

## Stability Characteristics of Trisodium Citrate–Stabilized Silver–Water Nanofluids

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### Abstract

Silver nanoparticles (AgNPs) exhibit special properties due to their large surface-area-to-volume ratio. Silver–water nanofluids (NFs, dispersion of AgNPs in water) can be utilized in mass transfer applications in 3D printing industries for printed electronics, heat transfer, and drug delivery, among other uses. This paper reports the preparation and stability characteristics evaluation of 2.0 vol% silver–water NFs. First, size-controlled one-pot facile synthesis of AgNPs was carried out by chemical reduction method using  $\text{AgNO}_3$  as a precursor (P), hydrazine as a reducing agent (RA), and trisodium citrate as a capping agent (CA) with different molar ratios of P: RA: CA=1:3:0, P: RA: CA=1:3:3, P: RA: CA=1:3:6, P: RA:CA=1:3:9, and P: RA: CA=1:3:12. Next, synthesized products were characterized by ultraviolet-visible spectroscopy (UV-vis), X-ray diffraction (XRD), scanning electron microscopy (SEM), energy dispersive X-ray (EDX) spectroscopy, and transmission electron microscopy (TEM). Crystalline spherical AgNPs with an average size of 35 nm were formed. Then, 2.0 vol% AgNPs–water NFs were prepared by adding the required amount of AgNPs to water, and the stability characteristics of the prepared NFs were evaluated by the comparison of sedimentation with time, UV–vis spectrum analysis, Zeta potential measurements, and dynamic light scattering (DLS). The prepared 2.0 vol% AgNPs–water NFs showed stability for at least two weeks. Results of this study indicate that the silver–water NFs have the potential for many industrial applications, such as heat transfer, mass transfer, drug delivery, etc.

**Keywords:** trisodium citrate capped silver nanoparticles, silver nanoparticles–water dispersion, stability assessment, ultraviolet-visible spectroscopy, dynamic light scattering

### 1. Introduction

Silver nanoparticles (AgNPs, sizes 1-100 nm) exhibit size-dependent special properties due to their large surface area-to-volume ratio. Silver-based nanofluids (NFs) offer a variety of new applications since these NFs exhibit enhanced properties in heat and mass transfer. The liquid state of AgNPs is required for such applications because the liquid state enables them to be handled well. Basically, NF is the dispersion of NPs in a base fluid (BF). The prime component of the NFs is NPs. The selection of NPs and BF in NF preparation depends on the purpose and suitability of applications. Silver–water NFs, dispersion of AgNPs in water<sup>1</sup>, can be used in mass transfer in three-dimensional (3D) printing industries for printed electronics<sup>2–4</sup>, heat transfer<sup>5,6</sup>, drug delivery<sup>7</sup>, including many other applications. The possible areas of application of NFs are increasing with the expanding magnitude of research. It is expected that silver-based dispersions will be more lucrative materials for various fields shortly. However, the good dispersion and stability characteristics of AgNPs in liquid are the primary issue for such applications.

Before going for a stability characteristics study, preparation of NFs is mandatory. NF formulation method is important because it does not mean only mixing the solid particles in a liquid, rather something else. To prepare NFs, mostly the two-step method is followed, in which first NPs are synthesized, and then NFs are prepared by dispersing them in BFs<sup>8,9</sup>. For feasibility, a one-step method is also used in which the NPs synthesis and NF preparation is done in a single step, although this method has limitations. Often,

NPs of NFs tend to agglomerate shortly after their preparation. Therefore, the stability characteristics of NF is a great concern. Although a good amount of research is available on the synthesis and applications of AgNPs, however, very limited paper is found on the stability study of AgNPs in liquid media.

The stability of NFs mainly depends on the particle size and concentration<sup>10</sup>. The stability of NFs can be attained by some means like pH adjustment, zeta potential, ultrasonification, surfactant usage, surface modification of NPs, etc.<sup>11</sup> Adding surfactant is frequently used to prepare stable NFs. Surfactants are attached by absorption on the surface of AgNPs, which prevents the attraction and favors the repulsion among NPs. However, this technique has some disadvantages because of the physisorption of the surfactant molecules on NPs. Conversely, the chemisorption of surface capping agents can overcome such disadvantages. In-situ chemisorption, the chemical attachment of surfactant molecules on the surface of NPs in the reactor vessel during NPs growth and synthesis, is an effective way of adding surface capping or surface-modifying molecules on the surface of NPs<sup>12</sup>. Such bonds between the NPs and surface capping molecules are very strong. Surface capping agents remain stable if the NPs are dispersed again in the liquid media.

