Aloe Vera Gel Mediated Green Synthesis of Ɛ-Fe2o3 Nanoparticles

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Abstract – A green synthesis method for the preparation of Ɛ-Fe_2O_3nanoparticles had been developed using Aloe Vera leaf gel extract as reducing and stabilizing agent and Ferric Chloride ($FeCl_3$) as precursor. The significant change in colour and pH of the prepared mixture solution indicates the formation of Ferric Oxide nanoparticles. Synthesized nanoparticles were characterized by XRD and FTIR methods. The X-Ray diffraction (XRD) analysis revealed that the synthesized Fe_2O_3 nanopowder has the Ɛ-phase orthorhombic structure having crystallite size 10.2 nm and further morphological studies were done. FTIR spectrum reveals that the characteristic band at 585 cm^{-1} is consigned to Fe–O of Fe_2O_3. The presence of phenol compounds, N-H bending of amide group and the pectins are the dominant species in the Aloe Vera gel extract which were responsible for the reduction of ferrous ion and stabilization of the Ɛ-Fe_2O_3 nanoparticles.

Keywords: Aloe Vera extract; Ferric Oxide nanoparticles; Green synthesis; X-ray techniques; FTIR

I. INTRODUCTION

Metal and metal oxide nanoparticles have been entrancing significant interest because of their wide applications.[1] Among the metal oxide nanoparticles, Ferric (III) oxide is a polymorphic compound. Under ambient conditions, the four different crystalline polymorphs of Ferric(III) oxide have been discovered and characterized in details[2-6] (i) α-Fe_2O_3 recognized as hematite has rhombohedral structure (ii) β-Fe_2O_3 has a cubic body-centred crystal structure (iii) γ-Fe_2O_3 recognized as maghemite has cubic structure, and (iv) ε-Fe_2O_3 has an orthorhombic structure. Ferric oxide nanoparticles have recently gained vast interest in various processes such as wastewater treatment [7], analytical instruments and biocatalysts [8-11] because of their large surface area, high sorption capacity and high surface reactivity.

Ferric oxide nanoparticles were synthesized by various physical and chemical methods. To escape the environmental contamination, the development of non-toxic, cost-effective, faster and eco-friendly process was required for synthesizing Ferric Oxide nanoparticles [12, 13].

Recently, green synthesis of Ferric Oxide nanoparticles of different size and shape has been developed using soya bean sprouts[14]; green tea[15-17] and sorghumbran extracts[18] .
The present paper reports the study of Aloe Vera leaf gel extract for the reduction and stabilization of Ferric oxide nanoparticles. The influence of pH during sample preparation and the effect of washing on the Ɛ-Fe_2O_3 nanoparticles were studied to know the characteristics of biomolecules present in sample during reaction which reduces and stabilizes the Ferric Oxide nanoparticles.

II. EXPERIMENTAL

Preparation of Plant Extract

For the synthesis of Ferric Oxide nanoparticles, 0.2 M $FeCl_3$ solution was prepared by using triple distilled water. Aloe Vera leaf gel extract were prepared by taking 2-3 leaf of Aloe Vera plant from the local garden that were thoroughly washed with distilled water and dried. Then collect their gel for fine crushing with the help of mortar and pestle by adding 5-10 ml of deionised water gradually. The mixture was poured in a beaker and heated for 10 minutes at 80^0C before finally decanting it. The mixture was sieved using Whatman No. 1 filter paper and then collected as the Aloe Vera leaf gel extract and used for further process.

Synthesis of Ferric Oxide nanoparticles

During the synthesis of Ferric Oxide Nanoparticles both the precursor and reducing agent were mixed in a clean sterilized flask in 2:1 proportion. For the reduction of Fe ions, 50ml of plant extract was mixed to 100 ml of 0.2 M aqueous $FeCl_3$ solution with constant stirring at 60-70^0C. After 10-15 minutes we observe the colour change from pale orange to dark red that indicates the formation of Ferric oxide nanoparticles. Then centrifuge the sample at 10000 rpm for 15 minutes and collect the supernatant for drying in hot air oven at 80^0C for 6 hours.

