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Microwave-assisted rapid synthesis of alumina nanoparticles using tea, coffee and triphala extracts

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Abstract Alumina nanoparticles (AlNP) were synthesized from aluminium nitrate using extracts of tea, coffee and triphala—a well known herbal plant as well as a non-toxic and eco—friendly green material. The synthesis was carried out taking 1:4 ratio of metal salt and these extracts under microwave irradiations at 540 W, which gave better yield of nanoparticles. Water was taken as solvent medium. The formations of AlNP were initially monitored by the colour changes occurring in the reaction mixture during the incubation period. As synthesized nanoparticles were characterized by scanning electron microscope (SEM), UV–Visible (UV–Vis) spectroscopy and Fourier transform infrared spectroscopy (FTIR). The AlNP were found to be spherical in shape in case of tea and coffee extracts with a size of 50–200 nm and to be oval shaped in case of triphala extract with an average size of 200–400 nm. The formation of AlNP with the microwave-assistance using these plant extracts has proved to be very faster than any other methods. In addition, excellent reproducibility of these nanoparticles, without the use of any additional capping agent or stabilizer will have great advantages in comparison with microbial synthesis, avoiding all the tedious and hygienic complications.

Keywords Alumina nanoparticles · Microwave · Tea · Coffee · Triphala

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1 Introduction

Nanocrystalline metal oxides are found to have immense applications in the fields of catalysts, semiconductors, medical science, capacitors, batteries, absorbents, chemical and biological sensors, optoelectronics, information storage photonic, electronic devices and in preparation of polymer nanocomposites with improved mechanical properties [1–4]. Various methods, such as wet chemical reduction, reverse micelles, electrochemical and non-electrochemical techniques, microwave assisted process and nowdays via green chemistry route [5–9] have been developed to synthesize metal nanoparticles because of the diversity and importance of these applications. Use of plant sources or extracts in synthesis of nanoparticles is quite novel leading to truly green chemistry, which provides advancement over chemical and physical methods [10–12]. It is costly effective, environment friendly and can be easily scaled up for large scale synthesis and most importantly, there is no need in this method to use high pressure, energy, temperature and toxic chemicals. Biosynthesis of metal nanoparticles extracted from different parts of the plant is the most effective process of synthesis at a very affordable cost.

According to the researchers, the polyol components present in the plant extract are responsible for the reduction of metal ions, whereas water soluble heterocyclic components stabilize the formed nanoparticles. The synthesis of metal oxide nanoparticles by reduction of the corresponding metal salts via green chemistry mainly depends upon the choice of solvent, reducing agent employed and the capping agent. Silver, gold, platinum and palladium have been rottenly used for the synthesis of nanoparticles [13–16]. In general, alumina has many interesting properties such as catalyst, as high stability and hardness,

insulation, surface protective coatings, as composite materials with tuneable mechanical properties, etc [4, 17]. In the recent years, AlNP were synthesized in liquid using a short pulse laser with the pulse width in the range of nanosecond [18]. In continuation of our earlier studies to synthesize metal nanoparticles [19], we now have reported the microwave assisted green chemistry to synthesize AlNP using tea, coffee and triphala extracts. To the best of our knowledge, the preparation of AlNP in water using these extracts is so far unexplored under microwave irradiation. This approach has the basic advantage that no other stabilizing agent or capping agent is required to stabilize these nanoparticles.

2 Materials and methods

2.1 Preparation of tea, coffee and triphala extracts

Three plant extracts were used to produce AlNP. 10 g fine grounded powders of tea, coffee and crushed triphala were dissolved in 100 ml water and boiled for around 2 h. These were then centrifuged, filtered and stored in a cool place. After cooling at room temperature, these were centrifuged for 15 min and filtered. The filtrates were stored at 5–10 °C and used as reducing and stabilizing agents.

2.2 Synthesis of AlNP using tea extract

Aluminium nitrate was used as precursor for the synthesis of AlNP. To make sure a complete reduction, AlNP was dissolved in aqueous tea extract taking 1:4 ratio (*w*/*w*) with stirring at room temperature. It was then subjected to microwave heating at 540 W, which produced yellowish-brown precipitate after 6–7 min. The precipitates were centrifuged and washed with distilled water and methanol. These were further kept for drying at 100 °C for 3–4 h.