Another important property related to the stability of NF is dispersion. Generally, the smaller the particle size, the higher the possibility of dispersion. Of course, there might be agglomerations in liquid media as well. Dispersion of NPs in BFs depends on various properties such as the

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surface nature of NPs, the volume percent of NPs, properties of solvents, solvents-solvents, solvents-NPs, NPs-NPs interactions, etc., and, of course, importantly, on the size of the NPs. Therefore, synthesizing AgNPs by keeping the above-mentioned issues and small particle size is in particular, is key technique for obtaining stable NFs with a high percentage of particle concentration, which may intensify many industrial processes and may have better performance in many areas, including 3D inkjet printing.

AgNPs can be synthesized by several routes including physical, chemical, and biological<sup>13</sup>. Among these, the chemical route is widely used because of its easiness<sup>14</sup>. The chemical route has some advantages over other routes, such as it requires simple instruments, being economically cheap, short reaction time, and high yield percentage. Chemical reduction is one of the common methods of NPs synthesis<sup>14</sup>. Generally, NPs are very unstable, and aggregation occurs, i.e., particle size becomes larger very fast due to the high surface energy of nanoparticles<sup>15</sup>. To prevent the aggregation, the synthesis of NPs using CAs has been reviewed and reported.<sup>16</sup> Additionally, the attachment of CAs to the surface of the AgNPs suppresses the particle growth; consequently, small particles are obtained<sup>17</sup>. To synthesize AgNPs, most chemical reduction methods use silver nitrate, sodium borohydride/hydrazine, TSC, and water as a precursor (P), reducing agent (RA), capping agent (CA), and reaction media, respectively<sup>13</sup>. One-pot facile synthesis of AgNPs is possible by mixing the precursor, reducing agent, capping agent, and water, and by the reduction of AgNO<sub>3</sub>, which involves several stages such as atomization (from silver ion), aggregation, nucleation, particle growth, and NPs formation. In this way, surface modification of AgNPs enables them to be produced in real nanometer sizes. Some researchers focused on the preparation of AgNPs by the chemical reduction method using TSC as capping agents; however, no paper according to our survey investigated the effect of the CAs' molar ratios on the stability of NPs dispersion.

Following AgNPs synthesis, NF preparation is necessary. If the NF formulation is completed, the stability characteristics need to be evaluated. Prior to every application, the prepared NF's stability must be evaluated. According to Ilyas et al.<sup>18</sup>, one of the methods for evaluating stability is the visual inspection of NFs whether in a clear solution, agglomerations, or sedimentation. Quick sedimentation suggests that the prepared NFs are unstable. NPs frequently agglomerate into huge clusters in liquid media that eventually settle down at the bottom of the container, as can be seen in a transparent glass vial. Another way to assess the stability of NFs is to compare the photos that are taken using a camera. Zeta potential measurement is one useful method to verify stability. An NF's Zeta potential value between +60 mV and -60 mV typically denotes stability, and NFs are often unstable when their zeta potential is larger than +60 mV or less than -60 mV<sup>19</sup>. A different method for evaluating the stability of NFs is DLS measurement with light scattering theory. By comparing the

sizes of pristine and liquid phase particles, DLS yields the diameter of the former. The sizes of NPs in liquids can reveal whether or not the NF is stable. This allows for the evaluation of the prepared NFs' stability. In this paper, we report the synthesis of AgNPs from AgNO<sub>3</sub> with various ratio of TSC, the preparation of AgNPs-water NFs at different concentrations, and tested the stability characteristics of silver–water NFs using sedimentation observation by image capturing with time intervals, UV-vis, and DLS analysis.

## II. Materials and Methods

### *Materials*

Silver nitrate (AgNO<sub>3</sub>, 99.5%) and hydrazine hydrate (H<sub>4</sub>N<sub>2</sub>·H<sub>2</sub>O, 80%) were used as a precursor and reducing agent, respectively, and were purchased from Merck, Germany, and used as received. Trisodium citrate (TSC), a capping agent, was purchased from Merck, India. De-ionized water (DIW) was used as a solvent for all solution preparation and other purposes.

