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Synthesize of green silver nanoparticles by one pot microwave-assisted technique: Modeling and optimization

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ABSTRACT

The use of nanoparticles (NPs) is recently increased due to their many applications in many different sectors. The majority of the methods used to manufacture of nanoparticles is mostly harmful to the environment and have high costs. The aim of the current work is to step forward in production silver NPs in a way with less cost and harm to environment using the green biosynthesis route. The Silver NP_S colloidal suspension is produced based on the reaction of the metal precursor $AgNO_3$ and a Cactus extract using Microwave instead of thermal heating. Optimization and modeling of NPs synthesis at lab scale is carried out throughout 10 experiments designed using software for experimental design and treating the responses statistically. The effect of concentration the metal precursor and power of microwave on the formation time of the NPs is investigated using Response Surface Methodology. The statistical results showed that the microwave power is more significant than the metal ions concentration, and the Ag NPs formation time decreased with increasing the microwave power and metal ions concentration. The optimum value for NPs formation time estimated is 10.27 minute. This formation time could be achieved using microwave power of 129.05 Watt and 1.8 ml of $AgNO_3$ solution. The equilibrium adsorption data of methylene blue dye on the synthesized silver NPs were mathematically modeled by employing the pseudo-first-order kinetics equation and the photo-catalytic performance was inspected throughout the degradation of methylene blue under irradiation by sunlight. The dye was effectively nearly 99% degraded by the green synthesized silver nanoparticles after 72 hours of exposure to sunlight.

Keywords: Silver nanoparticles, Modeling; Optimization, Green synthesis; Microwave, Adsorption, and Photo-catalytic performance

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1. Introduction

Metallic nanoparticles are among nanomaterials that exhibit higher chemical activity due to their crystallographic structure and surface/volume ratio and it had been used in many applications that related to manufacturing of pharmaceutical ingredient and fine chemicals[1, 2]. Silver is one of the most noteworthy metals used in the preparation of NPs and nanomaterials. Silver nanoparticles have received attention due to their physical, chemical, and biological properties. They showed unique optical, thermal, antimicrobial and catalytic characteristics which are strongly influenced by their size and shape. They attract increasing attention for the wide range of applications in nanotechnology fields including their use in sensors, optical probes, medicines, antimicrobial agents and catalyst [3, 4]. As demonstrated in [5, 6] several methods are found in literature for the synthesis of Ag NPs, the most conventional method is the chemical methods which involve the salt metal ions reduction by organic and inorganic chemical reducing agents like sodium citrate, hydrazine, sodium borohydride ($NaBH_4$), ammonium formate, elemental hydrogen, N,N-dimethylformamide (DMF), and poly(ethylene glycol)-block copolymers which are capable to reduce silver ions (Ag^+) in aqueous or non-aqueous solutions [7, 8]. Silver nitrate salt is the inorganic precursor of silver ions. The reducing agent used first reduced silver ions to atoms, then small clusters of the nucleated atoms are formed that grow after into particles of nano size[9]. However, most of the chemicals used as reducing agent are expensive and chemical agents associated with environmental toxicity [10, 11]. To protect the environment from the hazardous impact

of nanoparticles, researchers are interested towards the green synthesis of Ag NPs. Numerous studies have carried out by green biosynthesis routes to produce silver nanoparticles from plants [12, 13], microorganisms like bacteria and fungi On another hand, Microwave radiation (MW) is developing a rapid and environmentally friendly method of heating to generate nanomaterials[2, 14]. Nanostructures with higher degree of crystallization, narrower size distributions and smaller sizes are consistently yielded by Microwave heating rather than heating by conventional oil bath [15]. Heating by Microwave Offers quickly and volumetric heating of solvents, reagents and intermediates, which provides uniform and growing nuclear conditions for synthesis of nanomaterials [16, 17]. The synthesis of Ag NPs using microwave technology allowed for improvement over traditional heating to produce Ag NPs while significantly reducing reaction time and thereby significantly improving experimental efficiency short reaction time, lower energy consumption, relatively small volume and better product yield and reduced chemical waste [15, 18-20]. Basically, microwaves interact with materials throughout different ways. One way is by reflection which carried out with conducting materials like metals which do not absorb microwaves but reflect it. The second way is absorption; dielectric materials like acids, water and polar organic solvents can absorb microwave and be polarized by an electric field. On another hand, insulating materials like polymers, quartz or ceramics are transparent to microwaves. In the current work, green Ag NPs were synthesized using microwave technique and were applied to dye adsorption in dark and dye degradation under sunlight irradiation.