2.3 Synthesis of AlNP using coffee extract

Aluminium nitrate and coffee extract taken in 1:4 ratio (*w*/*w*) were subjected to microwave heating at 540 W in a similar way mentioned above. After 5–6 min, yellowish-brown precipitate was observed. The precipitate was centrifuged, washed with distilled water and methanol, and then kept for drying at 100 °C for 3–4 h.

2.4 Synthesis of AlNP using triphala extract

Aluminium nitrate was added in triphala extract 1:4 ratio (*w*/*w*), and the solution was subjected to microwave heating at 540 W. Within 2–3 min light brown coloured precipitate was formed. The precipitate was centrifuged, washed with distilled water, and methanol and kept for drying at 100 °C for 3–4 h.

3 Results and discussion

In conventional heating, reactants are slowly activated by a conventional external heat source. Heat is driven into the substance, firstly passing through the walls of the vessel in order to reach the solvent and the reactants. On the other hand, microwave penetrates inside the material and heat is generated through direct microwave–material interaction. Since microwaves couple directly with the molecules of the entire reaction mixture, it leads to a rapid rise in the temperature. In this case, only the reaction vessel contents are

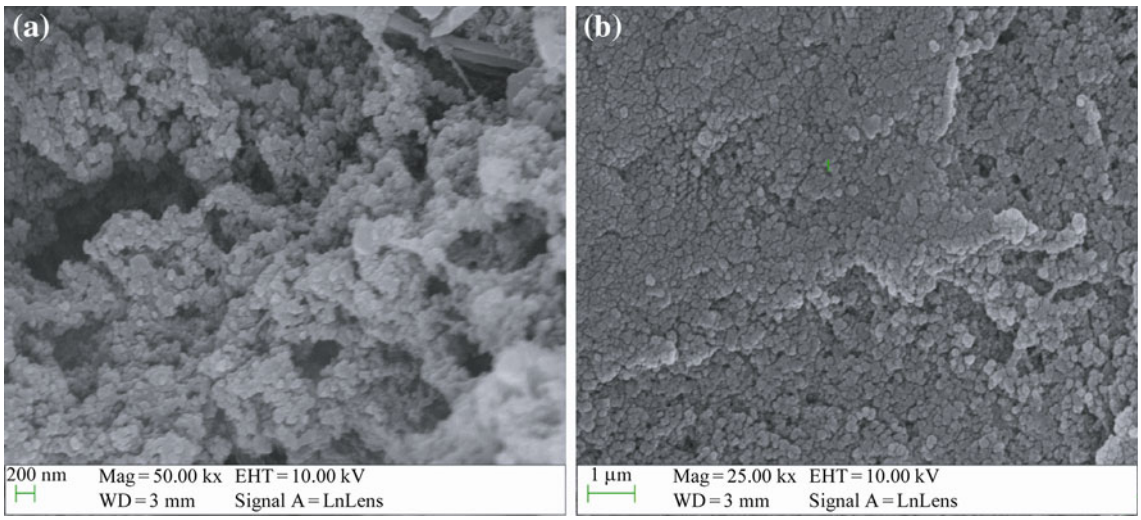

Fig. 1 SEM images of AlNP using tea leaves extract

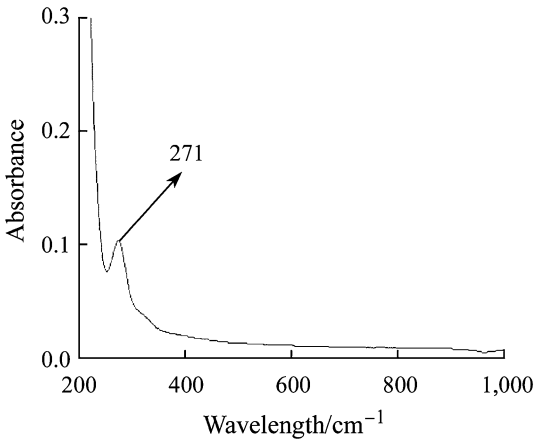

Fig. 2 UV–Vis of AlNP using tea leaves extract

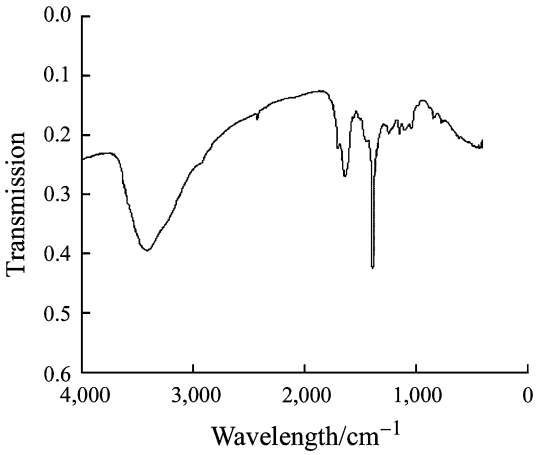

Fig. 3 FTIR of AlNP using tea leaves extract

heated and not the vessel itself, hence better homogeneity and selective heating of polar molecules should be achieved. Therefore, for microwave heating, the substance must possess a dipole moment [20].

