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Synthesis of Silver Nanoparticles Using Response Surface Methodology

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Silver nanoparticles have become a popular area of research in the field of nanotechnology due to their unique properties, including excellent antimicrobial activity. The synthesis of silver nanoparticles can be optimized using response surface methodology (RSM), which provides a systematic and efficient approach to determine the ideal process parameters for obtaining the desired nanoparticles. In this study, the Response Surface Methodology (RSM) - Faced-Centered Central Composite Design (FCCD) was utilized to optimize the synthesis conditions for the preparation of silver nanoparticles. The variables affecting the synthesis of silver nanoparticles, including precursor concentration, reducing agent concentration, and reaction time, were investigated. The synthesized silver nanoparticles obtained in this study had a size of 10.99 nm and a maximum wavelength of 450 nm. Through the resulting 3D plots, the impact and interaction between silver nitrate concentration, ascorbic acid concentration, and reaction time could be analyzed. Overall, this research provides valuable insights into the optimization of silver nanoparticle synthesis using response surface methodology, which can lead to the development of more efficient and effective methods for producing nanoparticles with desired properties.

Keywords: Silver nanoparticles, Response surface methodology, Central composite design.

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1. INTRODUCTION

Nanoparticles are defined as particles with dimensions ranging from 1 to 100 nm [1]. Due to their size, nanoparticles are more reactive than larger particles, as the atoms on their surface are in direct contact with other materials, influencing their material reactivity. This property is advantageous and has led to the development of nanoparticles for various purposes [2]. Among the various types of noble metal nanoparticles, silver nanoparticles (AgNP) have significant potential in various applications, including as anti-bacterial agents, biomedical device coatings, and drug carriers [3].

The chemical reduction of aqueous solutions of silver salts or organic solvents using external reducing agents into colloidal suspensions is the most common method for producing silver nanoparticles [4]. This method has a large-scale production capacity [5]. A variety of reducing agents, such as sodium borohydride, ascorbic acid, aspartic acid, citric acid, and hydrazine hydrate, have been utilized to convert Ag^+ to Ag^0. To regulate the size and stability of the nanoparticles, a stabilizing agent can be incorporated [6]. Polymers such as polyvinyl pyrrolidone (PVP) are excellent hosts for stabilizing metal nanoparticles due to their ability to act as stabilizers or stabilizing agents [7].

The production of AgNP has recently been progressing with different characteristics and effects of physiochemical properties, so characterization techniques have been developed. These techniques allow the analysis of the morphology, structure, composition, and behavior of AgNP using an instrument such as visible ultraviolet spectroscopy (UV-Vis), dynamic light scattering (DLS), and transmission electron microscopy (TEM) [8].

Various factors can affect the synthesis of silver nanoparticles, such as precursor, reducing agent and stabilizing agent concentrations, temperature, *pH*, and reaction time [9]. However, the conventional approach to finding optimal conditions for silver nanoparticle synthesis is through the one factor at a time (OFAT) method, which can only evaluate the interaction of up to two factors and requires a large number of experiments and time. Experimental design is a more efficient approach that reduces the amount of data collected in the fewest number of trials while focusing on relevant statistical assessments and interactions between studied factors within a realistic range [10]. Among the experimental designs, the CCD is the most commonly used second-order design. These designs usually involve three types of experimental points: factorial, center points, and axial points [11]. The advantage of this type of optimization model is that it is more accurate and there is no need to carry out a three-level factorial experiment to build a second-order quadratic model [12].

In this study, FCCD was adopted using response surface methodology (RSM) to minimize the number of synthesis trials and investigate the relationship between independent variables and response variables to determine suitable experimental formulations for the synthesis of silver nanoparticles using ascorbic acid as a reducing agent and polyvinyl pyrrolidone as stabilizing agent.