2. Response surface methodology (RSM)

While there are a number of papers in the literature concerning the biosynthesis of Ag NPs, there has been less work reported on the biosynthesis using microwave technology, also optimization and modeling the effect of the synthesis parameters on NPs formation time is rare. Response Surface Methodology was employed for that purpose. Response surface methodology RSM could be defined as a set of mathematical and statistical methods based on the suited of a polynomial equation to depict the practical data of dependent variables versus number of independent variables (factors). RSM is normally applied by selecting the operating parameters and their own ranges then implement an adequate design for the experiments of [21, 22]based on running some preliminary experiments for data collection. The statistical Stat graphics software version 5.1, SIGMA PLUS Neuilly sur Seine, France was used for designing the experiments and treating the responses statistically.
The general second-order polynomial empirical model proposed for the response surface analysis is given as follows:

$$Y = \beta_0 + \sum_{i=1}^{n} \beta_i \chi_i + \sum_{i=1}^{n} \beta_{ii} \chi_i^2 + \sum_{i=1}^{n-1} \sum_{j=2}^{n} \beta_{ij} \chi_i \chi_j \quad (1)$$

Where Y is the response or dependent variable; χ_i and χ_j are the independent variables; β_0, β_i, β_{ii}, and β_{ij} are the regression coefficients for intercept, linear, quadratic and interaction terms, respectively estimated by the model. In the present study, RSM is used as optimization and modeling tool for studying the effect of microwave intensity and concentration of $AgNO_3$ solution on the formation time of silver NPs. For optimization and mathematical modeling of the effect of microwave intensity and concentration of $AgNO_3$ solution, two-factorial central composite design with 2^2 =4 factorial points, 2*2 star-points and 2 repetitions of central point is used. The coded and actual levels of the operating parameters (independent variables) and the Ag NPs formation time for the 10 experiments are listed in Table 1.

Table 1. The Coded and actual levels of the operating parameters (independent variables) and the Ag NPs formation time for the 10 experiments

Coded level	-α	-1	0	1	+α
Microwave power (Watt)	0.10	150	0.28	590	0.45
AgNO3 volume (ml)	0.57	2.5	3.75	5.5	6.93

α is the axial distance

Experiment number	1	2	3,4*	5	6	7	8	9	10
Microwave power (Watt)	150	730.0	370.0	590.0	60.00	150.0	680.0	370.0	590.0
AgNO3 volume (ml)	5.5	1.9	4.0	5.5	4.0	2.5	4.0	6.1	2.5

Experiment number	1	2	3,4*	5	6	7	8	9	10
Ag NPs formation time (min.)	6.2	9.1	4.0	1.0	2.9	9.0	0.4	5.0	2.5

*Repetitions of central points

3. Experimental part

3.1 Preparation of plant extract

Preparation of plant extract Fresh leaves of cactus were collected from local farm of Soran city. The pins were removed and the cactus leaves were washed properly with distilled water. The clean leaves (200 g) were clipped finely into small pieces which were then dipped into 400 ml distilled water. The mixture was then refluxed for 3 hr. After that, filtration of the mixture was conducted to separate and collect the filtrate. 3.2 Synthesis of Silver Nanoparticles Set of preliminary experiments were conducted. Aqueous solution of 0.12 M silver nitrate was prepared. Silver nitrate was brought from Merk. The extract of cactus leaves was mixed with the aqueous solution Silver nitrate (1:4) volume ratio in conical flask (6 samples). The pH of the mixture was set to 8.0. The solutions were exposed directly to microwave irradiation each at different intensity. Color change was observed indicating the formation of Ag NPs [23]. For optimization purposes 10 experiments were designed based on RSM methodology using central composite design to modeling and optimization the impact of two operating variables of the biosynthesis reaction which are the microwave radiation (MW) power (GHz) and the concentration of Ag ions. Different volumes 1.9 ml to 6.1 ml of silver nitrate solution (0.12M) were treated with 1 mL of cactus extract and exposed to microwave irradiation at different intensity (59-680 Watt). Figure 1 shows the biosynthesis starting materials and a sample of the prepared Ag NPs.

