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Microwave-Mediated Green Synthesis of Silver Nanoparticles Using *Ficus Elastica* Leaf Extract and Application in Air Pollution Controlling Studies

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ABSTRACT

Silver Nanoparticles are applied in various fields due to its anti bacterial properties. A conventional method for synthesis of AgNP requires dangerous chemical and large amount of energy is released in the process. Environmental friendly techniques are adopted for the synthesis of nanoparticles of silver. The present research work summarizes the green synthesis of silver nanoparticles by using leaf extract of *Ficus Elastica* and alternative energy sources micro wave irradiation. The synthesized Nanoparticles are characterized by uv-visible spectroscopy and by SEM. The synthesized nanoparticles are applied for controlling SO_2 and NO_2 from aqueous solution of SO_2 and NO_2. Batch adsorption studies are carried out. The effect of the temperature on adsorption of aqueous solution is studied at different temperature. A comparison of kinetic models applied to the adsorption of on silver Nanoparticles was evaluated for the pseudo first order, pseudo second order, Elovich and intraparticle diffusion models respectively. Results show that pseudo second order model was found to correlate the experimental data. Data fitted perfectly into and Freundlich adsorption isotherms.

***Key words*:** Batch adsorption, green synthesis, silver Nanoparticles, adsorption kinetics, adsorption isotherms.

I. Introduction

"Pollution" is the unwanted alteration in environmental parameters by means of mixing of certain objects, called as pollutants. Among all the kinds of pollution, air pollution is one, and has the deploying impacts over humans and atmosphere. There are several techniques available to control air pollution but application of nanotechnology in environmental pollution control even in initial stage only. Nanotechnology is one of the modern and sophisticated technologies which create waves in the modern times. The opportunities for novel application continuous to grow as more and more knowledge are obtained about the Nanoparticles of Ag. The promise of these developments appears to be able to enhance the quality of life through novel applications of nonmaterials. The ability to produce efficiently and light weight of significant interest of research. Taking that factor into consideration, the present study and application of air pollutant dissolved in aqueous solution.

There are so many ways to synthesize nanoparticles, but greater emphasis is given on "*Biobased microwave synthesis*". In present study, the AgNPs were synthesized by using leaf extract of *Ficus elastica* with reference of previously done studies such as by using leaf extract of *Acalypha indica* [1], *Helianthus annus* [2], Pine [3], *Moringa oleifera* [4], *Camellia sinensis* [5], *Trianthema decandra* [6]. The physical characterization of synthesized AgNPs was done by scanning electron microscope (SEM) and UV- Vis, Spectrophotometer.

The present study deals with the removal of NO_2 & SO_2 from atmosphere. The reason behind selection of these gases is their carcinogenic trait. These gases are found to cause cancer especially breast cancer [7], lungs cancer & cardiac disorders [8], mainly in the industrial surrounding localities.

II. Materials and Methods

Preparation of Adsorbent (Ag Nanoparticles)

Source of $AgNO_3$

Chemicals used in the present study all are including silver nitrate are analytical grade and used as it is. There is no further purification procedures are followed.

Preparation of Leaf Extract:

The fresh leaves of Ficus elastica were collected from vikarabad forest, located 40 km away from Hyderabad. The leaves were washed with tap water as well as distilled water to remove dust particles which present on the surface of leaves. Then leaves were allowed for dry for 20 minutes then weighed 25 grams of leaves and grinded it by using laboratory mortar and pestle. The leaf extract collected in a 25 ml standard flask and kept in refrigerator for further use.

Preparation of Silver Nanoparticles:

Many techniques, including chemical and physical means, have been to prepare metal nanoparticles, such as chemical reduction using a reducing agent, electrochemical reduction, photo chemical reduction and heat evaporation method. Not only physicists and chemists, but also the biologists are highly interested in synthesizing nanoparticles of different shapes and sized by employing bio-based synthesis of nanometals using plant leaf extracts. In the present study to the 25 ml leaf extract 100 ml of 0.1 M $AgNO_3$ solution was added and kept it in microwave oven at 180 watts for 30 minutes. Formations of silver nanoparticles were checked by using UV-vis spectrophotometer (Lambda Scientific Spectrophotometer) at regular intervals. This naturally occurring nanoparticles are generated by the erosion and chemical degradation of plants [9].

Characterization of Silver Nanoparticles:

The characterization technique involves UV-vis absorption spectra, Scanning Electron Microscope (SEM), Fourier transform infrared spectroscopy (FT-IR).

Preparation of Standard Samples:

A 100 ppm stock solution of Sulphite prepared by dissolving 0.1575 gm of Analytical grade Sodium sulphite in double distilled water and made up to 1000 ml with double distilled water. A 100 ppm stock solution of nitrite prepared by dissolving 177 mg of Analytical grade sodium nitrite in 1000 ml standard flask and made up to the mark with double distilled water. From the stock solution of Sulphite and Nitrite the working standards are prepared by taking appropriate quantities to get the desire concentration of SO_2 and NO_2. The working standards are treated with silver nanoparticles and checked for percentage removal by using Uv-Vis Spectrophotometer. Batch Adsorption Studies are carried out for the removal of SO_2 and NO_2. The percentage removal of SO_2 and NO_2 is calculated by using the following formula.

$$\% \text{ Removal} = \frac{\text{Initial Concentration} - \text{Final Concentration}}{\text{Initial Concentration}} \times 100$$

Experimental Procedure:

In the present study an attempt has been made to suggest a new method application on green synthesized silver nanoparticles in air pollution controlling technologies. Batch adsorption experiments were adopted for the removal studies. The estimation of SO_2 is done by west geak method and NO_2 estimation by NEDA method. The experiments are conducted with respect to contact time, effect of SO_2 concentrations, silver nanoparticles dosage and temperature effect.

