration	[31]
2925	2891	2891	C—H stretching of sp^3 carbon	[32]
1737	–	–	Either acetyl & uronic ester linkage of carboxylic group of the ferulic and p-coumeric acids of lignin and/or hemicelluloses	[28,33]
1626	1626	1626	O—H bending vibration of absorbed water	[34]
–	–	1588	—COO^- of carboxylic acid salt	[35]
1419	1419	1419	CH_2 bending	[36]
1514	–	–	C=C stretch of the aromatic ring in lignin	[30]
1366	1366	–	C—H··· deformation	[37]
1315	1315	1315	CH_2 wagging	[36]
1231	–	–	CH and/or OH deformation	[37]
1201	1201	1201	OH in-plane bending	[36]
1156	1156	1156	Antisymmetric bridge oxygen stretching	[36]
1100	1100	1100	Antisymmetric in-phase ring stretching	[36]
1030	1023	1006	C—O—C stretching of pyranose ring skeletal	[30]
890	898	890	C_1 group frequency	[36]

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