

COMPARISON STUDY BETWEEN HYDROTHERMAL AND CO-PRECIPIATION METHOD FOR GREEN SYNTHESIZE OF MAGNETIC SILVER NANOPARTICLES

MOHAMMAD AIMAN HAKIM ABDULLAH¹, AINUL HUSNA ABDUL AZIZ¹,
NURUL SAKINAH ENGLIMAN^{1*}

¹*Department of Chemical Engineering & Sustainability, Kulliyah of Engineering,
International Islamic University. Malaysia (IIUM), Jalan Gombak, 53100 Kuala
Lumpur, Malaysia.*

* Corresponding author: sakinahengliman@iium.edu.my

ABSTRACT: Nanoparticle research has been attractive for the past decade due to its unique electronic, mechanical, optical, chemical, and magnetic properties, which can be used in various applications, including sensors, medical, food, and others. Yet, the use of toxic chemicals in the synthesis of nanoparticles limits their potential use in the medical and food industries. The green synthesis of nanoparticles is eco-friendly and well-suited for many applications. However, there are some issues related to it as there are limited comparisons made between nanoparticles synthesized through different routes, and even the physiochemical and morphological properties are also not compared. Therefore, this study attempts to synthesize magnetic silver nanoparticles using the greener technique, which utilized the banana peel waste extract as the reductant during the synthesis of nanoparticles. The banana peel waste extract and the nanoparticles were characterized using a UV-VIS spectrometer, Fourier transform infrared spectroscopy (FTIR), and field emission scanning electron microscope (FESEM) to analyze the properties of the extract and the fabricated nanoparticles. The results found that the -OH group was present in both banana peel extract as well and the synthesis of silver nanoparticles in FTIR analysis, which is believed to come from the phenolic group that helps in the reduction of silver ions to silver nanoparticles during the synthesis process. VSM analysis indicates that the synthesized silver nanoparticles had ferromagnetic properties of 2.83 emu/g for the coprecipitation method and 3.91 emu/g for a hydrothermal method, which is considered stronger to be utilized for further application. In addition, FESEM analysis shows that the hydrothermal could synthesize the uniformly distributed and mono-dispersed spherical shape compared to the coprecipitation method, which produces uneven shapes like rods, pellets, and spheres. The study concludes that the green-synthesized silver nanoparticles using banana peel waste extracts could produce medium-strength magnetic silver nanoparticles, especially through a hydrothermal process when the diluted precursor ions were used compared to concentrated ones.

KEY WORDS: *Silver nanoparticles, Green synthesis, Hydrothermal, Coprecipitation, and Magnetic Properties.*

1. INTRODUCTION

The emergence of nanotechnology has introduced the world to nanoparticles that have a lot of benefits due to their unique chemical, mechanical, electronic, magnetic, and optical properties, which can be applied in many applications [1]. The unique properties of

nanoparticles are due to two physical effects related to the quantization of electronic states of the material that lead to a very sensitive size-dependent effect, which can be analyzed in terms of magnetic and optical properties and due to increased surface-to-volume ratio of the synthesized nanoparticles that influence the mechanical, chemical, and thermal properties of that material [2]. Hence, the nanoparticles are a suitable candidate for certain applications related to biosensors and drug delivery [3]. Different physical and chemical methods have been implemented in synthesizing metallic nanoparticles, which are capable of synthesizing nanoparticles of specific sizes [4,5]. However, some of the synthesis methods for metallic nanoparticles involved the utilization of hazardous and toxic chemicals. This will reduce the chance for the fabricated nanoparticles to be used in sensitive applications such as for the food industry or for medical purposes due to the concern about the trace of this toxic chemical [6]. Therefore, green synthesis methods are introduced to solve the problem by the conventional method.

The green synthesis method, which is also called the biosynthetic process, is the same as the chemical reduction process. However, the plant extracts or microbes will be used as a replacement for the costly and toxic reagents during the synthesized process [7]. Natural chemicals extracted from plants and microorganisms known as phytochemicals can replace the use of chemical reagents to act as reductants for the precursor during the synthesis process. This is because the phytochemicals from plant or bacterial extract have -OH functional groups that can reduce the metal ions during the priming step in the synthesis process [8].

Among the nanoscale materials being investigated for biomedical use, magnetic nanoparticles have gained significant attention since they can be traced through magnetic resonance imaging. Magnetic nanoparticles have the potential to be used in biomedical applications not only to promote tissue imaging but also to control drug release, cell separation, and localized magnetic hyperthermia therapy since they have good biocompatibility and low toxicity [24].

In general, the magnetic properties of nanoparticles are strongly dependent on their size and morphology. Several methods have been used aiming for fine control of size and morphology, such as co-precipitation, microemulsion, sol-gel, solvothermal, and hydrothermal synthesis [1-3, 5-8, 24, 25]. Among the cited methods, the co-precipitation method stands out due to its simplicity and environmentally friendly reactants. It is based on the formation of metal oxides from inorganic salts and their respective bases under an inert atmosphere at room temperature or mild temperatures. The nanoparticles obtained from this method have high reproducibility when the experimental conditions are fixed. However, the yield of the synthesis nanoparticles under the co-precipitation method is lower than that of other methods. The hydrothermal method has been used widely in the large-scale production of nanoparticles due to high-yield nanoparticle production and the control of nanoparticle morphology with remarkably narrow size distribution [25].

Although the underlying antimicrobial processes and mode of action for silver nanoparticles have not been well elucidated, they have been widely used as the universal germicidal against various microorganisms [13,14]. This is because silver nanoparticles may bind to sulfur-containing proteins on the cell membrane, causing structural distortion through membrane breakdown. The release of silver ions (Ag^+) from silver nanoparticles may reach the cytosol via the vulnerable membrane, resulting in cell death. However, the dose-dependent toxicity of silver nanoparticles has been identified as a significant environmental concern. Researchers have worked on reducing the concentration of silver

nanoparticles needed while increasing its germicidal efficacy by modifying the size, shape, and surface modification of silver nanoparticles. [13, 14, 25].