The formation of AlNP by the supernatant of the tea, coffee and triphala extracts were initially observed through colour change. The SEM of AlNP using tea extract clearly indicates the formation of spherical AlNP with the size ranging between 50 nm and 100 nm as shown in the Fig. 1a, b. The formation of nanoparticles by the supernatant of the tea extract was monitored by periodic sampling of aliquots (1 ml) and subsequently measuring UV–Vis spectra of these solutions between 200 nm and 1,000 nm. Distilled water was used to adjust the baseline. Thus, the UV–Vis spectrum of AlNP from tea extract shows the peak at 271 nm (see Fig. 2). Figure 3 shows the Fourier transform infrared spectroscopy (FTIR) peaks of bio-functionalized AlNP at 1,630, 1,383, and 1,090 cm^{-1}. The peak at 1,090 cm^{-1} indicates the presence of C—N stretching frequency, whereas the geminal methyl group at 1,383 cm^{-1} and peak at 1,630 cm^{-1} represent the unreacted ketone group, suggesting the presence of flavonones adsorbed on the surface of AlNP. This suggests that influence of water soluble organic moieties of tea is responsible for the synthesis of AlNP and surface modification. The FTIR spectrum also shows a broad absorption band at 3,440 cm^{-1} is mainly ascribed to OH— groups on the surface of the AlNP nanostructure. The peak at 523 cm^{-1} indicates the formation of the AlNP nanostructures.

Figure 4a, b show the SEM images of AlNP using coffee extract, and the results indicate the formation of spherical shaped nanoparticles below the size of 100 nm. Absorption spectrum of AlNP has an absorbance peak at 275 nm (see Fig. 5). The FTIR spectrum shown in Fig. 6

