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Synthesis of colloidal alumina nanoparticles using green method

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Abstract. Alumina nanoparticles were synthesized using aluminium nitrate and lemon extract, lemon being an eco-friendly and non-toxic material. The synthesis was carried out using salt solution and the above extract in 1:1 ratio, radiated with the microwaves at 600W for 5 minutes. The synthesis was carried with and without the stabilizing agent to know the role played by it. The stabilizing agent used was sodium citrate which is environmental friendly. The amount of stabilizing agent used was varied and the optimum amount for better stability was found out. The nanoparticles thus formed were characterized by using UV Visible (UVVis) Spectroscopy, Dynamic Light Scattering and Zeta Analyzer. The nano particles had an average size of 460nm. This method of synthesis has proven to be faster than any other method because of the involvement of microwave heating. Above all, the synthesized nano particles may have anti-bacterial and anti-fungal properties which have a scope for future study.

1. Introduction

As a result of the development in the field of nanotechnology, a new kind of material has emerged which typically falls in the nano range. The material which is known as nanoparticles has wide range of applications. Metal oxide nanoparticles have been widely used in many applications such as surface coating, medical science and also as a heat transfer fluid. Aluminium Oxide or alumina generally refers to corundum, which is a white oxide. Alumina has several phases such as gamma, delta, theta and alpha phases. However, the alpha alumina phase is the most thermodynamically stable phase. In general, alumina has many interesting properties, for example high hardness, high stability, high insulation and transparency [1]. Alumina nanoparticles can be synthesized by other methods like chemical method, physical method, biological method and green method. Other methods like wet chemical reduction, reverse micelles, electrochemical and non-electrochemical methods are still used, but microwave assisted green methods are being widely used because of its diversity and importance of applications [2-6]. Synthesis of nanoparticles from plant extract is an approach of green chemistry that connects nanotechnology and plant biotechnology. Plant extracts are used for the reduction of metal ions and this process is known as bio reduction which helps to form nanoparticles. It has been demonstrated that plant metabolites like sugars, terpenoids, polyphenols, alkaloids, phenol acids and proteins play an important role in metal ions reduction into nanoparticles and in supporting their subsequent stability [7]. The conventional methods that are used for the synthesis of nanoparticles especially the chemical method where chemicals used create a toxic nature to the synthesized nanoparticle which will eventually be a threat to the environment. The green synthesis method aims at eradicating this threat to the environment by using plant extract for synthesizing process where nanoparticles can be synthesized more effectively and easily.

Conventional heating method can be used while synthesizing. In this method the container or the reactor is heated either by conduction or convection. But the problem with this method is that it would take time for uniform temperature distribution. This problem with non-uniform temperature distribution can be tackled with the use of microwave heating. Microwave heating is a propitious method in both organic and inorganic methods. The advantage with this method is that uniform heating of the mixture can be obtained which helps in saving time and energy [8].

2. Experiment

In the synthesis process, initially 20 grams of lemon was taken and then the extract was prepared by grinding the lemon and filtering it using vacuum filtration method. The vacuum filtration process involves a Buchner funnel conical flask and a vacuum pump. The conical flask consists of a port through which a connection can be made to the vacuum pump with the help of a pipe. This filtering process is chosen because it is faster than a simple gravity filtration process. In the succeeding process, aqueous solution of 0.1% (w/v) of aluminum nitrate was prepared and also aqueous solution of 2% (w/v) of sodium citrate which was used as a stabilizing agent. The extract thus prepared and the aluminum nitrate was mixed at a ratio of 1:1 and the mixture was then taken for analysis using UV spectrometer.