In our study, the banana peel waste extract was used in synthesizing the silver nanoparticles as the study shows that large amounts of phenols can be found in banana peel, which helps in the formation of metallic silver nanoparticles [9]. Two different methods, hydrothermal and coprecipitation, were used to synthesize silver nanoparticles with two different parameters: the ratio of banana peel extract to silver nitrate solution and the concentration of silver nitrate solution. Synthesized silver nanoparticles were characterized with UV-Vis, FTIR, VSM, and FESEM analysis to further study their optical, functional group, magnetic properties, and morphologies.

2. MATERIALS AND METHODS

2.1 Chemical and Reagents

This study involved the chemicals silver nitrate (AgNO_3), hydrochloric acid (HCl), sodium hydroxide (NaOH), and distilled water.

2.2 Preparation of the Banana Plant Peel Extract

Healthy and fresh banana peels from the species of *Musa acuminata* were collected and washed carefully with tap water, then distilled water to remove any undesirable residue. The clean peels were dried using clean tissue to remove the leftover water. About 15 g of banana peel was added to 300 mL of distilled water, which was later boiled at 90 °C, and the solution was stirred on the magnetic stirrer at 245 rpm for 20 minutes. The solution was filtered and stored in a 4 °C refrigerator for further use. The Folin-Ciocalteu method was used to determine the total phenolic content of the extract.

2.3 Synthesis of nanoparticles using hydrothermal method

The hydrothermal method of magnetic silver NPs was prepared by adding 100 ml of banana peel extract dropwise into 200 mL of varying concentrations of AgNO_3 solution (0.01M, 0.05M, and 0.1M). The mixed solution was continuously stirred at 200 rpm, 30 °C, using a hot plate magnetic stirrer for 30 min to obtain a blackish-brown solution. The solution was transferred to a Teflon autoclave tube inside a stainless-steel hydrothermal reactor vessel and placed inside the pre-heated oil bath at 120 °C. After 2 hours of heating, the black sediment started to form. The black sediment was collected by centrifuge at 4000 rpm for 10 minutes, and the final washing was done using deionized water. After that, the pellet was stored at -80 °C before being sent to freeze dry.

2.4 Synthesis of nanoparticles using coprecipitation method

The magnetic silver nanoparticles were synthesized using the coprecipitation method by adding 100 ml of banana peel extract dropwise into 200 mL of 0.01M, 0.05M, and 0.1M of AgNO_3 solution (Table 1). The solution was continuously stirred at 200 rpm using a magnetic stirrer and heated at 60 °C on the hotplate for 2 hours to obtain a blackish-brown solution. The solution was cooled at room temperature for 2 hours, and the colloidal suspension was obtained. The colloidal suspension was centrifuged at 4000 rpm for 10 minutes and washed with deionized water. After that, the pellet was stored at -80 °C before sending it to freeze dry.

Table 1: Parameters for optimization of synthesis of magnetic silver NPs

	The ratio between banana peel extract to 1 mM of AgNO ₃ [v:v]	Concentration of AgNO ₃ M [M]
Hydrothermal	1:3, 1:2, 1:9	0.01, 0.05, 0.1
Coprecipitation	1:3, 1:2, 1:9	0.01, 0.05, 0.1

2.5 Characterization of synthesized nanoparticles.

The functional groups' surface absorption for the AgNPs was observed using Fourier Transform Infrared Spectroscopy (FTIR) analysis at a range of 400 cm⁻¹ to 4000 cm⁻¹. The UV-Vis analysis was done to study the optical properties of AgNPs where the spectra were read between 300-600 nm at time intervals up to 60 minutes for coprecipitation and two times taken, minute 15 and after cooling down. For the morphology of AgNPs, Field Emission Scanning Electron Microscopy (FESEM) was used to determine the shape, size, and distribution. The magnetic properties of AgNPs were studied using the vibrating sample magnetometer (VSM) analysis that detects the macroscopic magnetization moment of the synthesized nanoparticles.

2.6 Identification of banana peel extract's composition using GC-MS

The compositions of the coffee extract samples were analyzed using an Agilent 7890A gas chromatography equipped with an Agilent MSD 5975C mass spectrometer. A 30 m x 0.25 mm capillary column with an internal diameter of 0.25 µm HP-5MS was used. Before analyzing the samples, the retention time was locked by changing column pressure using standard samples. A constant pressure model was then used for the entire analysis process. The gas chromatography (GC) oven temperature was programmed from 40 to 300 °C via a ramp of 10 °C min⁻¹ and maintained at 40 °C for 2 min and at 300 °C for 15 min. The mass spectrum (MS) was operated in full-scan mode from m/z, 50–700 for qualitative analysis or selected ion monitoring (SIM) mode for quantitative analysis. The inlet and MS transfer line temperatures were maintained at 250 °C, and the ion source temperature was 300 °C. Sample injection (1 µL) for each test was done using splitless mode.

3. RESULTS AND DISCUSSION

3.1 Banana Peel Waste Extract

The banana peel waste extract was tested using the Folin-Ciocalteu method to check the total phenolic compound. From this analysis, the total phenolic compound in the banana peel extract was found to have 100 mg GAE/L of gallic acid, and this amount was capable of quite enough to act as a reductant during the synthesis of silver nanoparticles. This was correlated with the previous research, where the polyphenol compounds and the water-soluble constituents are mainly responsible for reducing carbonyl groups with silver ions and later stabilizing the nanoparticle formation [10]. The GC-MS analysis of the banana peel extract showed that there were four main phenolic compounds in the extract: astaxanthin, carotenoid, tetracycline, and polyphenols (Fig. 1). The details of the major compounds can be seen in Table 2. Among these four compounds, it was believed that the presence of tetracycline in the extract acts as the reductant and the capping agent during the silver nanoparticle formation. Based on the structure of tetracycline (Fig. 2), the reduction of Ag⁺ to Ag⁰ may be assisted by tetracycline molecules by contributing their electrons from the hydroxyl groups and the nitrogen atom in the rings [11]. For example, it was reported

that secondary metabolites such as tetracycline also function as a capping agent in the synthesis of AgNPs, whereby the presence of the rings improves the stabilization of the AgNPs and prevents aggregation or inversion into Ag ions [12].

