

## *Retraction*

# **Retracted: Role of Surfactant in the Formation of Gold Nanoparticles in Aqueous Medium**

### **Journal of Nanoparticles**

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The paper titled “Role of Surfactant in the Formation of Gold Nanoparticles in Aqueous Medium” [1], published in *Journal of Nanoparticles*, has been retracted as it was accepted for publication on the basis of peer review reports that were submitted from fraudulent reviewer accounts.

In late 2014, a number of publishers discovered widespread abuse of the peer review process, including cases of identity theft and faked review reports. In July 2015, Hindawi concluded an extensive investigation into peer review fraud and identified a number of articles that had been accepted on the basis of fraudulent peer review reports. In accordance with the recommendations of the Committee on Publication Ethics (COPE), Hindawi sent these manuscripts for re-review using independent Editorial Board Members. Following this re-review process, this article has been retracted as it was deemed unsuitable for publication.

### **References**

- [1] A. Das, R. Chadha, N. Maiti, and S. Kapoor, “Role of surfactant in the formation of gold nanoparticles in aqueous medium,” *Journal of Nanoparticles*, vol. 2014, Article ID 916429, 7 pages, 2014.

## Research Article

# Role of Surfactant in the Formation of Gold Nanoparticles in Aqueous Medium

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The stability of gold nanoparticles is a major issue which decides their impending usage in nanobiotechnological applications. Often biomimetically synthesized nanoparticles are deemed useless owing to their instability in aqueous medium. So, surfactants are used to stabilize the nanoparticles. But does the surfactant only stabilize by being adsorbed to the surface of the nanoparticles and not play significantly in moulding the size and shape of the nanoparticles? Keeping this idea in mind, gold nanoparticles (GNPs) synthesized by L-tryptophan (Trp) mediated reduction of chloroauric acid ( $\text{HAuCl}_4$ ) were stabilized by anionic surfactant, sodium dodecyl sulphate (SDS), and its effect on the moulding of size and properties of the GNPs was studied. Interestingly, unlike most of the gold nanoparticles synthesis mechanism showing saturation growth mechanism, inclusion of SDS in the reaction mixture for GNPs synthesis resulted in a bimodal mechanism which was studied by UV-Vis spectroscopy. The mechanism was further substantiated with transmission electron microscopy. Zeta potential of GNPs solutions was measured to corroborate stability observations recorded visually.

## 1. Introduction

Gold nanoparticles are important to bionanotechnology, owing to their easy synthesis, inert nature, good biocompatibility, and unique optical properties [1]. The tuning of optical properties of gold nanoparticles can be achieved by controlling the size of the nanoparticles [2]. Factors controlling the size of the gold nanoparticles are, namely, surface charge and double layer thickness [3]. Surface charge is again controlled by two criteria, nature and charge of ions adsorbed on the particles and surfactants used to stabilize the particles [4, 5]. Surfactants are used in the synthesis of gold nanoparticles when the reducing agent is not able to stabilize the particles [6]. The standard reducing agents which stabilize the gold nanoparticles in absence of any surfactant are sodium borohydride [7] and sodium citrate [8]. But for *in-vivo* applications these nanoparticles are of little use since the oxidised products of the reducing agents themselves show cytotoxicity [9, 10]. Thus, biomimetic routes of synthesis of gold nanoparticles are often implemented wherein a biomolecule is used as a reducing agent [11].

In recent years, many biomimetic synthetic [12–14] routes have been proposed to make the synthesized GNPs viable for bioapplication. For example, a variety of amino compounds have been explored as reducing agents which includes amino acids [15, 16], primary amines [17], peptides [18–22], and polymers [23]. Amino acids being the building blocks of proteins, which are ubiquitous in organisms, have collected a lot of interest to serve as a reducing agent. In some cases, amino acids have been also used as a capping agent to stabilize GNPs [24] since amino acids show strong interaction with gold nanoparticles [25]. Jacob et al. have shown that replacement of  $\text{BH}_4^-$  ion by Trp at silver nanoparticles surface resulted in particle aggregation [26]. Recently a simple, facile single-step technique for synthesis of gold nanowires and nanochains has been demonstrated using amino acids like histidine and glutamic acid [27]. Wangoo et al. have demonstrated the successful synthesis and capping of GNPs by glutamic acid [28]. Glutathione stabilized gold nanoparticles were found as an alternative to polyethylene glycol (PEG) stabilized GNPs that increase the stealth properties in *in vivo* studies for application in therapeutics [29]. It was also demonstrated