### *Synthesis of TSC-coated and uncoated AgNPs*

AgNPs were synthesized by a chemical reduction method following the procedure mentioned in our previous paper<sup>20,21</sup>. Samples were prepared using AgNO<sub>3</sub> salt as a, hydrazine as the RA, and TSC as a CA. Five types of AgNPs samples were prepared with a molar ratio of (a) P: RA: CA=1:3:0, (b) P: RA: CA=1:3:3, (c) P: RA: CA=1:3:6; (d) P: RA: CA=1:3:9, and (e) P: RA: CA=1:3:12. To prepare TSC coated AgNPs, typically 50 mL of 0.001 mmolL<sup>-1</sup> AgNO<sub>3</sub> aqueous solution was prepared simply by dissolving the required amounts of AgNO<sub>3</sub> salt into DIW. Separately, an aqueous solution of hydrazine was prepared in DIW. Both solutions were mixed, followed by the addition of TSC dropwise with rigorous magnetic stirring. Next, the solution mixture was vigorously magnetically stirred for ~4 h. Black coloration was observed in the beginning. After completion of the reaction, the solution turned into a brown/orange color, which indicates the formation of AgNPs. Particle formation occurs here by the 'bottom-up' approach. It is assumed that the silver ions (Ag<sup>+</sup>) of the solution are atomized, atoms form aggregates due to the friction of the stirrer, several aggregates combine by nucleation for NPs form, and surface capping is attached. Thus, synthesized AgNPs are obtained as colloidal dispersions in a mixture of water, unreacted hydrazine, and the precursor solution. For analysis purposes, the solid NPs were collected. To do so, the unreacted hydrazine and unreacted silver ions (if any) were removed by centrifugation at 8000 rpm for 20 min with DIW. The particles were re-dispersed in DIW and collected after centrifugation at 8000 rpm for 20 min. To purify the products properly, the process was repeated and dried well in the oven at 105 °C for 3 hours. Finally, the synthesized products were stored in desiccator for further characterization.

### Characterizations of synthesized AgNPs

To get the primary idea of whether the AgNPs were formed or not, a UV-vis spectrum was taken using a spectrophotometer (UV-1800, Shimadzu, Japan) with a variable wavelength ranging from 200 nm to 800 nm in quartz cuvettes having a path length of 10 mm. The crystallinity of the synthesized products was examined using the X-ray diffractometer (SmartLab 9MTP; Rigaku Corporation) with the CuK $\alpha$  radiation in a 2 $\theta$ - $\theta$  setup. The 2 $\theta$  angle was scanned between 10° and 90° at a scanning rate of 0.02°/0.6 s. The measured data were compared with the data from the Joint Committee for Powder Diffraction Studies (JCPDS) file for silver. The average crystallite size was calculated based on Debye-Scherrer's equation with the two vital peak patterns. SEM was taken to see the particle morphology using an electron microscope (FESEM, 7610F, JEOL, USA), and EDX was taken to know the elemental configuration using an energy-dispersive X-ray spectrophotometer section of the same instrument. Particle size, shape, and morphology were examined using a transmission electron microscope (TEM, HD 2700, Hitachi Corporation, Japan) with 200 kV acceleration voltage. The mean particle size was obtained by measuring the size of 100 particles in the TEM images.

### 2.4 Preparation and Stability Assessment of AgNPs–water NFs

After the synthesis of a typical batch, AgNPs were first dried (at 105 °C) in the oven, and the weight was recorded. Similarly, the weight of 10 batches of AgNPs was taken, and the average weight was obtained as 16 mg. In this case, AgNPs were not dried before the preparation of NFs. Rather, after washing and centrifugation, the required amount of product of batches was dispersed in 10 mL of DIW to obtain 2.0 vol% of AgNPs–water NFs. This NFs preparation is similar to the two-step method. The following equation (1) is used to calculate the vol% of the AgNPs–water NFs.

$$\text{Vol}\%, \Phi = \left[ \frac{\left( \frac{W_{\text{Ag}}}{\rho_{\text{Ag}}} \right)}{\left( \frac{W_{\text{Ag}}}{\rho_{\text{Ag}}} + \frac{W_{\text{bf}}}{\rho_{\text{bf}}} \right)} \right] \times 100 = \left[ \frac{\left( \frac{W_{\text{Ag}}}{10,800} \right)}{\left( \frac{W_{\text{Ag}}}{10,800} + \frac{10}{1000} \right)} \right] \times 100 \quad (1)$$

Here,  $\Phi$  is volume fraction (%),  $W_{\text{Ag}}$ =weight of Ag,  $\rho_{\text{Ag}}$ =density of Ag,  $W_{\text{bf}}$ =weight of bf,  $\rho_{\text{bf}}$ =density of bf.