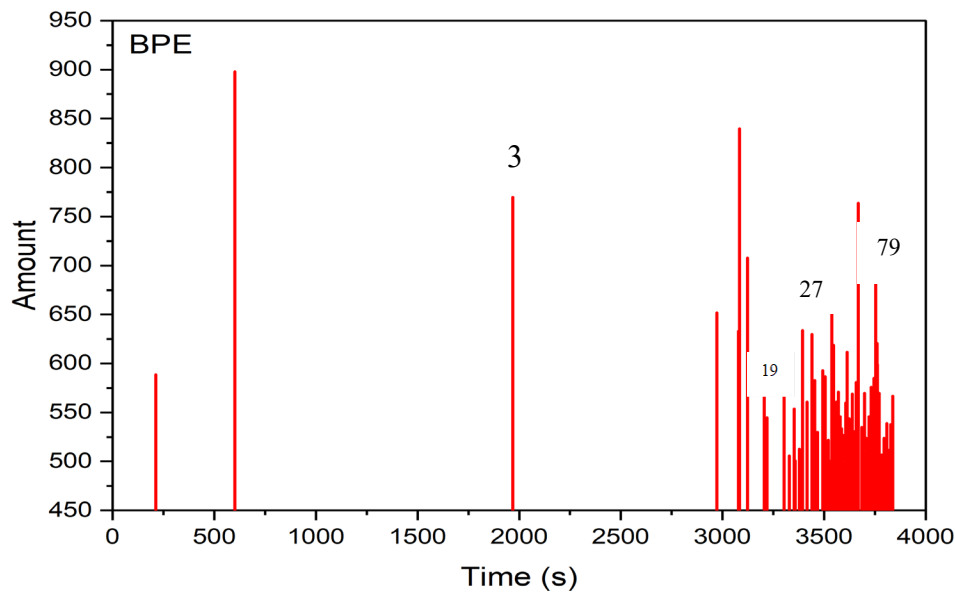


Fig. 1. The graph showed the GC-MS results on the banana peel extract.

Table 2: Phenolic compounds detected in banana peel extract using GC-MS method

No	Compound name	Retention time [s]	Type of phenolic/alkaloid	Peak
1	Phenylephrine	1968.42	Phenolic	3
2	Chlortetracycline	3446.15	Phenolic	19
3	Astaxanthin	3521.35	Phenolic	27
4	Polyphenol	3828.22	Phenolic	79

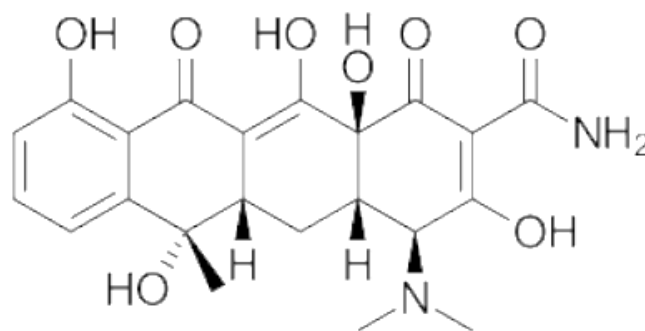


Fig. 2. Chemical structure of tetracycline.

3.2 Synthesis of magnetic silver nanoparticles

3.2.1 Ratio of banana peel extract to $AgNO_3$ solution

Initially, to determine the best ratio of banana peel extract to $AgNO_3$ solution, the experiment was carried out using the coprecipitation method and the fixed concentration of

AgNO₃ solution (1 mM). The results showed that the formation of silver nanoparticles had completely occurred when the ratio was 1:2, as in this ratio, the UV-Vis spectrum of the colloidal solution obtained showed a strong absorption peak with a peak maximum of 435 nm. This result indicates that the prepared colloidal solutions (Fig. 3) were an optically active material that could be speculated to be silver nanoparticles. Recently, it has been reported that silver nanoparticles show UV-Vis absorbance peak maximum at a wavelength between 400–500 nm due to the surface plasmon resonance [13]. The results of the UV-Vis spectrum of our studied material showed good agreement with the optical nature of the reported silver nanoparticles [13], [14]. So, it can be concluded that silver nanoparticles were synthesized through the reaction of AgNO₃ solution and banana peel extract, and the color change from yellow to grey might be due to the reduction of the Ag⁺ ion to Ag⁰, which correlates with the excitation of the surface plasmon resonance vibration that occurred in the Ag NPs. The mechanism of green synthesis of Ag NPs using banana peel extract can be described through the presence of the hydroxyl group in the extract where Ag⁺ ions were mixed with the extract, an ion-exchange process occurred, where the carboxylate group of the extract glycoprotein was transformed into –COOAg. Afterward, these –COOAg were converted into AgNPs and stabilized by polysaccharide polymer chains of the extract. The color change from yellow to grey was observed through surface plasmon resonance during this reduction process.



Fig. 3. The different ratios tested in synthesizing magnetic silver nanoparticles. 1:9 (left), 1:2 (middle), 1:3 (right).

3.3 Comparison between coprecipitation and hydrothermal methods in producing silver nanoparticles.

For both coprecipitation and hydrothermal methods, the optimized experiments were carried out using two different concentrations of precursor (AgNO₃ solution), which were 0.1 M and 0.05 M, respectively. The reaction time was believed to be one of the crucial parameters in increasing the yield of silver nanoparticles. This is because the longer reaction time will allow more Ag⁺ to be reduced for the formation of silver nanoparticles. Color changes of the AgNO₃ solution from crystal clear into yellowish-brown to grey can be clearly observed in the coprecipitation method, while for the hydrothermal method, the color changes hardly be monitored in which the final color of the solution can only be observed after finishing the experiment. Color changes indicate the formation of silver nanoparticles where the reduction of the silver ions with (Ag⁺) to the silver nanoparticles (Ag⁰). This was aligned with the earlier study, which reported that the change in color was due to the combined oscillations of conduction electrons in nanoparticles [4].