Effect of Contact time and Kinetic models:

Contact time plays an important role in designing the system [10]. The initial (before batch adsorption) and final (after batch adsorption) concentrations of SO_2 and NO_2 are determined at regular time intervals i.e. 5, 10, 15, 20, 30, 40, 45, 50 and 60 minutes. In order to investigate the mechanism of adsorption several kinetic models were tested including pseudo first order kinetic model, the Elovich model and pseudo second order kinetic model for batch adsorption studies [11].

Effect of Initial concentrations of SO_2 on Silver nanoparticles:

Different concentrations of aqueous solutions of SO_2 were studied with a constant amount of adsorbent (silver nanoparticles) and constant contact time. The initial and final concentrations of SO_2 were determined by spectrophotometer. The results are given in figure-12.

Effect of Adsorbent Dosage:

To a series of constant concentrations of SO_2 solutions, different amount of adsorbent were added and allowed it for batch adsorption for 60 minutes to get equilibrium. After batch adsorption samples were tested for percentage removal by using spectrophotometer.

Effect of Temperature

To study the effect of temperature, the experiments are conducted at following temperature 0, 20, 40, 60, 80 and 100 ^{O} C.

Initial screenings have been done by taking both SO_2 and NO_2 separately. Finally it was observed that decrease in SO_2 concentration but there is no change in NO_2 concentration. It means silver nanoparticles do not have the capacity to remove aqueous NO_2 solution. So effects of contact time, concentration and adsorbent dosage have done by taking only with SO_2 solution.

III. Results and Discussion

UV- Visible Analysis of Silver Nanoparticles

The distinctive colors of colloidal silver are due to a phenomenon known as Plasmon absorbance. Incident light creates oscillation in conduction electrons on the surface of the nanoparticles and electromagnetic radiation is absorbed. The spectrum of clear brownish green colloidal silver from the synthesis. The Plasmon resonance produces a peak near 400 nm. It is already been reported that the absorption spectrum of aqueous $AgNO_3$ only solution exhibited λ max at about 220 nm where as silver nanoparticles λ max at about 430 nm [2]. The wavelength of the Plasmon absorption maximum in a given solvent can be used to indicate particle size. If the λ max in between 395 nm- 405 nm then the size of the particle is in between 10-14 nm, if the λ max 420 nm then the particle size is in between 35-50 nm and the λ max 438-440 indicates that the formed nanoparticles are around 60-80 nm in size.

Thus, color change of the solution clearly indicated the formation of AgNPs. The color intensity of the cell filtrate with $AgNO_3$ was sustained even after 24 h incubation, which indicated that the particles were well dispersed in the solution, and there was no obvious aggregation .All these reactions were monitored by ultraviolet-visible spectroscopy of the colloidal AgNPs solutions. The ultraviolet-visible spectra of the cell filtrate with $AgNO_3$ showed a strong broad peak at 430 nm which is surface Plasmon resonances (SPR band), which indicated the presence of AgNPs (Figure). These results were consistent with the reports of Verma et al 2010 [12]. The intensity of the SPR band steadily increased from 15 min to 25 min as a function of time of reaction.

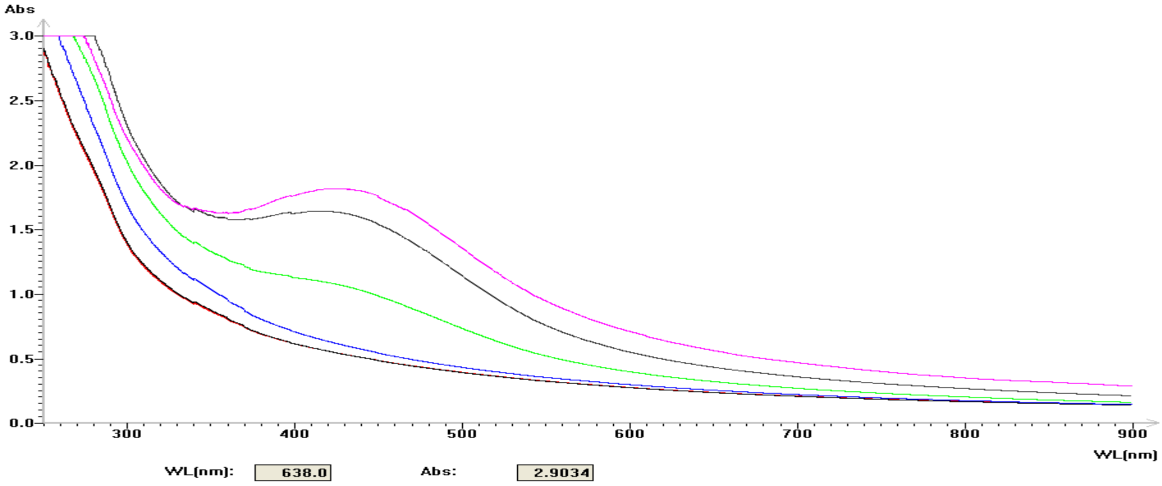

Figure-1: Graphical form of absorbances for synthesis of AgNPs by using Ficus elastica leaf extract