that tyrosine plays an important role in reduction of  $\text{Au}^{3+}$  to  $\text{Au}^0$  by a number of tyrosine containing peptides, but mostly limited to dipeptides and tripeptides [19]. Oligopeptides containing l-tryptophan at C-terminus are used by Si and Mandal to synthesize gold and silver nanoparticles at high pH [22]. Selvakannan et al. have reported the synthesis of lysine and tryptophan capped GNPs by spontaneous reduction of  $\text{HAuCl}_4$  or reduction by  $\text{NaBH}_4$  followed by capping with tryptophan [15, 30]. Iosin et al. have reported the synthesis of GNPs using tryptophan as reducing agent and have shown that with changing temperature the morphology of the GNPs synthesized changes from spherical to anisotropic shapes [31]. Tryptophan exists in our body as free tryptophan, dipeptides and in peptide chains [32]. Tryptophan is easily detectable by UV-Vis spectroscopy and fluorescence owing to the indole ring in its structure. Tryptophan is known to reduce gold nanoparticles [31], but it is inadequate in stabilizing the nanoparticles. Thus, in this study, the use of stabilizing agent becomes imperative. In this work, chloroauric acid ( $\text{HAuCl}_4$ ) is reduced by l-tryptophan (Trp) in presence of a stabilizing agent, sodium dodecyl sulphate (SDS).

## 2. Materials and Methods

**2.1. Chemicals.** Chloroauric acid (Sigma-Aldrich), l-tryptophan (Fluka AG), and sodium dodecyl sulphate (Sigma, USA) were used as received. All experiments were performed in Millipore purified water.

**2.2. Synthesis and Characterization of Au Nanoparticles.** Gold nanoparticles are produced in absence of any stabilizer by the reaction of chloroauric acid ( $\text{HAuCl}_4$ ) with l-tryptophan (Trp) mixed in different ratio. The UV-Vis spectra of the produced nanoparticles were then recorded in JASCO V-650 spectrophotometer. The same experiment was performed in presence of 1 mM sodium dodecyl sulphate (SDS). The stable gold sols were then characterised by scanning electron microscopy (SERON INC, South Korea, AIS 2100), energy dispersive X-ray spectroscopy (Oxford Instruments, UK, INCA E350), transmission electron microscopy (Zeiss, Libra 12), and zeta potential measurements (Zetasizer, Malvern, Nano-Z).

## 3. Results and Discussion

UV-Vis spectroscopy is an important tool for analysing formation of gold nanoparticles as gold nanoparticles have high molar extinction coefficient. The UV-visible absorption spectra of the fairly dilute dispersion of colloidal particles can be calculated from the "Mie theory" [5] and this technique serves as a powerful tool to characterize metal nanoparticles. The absorbance  $A$  of a colloidal solution containing  $N$  particles per unit volume is given by

$$A = \left( \frac{N\sigma_{\text{abs}}L}{\ln 10} \right), \quad (1)$$

where  $\sigma_{\text{abs}}$  (given by  $\sigma_{\text{abs}} = \sigma_{\text{ext}} - \sigma_{\text{scattering}}$ ) and  $L$  are absorption cross-section and optical path length, respectively.

So, it can be clearly understood that the magnitude of absorbance for resonant peak indicates the concentration of nanoparticles.

Again, the particle size determines the peak position. At nanoregime, the variation in surface to volume ratio with increasing size shows a gradually decreasing trend. As a result, on increasing size the fraction of surface free electrons decreases. So less energy is required to polarize them. Thus, red shift (shift to longer wavelength) is observed on increasing size. Moreover, plasmonic dipolar coupling leads to red shift of the plasmon band on agglomeration.

GNPs were synthesized by the reaction of  $\text{HAuCl}_4$  and Trp in absence of any stabilizing agent. UV-Vis spectrums of the GNP solutions were recorded, as shown in Figure 1. When 0.5 mM  $\text{HAuCl}_4$  reacted with 0.5 mM Trp (Figure 1(a)) red coloured GNPs which showed maximum absorbance at 577 nm ( $\lambda_{\text{max}}$ ) were formed. When 0.5 mM  $\text{HAuCl}_4$  reacted with 1 mM Trp (Figure 1(b)), red coloured GNPs were formed with  $\lambda_{\text{max}}$  at 569 nm. GNPs formed by the reaction of 1 mM  $\text{HAuCl}_4$  and 0.5 mM Trp (Figure 1(c)) showed  $\lambda_{\text{max}}$  at 530 nm. GNPs formed by the reaction of 1 mM  $\text{HAuCl}_4$  and 1 mM Trp showed absorption maxima ( $\lambda_{\text{max}}$ ) at 570 nm, as shown in Figure 1(d). All the above GNP solutions precipitated within 1 hour of formation of gold nanoparticles. In all the above GNPs solutions,  $\lambda_{\text{max}}$  ranges from 530 nm to 577 nm.