The color changes due to the reduction of Ag^+ to Ag^0 were clearly observed in the coprecipitation method, where the intensity of the brown color was increased when the different concentration of precursor (AgNO_3 solution) was used during the synthesis process, in which the darker solution of silver nanoparticles was produced when the concentration of precursor was at 0.1 M, and the color become lighter at lower concentration (0.01 M). The color changed from the different concentrations of silver nanoparticles, where the hydrothermal-synthesized silver nanoparticle with 0.1 M of AgNO_3 showed the clearest grey color, followed by 0.05 M and 0.01 M. The color changes for different concentrations of hydrothermal methods are shown in Figs. 4, 5, and 6, respectively.

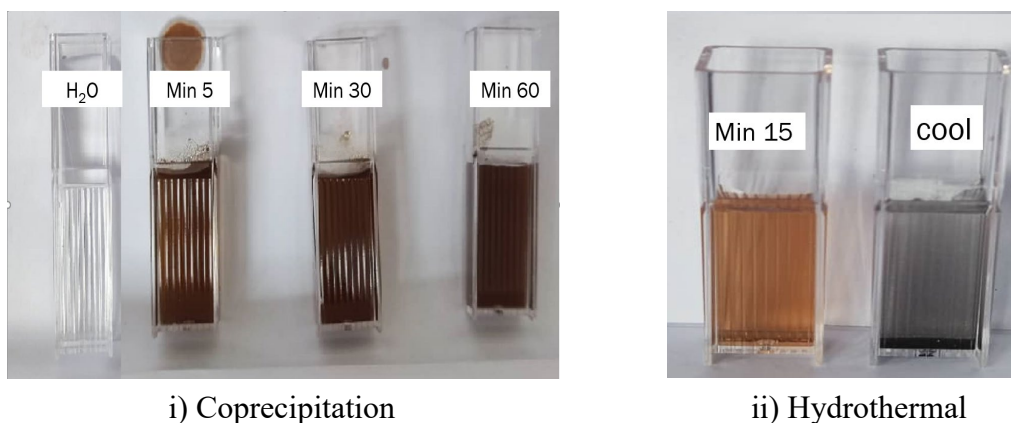


Fig. 4. Color changes during the formation of silver nanoparticles for i) coprecipitation and ii) hydrothermal methods with 0.01 M AgNO_3 .

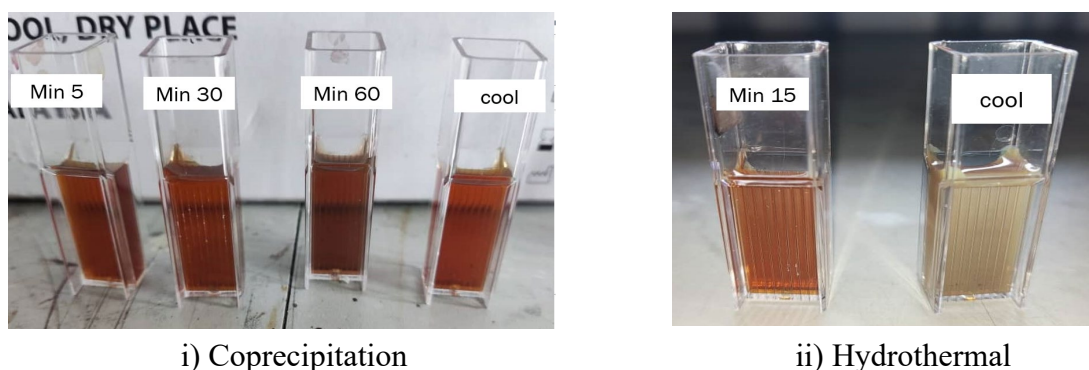


Fig. 5. Color changes during the formation of silver nanoparticles for i) coprecipitation and ii) hydrothermal methods with 0.05 M AgNO_3 .

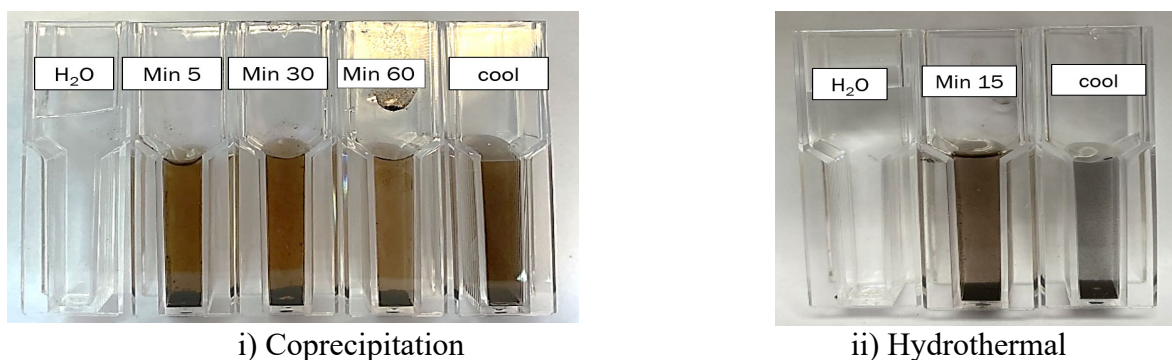


Fig. 6. Color changes during the formation of silver nanoparticles for i) coprecipitation and ii) hydrothermal methods with 0.1 M AgNO_3 .

The UV-Vis results depicted that the maxima UV-Vis absorbance of synthesized magnetic silver nanoparticles for different concentrations and methods was in the range of 400-500 nm. The results of synthesized magnetic silver nanoparticles were according to the nature of the reported silver nanoparticles, where the study stated that due to the surface plasmon resonance, the UV-Vis highest peak absorbance had a wavelength in the range of 400-500 nm for the silver nanoparticles [15]. Table 3 shows the UV-Vis spectrum for coprecipitation and hydrothermal methods at various concentrations of precursor. It was found that the maximum peak for all observations was between 427 nm and 473 nm, in which the highest peak was observed for the colloidal solution under hydrothermal process at 0.1M, which indicates that the formation of silver nanoparticles had completely occurred in that solution. The UV-Vis peak can also indicate the variation in size and shape of the nanoparticles formed, which required further observation using a field electron scanning microscope (FESEM). This is because the higher absorption intensity of the UV-Vis peak shows that the number of AgNPs formed in the solution increases with increasing reaction time.