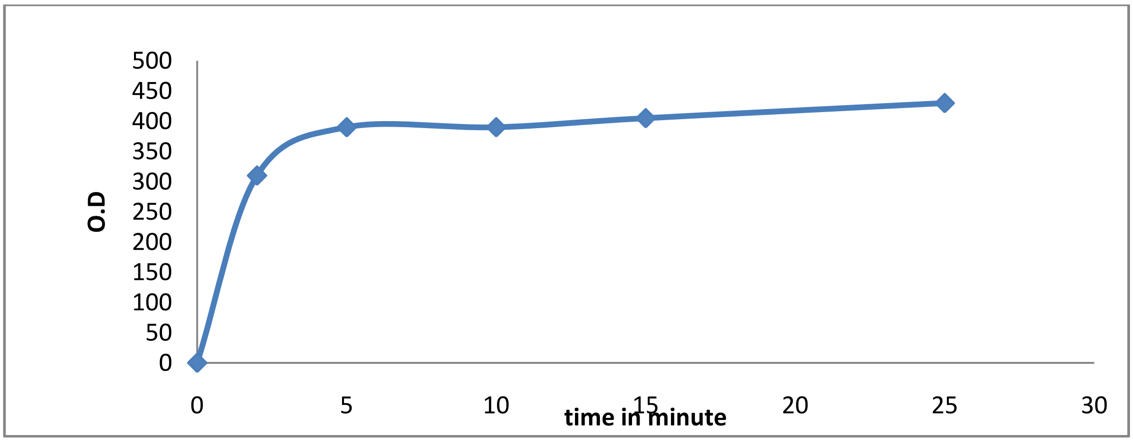

Figure-2: Graphical form of Relationship between λ$_{max}$ and time of introduction

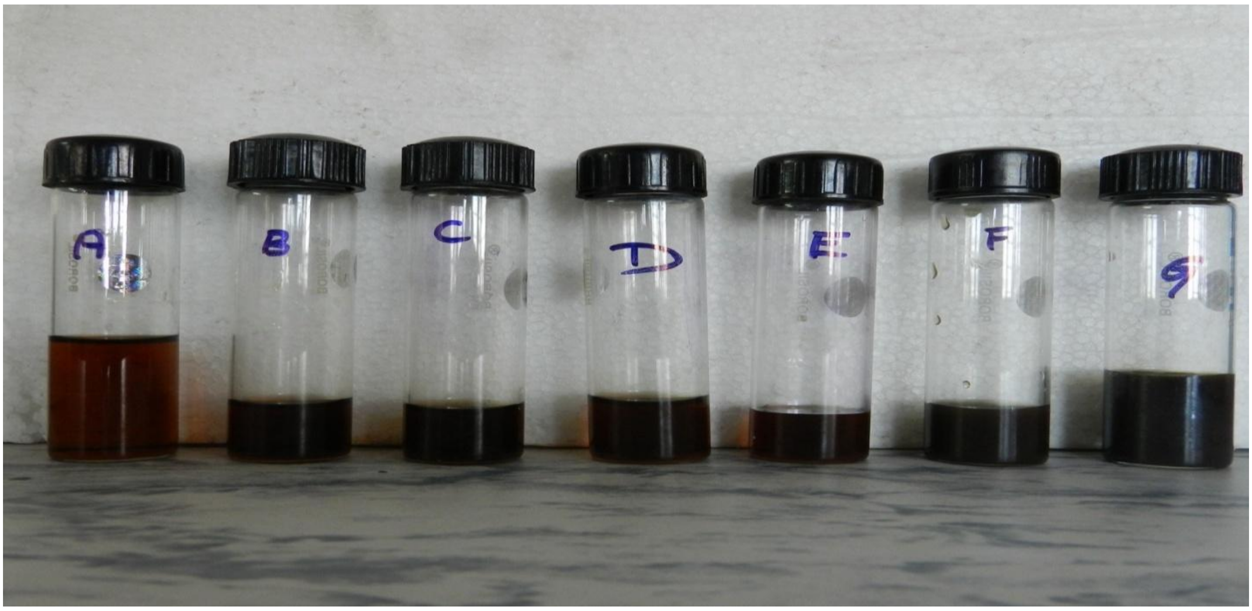

Figure-3: Change in colour of Ficus elastica leaf extract during nanoparticle synthesis

From the figure-3 and graph it was concluded that the formed particles are silver nanoparticles with the size around 50-60 nm. The application of AgNPs was highly dependent on the chemical composition, shape, size, and monodispersity of particles [13] to broaden the application scope; the AgNPs obtained were systematically characterized using SEM and FT-IR analysis. Through the SEM analysis, the particles were spherical and polydisperse with an average size of 50 nm, and the majority of the particles were less than 50 nm.

Particle Size Measurement using SEM

Silver nanoparticles that produced the spectrum were examined using Scanning Electron Microscopy. A sample of silver nanoparticles from freshly prepared clear brownish green solution was centrifuged at 8000 rpm for 5 minutes and redispersed in distilled water. This procedure was repeated three times and the remnant pellets were dried and powdered for SEM analysis. A thin film of the sample was prepared by dissolving a portion of the powdered particle in sterile distilled water on small glass cover slip and set on a copper stab for electron microscopy. The results are given in figure-4.