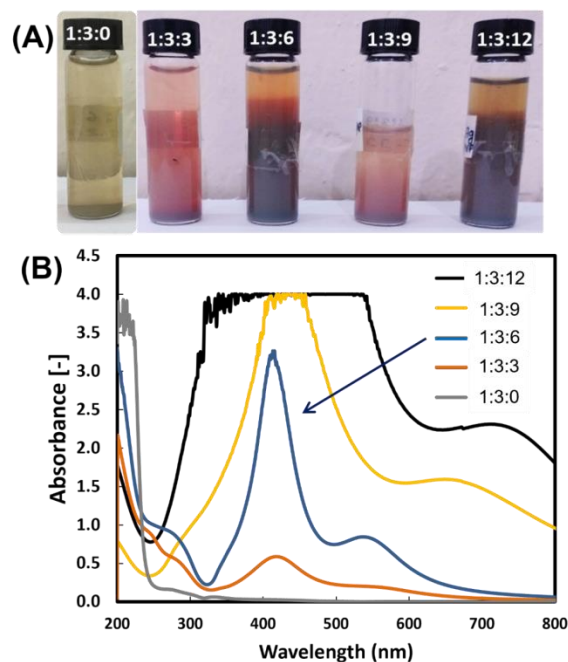
As mentioned earlier, AgNPs–water NF was prepared in two steps. That means NPs were synthesized, washed, purified, centrifuged, and collected. Then, for NFs preparation, centrifuged NPs are re-dispersed in water, and NFs are prepared. The NFs preparation does not require any further treatment. First, NFs' stability was observed by the naked eye. Images of the prepared NFs were captured by a camera. Next, A UV-1800 spectrophotometer from Shimadzu, Japan, with a variable wavelength between 200 nm and 800 nm, along with quartz cuvettes with a 10 mm path length, was used to perform the stability assessment of the NF at room temperature. A Zeta seizer ZS-nano

Malvern, UK, is also used by DLS to measure zeta potential and zeta size.

## III. Results and Discussion

### UV-visible spectrum analysis

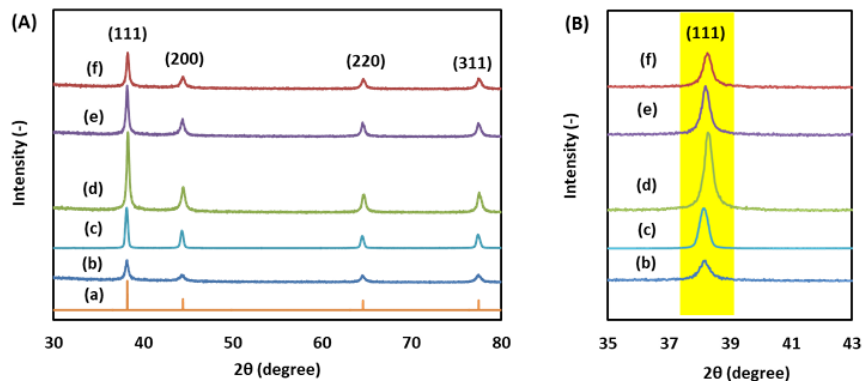
Figure 1 depicts the UV vis spectrum of the four AgNPs samples synthesized with five different molar ratios (P: RA: CA=1:3:0, P: RA: CA=1:3:3, P: RA: CA=1:3:6, P: RA: CA=1:3:9, and P: RA: CA=1:3:12) of TSC as CA. In every synthesized product, the molarity of P and RA remained the same; however, only the molarity of CA changed. Obtained  $\lambda_{\text{max}}$  from the UV vis spectrum for the products with the molar ratio of P: RA: CA=1:3:0, P: RA: CA=1:3:3, P: RA: CA=1:3:6, P: RA: CA=1:3:9, and P: RA: CA=1:3:12 were 413, 416, 420, and 426 nm, respectively, which are close to the literature values<sup>14,22</sup>. Observe  $\lambda_{\text{max}}$  values indicate the formation of the above four products. Shifting of the  $\lambda_{\text{max}}$  may be due to the size differences of AgNPs. Notably, Fig. 1 depicts the most intense peak for the sample with the molar ratio of P: RA: CA=1:3:6. On the other hand, the product without CA does not have any  $\lambda_{\text{max}}$  as seen in the lowest curve of Fig. 1. It is, probably, due the rapid agglomerations AgNPs and then sedimentation of the agglomerated or clusters at the bottom of the sample vial. Peak intensity data is significant as the peak intensity may relate to the particle growth and their stability in a solvent. In the same way, the stability of AgNPs dispersions in water could be monitored using the UV vis method. Peak intensity and peak shifting depend mainly on the particle size, which is also related to stability.