For the morphology of the fabricated magnetic silver nanoparticle, field emission scanning electron microscope (FESEM) analysis was conducted to show the size, shape distribution, and surface structure of the synthesized nanoparticles. Table 4 shows the shape and size distribution of the silver nanoparticles produced by coprecipitation and hydrothermal methods under various concentrations of precursor. The shape of AgNPs formed for both methods was spherical, and the size varied depending on the concentration of precursor where for 0.1M AgNO₃, the average size of AgNPs was ± 56 nm for the coprecipitation method while ± 49 nm for the hydrothermal method. In the meantime, the average size for AgNPs was found to be ± 21 nm and ± 24 nm for coprecipitation and hydrothermal methods, respectively. Size and shape distribution for the synthesized silver nanoparticles in this study were all in the same range as previously reported studies where they successfully fabricated the silver nanoparticles using the *Cajanus cajan* leaf extract using coprecipitation [16]. The hydrothermal method showed better size and shape distribution for both 0.1M and 0.05M compared to the coprecipitation methods. These results of the hydrothermal method adhered with the study reported before, where they got the well-dispersed shape of synthesized nanoparticles using the hydrothermal method via Arabic gum aqueous solution [17]. This shows the advantage of the hydrothermal method, where consistent shape could be achieved [18]. However, all the synthesized silver nanoparticles for both methods have almost uniform dispersity of nanoparticles over the surface and appear to be less agglomerated. The minimal aggregation of the Ag NPs might be due to the encapsulation of Ag NPs with biomolecules. Thus, FESEM analysis depicts the potentiality of banana peel extract to minimize the aggregation of silver nanoparticles formed.

Table 3: UV-Vis spectrum for coprecipitation and hydrothermal method at different concentrations of precursor