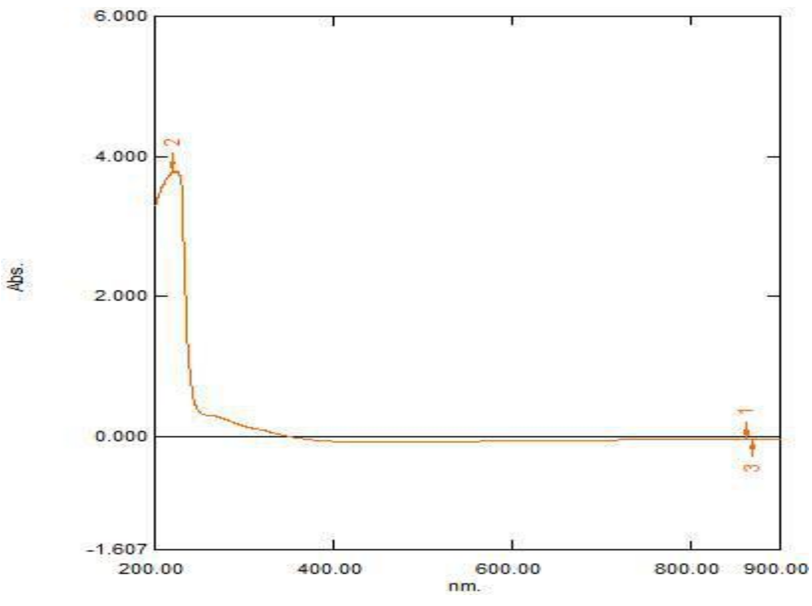

Figure 1. UV Spectrum of lemon extract and aluminum nitrate mixture

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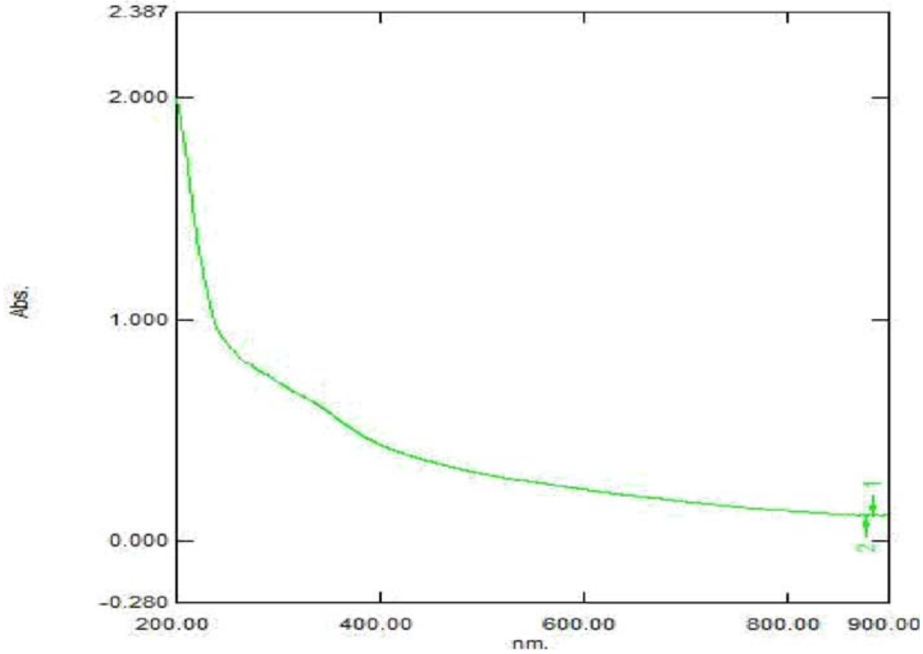

Figure 2. UV Spectrum of heated lemon extract and aluminum nitrate mixture

From the UV Spectrum it can be said that simple mixing of solutions is not enough for the spontaneous reduction of the mixture. Further the mixture was heated with microwaves at 600 W for 5 minutes. Then the heated sample was taken for UV spectrometer test and a peak at 271.50nm was observed. The UV Spectrum thus obtained was compared with the UV Spectrums of other research works to ascertain the formation of alumina nano particles. Later the sample was centrifuged at 3500 rpm and there was a sediment formation. This sediment was ultrasonicated for 30 minutes which had an operating frequency 33+/-3 KHz and then the sediment was taken for UV test. From the UV Spectrum of the sediment it can be concluded that there are no traces of nanoparticles in the sediment which clearly indicates that only the supernatant contains the nanoparticles. Another inference that can be made from this is that since aluminium being a lighter metal, centrifuge that has been used is insufficient to separate the particles and therefore centrifuge of higher rotational speed is required. The supernatant was kept for analysis under static state.

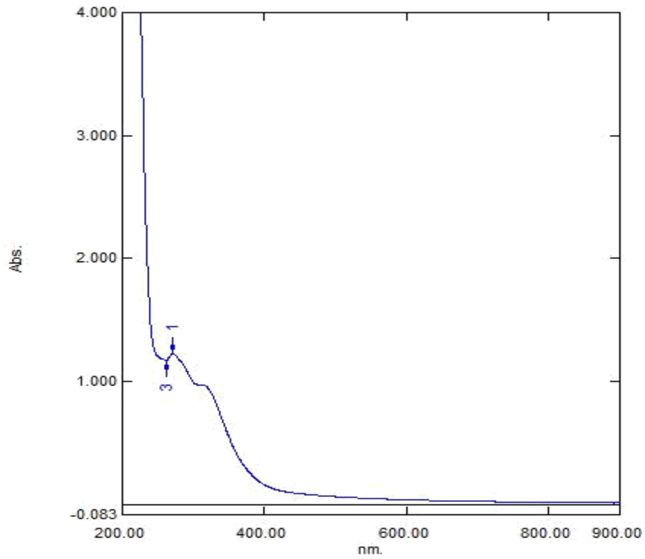

Figure 3. UV Spectrum of the sediment.