III. RESULTS AND DISCUSSION

pH analysis:-

The pH value of the solution plays a significant role in the mechanism of formation and stabilization of synthesized Ferric Oxide nanoparticles by green synthesis method. Aloe Vera leaf gel has shown a remarkable colour change from light orange to dark red with concerned change from 5.5 to 2.5 in pH of the Ferric solution.

X-Ray Diffraction analysis: -

The XRD pattern of as-prepared Ɛ-Fe_2O_3 nanoparticles was shown in fig-1. The XRD peaks was easily indexed to orthorhombic structure of Ɛ-Fe_2O_3 with lattice parameters a=5.095(Å), b=8.789(Å) and c= 9.437(Å); which were in good agreement with the reported values (JCPDS 521449). The average crystallite size (t) was calculated by using Scherer equation $t=0.9\,\lambda/\beta\cos\theta$, where β =broadening of the diffraction line (Bragg peak) measured at full width at half of its maximum intensity (FWHM), λ=X-Ray wavelength, θ= diffraction angle of the XRD spectra. The average crystallite size was calculated to be 10.2 nm.

The X-ray density was measured to be 5.02g/cc using the formula ($d = 8M/N_AV$) where *M* is the molecular weight, N_A is Avogadro's number and V is the cell volume.

The dislocation density; a crystallographic defect within a crystal structure was calculated by formula (D =1/t) where D =dislocation density and t =crystallite size. The dislocation density was found 9.61 x 10^{15} lines/m^2.

Specific Surface Area was found to be 112.26(m^2/g) using Sauter formula S= (6x1000)/(txd); where S =specific surface area; d =density of the material; t =crystallite size. SSA has particular significance in case of adsorption along with reactions on surfaces. [19]

FTIR :-

The infra red spectra were recorded on the Fourier transform spectrometer in mid-infrared range (400-4000 cm^{-1}).The fig-2 illustrates the FTIR spectrum of Aloe Vera leaf gel extract. The broad peak exhibited at 3406 cm^{-1} indicates the stretching of –OH groups of phenols and carbohydrate monomers which includes mannose and uronic acid. The peak at 2891 cm^{-1} was assigned for asymmetrical and symmetrical C–H stretching of aliphatic –CH2 and –CH groups.

The absorption peaks at 1637 and 1475 cm^{-1} are the characteristic of pectins present in Aloe Vera and are respectively due to the COO stretching of carboxylic ester (–COOCH3) and the asymmetrical and symmetrical –

COO^- of carboxylic acid (–COOH) groups. The C–O–C stretching of –COCH3 groups was observed at 1190 cm^{-1}. The peak at 967 cm^{-1} was due to C–O stretching associated with rhamnogalacturonan, a side-chain constituent of pectins [20]. The peak at 708 cm^{-1} represents C–H out-of-plane deformation of carbohydrate monomers.

In fig-3 the FTIR of synthesized Ɛ-Fe_2O_3 nanoparticles were shown. The peak at 3304 cm^{-1} represents the -OH stretching, a characteristic of the carbohydrate monomers that includes mannose and uronic acid. The peak at 2132 cm^{-1} was attributed for C=O stretching of carbonyl groups present in the Aloe vera samples. The presence of pyridine nucleus ring with N-H bending of amide group was observed at 1637 cm^{-1}. The peak at 1020 cm^{-1} may be due to C–O stretching associated with rhamnogalacturonan which was a side-chain constituent of pectins. The strong peak at 585 cm^{-1} attributes the Fe-O stretching vibration mode.