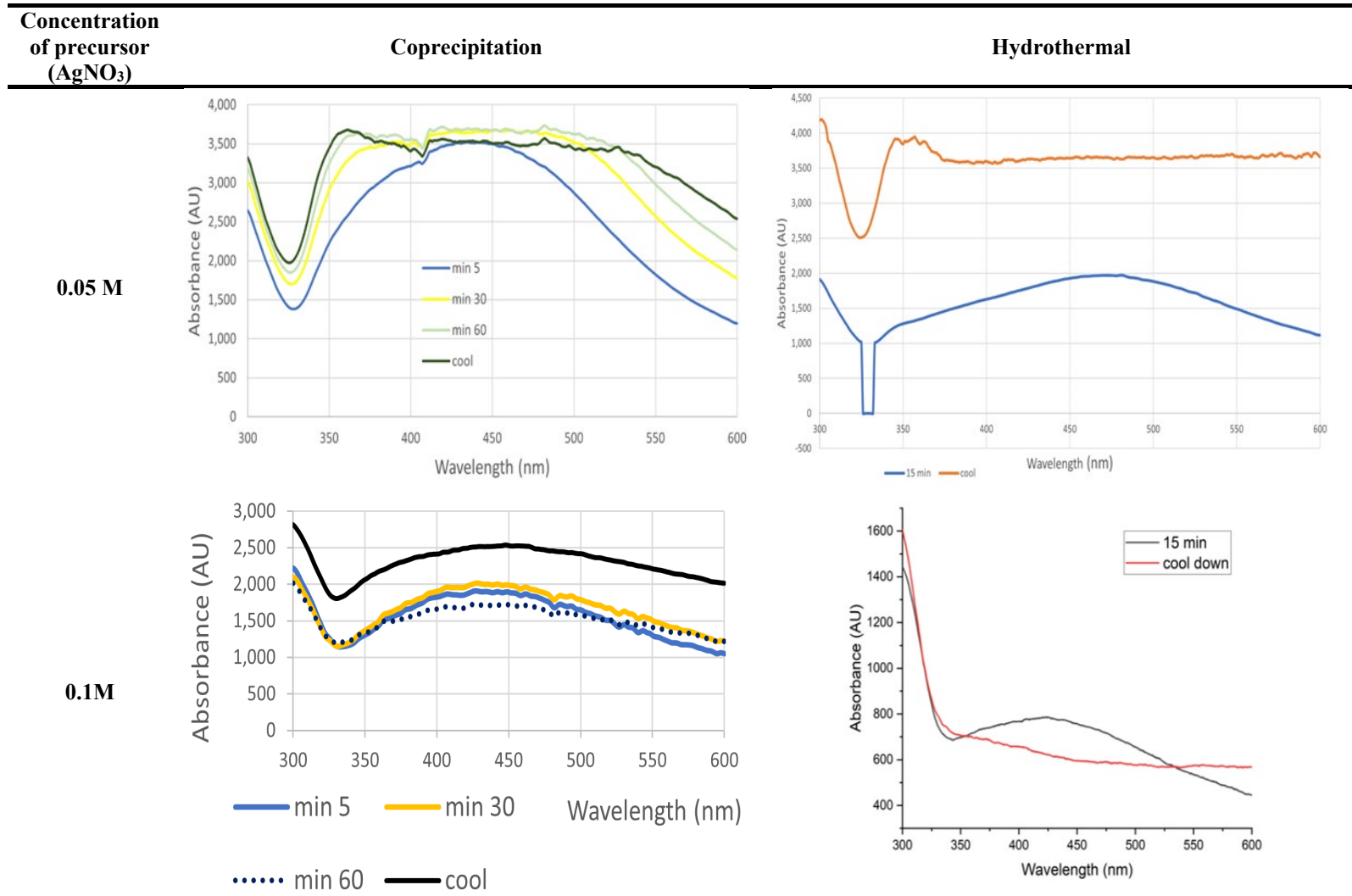
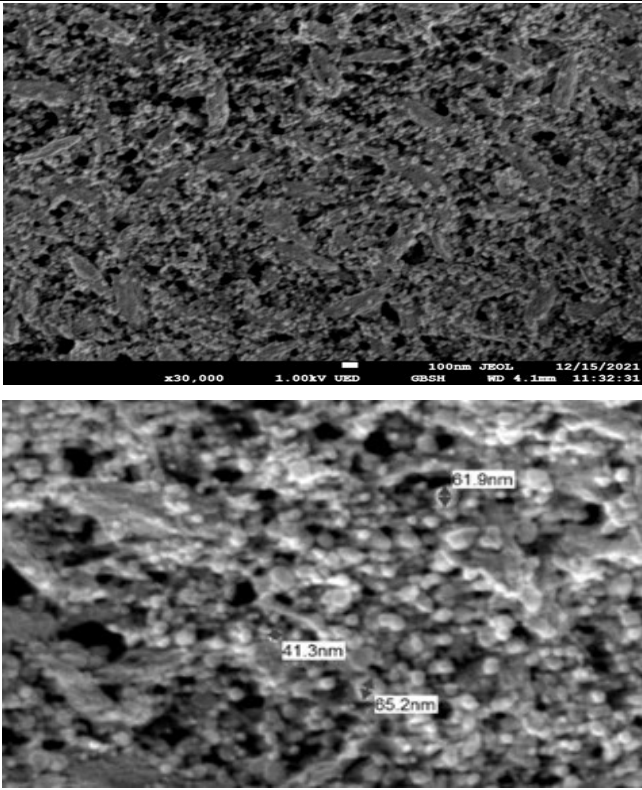
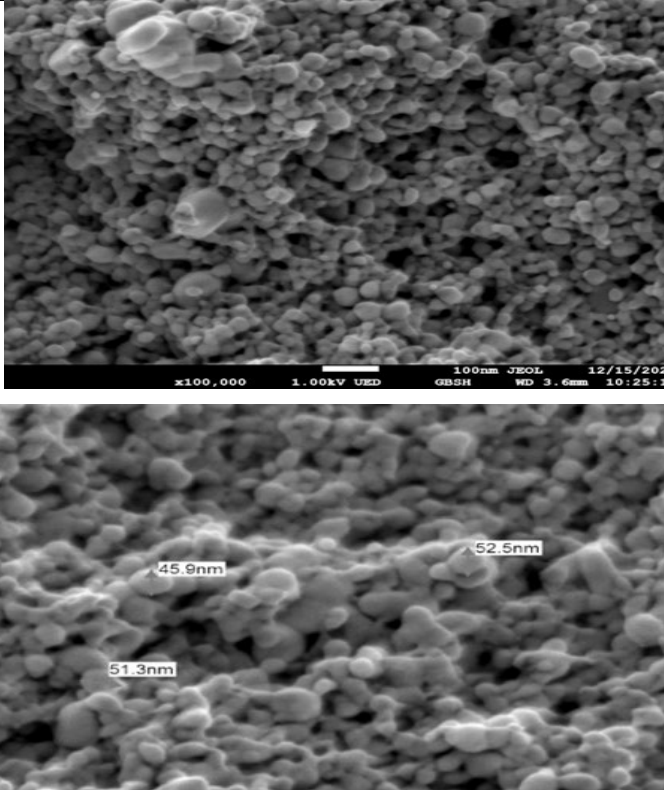
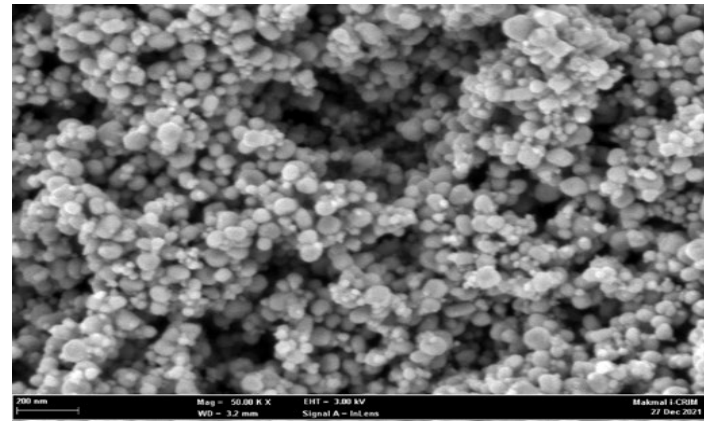
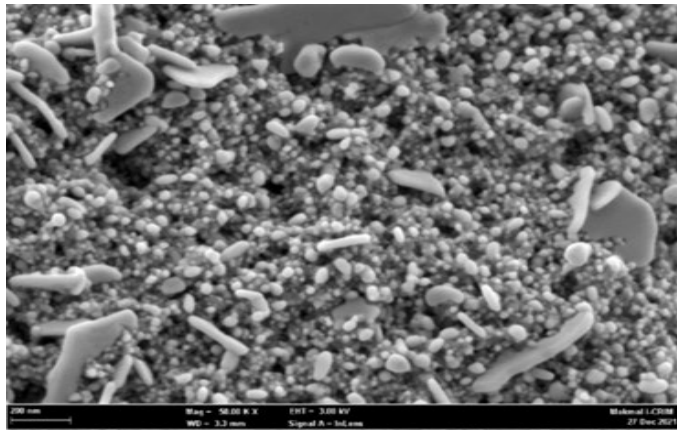
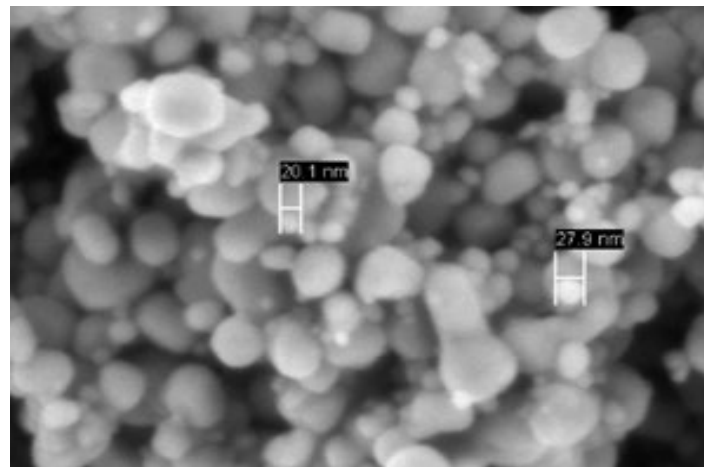
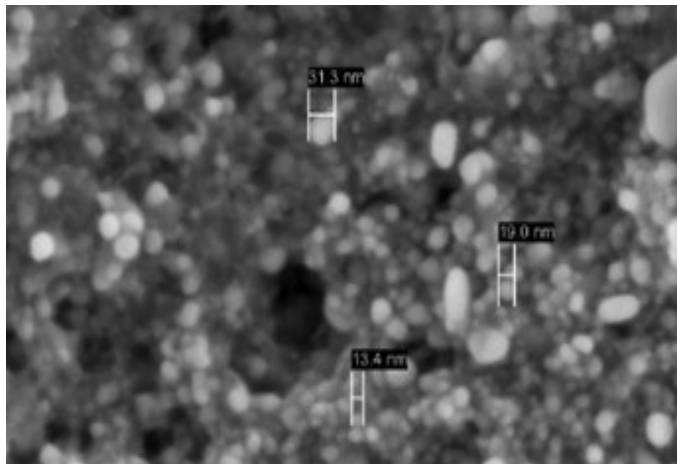