**Fig. 1.** UV-vis spectrum of the trisodium citrate capped AgNPs water NFs at a concentration of 2.0 vol% with the molar ratio of (a) P: RA: CA=1:3:0; (b) P: RA: CA=1:3:3; (c) P: RA: CA=1:3:6; (d) P: RA: CA=1:3:9 and (e) P: RA: CA=1:3:12.

The obtained XRD patterns for the synthesized AgNPs are displayed in Fig. 2. XRD patterns of synthesized AgNPs with the molar ratio of (a) JCPDS (Card No. 00-004-0783), (b) P: RA: CA=1:3:0; (c) P: RA: CA=1:3:3; (d) P: RA: CA=1:3:6; (e) P: RA: CA=1:3:9 and (f) P: RA: CA=1:3:12 are displayed in Fig. 2 (A). The XRD patterns clearly show four diffraction peaks of planes (*hkl* values) of (111), (200), (220), and (311) at corresponding different ( $2\theta$ ) values, respectively, which indicates the crystalline nature of the synthesized products of all types. Compared with JCPDS (Card No. 00-004-0783), the patterns of the chemically

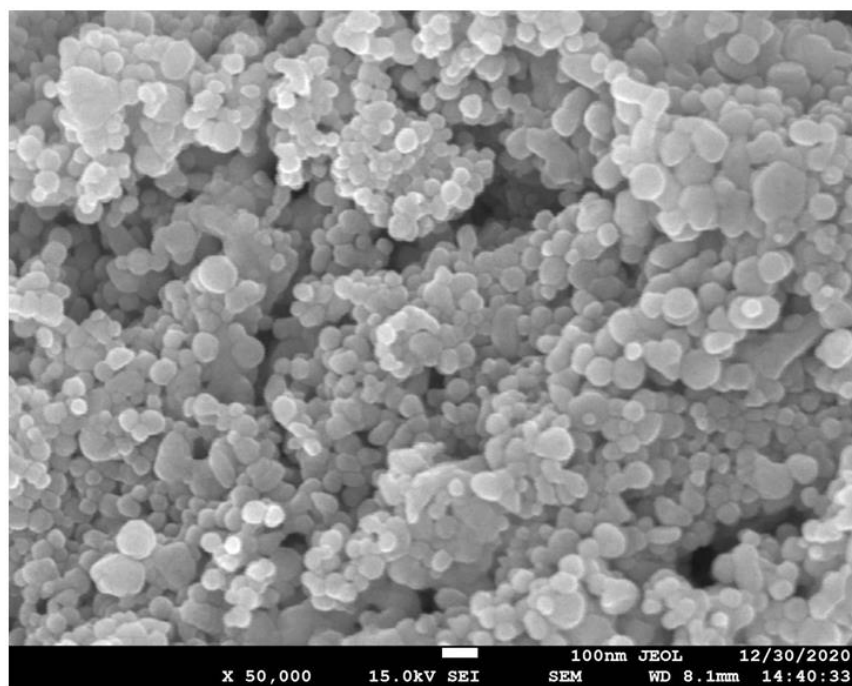
synthesized AgNPs indicate a face-centered cubic (fcc) structure with the  $Fm\bar{3}m$  space group as seen in Fig. 2. It is seen that the most intense peak is at the (111) plane varies. Fig. 2(B) shows the relative peak intensities and also indicates that the most intense peak was found for the product of the ratio P: RA: CA=1:3:6. It is evident that from both UV vis and XRD analysis, the AgNPs with the molar ratio of P: RA: CA=1:3:6 provide the best products. Therefore, for further analysis and application, a product with a molar ratio of P: RA: CA=1:3:6 was used.



**Fig. 2.** XRD patterns of the products (A); (a) JCPDS file No. 00-004-0783, and AgNPs synthesized with the molar ratio of TSC as capping agents (b) P:RA: CA=1:3:0 (c) P:RA: CA=1:3:3 (d) P:RA: CA=1:3:6, and (e) P:RA: CA=1:3:9 and (f) P:RA: CA=1:3:12; (B) Magnified peak intensity and width at (111) position of five products.