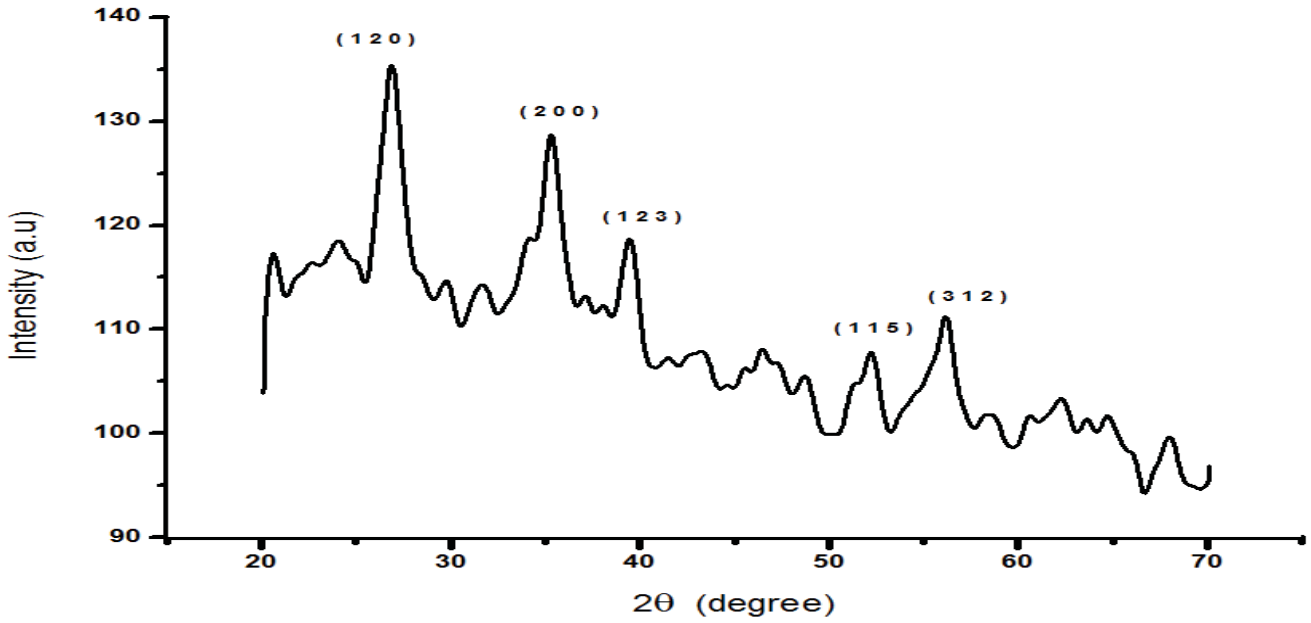

Fig 1:- XRD analysis of Ɛ-Fe_2O_3 nanoparticles

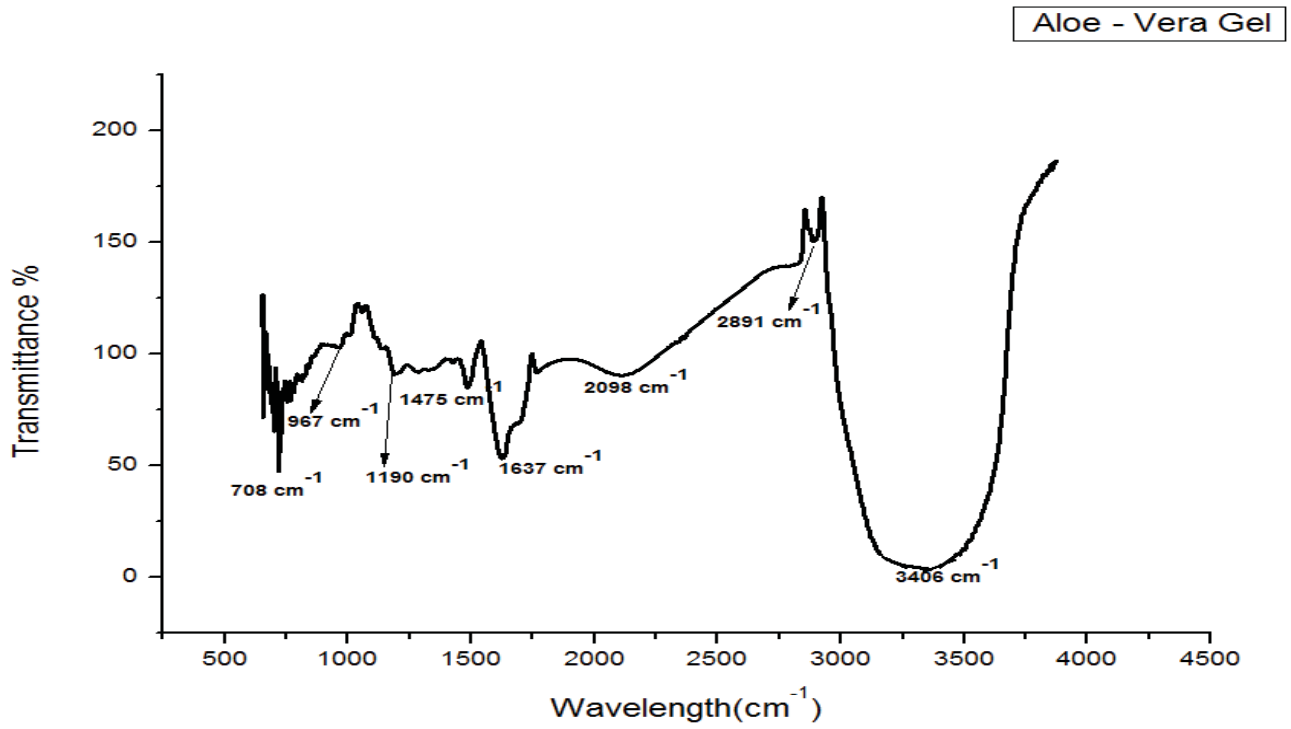

