PHYTOCHEMICAL PREPARATION, CHARACTERIZATION AND PHOTOCATALYTIC APPLICATION OF Ag - SiO_2 NANOCATALYST

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ABSTRACT

Silver nanoparticles (Ag NPs) were synthesized using Aloe vera extract as a reducing agent. Formation of Ag NPs were confirmed from their surface Plasmon resonance bands. Prepared nanoparticles were stabilized using a porous silica support (SiO_2) and characterized by various physio chemical techniques like UV-visible spectroscopy, Fourier-Transform Infra red spectroscopy (FTIR), powder X-ray diffraction (XRD), scanning electron microscopy (SEM) and atomic absorption spectroscopy (AAS). Catalytic efficiency of the prepared catalyst towards the degradation of crystal violet dye was tested through UV light irradiation. Effect of various parameters like pH of the medium, quantity of the catalyst etc., on the degradation efficiency were also studied and compiled in this paper.

Keywords: Photocatalysis, Phytochemical Synthesis, Silver Nanoparticles

I INTRODUCTION

Manifestation of size of a material to nano scale causes appreciable change in its properties, which can be used in practice for development of novel materials and technologies. Passion in the preparation of metal nanoparticles increases dramatically with the increase in number of publications for the past few years. However, the properties of NPs purely depend on their size as well as structure, shape and environment. Thus, control over the size and size distribution is an important task. Generally, specific control of shape, and size is often achieved by varying the synthesis methods, reducing agents and stabilizers [1]. Among the well known synthetic processes, use of hazardous reducing agents like borohydride salts, thiols etc.,[2] mounts a bias for the expected eco friendly approach. Hence a green chemical approach for the nanoparticle synthesis was adopted by various research groups [3-6] some of them include the use of microbes, plant extracts and so on. Among the plasmonic metal nanoparticles, silver nanoparticles (Ag NPs) have attracted much attention for various applications like catalysis, optoelectronics, bactericides, sensing probes for biological systems, information technology etc.,[7]. Despite the above mentioned applications, NPs find their own limitations when they are used as catalysts i.e., the difficulty in recovery and reusability. These colloidal NPs tend to aggregate or dissolve due to their low temperature stability during the course of reaction that leads to the difficulty in reuse. Hence it

is necessary to prevent the NPs from aggregation by using a suitable stabilizing agent. Herein we report the preparation of a Ag NPs using an aqueous extract of Aloe vera, stabilization of the prepared NPs using an inorganic support (SiO_2) and the preliminary photocatalytic behaviour of the stabilized catalyst towards the degradation of an organic pollutant crystal violet.

II EXPERIMENTAL DETAILS

Silver nitrate, Silica and Crystal violet were purchased from Rankem and were used as such without any further purification. Aloe vera is collected from the neighbouring irrigation field. Aqueous extract of Aloe vera was obtained by refluxing 20g of Aloe vera in 100mL of double distilled water. To an aqueous solution of $AgNO_3$ (0.1M), calculated quantity of extract was added and stirred under dark at room temperature and frequently monitored by UV-Visible spectroscopy for the formation of NPs. To load the Ag NPs on SiO_2 two different approaches were made. In the first approach calculated quantity of SiO_2 was dispersed in 0.1M $AgNO_3$ solution under dark. Then it was filtered, washed with distilled water and the Ag^+ loaded SiO_2 was redispersed with distilled water, reduced by adding optimised quantity of extract and stirred at room temperature for 6 hrs. In the second approach SiO_2 was added to the already prepared AgNps and stirred at RT. Finally the samples were filtered, washed with double distilled water, dried and named as Ag-SiO_2 I and Ag-SiO_2 II respectively.

Above prepared Ag NPs and the Ag-SiO_2 composites were characterised by the following state of the art techniques. UV- visible absorption spectrum was recorded at room temperature with JASCO- UV VIS spectrophotometer. The powder X- ray diffraction patterns were recorded using PANALYTICAL X – Ray diffractometer (Cu- Kα radiation, λ= 1.54Å) in 2θ range from 20- 80°. The SEM images of the samples were recorded on JEOL JSM- 6490L A scanning electron microscope. Concentration of silver in the composites was estimated using PERKINELMER atomic absorption spectrophotometer. Particle size of the materials were determined from the HORIBA particle size analyser.