For further experimental analysis and to study the role played by the stabilizing agent in the stability of alumina nanoparticles, stabilizers were used. Either of two chemicals i.e. SDS (Sodium Dodecyl Sulphate) or Sodium Citrate can be chosen. SDS may be fatal or it causes serious damage to living beings when it comes in direct contact. It may cause moderate inflammation or irritation to the skin. Considering the above factors, SDS cannot be chosen which in turn favours using sodium citrate. In the current experiment, 2% (w/v) of Sodium Citrate was prepared and different volumes of the solution this prepared was added (1ml, 3ml, 5ml) in 1:1 ratio along with lemon extract and aluminium nitrate. Each solution was heated separately using a microwaves at 600W for 5 minutes, the UV of the samples were taken and it has been found that they are similar to those of the previous ones.

The solution was kept undisturbed for static analysis. The samples were taken for analysis after two days and there was no sign of agglomeration. Then on the 4th day it was observed that in the sample containing 1ml of sodium citrate solution there was separation of aluminium hydroxide which was understood by the presence of jelly like sediment and 5ml solution was affected by fungus. Only the sample containing 3ml of sodium citrate solution was available for further study. The UV Spectrum of the sample was taken on 4th and 19th day. From the UV Spectrum it can be concluded that stabilizing agent plays an important role for the stable behavior of the nanoparticles.

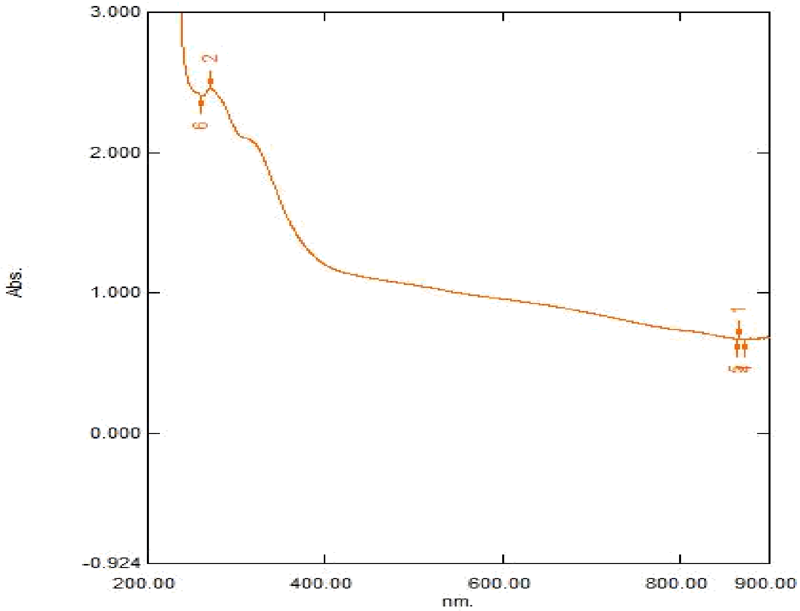

Figure 4. UV Spectrum of colloidal nanoparticles after 4 days.

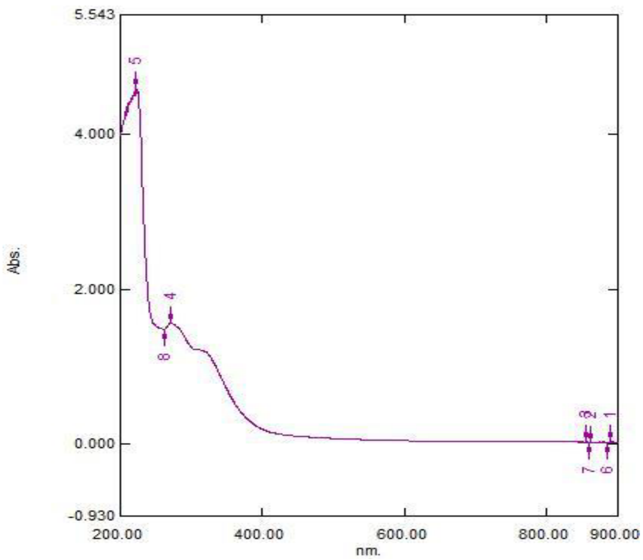

Figure 5. UV Spectrum of colloidal nanoparticles after 19 days.