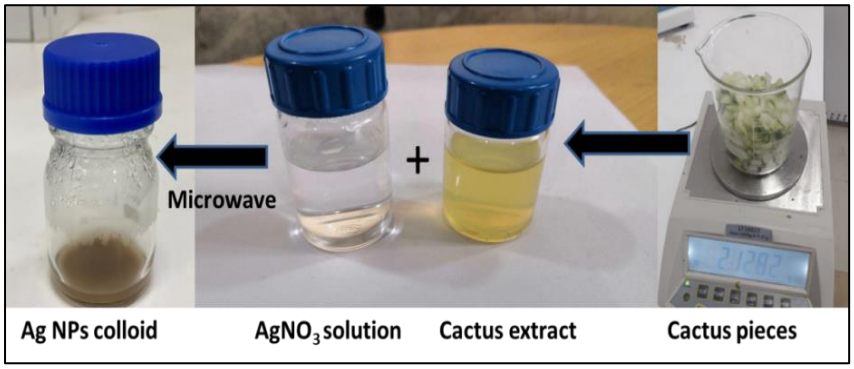

Figure 1. The biosynthesis starting of Ag NPs

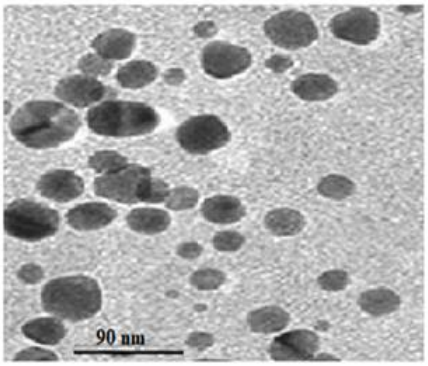

Figure 2. TEM micrograph of Ag NPs

3.3 Characterization of Ag NPs

One of the simplest methods to characterize Ag NPs is the electronic spectroscopy. The bio reduction of silver ions in aqueous solution is monitored by UV-visible spectra of the solution between 300 to 600 nm using UV-visible spectrophotometer. Distilled water is taken to adjust the baseline. Typical spectra of Ag NPs are presented below. The Surface Plasmon Resonance (SPR) peak corresponding to the cumulative oscillation of the electrons at the surface of metallic silver, is detected in all the tested samples in the range of 415 to 430 nm. The development of Ag NPs was simply identified by change the color of the reaction mixture from light yellow to dark brown as shown in Figure 1.

The formation of Ag NPs was also confirmed using Transmission Electron Microscopy (TEM) (see Figure 2). The TEM micrograph showed that the NPs are approximately spherical in shape with an average diameter of 47 nm. Similar results have been reported in literature where different green methods have been used to synthesize spherical shaped Ag NPs with nano diameter [24] The results indicate that cactus extract served as a good reductant and a capping agent in the synthesis of Ag NPs.

3.4 Methylene blue adsorption by Ag NPs

Adsorption experiment carried out by batch mode was performed to realize the adsorption performance of the Ag NPs. The uptake capacity was investigated by UV-vis spectrometry. 50 ml of suspension of Ag NPs with methylene blue was prepared at different pH (4, 7 and 9). The suspensions were agitated for 120 min in the dark. At each 20 minutes' time intervals, 5 ml of the solution was collected, filtered and then analyzed.

3.5 Photocatalytic degradation of methylene blue

500 mL of double distilled water containing 5 mg of methylene blue dye was used as stock dye solution. A mixture of 1 ml of biosynthesized silver nanoparticles colloid and 10 ml of methylene blue dye solution was prepared. The dye solution without addition of silver nanoparticles was used as control sample. The pH of the reaction mixture was adjusted to be 9, then the mixture was soundly mixed by magnetic stirring for 30 min to achieve adsorption–desorption equilibrium between the dye and photo catalyst of the working solution. Afterwards, the suspension was put under the sunlight and monitored from morning to evening sunset. At certain time intervals, about 2-3 ml of the reaction mixture were filtered and utilized for evaluation the photo catalytic degradation of the dye. The supernatant was tested using UV-Vis spectrophotometer and the absorbance spectrum was subsequently measured. The absorbance value at 660 nm is used to calculate the initial concentration of the dye and the concentration of dye during degradation. The Percentage of dye degradation was estimated using equation 2:

$$\%\ Dye\ degradation\ = \frac{(C_0 - C)}{C_0} \times 100 \quad (2)$$

where C_0 is the initial concentration of dye solution and C is the concentration of dye solution after photocatalytic degradation.