III RESULTS AND DISCUSSION

Figure -1a shows the UV- Visible spectrum of mixture of 100 mL aqueous 0.1M $AgNO_3$ and 1mL or 2mL of extract at different reaction time. When the reaction was performed with 1mL extract, formation of Ag NPs was not observed even after 6 h but for the reaction with 2mL extract possesses a broad surface Plasmon resonance absorption between 410 nm and 590 nm after 4 hrs of the reaction and intensifies after 6 hrs, which confirms the formation of Ag NPs [6,8]. Powder XRD pattern of Ag NPs loaded on SiO_2 is given in figure – 1b. In both the methods adopted for loading of NPs on the support, the diffraction peak is originated at 2 θ = 23.4˚ corresponds to the support (SiO_2). No peaks for the elemental silver (predominant one at 2 θ = 38.1˚) is appeared in the difractogram, probably due to detection limit of the instrument [9]. But the presence of Ag in the composite was quantified by AAS and it was found to be 12% and 9.6% correspondingly for Ag-SiO_2 I and Ag-SiO_2 II.

Fig – 2 (a & b) is the SEM images of Ag-SiO_2 I and Ag-SiO_2 II respectively. Patches of Ag NPs formed were dispersed on the rock like porous SiO_2 support is clearly identified. Images of the dispersed Ag NPs were given as inset. Since the particle size of the composites was not identified from the SEM, but was obtained from the particle size analyser and the results are given in TABLE – 1. From the results, the average particle size of the

composite is greater than the prepared Ag NPs as well as SiO_2, which clearly indicates the loading of Ag on SiO_2 resulting in the formation of a composite (Ag-SiO_2).

3.1. Photocatalytic activity

Among the prepared composites, amount of Ag loaded was found to be maximum in the case of Ag-SiO_2 I and hence the photocatalytic efficiency of the prepared composite was tested with crystal violet dye (1 X 10^{-4}M) under UV irradiation and the efficiency of the degradation was calculated as per the literature [10]. Effect of catalyst concentration on the degradation was studied. Reaction did not proceed at all in the absence of the catalyst. But upon increasing the catalyst quantity from 5mg to 10mg, the degradation efficiency increases from 59% to 92% and further increase upto 20mg didnot show any appreciable change (only 95%). Hence, the optimum catalyst quantity for the heterogeneous photodegradation of crystal violet was found to be 10mg. Lower catalyst concentration suffer lack of reactive sites but higher concentration lead to the backscattering of radiation rather than the interaction with the dye. Effect of pH of the reaction medium on the degradation efficiency was also tested and it was found that pH-9 is the optimum one for the effective degradation.