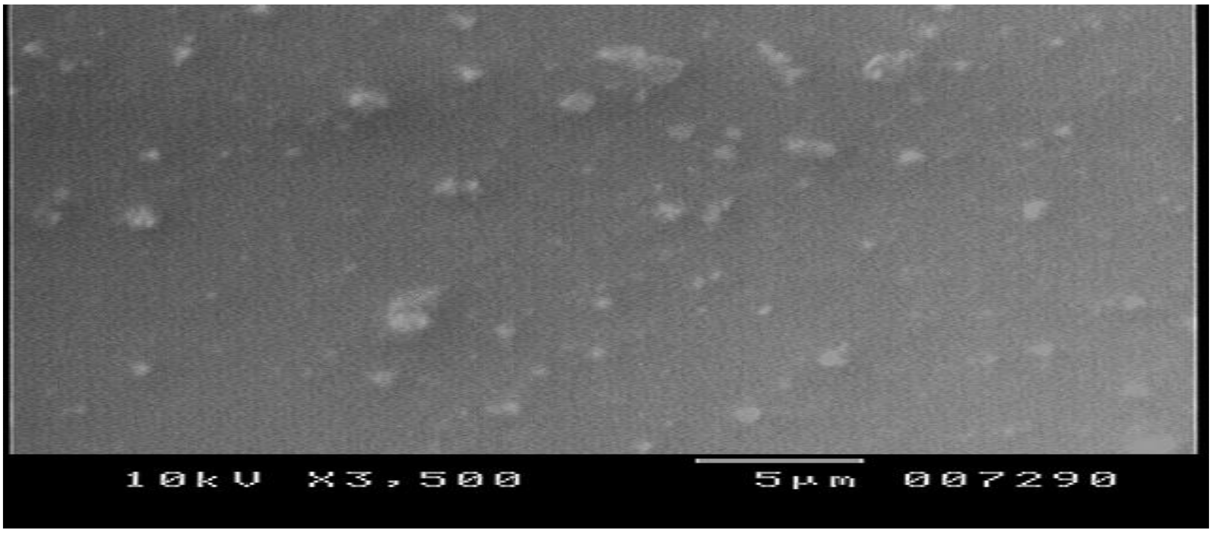

Figure-4: SEM image of synthesized silver nanoparticles. Fourier transforms infrared spectroscopy (FTIR) Analysis

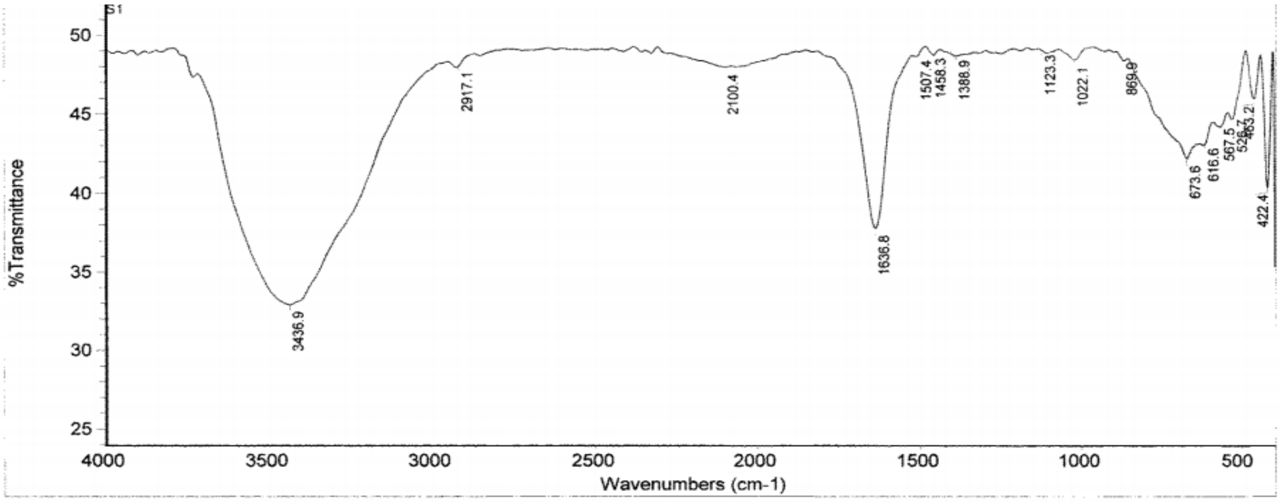

Figure-5: FT-IR analysis plot of Synthesized Silver Nanoparticles:

FTIR studies are carried for identifying the bio molecules responsible for capping and reducing AgNPs from the extract. The peaks at 3436.9 and 2917.1 were due to the stretching of primary and secondary amides respectively [14]. The peaks at these two regions are characteristic of proteins that are responsible for the reduction of metal ions when using leaf extract. The peaks arise due to carbonyl stretch and N-H stretch in vibrations in the amide linkage of the proteins. The study confirms amino groups and peptide bonds of proteins form a capping agent and maintaining the stability [15-17].

3.1 Effect of Contact time and Kinetic studies

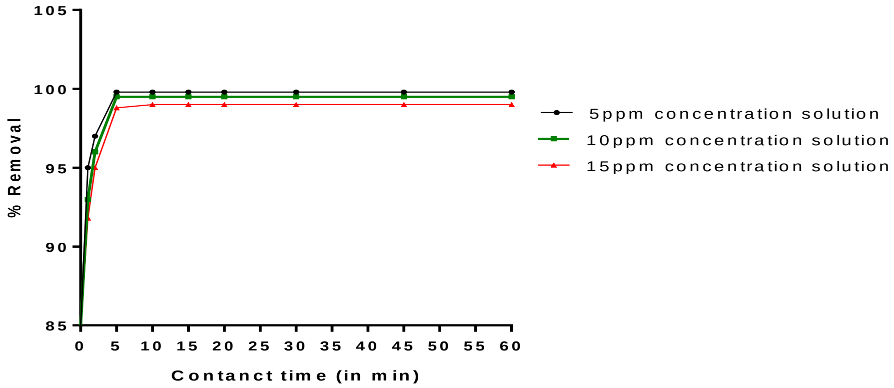

Fig- 6: Variation of contact time on SO_2 removal

From the figure-6, it was observed that the percentage removal of aqueous SO_2 is increasing with increase in contact time and it became constant at optimum contact time [18]. The experiment was performed taking three different concentration of SO_2 solution and checked for contact time effect and it is showing same results for all three different concentrations. The initial rapid adsorption showed a very slow approach to equilibrium. The nature of adsorbent and its available sorption sites affected the time needed to reach equilibrium [19], for silver nanoparticles this time was 5 minutes.

Kinetic Studies

In order to check the kinetic parameters of removal of SO_2 using silver nanoparticles, the experimental data were tested with pseudo first order kinetic model, pseudo second order kinetic model and Elovich kinetic model. This adsorption kinetics is used to investigate the mechanism and the rate controlling steps of adsorption. The mechanism of adsorption involves the chemical reaction of functional groups present on the surface of the adsorbent and the adsorbate, temperature [20].