2. MATERIALS AND EXPERIMENTS

The materials utilized in this study include polyvinylpyrrolidone (PVP), ascorbic acid (AA), silver nitrate ($AgNO_3$), and distilled water. The preparation of 1% PVP solution, AA solution (0.005; 0.025; 0.05 M), $AgNO_3$ solution (0.005; 0.01; 0.02 M), and the synthesis of silver nanoparticles were conducted in the Laboratory of Instrumentation and Analytical Sciences, Chemistry Department, Faculty of Science and Data Analytics,

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Institut Teknologi Sepuluh Nopember. The FCCD was optimized using Jupyter Notebook software with three parameters, namely $AgNO_3$ concentration (0.005 – 0.02 M), AA concentration (0.005 – 0.05 M), and reaction time or reaction time (0 – 20 minutes), with the response variable being the absorbance at the highest wavelength of UV-Vis spectrophotometry. The values of the variables used in the experiment are presented in Table 1.

The silver nanoparticles were synthesized by reacting 50 *mL* of 1% PVP solution with 30 *mL* of $AgNO_3$ solution in an Erlenmeyer flask, followed by stirring at 350 rpm. The mixture was then added with 0.5 *mL* of AA solution under reaction conditions, according to the variations in synthesis. The resulting silver nanoparticles were tested for their wavelength using a UV-Vis spectrophotometer and their particle size distribution using a particle size analyzer (PSA).

Table 1 – Variation of silver nanoparticle synthesis

Code	$AgNO_3$ concentration (M)	AA concentration (M)	Reaction time (minutes)
0	0.005	0.005	0
1	0.005	0.05	0
2	0.02	0.005	0
3	0.02	0.05	0
4	0.005	0.005	20
5	0.005	0.05	20
6	0.02	0.005	20
7	0.02	0.05	20
8	0.01	0.005	10
9	0.01	0.05	10
10	0.005	0.025	10
11	0.02	0.025	10
12	0.01	0.025	0
13	0.01	0.025	20
14	0.01	0.025	10

The independent and collaborative relationships of the variables were evaluated using a faced-centered central composite design (FCCD) using Python software. The developed models were assessed using statistical tools such as analysis of variance (ANOVA) and F scores. The level of fit of the model equation was determined by the value of the correlation coefficient, R2. Finally, the fitted model was presented as a three-dimensional surface plot to visualize the relationship between the results.

3. RESULTS AND DISCUSSION

The absorption spectrum of the prepared sample was obtained using a UV-Vis spectrophotometer. This spectrum is indicative of the formation of colloidal silver nanoparticles as well as the quality of the sample. Fig. 1 shows the absorption spectrum of the AgNP formed with the addition of AA. The measurements revealed an absorption peak at a wavelength of approximately 450 nm, which is a characteristic feature of silver nanoparticles and corresponds to surface plasmon resonance [14]. These results are consistent with previous studies regarding the synthesis of silver nanoparticles with ascorbic acid which showed absorption peaks of around 453 *nm* with variations in *pH* [15]. Fig. 2 shows a typical absorption spectrum of colloidal silver nanoparticles.