Fig 2:- FTIR analysis of Aloe–Vera leaf gel extract.

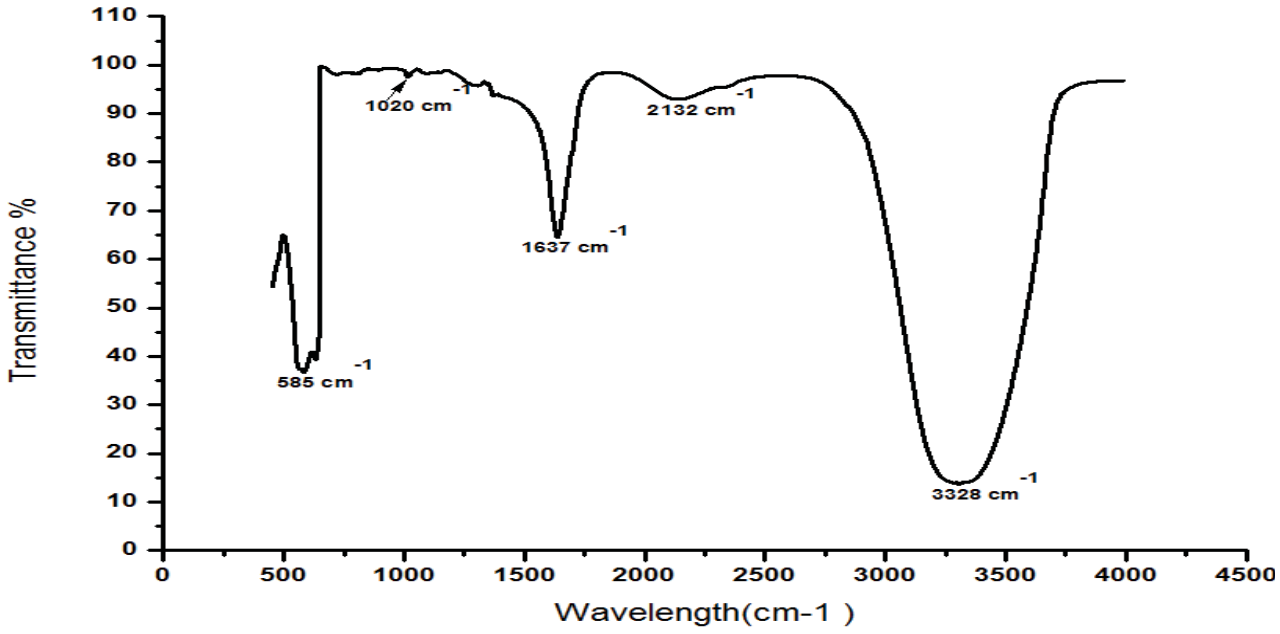

Fig 3:- FTIR analysis of Ɛ-Fe_2O_3 nanoparticles.