4. Result and Discussion

The effect of microwave power on Ag NPs formation time is shown in Figure 3. It is worthy to observe that as microwave power increases the formation time of NPs decreases. More microwave power means more energy for promoting the reduction rate of Silver ions to Silver NPs.

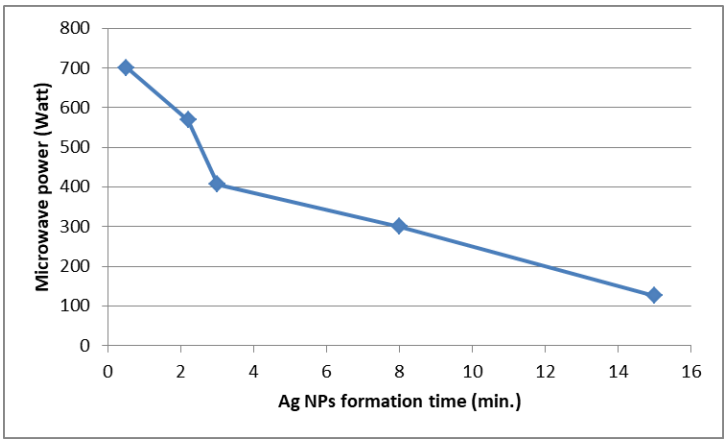

Figure 3. MW power versus formation time of Ag NPs

It was figured out that the components of the bio reducing and capping agent may be attached to the prepared Ag NPs via chemical bonding (chemisorption) or physical forces (physisorption). In chemisorption, the S containing molecules of the organic capping agent like proteins, peptides and some amino acids, also N and O containing molecules like carboxyl, carbonyl, amide, amino and hydroxyl groups possess high affinity to be adsorbed on the surface of Ag NPs. The adsorption may take place via coordinate-covalent chemical bonds, covalent or ionic. In physical desorption, the attachment is usually occurring throughout the weak Van der Val forces [22].

4.1 RSM analysis and numerical modeling results

A 2-operating parameter central composite design was used to investigate the impact of concentration of the inorganic metal salt $AgNO_3$ solution (1.9-6.1) ml, and Microwave power (58.9-590.0) Watt on Ag NPs formation time (see Table 1). The experimental data for the preparation of Ag NPs were analyzed using the computer software based on RSM. The ANOVA (Analysis of variance) for the response parameter (NPs formation time) are illustrated in Figure 5. The results estimated from ANOVCA are displayed within seven plots. The first plot is the Pareto chart (Figure 4A) which showed that the microwave power has significant effect on NPs formation time, while less significance effect was observed for the concentration of the metal ion precursor ($AgNO_3$).

However, NPs formation time found to decline with augmentation of the metal ion precursor ($AgNO_3$) concentration and the decreasing of microwave power as shown in the second plot; the standardized effect plots (Figure 4B). The interaction effect plot (Figure 4C) indicates the non-interactive effect of the two operating variables. Graphically, non-parallel lines suggest that the two parameters interact together, while two parallel lines of parameters indicate no interaction between parameters. The three-dimensional (3D) response (surface plots; Figure 4D) can help to visualize the response surface and showed how NPs formation time relates to two factors in term of response surface. Normal probability plot is presented in the (fifth plot; Figure 4E), It is seen that the points are almost close to the straight line and hence the model is considered to be effective. The two-dimensional (2D) contour plot (the sixth plot; Figure 4F) showed the independent variables plotted on x- and y-scales and response values represented by contours (z-scale). In general, different interactions between the variables reflect different shapes of the contour plots. An elliptical contour plot indicated a significant interaction between the variables, while a circular contour plot means otherwise. In the current case it can be observed that at low concentration of $AgNO_3$ and high dose of microwave radiation a minimum % NPs formation can be obtained in the range of the adopted experiments. The plots of the residuals as shown in (the seventh plot: Figure 4G) verify the validity of the regression. The plot shows that the Residuals vs. Predicted are scattered randomly, such as the errors are normally distributed (the points fall randomly on both sides of (0) and are independent of each other. From the results, it can therefore be concluded that the model is suitable for use and can be used to identify the optimal NPs formation time. Also, numerical modeling is estimated by analyzing all the experimental results using RSM to estimate an empirical developed mathematical model for the optimum response. An empirical regression model equation 3 (second-order) is generated based on experimental data of Table 1. The soundness of the fit of the model is expressed by the regression coefficient R^2. The Ag NPs formation time was predicted with an R value of 89.5204 %. The high Regression Factor R-squared calculated by the response surface analysis of the experimental values reflects the adequate explanation of the experimental data by the developed model. The multinomial equation derived from the RSM analysis is displayed in equation 3. Mathematical model:

$$T = 20.317 + 0.004\ M - 6.87\ V - 0.00002\ M^2 + 0.0011\ M\ V + 0.7\ V^2 \quad (3)$$