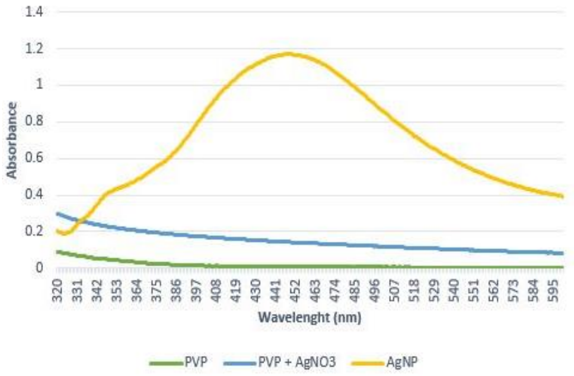

Fig. 1 – UV – Visible spectrum of PVP, PVP + $AgNO_3$ and AgNP

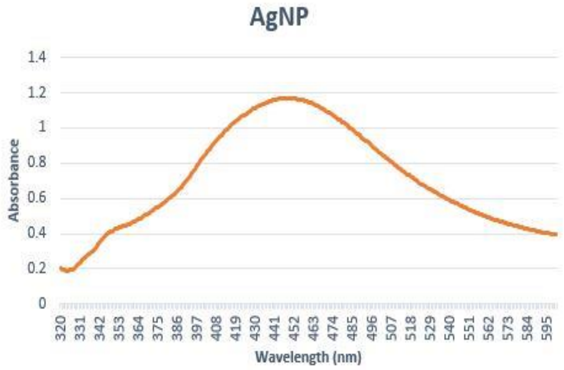

Fig. 2 – UV – Visible spectrum of silver nanoparticles

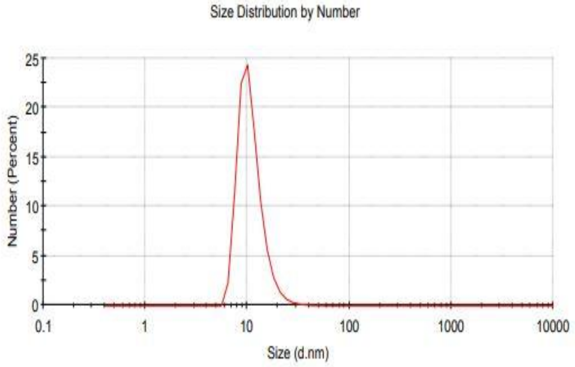

Fig. 3 – Size distribution of silver nanoparticles

Nanoparticle size was analyzed using a particle size analyzer with a dynamic light scattering technique and the size distribution of one sample is presented in Fig. 3. The results indicate that most of the prepared nanoparticles have a radius of less than 100 *nm* with an average size distribution of 81.20 *nm*. Furthermore, over 22.5% of the particles had a size of ~10.99 *nm*, demonstrating the successful synthesis of nano-sized silver particles as observed in previous studies [16].

The statistical significance of the variables on AgNP*s* absorbances was confirmed by the Model F value of 17.59 and a Prob > F value of 0.00283, indicating that the input factors have a significant impact on the response. The correlation coefficient of the model (R2) and adjusted R2 (R2 adj) also demonstrated the model's significance and goodness of fit. The obtained R2 value of 0.969 indicates excellent agreement between experimental data and model prediction results, while the adjusted R2 value of 0.914, which is only 0.055 lower than the R2 value, highlights the model's robustness.

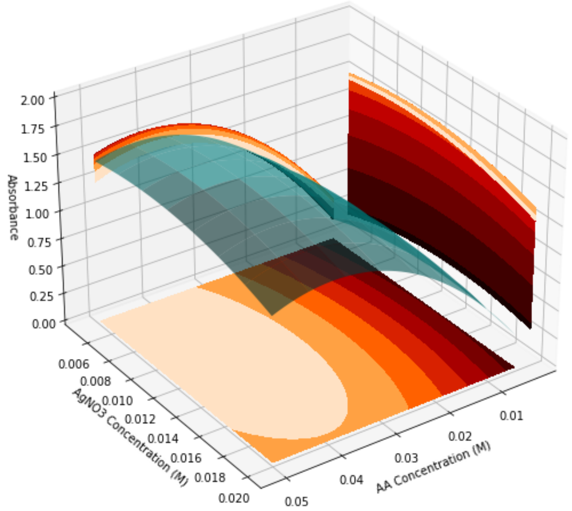

Fig. 4 – The 3D surface plot of AgNP synthesis via FCCD approach for effects of AA concentration and $AgNO_3$ concentration

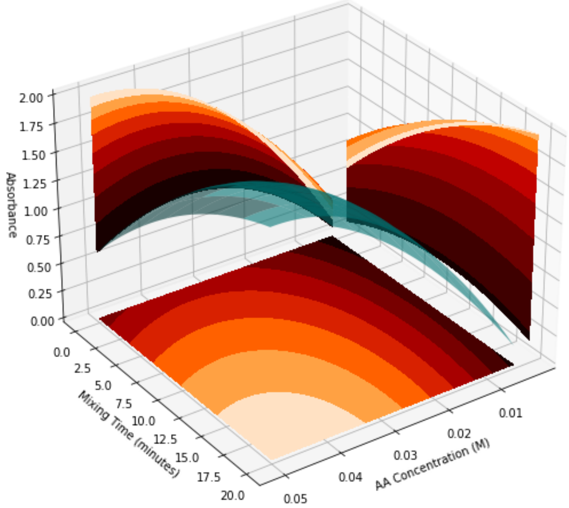

Fig. 5 – The 3D surface plot of AgNP synthesis via FCCD approach for effects of AA concentration and reaction time