Table 4 : Comparison of size and shape of synthesized silver nanoparticles for coprecipitation and hydrothermal method

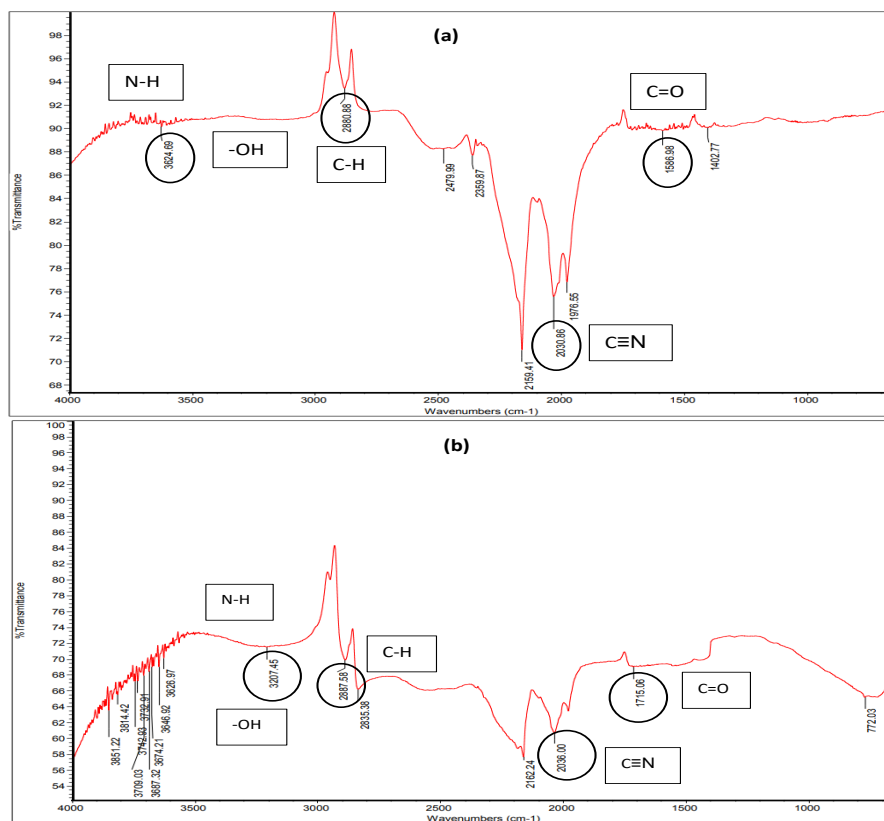
Concentration of precursor	Coprecipitation	Hydrothermal
0.05 M		



0.1M



FTIR analysis was used to study the functionalized group attached to the surface of synthesized silver nanoparticles (Fig. 7). The analysis showed that all the synthesized magnetic silver nanoparticles gave the same pattern but with different intensities. This was due to the same banana peel extraction and silver nitrate solution used for all methods. The peaks which were observed at 3624.69 cm^{-1} , 3207.45 cm^{-1} , 3631.07 cm^{-1} , and 3621.76 cm^{-1} for 0.1 M coprecipitation, 0.1 M hydrothermal, 0.05 M coprecipitation and 0.05 M hydrothermal, respectively, belonged to the -OH stretch of hydrogen-bonded for a phenolic group that was present in the banana peel extraction. Besides, the presences of methyl group were spotted at the peak of 2880.88 cm^{-1} , 2887.58 cm^{-1} , 2883.50 cm^{-1} for 0.1 M coprecipitation, 0.1 M hydrothermal, and 0.05 M coprecipitation. In addition, it could be observed at the peak of 2030.86 cm^{-1} , 2036.00 cm^{-1} , 2033.62 cm^{-1} , 2032.86 cm^{-1} for 0.1 M coprecipitation, 0.1 M hydrothermal, 0.05 M coprecipitation and 0.05M hydrothermal correspondingly which indicated the functional group of nitriles in the synthesized of silver nanoparticles. Finally, the presence of the aldehydes, ketones, or carboxylic acids could be spotted at the peak of 1586.98 cm^{-1} , 1715.06 cm^{-1} , 1501.36 cm^{-1} , 1492.14 cm^{-1} for 0.1 M coprecipitation, 0.1 M hydrothermal, 0.05 M coprecipitation and 0.05 M hydrothermal, respectively. The presence of the -OH group from the phenolic groups could be the reducing and stabilizing agents for the synthesized magnetic silver nanoparticles with the help of other phytochemicals present in the banana peel, like carboxylic acids and nitriles. These results were quite similar to the prior study, which reported the same presence of functional groups during the synthesized of silver nanoparticles using *Artocarpus heterophyllus* leaves [15].