3. Characterization

Nanoparticle characterization is used to determine the size and morphology and surface charge using techniques like Atomic Force Microscopy(AFM), Scanning Electron Microscopy (SEM) and Transmission Electron Microscopy(TEM). It can be said that the stability of the solution is determined by the van der Waals attractive and electrical double layer repulsive forces as the particles approach each other due to Brownian motion phenomenon, if the attractive force is greater than the repulsive force the two particles will collide and the suspension is not stable. . For the nanofluid to be stable the repulsive force must dominate the attractive force[9]. Stability also depends on synthesis method and it can also be increased by controlling the growth of particles . Silver nano paricles were synthesized from algae and Lignosulphonic acid it was found stable for 30 days[10-11] In the present work DLS and Zeta potential were measured. DLS actually measures the hydrodynamic diameter of the particle and zeta potential the surface charge of the particle. Here both DLS and Zeta potential test were carried out for an interval of 5 minutes. The hydrodynamic diameter was obtained about 460nm.

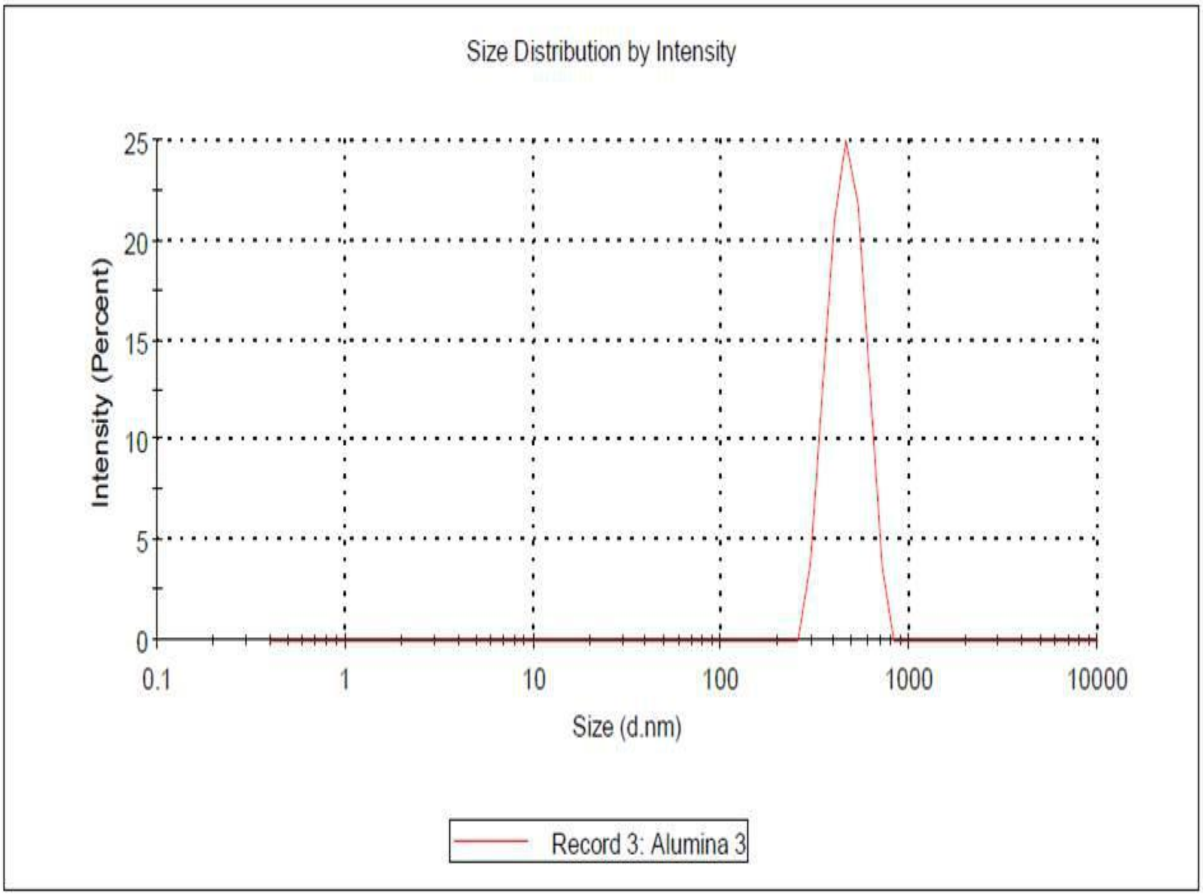

Figure 6. DLS of alumina nanoparticles.