Pseudo first order kinetic model

This model explains that the rate of change of solute uptake with time is directly proportional to difference in saturation concentration and the amount of solid uptake with time [21]. The plot of log ($q_e - q_t$) versus t should give a straight line with slope of –k/2.303 and intercept log q_e. Where qe and q_t are the amount of SO_2 adsorbed at equilibrium and at contact time t. From the slope and intercept of the plot the adsorption rate constant and equilibrium adsorption capacity were calculated [20]. From the graph (Figure- 7) and statistical report (Table-1) it was observed the present study removal of SO_2 by silver nanoparticles is following pseudo first order kinetic model.

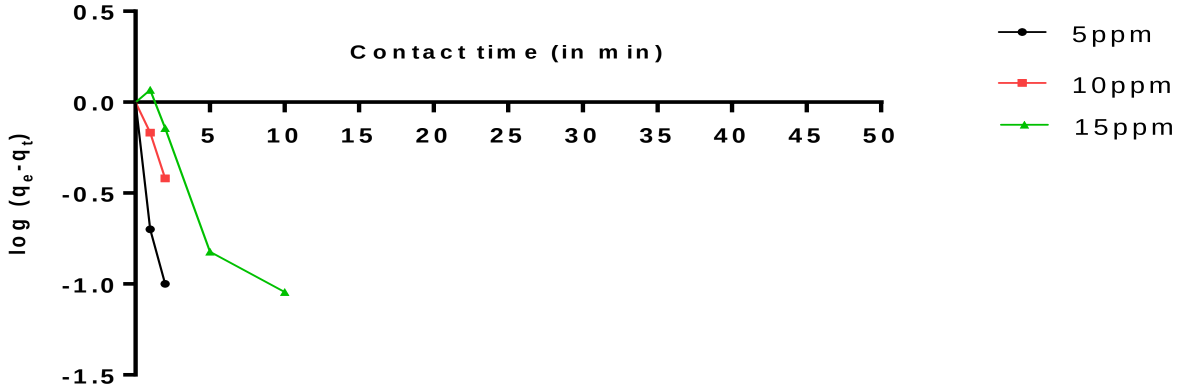

Figure- 7: Graphical representation of Pseudo first order kinetic model

Pseudo Second order kinetic model

The pseudo second order kinetic order equation expressed as

$$t/q_t = \frac{1}{k_2 q_e^2} + t/q_e$$

Where k_2 is the rate constant of pseudo second order adsorption (g/mg min) and q_e is the equilibrium adsorption capacity (mg/gm) [22]. Figure-8 shows the pseudo second order plot for the adsorption of SO_2 using silver nanoparticles at different initial concentration at room temperature.

When the pseudo second order kinetic is applicable, the plot of t/q_t against t should give a linear relationship from which k_2 and q_e determined respectively from the intercept and slope of the plot. The figure showing the plot of pseudo second order kinetic model with linear relation. The r^2 values are higher than the values of pseudo first order kinetic model. It is observed form the figure that the adsorption of SO_2 by silver nanoparticles perfectly fits to the pseudo second order kinetic model.

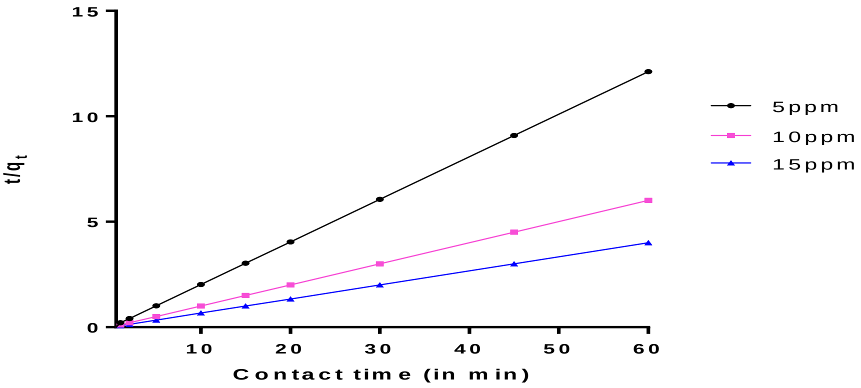

Figure- 8: Graphical representation of Pseudo second order kinetic model

Elovich Model

The Elovich model equation is generally expressed as [23-24]

$dq_t/d_t = \alpha \exp(-\beta q_t)$

Where:

α is the initial adsorption rate ($mg \cdot g^{-1} \cdot min^{-1}$)

β is the desorption constant ($g \cdot mg^{-1}$) during any one experiment.

If the adsorption of aqueous SO_2 solution by silver nanoparticles fits to the Elovich model, a plot of q_t versus In(t) should give a linear relationship with a slope of (1/β) and an intercept of 1/β In (αβ). The results of Elovich plot for the adsorption of SO_2 by silver nanoparticles at various initial concentrations are given in figure-9. Form the table-1 and figure-9 it was observed that the Elovich model does not fit for the adsorption of SO_2 by silver nanoparticles. The Elovich model mainly applicable for chemical adsorption kinetics [25].