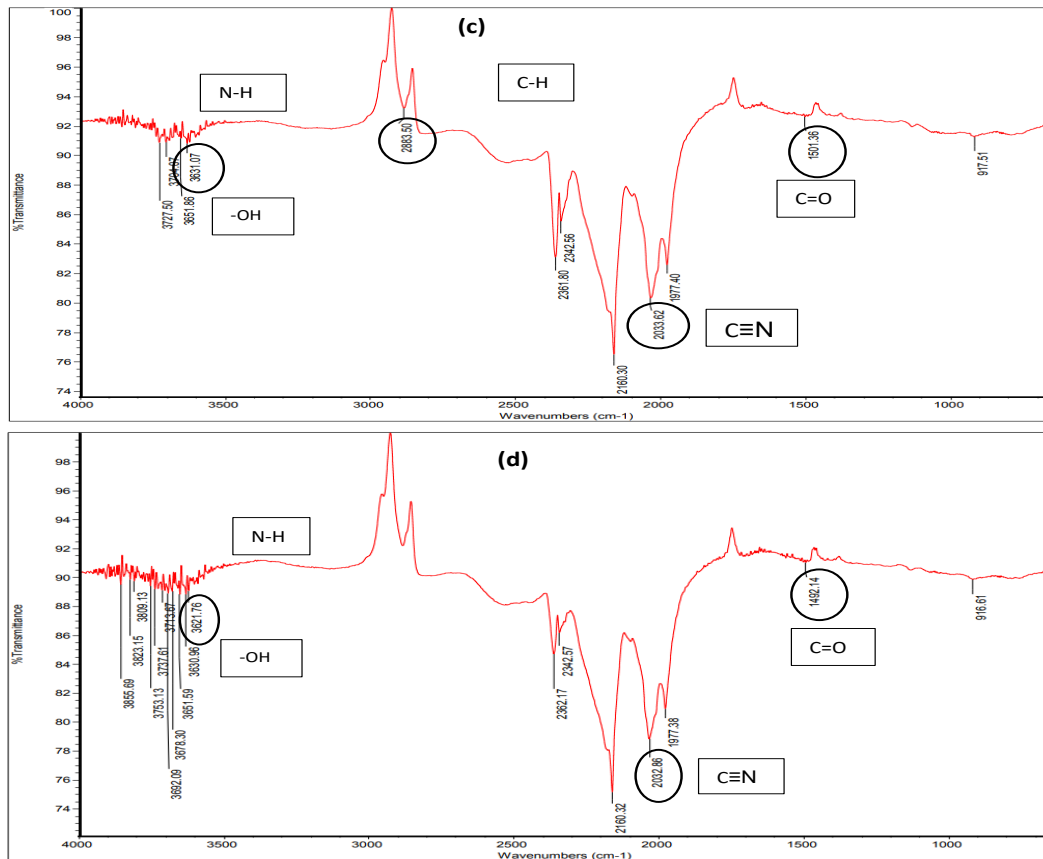


Fig. 7. FTIR analysis of silver nanoparticles with different methods and concentrations, (a) 0.1 M coprecipitation, (b) 0.1 M hydrothermal, (c) 0.05 M coprecipitation, (d) 0.05 M hydrothermal.

A vibrating sample magnetometer (VSM) analysis was performed to investigate the magnetism properties of the synthesized magnetic silver nanoparticles. In Table 5, all the synthesized magnetic silver nanoparticles showed weak ferromagnetic properties. However, both methods with a concentration of 0.05M showed higher saturation magnetism and coercivity compared to the concentration of 0.1M for hydrothermal and coprecipitation. The results shown were due to the bigger size of synthesized silver nanoparticles with 0.05M for hydrothermal and coprecipitation that led to the decreasing of crystallinity of the magnetic domain [19]. The results of VSM analysis in this study were slightly better than in the previous study, where they succeeded in synthesizing the magnetic silver nanoparticles using the *Artocarpus heterophyllus* leaves [15] Hence, the ferromagnetic behavior of the synthesized silver nanoparticles showed that this present experiment succeeded in synthesizing magnetic silver nanoparticles using banana plant extract and AgNO_3 solution.

It was predicted that ferromagnetic spin polarization could take place in 4D and 5D transition metals with reduced coordination geometry because of the large ratio of the number of atoms on the surface to the number in the core [20]. Consequently, it was speculated that the ferromagnetic nature of the silver nanoparticles might be due to the spin polarization. Moreover, the weak ferromagnetic nature of the silver nanoparticles might be due to the weak coupling between the capping agents and the silver surface [21]. The ferromagnetic nature of thiol-capped silver nanoparticles was reported earlier, where the authors claimed that charge transfer occurred from the surface metal to the S atoms, which induced the anisotropic orbital momenta, resulting in the magnetic nature of the silver nanoparticles [22]. The presence of oxygen in the nanoparticle sample predicts the existence

of a non-stoichiometric oxide layer on the surface, which results in the ferromagnetic nature of silver nanoparticles [23]. The ferromagnetic behavior of the synthesized silver nanoparticles indicates the success of our present study in synthesizing organic molecules capped magnetic silver nanoparticles using banana peel extract and AgNO₃ solution.

Table 5: VSM analysis results on the magnetic properties of synthesized silver nanoparticles

Method	Concentration [M]	Coercivity, H_c [Gauss]	Magnetization M_s [emu/g]
Coprecipitation	0.1	391.84	0.7318
Hydrothermal	0.1	280.99	0.9186
Coprecipitation	0.05	255.58	2.8275
Hydrothermal	0.05	537.02	3.9077

4. CONCLUSION

This study showed the comparison of two different methods, hydrothermal and coprecipitation methods, in green synthesis of magnetic silver nanoparticles by using the banana peel extract as a reductant and AgNO₃ as the precursor. The different ratios of banana peel extract and silver nitrate solution and different concentrations of AgNO₃ are also being used to show the comparison between these two methods. The best ratio was 1:2 because it could yield a high product in the end. The comparison between the hydrothermal and coprecipitation methods showed that the hydrothermal could synthesize the mono-dispersed, uniformly distributed spherical shape compared to the coprecipitation method. The magnetic properties of synthesized magnetic silver nanoparticles also depicted that hydrothermal was able to fabricate higher magnetism with 0.7318 emu/g compared to the coprecipitation method with 0.9186 emu/g. Hence, the hydrothermal method was favorable in synthesizing the magnetic silver nanoparticles compared to the coprecipitation method. Ferromagnetic silver nanoparticles achieved from the banana peel extract-mediated green synthesis have immense potential and might have a notable impact on the pharmaceutical, biomedical, and cosmetic industries. Moreover, these silver nanoparticles could become an alternative to chemically synthesized silver nanoparticles.

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