FIGURES

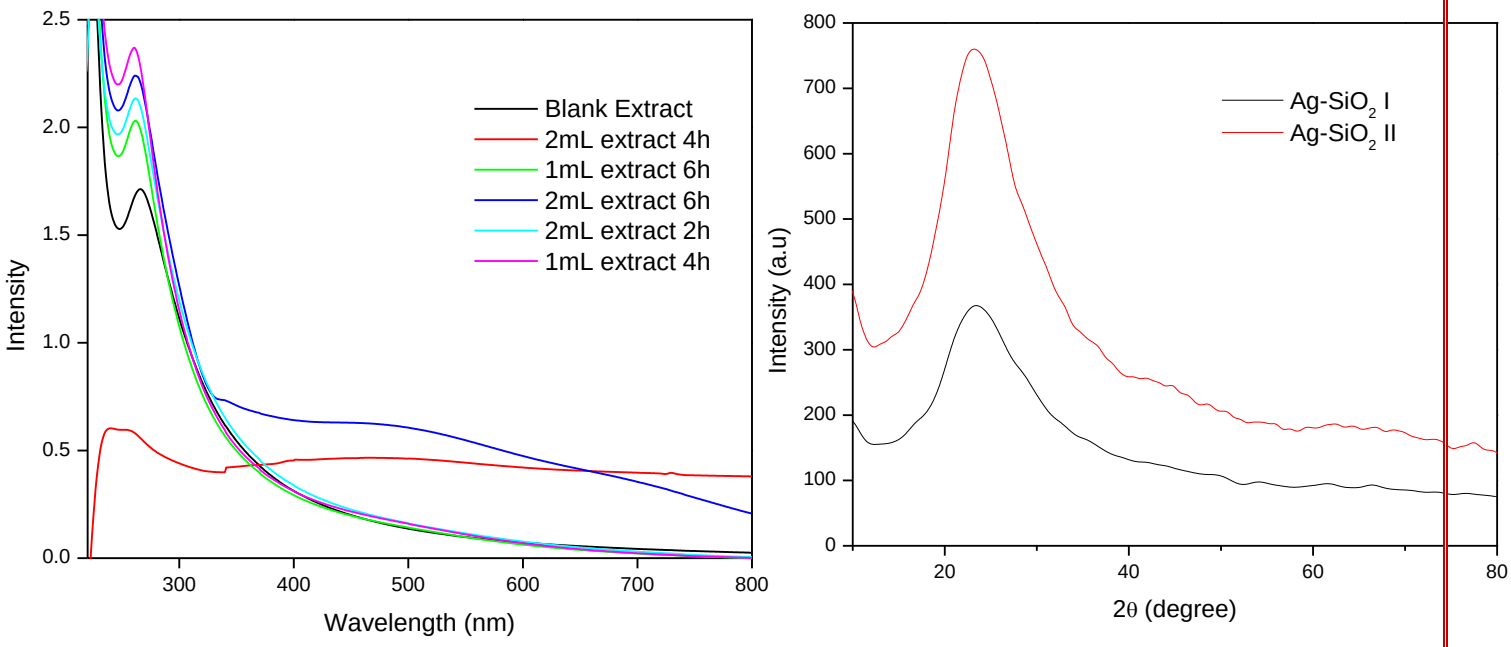

Figure – 1: a) UV-Visible spectrum of Ag NPs, b) Powder XRD pattern of composite

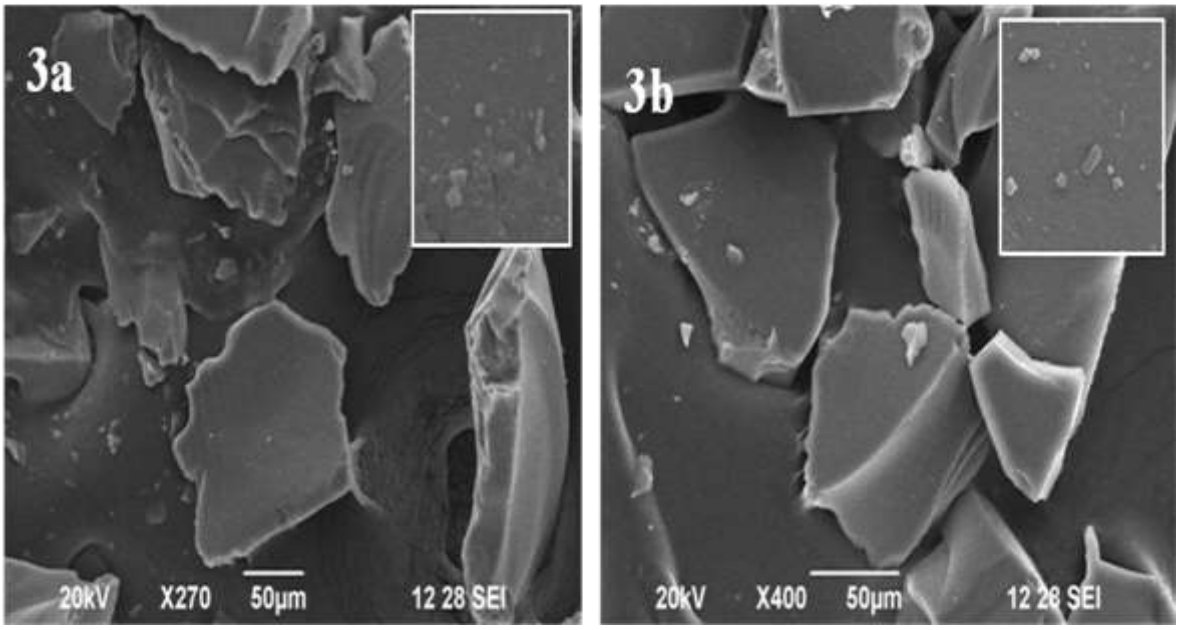

Figure – 2: SEM images of a) Ag-SiO_2 I and b) Ag- SiO_2 II

TABLES

Table 1 : Particle size data

S. No	Sample	Particle Size (nm)
1	Ag NP	104
2	SiO_2	205
3	Ag-SiO_2 I	321
4	Ag-SiO_2 II	396

IV CONCLUSION

Ag NPs were prepared by a simple eco-friendly approach using Aloe vera extract and loaded on SiO_2 support. The composite was characterized and tested for its activity towards heterogeneous photodegradation of a model substrate crystal violet dye. Since the composite is efficient in catalyzing a photochemical degradation, it can be further extended for its photocatalytic activity towards various important chemical reactions.

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