Where T= NPs formation time (min.), M= Microwave power (Watt), V= volume of metal salt ($AgNO_3$) ml. The optimum value for NPs formation time as estimated from ANOVA = 10.27 min. This formation time could be achieved using microwave power = 129.05 Watt and using 1.8 ml of $AgNO_3$ solution.

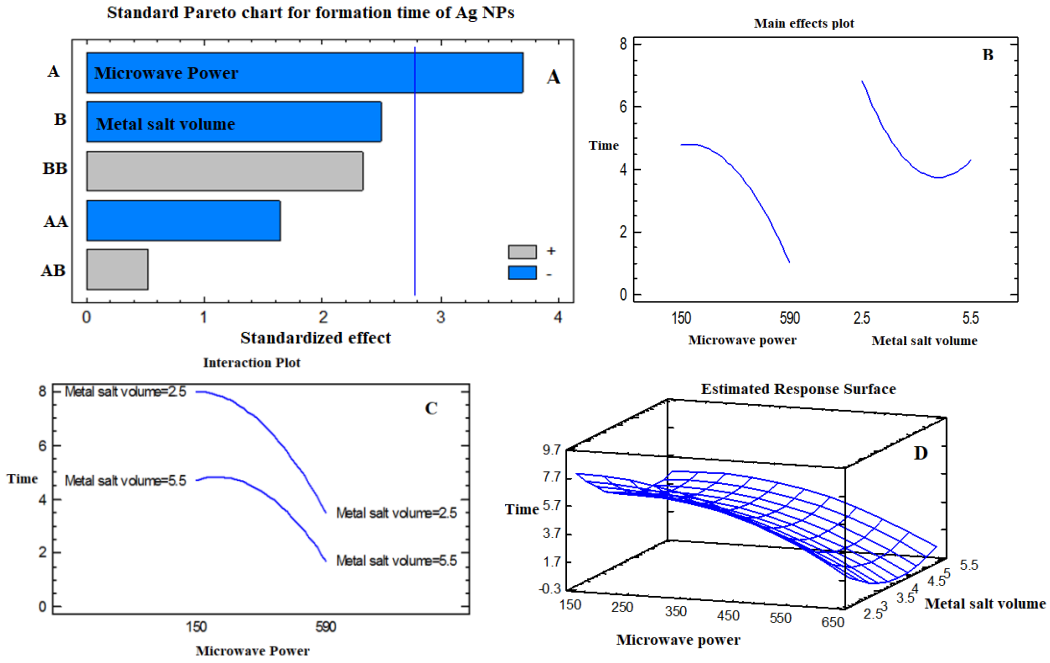

Figure 4. A: The standardized Pareto chart; B: The standardized main effect; C: The interaction plot; D: Estimated response surface;