The SEM images of the AgNPs with the molar ratio of P: RA: CA=1:3:6 is presented in Fig. 3. AgNPs exhibit a smooth surface, almost uniform small sizes, and spherical shape. Comparing the SEM sizes with those obtained from

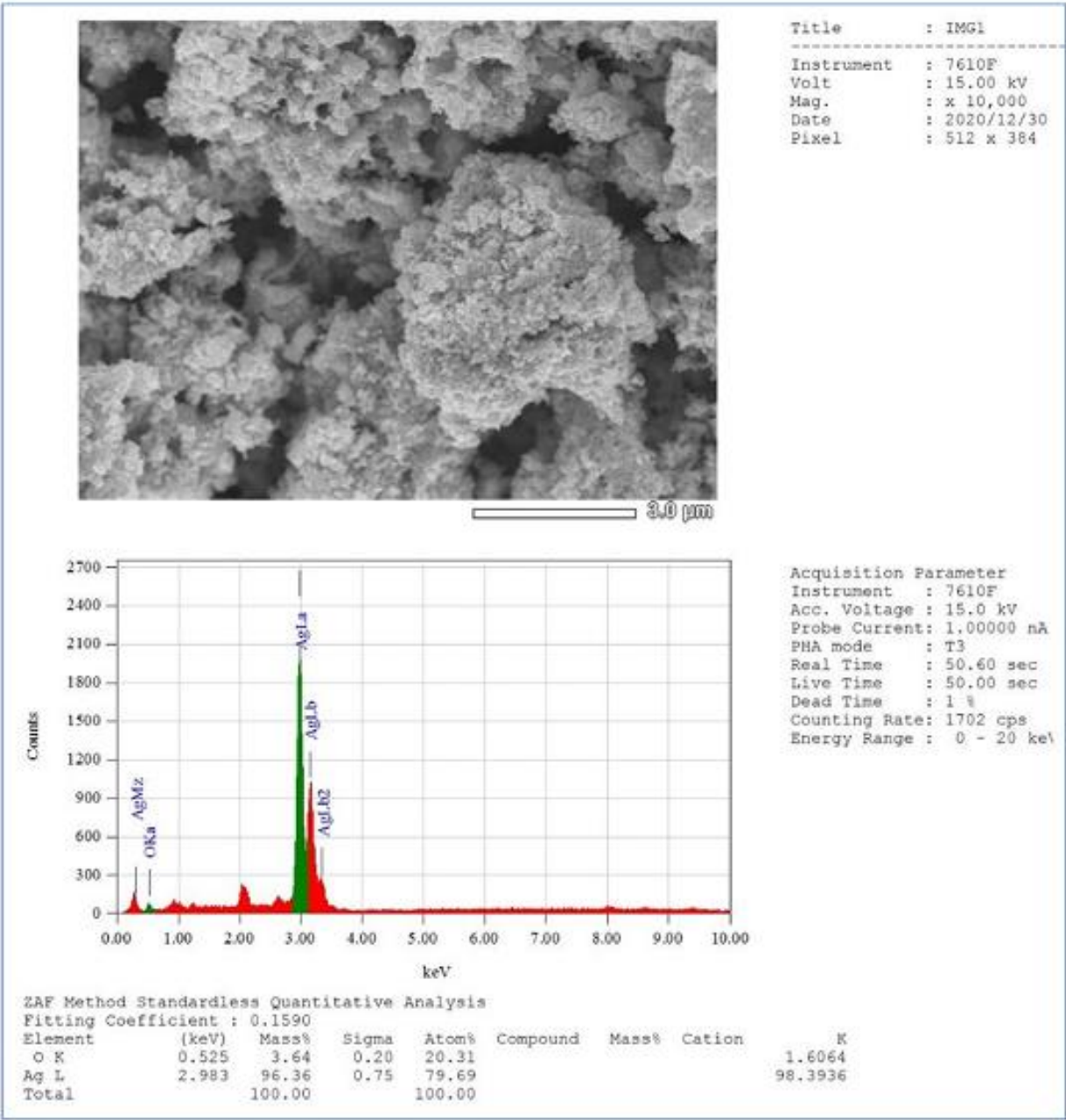
XRD, it can be said that the AgNPs are aggregated. The size of the particles/aggregates was <100 nm as seen in Fig. 3. Similar results were reported in the literature<sup>21</sup>. To get a clearer picture, TEM analysis may be worthwhile.



**Fig. 3.** SEM images of synthesized AgNPs with the molar ratio of P: RA: CA=1:3:6 at  $\times 50000$  magnification.

Figure 4 shows the EDX spectrums of the prepared AgNPs, which suggests the existence of silver (Ag) since it shows a typical strong signal peak at 3keV<sup>23</sup>. Peaks in Fig. 4 also reveal that mostly Ag elements exist, including a tiny silver

peak of elemental oxygen (O) peak which might be coming from the EDX chamber. Moreover, XRD analysis didn't suggest the existence of AgO/Ag<sub>2</sub>O. This result indicates a high percentage and purity of AgNPs.