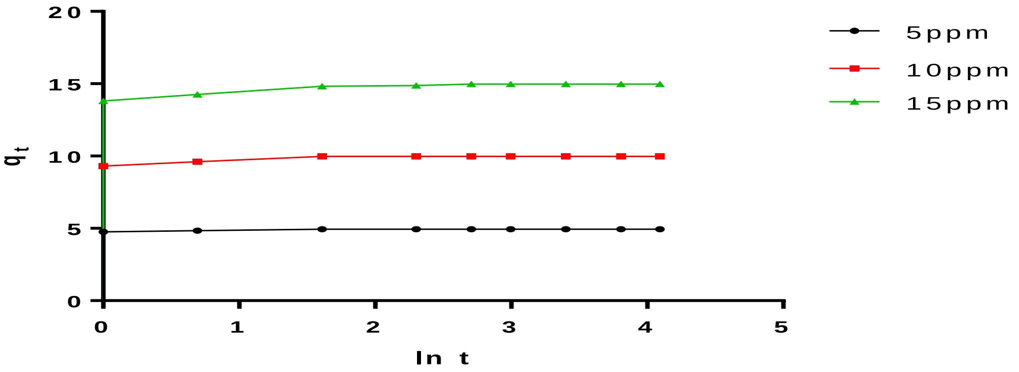

Figure- 9: Graphical representation of Elovich kinetic model

Intra particle diffusion model analysis

The adsorption of aqueous SO_2 solution by silver nanoparticles fits into pseudo second order kinetic model. To find out the adsorption mechanism the data is tested with intra particle diffusion model. Adsorption is a multi step process involving transport of the solute molecules from the aqueous phase to the surface of the solid particulates followed by diffusion into the interior of the pores [20]. Assuming that the rate is controlled by pore and intra particle diffusion, the amount adsorbed (q_t) is proportional to the $t^{1/2}$, as shown below

$$q_t = k_{id}t^{1/2} + I$$

where q_t is the amount of SO_2 adsorbed (mg/g) at time t (min), and I is the intercept (mg/g). k_{id} and I values are obtained from the slopes and intercept of the linear plot. Figure-10 represents the plots of q_t versus $t^{1/2}$ for adsorption of SO_2 by silver nanoparticles at various initial SO_2 concentrations. From the figure and table it was observed that the adsorption mechanism is not fits to intra particle diffusion model.

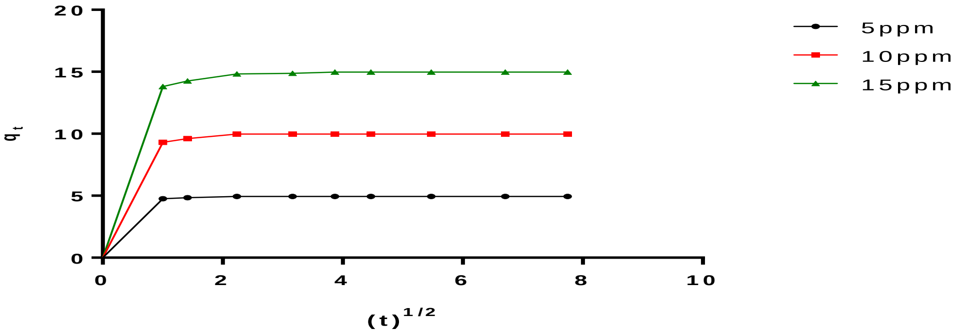

Figure-10: Intraparticle diffusion plots for the removal SO_2 by silver nanoparticles.

3.2 Effect of Initial Concentration of SO_2 on AgNps

The Adsorptive removal of SO_2 from aqueous solution is purely depends on the concentration SO_2 and available binding sites of adsorbent and its nature. If the concentration of SO_2 is increased the percentage of removal might be increase or decrease. In present study (Figure-11) the percentage removal of aqueous SO_2 solution is decreases with increase in initial concentration of SO_2 solution [26]. However at higher concentration the available sites of adsorption becomes fewer and the percentage of removal is decrease.

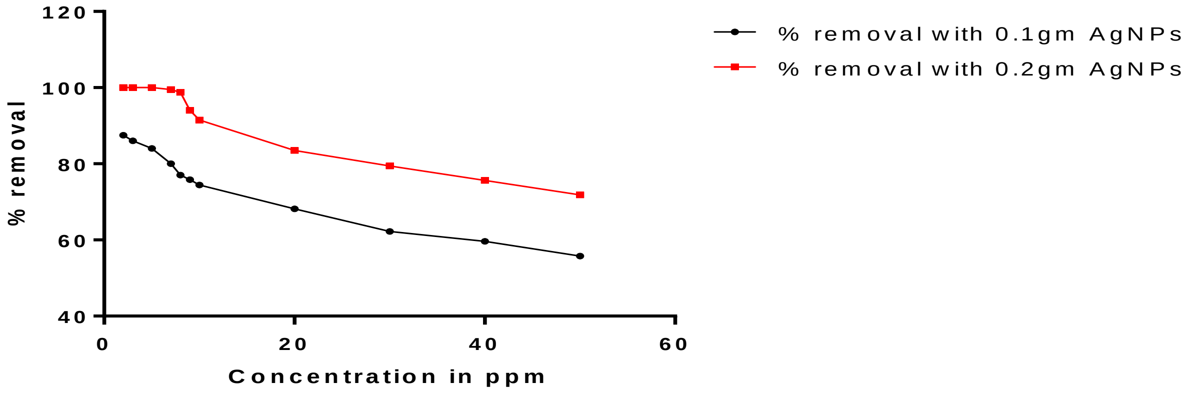

Figure-11: Effect of initial SO_2 concentration on adsorption process

3.3 Effect of Adsorbent Dosage

Figure- shows the effect of adsorbent dosage on the percentage adsorption. From the figure it was observed that the percentage removal of aqueous SO_2 is increasing with increase adsorbent dosage and it became constant at optimum adsorbent dosage of 0.3 gm/ 100 ml of 10 ppm concentrated solution. The reason behind this study the initial concentration of SO_2 is constant and the amount of silver nanoparticles are varies so the availability of binding sites are more.