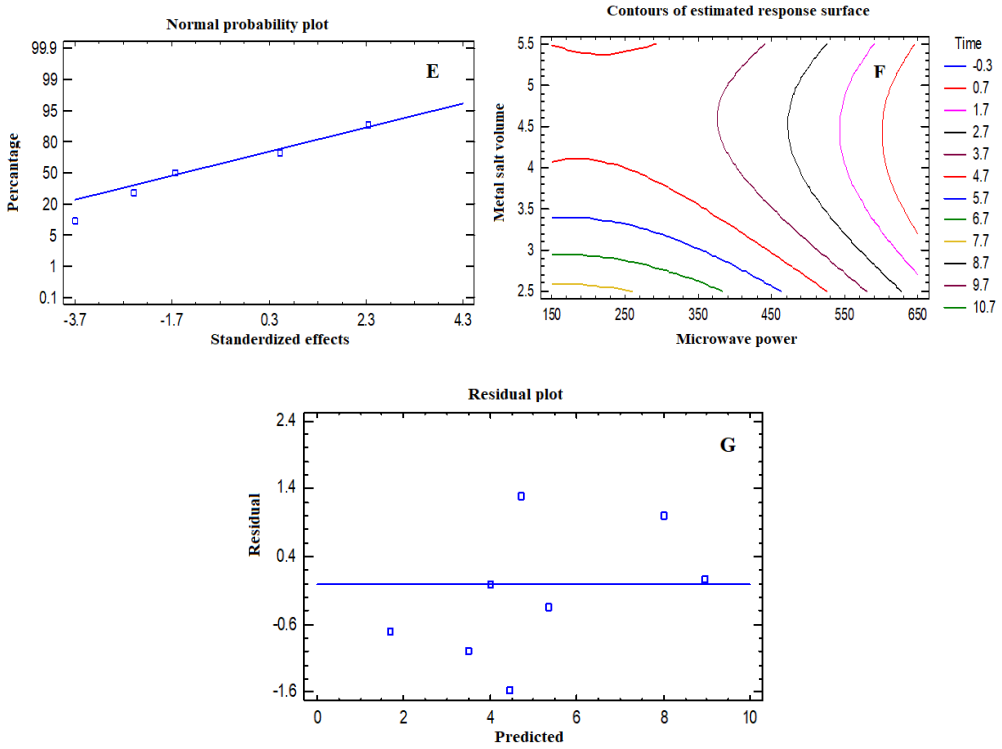

Figure 4. E: The normal probability plot; F: The contours of estimated response surface; G: Residual vs. predicted plot for formation time of Ag NPs

In the current work, one of the significant applications of metallic NPs as photo catalyst for removing organic contaminant from wastewater was employed for the biosynthesized Ag NPs prepared using microwave. Methylene blue (MB) dye was selected as the organic pollutant. Prior to studying the photo catalytic performance of the prepared nanoparticles for the degradation of methylene blue. The adsorption of Methylene blue dye on the Ag NPs in dark have been investigated firstly. Mixture of Ag NPs and MB were prepared at different pH (9, 7 and 4) and stirred in the dark and the uptake capacity of the NPs was kept under observation by measuring the absorbance at different time intervals in order to study the effect of pH on adsorption as well as studying the adsorption kinetics. The practical results were fitted to pseudo first order kinetic model as illustrated in Figure 6. The experimental results showed very remarkable fitting to the pseudo first order model with a regression coefficient 0.9879, 0.9272 and 0.9780 for the adsorption at pH 9, 7 and 4 respectively which indicates that pseudo-first-order model may represent one the adsorption routes. The rate constants for the adsorption of MB by Ag NPs was calculated from the slopes of the straight lines and found to be 0.0068, 0.0056 and 0.0032 min^{-1} for the adsorption at pH 9, 7 and 4 respectively. From the plot in Figure 5, it can be deduced that in alkaline pH range (pH 9), higher percentage of adsorption is observed. The percentage of adsorption was relatively lower in the acidic range (pH 4) reflecting that the dye adsorption is unfavorable at acidic pH values. The situation may be attributed to decline the number of negatively charged sites and increasing the numbers of positively charged surface sites on the adsorbent surface. The electrostatic repulsion between the positively charged dye cations and the positively charged surface sites of the adsorbent hinder the adsorption process. Also, at acidic pH the presence of excess H+ ions will compete with dye cations for the adsorption sites of the NPs [25]. The adsorption process could be described by diffusion of Methylene blue molecules on outer surface of the Ag NPs due to diffusion potential depicted by the concentrations of MB and NPs available external surface area. Posted to the diffusion of MB molecules on the external surface of the NPs, MB molecules are diffused on the available pores of the NPs. The uptake of MB molecules by all the available active site of the NPS may be driven by physisorption or chemisorption [26]. The adsorption study of the prepared Ag NPs

confirmed that the alkaline NPs colloidal suspension can be used directly as adsorbent for organic pollutants in wastewater treatment.