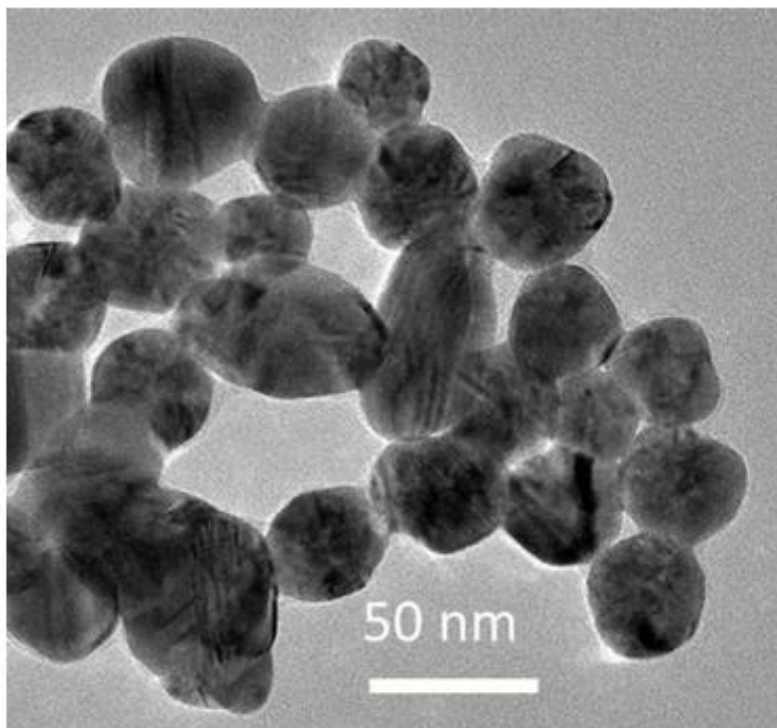


**Fig. 4.** Energy dispersive X-ray spectrum of the synthesized AgNPs with the molar ratio of P: RA: CA=1:3:6.

Figure 5 depicts the TEM image of AgNPs synthesized with the molar ratio of P: RA: CA=1:3:6. The TEM image in Fig. 5 shows small sizes with spherical shapes. The average particle size obtained from TEM calculation is 35 nm for TSC-coated AgNPs, which is comparable with the

value obtained from XRD calculation. The attachment of CAs to the surface of AgNPs suppresses the particle's growth. Consequently, smaller particles are obtained. In this way, the surface coating of AgNPs enables them to be produced in real nanometer sizes.





**Fig. 5.** TEM images of synthesized AgNPs with the molar ratios of P: RA: CA=1:3:6.

#### *Preparation and Stability Assessment of AgNPs–water*

Fig. 6 shows the images of prepared AgNPs–water NFs with concentrations of 2.0 vol%. No sedimentation was

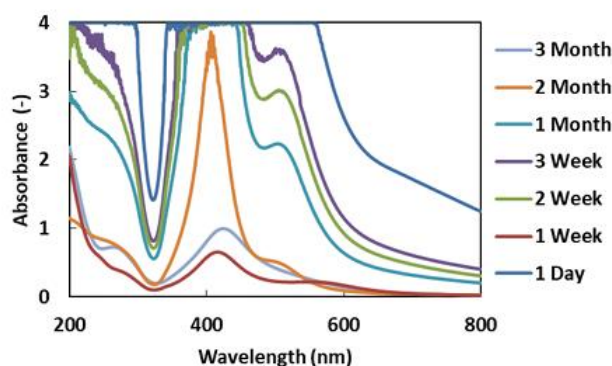
observed from the image, which indicates the stability of the prepared NFs. However, it is very difficult to understand stability only from picture observation.



**Fig. 6.** Images of the TSC-capped AgNPs–water NFs at a concentration of 2.0 vol% prepared with the molar ratio of P:RA: CA=1:3:6 at different time intervals, observation of 1 day, 1 week, 2 weeks, 3 weeks, 1 month, 2 months, and 3 months.

Next, the obtained absorbance spectra of synthesized samples prepared with the molar ratio of P: RA: CA=1:3:6 are taken as given in Fig. 7. Characteristic strong absorption peak maxima ( $I_{max}$ ) at around 400 nm of the colloidal product of surface capping whereas no peak is observed for uncapped particles as these sediments immediately after production as is seen in Fig. It is observed that the UV vis

peaks after 2 weeks have a small hump around 500 nm is probably due to the agglomeration formation with time increment. As it is seen from the spectrum, the peak intensity decreases with time increases. This phenomenon indicates the low stability at longer times from their preparation.