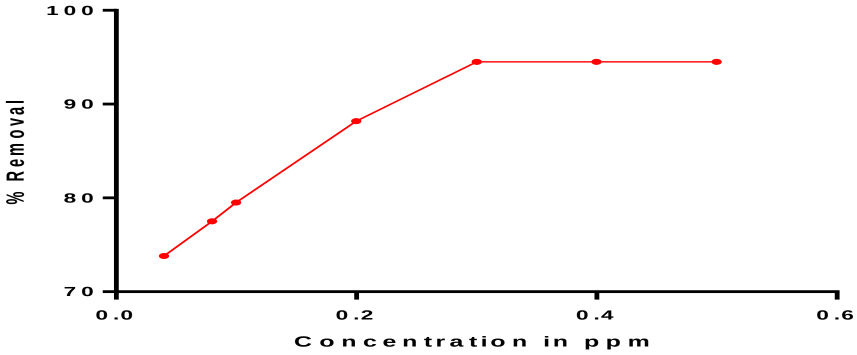

Figure-12: Effect of adsorbent dosage on SO_2 removal

3.4 Effect of Temperature

Temperature has an important effect on the process of adsorption. The percentage of SO_2 adsorption is studied as a function of temperature. The results obtained are presented in figure-13 at temperatures of 0°C, 40°C, 60°C, 80°C and 100°C. The decrease in percentage of adsorption with rise in temperature may be due to desorption caused by an increase in the available thermal energy. Higher temperature induces higher mobility of the adsorbate causing desorption [27].

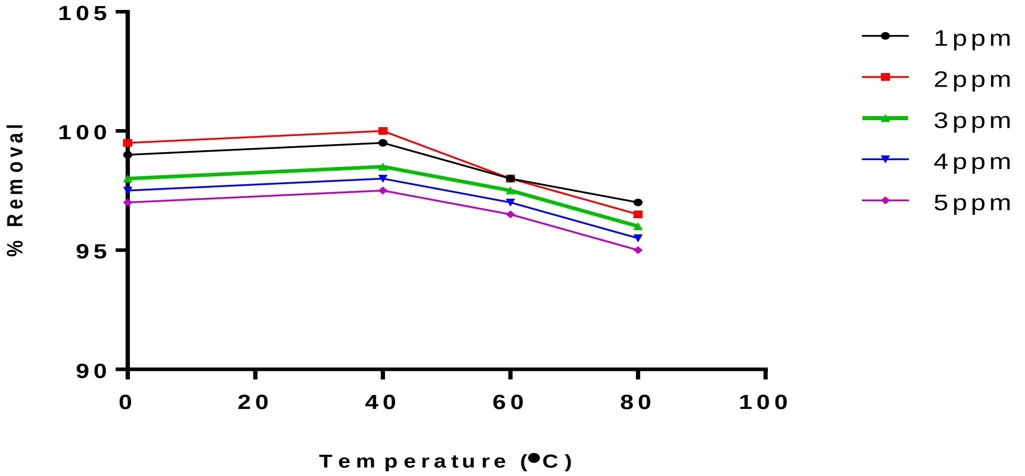

Figure- 13: Effect of Temperature on Adsorption of SO_2

3.5 Adsorption isotherms

3.5.1 Langmuir adsorption isotherm

The Langmuir adsorption isotherm is the best known linear model to determine the adsorption parameters. Langmuir model represented by following equation

$$q_e = \frac{K_L C_e}{1+ b_L C_e} = \frac{Q_0 b C_e}{1+ b_L C_e}$$

The linearized form of Langmuir adsorption isotherm is as follows

$$C_e = \frac{1}{Q_0 b_L} + (1/Q_0)\, C_e$$

Where q_e is the amount adsorbed at equilibrium (mg/g), Q_0 is the monolayer adsorption capacity (9mg/g), C_e the equilibrium concentration of adsorbate (mg/L) and b_L is the Langmuir constant related to energy adsorption. The figure-14 shows the plot of C_e/q_e against C_e. Values of Q_0 and b_L are in table-2. From the figure-14 and table-2 it was observed that the adsorption isotherm studies of removal of SO_2 by silver nanoparticles does not fit for the Langmuir isotherm.

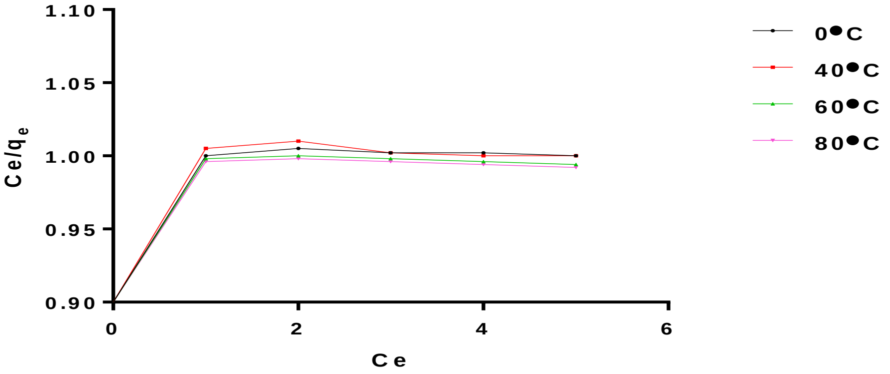

Figure-14: Langmuir adsorption plot for SO2 removal at different temperature

3.5.2 Freundlich adsorption isotherm

The Freundlich equation is an empirical relationship describing the adsorption of solute from liquid to a solid surface. The Freundlich equation is expressed as

$q_e = k_f C_e 1/n$

The linear form of Freundlich equation is as follows

$\text{Log } q_e = \log k_f + 1/n (\log C_e)$

Where k_f is the Freundlich adsorption capacity and n is the adsorption intensity. A plot of log q_e versus log C_e gives a linear line with slope of 1/n and intercept of log k_f given in table-2. From the figure-15 and table-2 it was observed that the adsorption of aqueous SO_2 at different temperature perfectly fits in to Freundlich isotherm. The r^2 value and ASS, values are more comparing to Langmuir isotherms.