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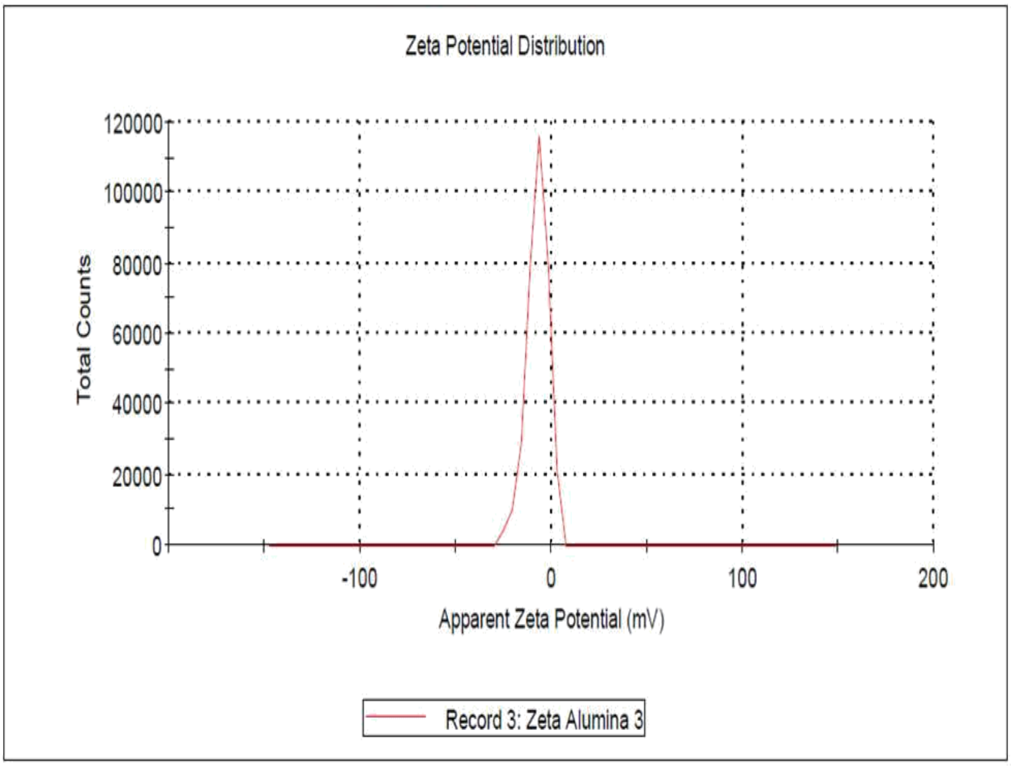

Figure 7. Zeta potential of alumina nanoparticles.

4. Results and Discussions

Conventional heating technique causes slow activation of the reactants due to slow driving speed of the heat through the container. Initially it has to pass through the walls of the container and then to the reacting compound which indeed is a time consuming process. Whereas, in microwave heating, the heat is uniformly distributed and it directly interacts with the molecules which helps in triggering a faster reaction. The formation of alumina nanoparticles was observed by analyzing the small color change. This was further confirmed with the help UV-spectrometer analysis by comparing the wavelengths with the existing literature. The wavelength obtained was 271.50 nm which was matching with the existing literature. From the experimental analysis it was confirmed that lemon played a role in the synthesis process. It was also observed that 3ml of (2% w/v) of sodium citrate showed a good result compared to 1ml and 5ml solutions of sodium citrate. Since lemon is rich in ascorbic acid, it can be said that the nanofluid prepared from this nanoparticle can have high temperature stability which can be studied further and will be useful for high temperature applications.

Figure 8. Synthesized colloidal Alumina nanoparticles

5. Conclusion

In the current paper, the synthesis of colloidal alumina nanoparticles has been carried out using microwave heating technique. It is a green chemistry approach, where lemon extract has been used for the reduction of aluminium nitrate into alumina nanoparticles. This method has completely eliminated the use of toxic chemicals. This method is proven to be faster and economical for the synthesis of the nanoparticles. The green chemistry approach can be a doorway for eco-friendly and economical production of nanoparticles which can help in faster commercialization. Research is still carried out to understand the heating effect on the particle size and ways to reduce it.

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