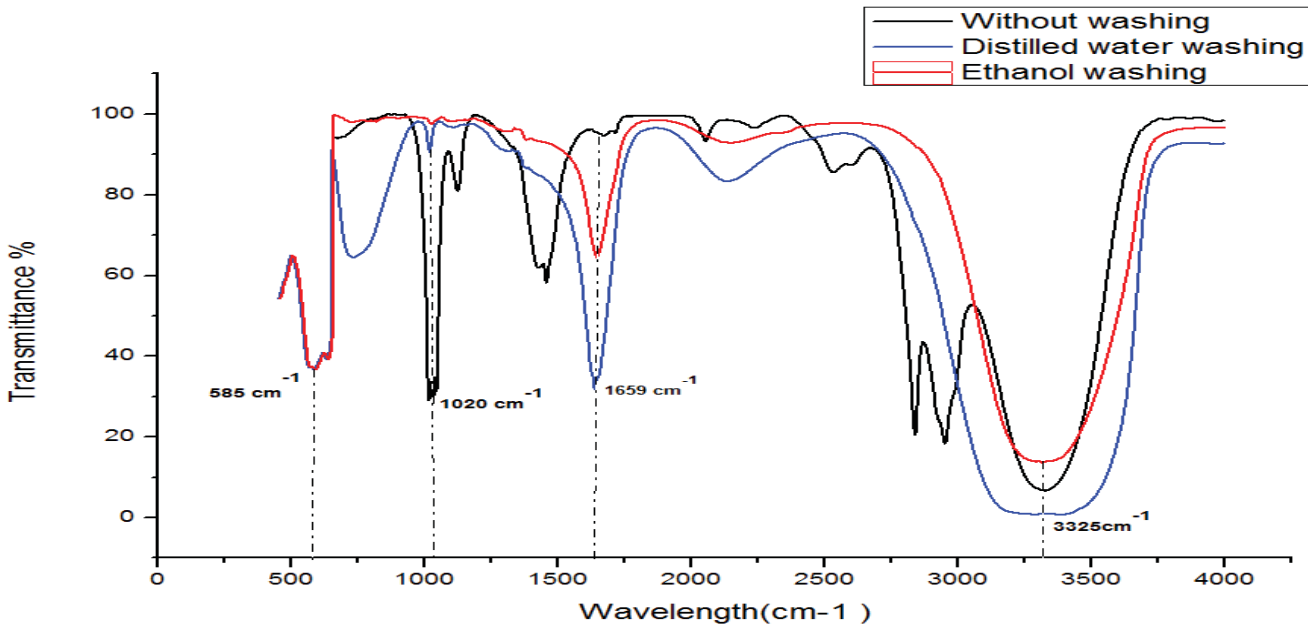

Fig 4:- Washing effect on the Ferric Oxide nanoparticles synthesis.

In fig.-4 the greater peak intensities at 3304 cm^{-1} and 1637 cm^{-1} with the peak at 1020 cm^{-1} that were retained after distilled water and ethanol washings. Suggest that the presence of –OH group of phenol compounds; the N-H bending of amide group and pectins are the dominant species in the Aloe Vera gel extract respectively were responsible for the reduction of ferrous ion and stabilization of the synthesized Ɛ-Fe_2O_3 nanoparticles.

IV. CONCLUSION

The Ɛ-Fe_2O_3 nanoparticles were prepared by using Aloe Vera leaf gel extract in a single-step green synthesis method. Synthesis mediated by Aloe Vera leaf gel extract is environmental benign, quite rapid and cost-effective also. The extract was capable of producing Ferric Oxide nanoparticles as the biomolecules present in the Aloe Vera gel extract reduces ferrous ion and was responsible for remarkable colour change with change in pH of the as-prepared solution. The size of particles formed were 10.2 nm studied by the XRD analysis. FTIR analysis identified the presence of phenol compounds, the N-H bending of amide group and the pectin are the dominant species in the Aloe Vera gel extract that were responsible for the reduction of ferrous ion and stabilization of the synthesized Ɛ-Fe_2O_3 nanoparticles.

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