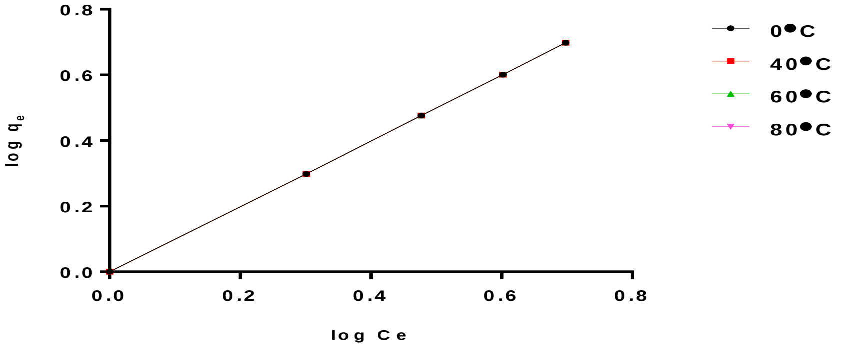

Figure-15: Freundlich adsorption isotherm plots of SO_2 removal at different temperature

3.5.3 Temkin adsorption isotherm

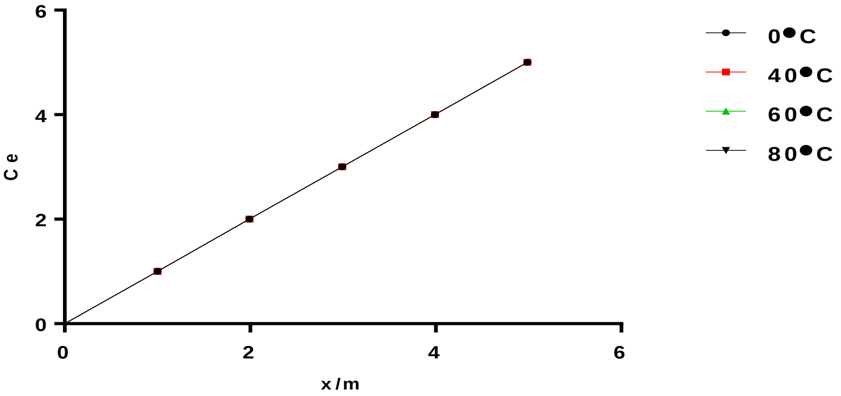

Figure-16: Temkin adsorption isotherm plots of SO_2 removal at different temperature.

Temkin adsorption isotherm is studied by takes into account he interactions between adsorbents and metal ions to be adsorbed and is based on the assumption that the free energy of sorption is a function of the surface coverage [28]. The linear form of the Temkin isotherm is represented as:

$q_e = B \ln A + B \ln C_e$

Where C_e is the equilibrium concentration of the adsorbate in mg/L, q_e is the amount of adsorbate adsorbed at equilibrium (mg/g), $RT/b_T = B$ where T is the temperature (K) and R is the ideal gas constant ($8.314\ J\ mol^{-1}K^{-1}$) and A and b_T are constants. Figure-16 showing relation between C_e and q_e, from the figure-16 and table-2 it was observed that the adsorption of SO_2 by silver nanoparticles is following Temkin adsorption isotherms.

Table-1: Kinetic parameters for adsorption of Aqueous SO_2 by Silver Nanoparticles

S.No	Parameters	SO_2concentration (5 ppm/L)	SO_2 concentration (10 ppm/L)	SO_2 concentration (15 ppm/L)
	Pseudo first order kinetic model			
01	R^2	0.999	0.999	0.8751
	ASS	0.0	0.0	0.1062
	K_1	0.0625	0.0835	0.1054
	Pseudo Second order kinetic model			
02	R^2	0.999	0.999	0.999
	ASS	0.0505	0.0389	0.0167
	K_2	3.8196×10^{-3}	10.2208×10^{-3}	5.4111×10^{-3}
	Elovich model			
03	R^2	0.6788	0.6921	0.7867
	ASS	0.01285	0.1484	0.3015
	α	3.067×10^{-6}	4.2026×10^{-6}	2.3743×10^{-6}
	β	92.2509	27.173	19.033

	Intraparticle diffusion model			
04	R^2	0.4286	0.4409	0.5370
	ASS	0.02285	0.2695	0.6546
	k_{id}	0.03969	0.1363	0.2124
	I	0.008685	0.02982	0.04648

Table-2: Isotherm kinetics for adsorption of Aqueous SO_2 by Silver Nanoparticles

S.No	Parameters	**Temperature °C**				
		0	40	60	80	100
	Temkin Adsorption Isotherm					
01	R^2	0.999	0.999	0.999	0.999	0.999
	ASS	0.0240	0.0240	0.0240	0.0240	0.0240
	a_T	0.003618	0.003618	0.003618	0.003618	0.003618
	b_T	0.001093	0.001093	0.001093	0.001093	0.001093
	Langmuir Adsorption Isotherm					
02	R^2	0.05357	0.05357	0.05357	0.05357	0.05357
	ASS	0.01071	0.01071	0.01071	0.01071	0.01071
	Q_0	0.002415	0.002415	0.002415	0.002415	0.002415
	b_L	0.0007280	0.0007280	0.0007280	0.0007280	0.0007280
	Freundlich Adsorption Isotherm					
03	R^2	0.999	0.999	0.999	0.999	0.999
	ASS	0.0147	0.0147	0.0147	0.0147	0.0147
	k_f	0.001236	0.001236	0.001236	0.001236	0.001236
	n	0.002558	0.002558	0.002558	0.002558	0.002558

IV. Conclusion

In this study, silver nanoparticles are synthesized by *Ficus Elastica* using the alternative energy source of microwave irradiation. The AgNPs are highly stable AgNPs synthesized in the present study are capable of adsorbing SO_2 molecules from aqueous solution and these AgNPs are inefficient in adsorbing NO_2. The formation of silver nanoparticles is confirmed by SEM and spectrophotometrically. Investigation carried out on adsorption studies revealed that it is following Pseudo second order kinetics. Optimum dosage for 98% removal of SO_2 is equal to ecofriendly nontoxic energy saving method is employed in the synthesis of nanoparticles. The present study gives a new direction for developing a catalytic tube for controlling air pollutant SO_2 caused by petrol-drove vehicles.

V. Acknowledgement

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