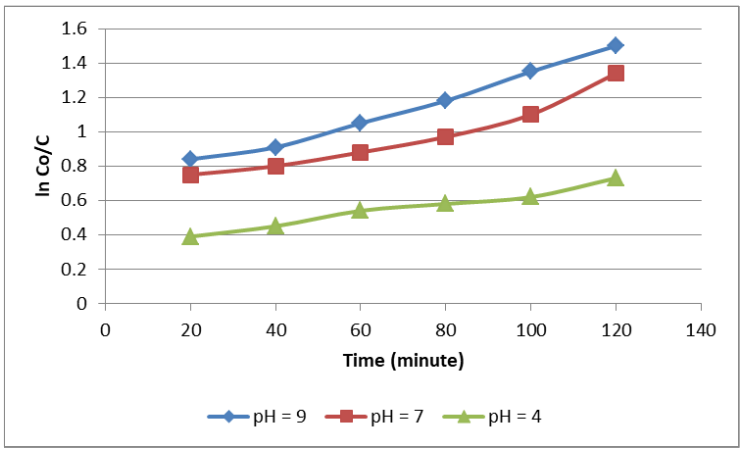

Figure 5. Adsorption kinetics of methylene blue by the Ag NPs at different pH

To confirm the photo catalytic capability of the prepared NPs, photo catalytic degradation of methylene blue was taken place by the utilization of the green synthesized silver nanoparticles under solar light. The visual indication of the dye degradation was initially identified by color change. The deep blue color of the dye solution in water changed into light blue then into very light green when exposed to sunlight. The change of reaction mixture color to approximately colorless was completed with 56 h. This discoloration is a perfect indication of degradation of the dye. The degradation process was followed up by UV-vis Spectrophotometer analysis. The absorption spectrum showed that the absorption peaks intensity at 660 nm for methylene blue dye declined gradually with increasing the exposure time to sunlight which pointed out the photo catalytic degradation reaction of the dye. The percentage of degradation efficiency of MB by silver nanoparticles was calculated and plotted against the exposure time of the solution of Ag NPs nanoparticles with methylene blue dye. Figure 6 shows the situation.

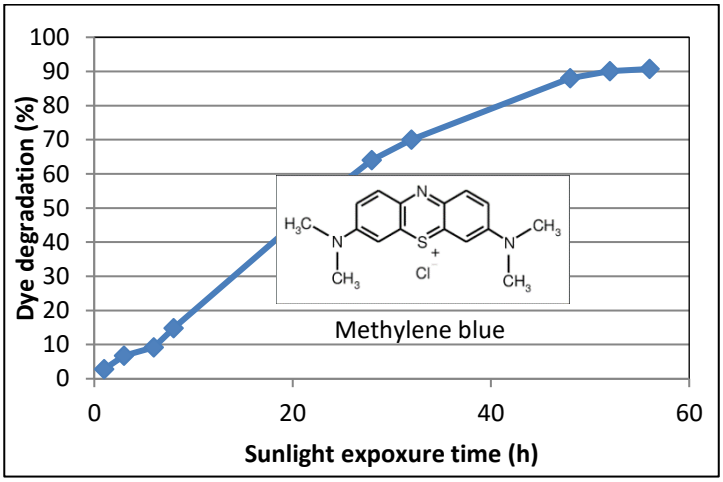

Figure 6. Sunlight degradation plot for methylene blue using Ag NPs colloidal suspension

On the other hand, MB dye was irradiated with UV light in the absence of the NPs throughout a control experiment. The results from the control experiment revealed that no significant alteration in the MB dye concentration was detected and only small amount (about 5.4%) discoloration of the dye was observed. This finding reflected that in the absence of the NPs, the direct photolysis of MB dye under sunlight was insignificant. The photo degradation mechanism of methylene blue is based on the transformation of the hazardous organic dye into benign compounds. It was reported that the initial step of methylene blue degradation is attributed to the formation of the reactive intermediate $OH^{\cdot}$ radicals that attack the $R–S^{+}=R$ functional group in MB followed

by the fission of the bonds of the R–S^+=R functional group in the dye into R–S(=O)–R, R–SO_2–R, R–SO_3H–R to bring out finally the formation of phenol and SO_4^{2-} (Houas et al., 2001, Azeez et al., 2018, Kumar et al., 2018).

5. Conclusions

Using microwave technique as environmentally friendly energy source instead of conventional heating to produce biosynthesized Ag NPs is an approach to minimize environment and global warming at the same time decrease the time needed for formation of the green nanoparticles. On the other hand, optimization the biosynthesis reaction variables using RSM is an effective tool to minimize number of experiments as well as identify the variables significance and its interactions as well as the mathematical modeling of the process. The results of the current work confirmed that the prepared Ag NPs colloid suspension can serve directly as adsorbents and photo catalyst for removing organic pollutant such as dyes from industrial wastewater. Nevertheless, minimizing the production cost of the green Ag nanoparticles could be achieved by directly loading of silver nanoparticles onto cheap supports. This will be covered in future study.

6. References

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