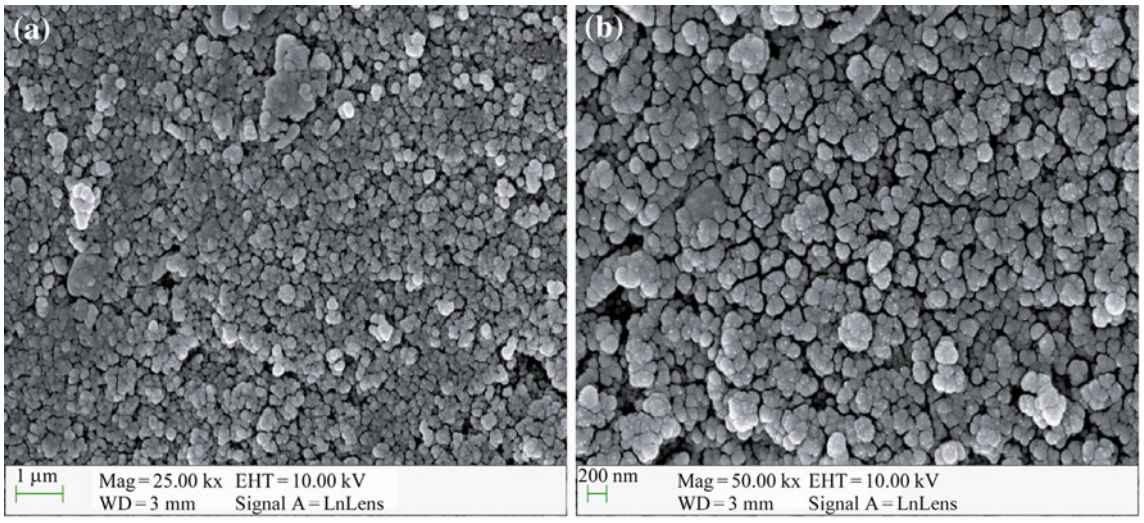

Fig. 4 SEM images of AlNP using coffee extract

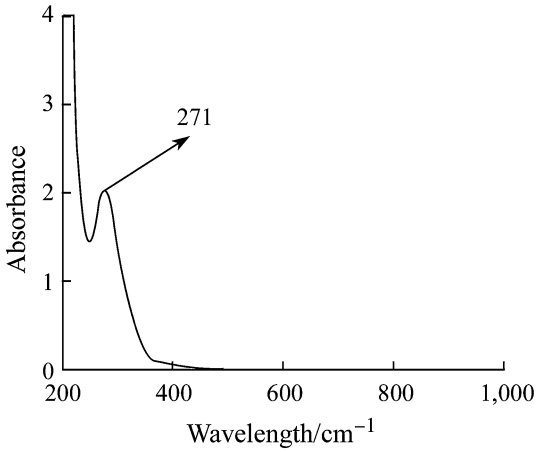

Fig. 5 UV–Vis of AlNP using coffee extract

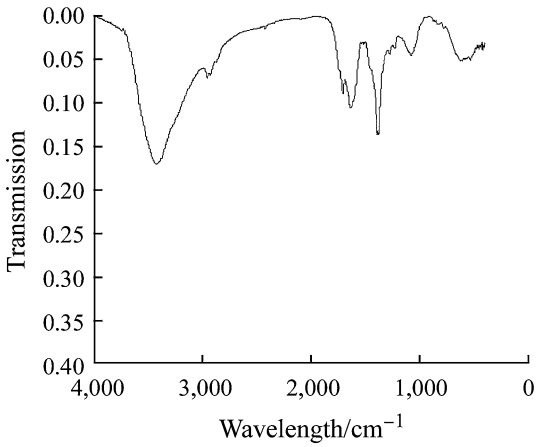

Fig. 6 FTIR of AlNP using coffee extract

again confirms the formation of AlNP in presence of coffee extract. The SEM images shown in Fig. 7a, b show the oval shaped structures of AlNP in 200–400 nm size, when triphala extract is used as reducing agent. The UV–Visible spectrum of AlNP from triphala extract shows the peak at

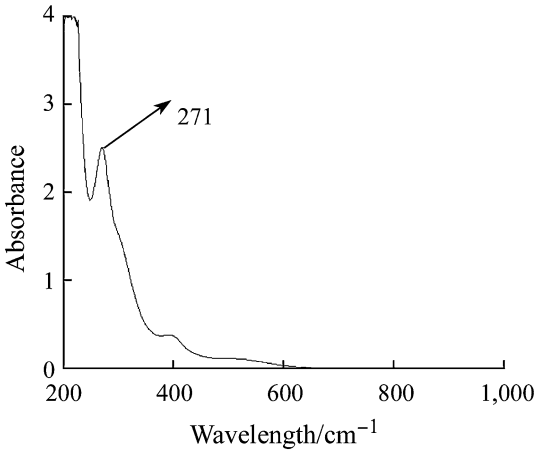

Fig. 8 UV–Vis of AlNP using triphala extract

273 nm (see Fig. 8). FTIR is shown in Fig. 9, showing peaks at 1,633, 1,380 and 1,080 cm^{-1}. This indicates the secretion of some water soluble organic components, which might contribute for the reduction of aluminium nitrate into AlNP.