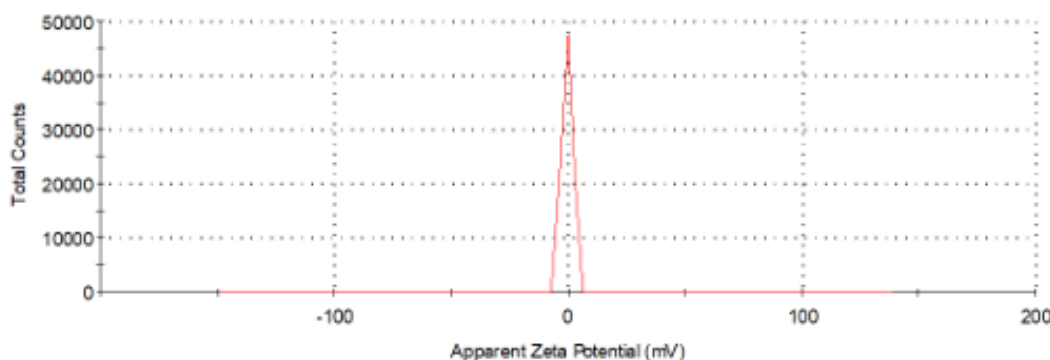


**Fig. 7.** UV vis spectrum of 2.0 vol% AgNPs water NFs with AgNPs synthesized with the molar ratio of P: RA: CA=1:3:6 at different time intervals of 1 day, 1 week, 2 weeks, 3 weeks, 1 month, 2 months, and 3months.

#### Zeta potential measurement

Zeta potential and zeta size of the prepared NFs were measured at 25 °C to assess the stability of the AgNPs–

water NF. Fig. 8 shows the zeta potential of the 2.0 vol% AgNPs-water NF. Zeta potential value between +10 mV to -10 mV i.e., near Zero which indicates the instability of the NF 2 months after the preparation.

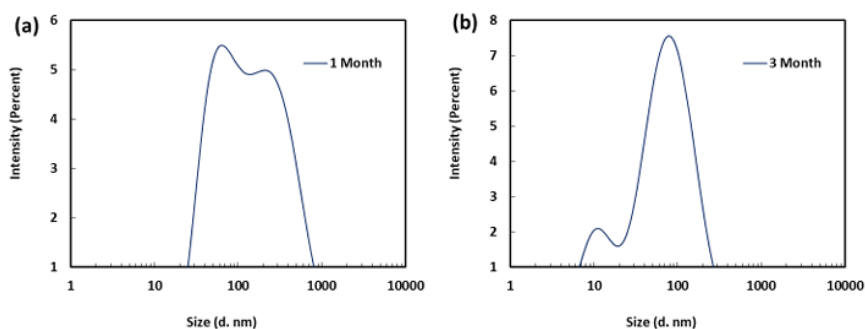


**Fig. 8.** Zeta potential of AgNPs water NF after 2 months of its preparation.

#### DLS analysis

Again, the diameter of the AgNPs in water was measured by DLS after 1 month (Fig. 9a) and 3 months (Fig. 9b) of its preparation. As is seen from Fig. 9, the AgNPs get

agglomerated followed by sedimentation with time increment. The size distribution ranges between several nm to five hundred nm. Of course, most of the particle size (84%) remained below 90 nm with an average of 45 nm as obtained from Fig. 9.



**Fig. 9.** Particle diameter of AgNPs water NF measured by DLS; (a) 1 month (b) 2 months after its preparation.

#### Conclusions

In this work, size-controlled spherical AgNPs were synthesized at different molar ratios of P: RA: CA=1:3:0, P:

RA: CA=1:3:3, P: RA: CA=1:3:6, P: RA: CA=1:3:9, and P: RA: CA=1:3:12 using the chemical reduction method, which is simple, fast, and cost-effective. XRD analysis

indicated the crystalline structure of AgNPs with an fcc structure the  $Fm\bar{3}m$  space group. SEM indicates the smooth surface, and TEM images revealed spherical AgNPs with a small particle with an average size of 35 nm. EDX elemental analysis confirmed the purity of AgNPs with a high percentage (~96 %) of silver. The stability of the synthesized AgNPs–water NFs showed a decrease with the time increment assessed by UV-vis, which was supported by the DLS measurement indicating the agglomerations of AgNPs in liquid media following the sedimentation. Also, prepared 2.0 vol% AgNPs–water NFs was stable at least for 2 weeks according to UV-vis analysis, which is a hope for many applications.

### Conflicts of Interest

There is no conflict to declare.

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