The effect of the parameters assessed on AgNP synthesis was depicted in Fig. 4-6 using counterplots and 3D surface plots, focusing on the impact of AA concentration, $AgNO_3$ concentration, and reaction time. The results indicated that higher concentrations of both AA and $AgNO_3$ lead to higher AgNPs absorbances, with an optimal $AgNO_3$ concentration of less than 0.02 M and an optimal AA concentration of more than 0.05 M. Moreover, the longer the reaction time and reaction time, the higher the AgNPs absorbances, with an optimal reaction time of more than 20 minutes. Importantly, the optimal point was observed within the experimental range based on the three plots obtained.

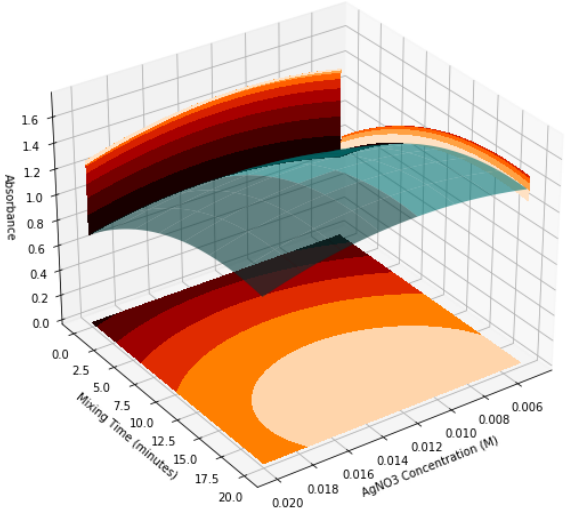

Fig. 6 – the 3D surface plot of AgNP synthesis via FCCD approach for effects of AA concentration and reaction time

4. CONCLUSIONS

The successful synthesis of silver nanoparticles using a chemical reduction method with silver nitrate as a precursor, ascorbic acid as a reducing agent, and PVP as a stabilizing agent has been accomplished using response surface methodology. The synthesized silver nanoparticles had a size of 10.99 *nm* and a maximum wavelength of 450 *nm*. The impact of silver nitrate concentration, ascorbic acid concentration, and reaction time can be analyzed through the resulting 3D plots. The 3D plot results indicate that the optimal point can be observed from the experimental ranges carried out [19].

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Синтез наночастинок срібла з використанням методології поверхні відгуку

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Наночастинки срібла стали популярною областю досліджень у галузі нанотехнологій завдяки своїм унікальним властивостям, включаючи високу антимікробну активність. Синтез наночастинок срібла можна оптимізувати за допомогою методології поверхні відгуку (RSM), яка забезпечує систематичний та ефективний підхід до визначення ідеальних параметрів процесу для отримання бажаних наночастинок. У даній роботі для оптимізації умов синтезу для отримання наночастинок срібла застосовувалася методологія поверхні відгуку (RSM) – центрований центральний композитний дизайн (FCCD). Було досліджено змінні, що впливають на синтез наночастинок срібла, включаючи концентрацію прекурсора, концентрацію відновника та час реакції. Синтезовані наночастинки срібла, отримані в цьому дослідженні, мали розмір 10,99 нм і максимальну довжину хвилі 450 нм. На основі тривимірних графіків можна проаналізувати вплив і взаємодію між концентрацією нітрату срібла, концентрацією аскорбінової кислоти та часом реакції. Загалом, це дослідження дає цінну інформацію щодо оптимізації синтезу наночастинок срібла за допомогою методології поверхні відгуку, що може призвести до розробки ефективних методів виробництва наночастинок з наперед заданими властивостями.

Ключові слова: Наночастинки срібла, Методологія поверхні відгуку, Центральний композитний дизайн.