Actually, the polyphenols in tea, coffee and triphala are responsible for the chemical reduction of aluminium nitrate. In tea, the polyphenol content is less than coffee and triphala. NMR and FTIR study show that it mainly contains gallic acid, which might have increased the reaction rate, and therefore it took less time (2–3 min) for the reduction of aluminium nitrate. The reaction does happen in absence of microwave heating, but it takes almost 7–8 h at room temperature. The particles shape are found to be uneven, much larger than those of microwave method. It can be inferred that the agglomeration of particles is prevented by the polyphenols content and thus confers stability of AlNP in the aqueous medium. This gives strong evidence for the polyphenols involvement in

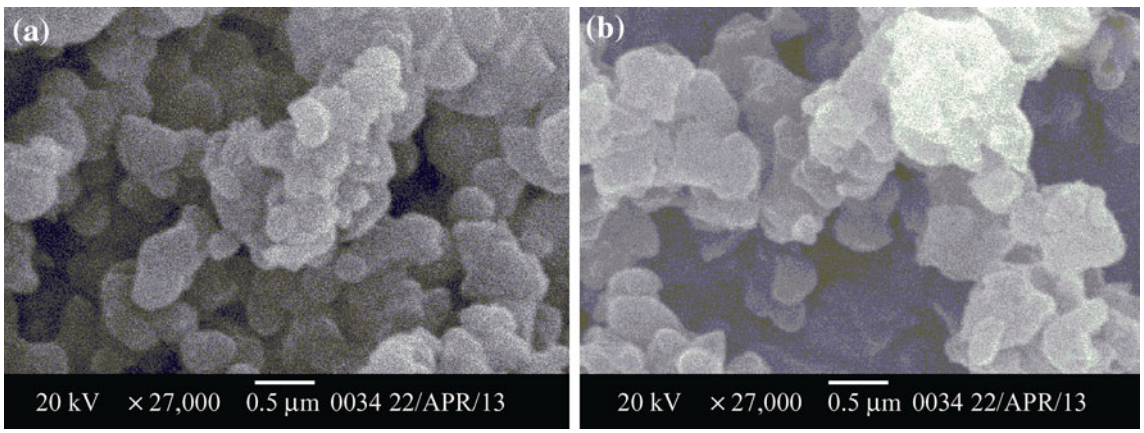

Fig. 7 SEM images of AlNP using triphala extract

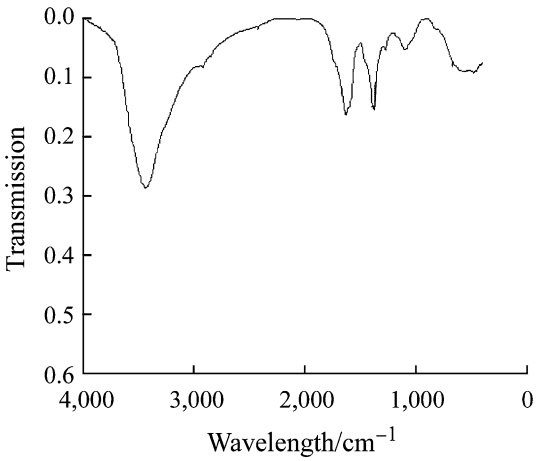

Fig. 9 FTIR of AlNP using triphala extract

the rapid biosynthesis and for the stability of metallic nanoparticles in the aqueous medium [21].

4 Conclusions

In this paper, microwave-assisted green chemistry has been used for the synthesis of AlNP. A facile approach has been reported using tea, coffee and triphala extracts, acting as reducing agents for the synthesis of AlNP of well-defined dimensions in bulk amount. This eliminates the need of toxic chemicals for the synthesis of nanoparticles. The formation of AlNP with the microwave-assistance using these plant extracts has proved to be very faster than any other method. These nanoparticles which are synthesized using green chemistry can be used for a variety of applications in the future. As triphala extract has medicinal importance, the results described here will also be useful for its biomedical applications.

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