Another set of similar reactions where the ratios of  $\text{HAuCl}_4$  and Trp were the same as above, was carried out in presence of 1 mM SDS as stabilizer. The UV-Vis spectrums of the gold nanoparticles solutions were recorded as shown in Figure 2(a–d).  $\lambda_{\text{max}}$  ranges from 533 nm to 543 nm. The narrow range of  $\lambda_{\text{max}}$  suggests that surfactant is controlling the shape and size of the GNPs formed. In reaction set where the ratio of  $\text{HAuCl}_4$  to Trp was 2 : 1, GNPs were found to precipitate (Figure 2(c)). This can be attributed to the presence of excess of  $\text{HAuCl}_4$  as compared to Trp, which reduced the double layer thickness, thereby facilitating faster agglomeration. In this set of reactions performed in presence of SDS, it was also observed that the surface plasmon peaks were quite sharper than that of the solution sets prepared in absence of SDS. This indicates that SDS is able to stabilize gold sol quite efficiently.

Transmission electron microscope images of GNPs formed in the above four reaction sets prepared in presence of 1 mM SDS were recorded as shown in Figures 3(a)–3(d). GNPs shown in Figures 3(a), 3(b), and 3(d) are approximately 20 nm in size. GNPs shown in Figure 3(c) show a size range of about 50–100 nm which indicates high agglomeration. Scanning electron microscope (SEM) image of GNPs synthesized by the reaction of 1 mM  $\text{HAuCl}_4$  and 1 mM Trp in presence of 1 mM SDS and drop casted on silica substrate revealed that the nanoparticles are well dispersed, as shown in Figure S1(a) (see the Supplementary Material available online at <http://dx.doi.org/10.1155/2014/916429>). Moreover, energy dispersive X-ray spectroscopy (EDX) of the above GNPs confirms the presence of gold in the sol as shown in Figure S1(b).

Zeta potential measurements were carried out in an electrophoretic cell to determine the long term stability and

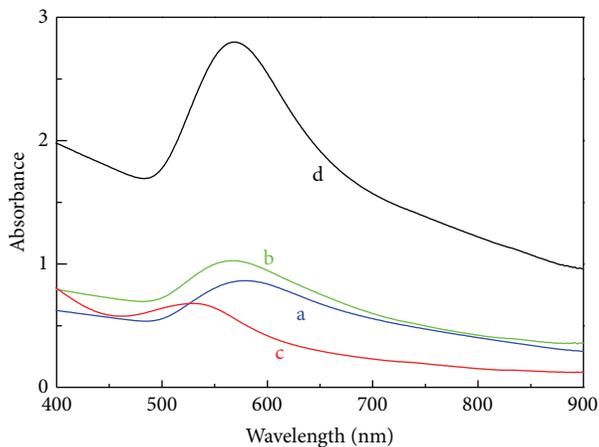


FIGURE 1: UV-Vis spectrum of gold nanoparticles in absence of any stabilizer in aqueous medium formed by the reaction of 0.5 mM HAuCl<sub>4</sub> with (a) 0.5 mM l-Trp and (b) 1 mM Trp and that of 1 mM HAuCl<sub>4</sub> with (c) 0.5 mM l-Trp and (d) 1 mM Trp.

to provide a basis to the earlier mentioned visual observations about the stability of different gold nanoparticles solutions. Zeta potential values recorded after formation of GNPs shown in Figures 3(a), 3(b), 3(c), and 3(d) prepared in SDS solution are  $-41.4$  mV,  $-47.7$  mV,  $-28.9$  mV, and  $-37.3$  mV, respectively. This clearly corroborates earlier observation that out of these four solutions, GNPs shown in Figure 3(c) are not stable in solution. Moreover the above negative zeta potential values also show that the colloids have a net negative charge on them. The reason behind the negative charge may be due to adsorption of negatively charged SDS molecules on the surface.

Studying of the kinetics involves plotting of UV-Vis  $\lambda_{\max}$  absorption value of GNPs with time from the beginning of the reaction. The reaction of  $5 \times 10^{-4}$  M Trp with  $1 \times 10^{-3}$  M HAuCl<sub>4</sub> shows saturation growth type of kinetics. An absorbance *versus* time spectra of the reaction, recorded at 530 nm ( $\lambda_{\max}$  for the GNPs synthesized), is shown in Figure 4. The above observation shows that Au<sup>3+</sup> and Trp show a simple reaction mechanism where GNPs of only one size distribution are formed. It has also been observed that the trace is concentration dependent.

All the solution sets of HAuCl<sub>4</sub> and Trp in presence of SDS were subjected to fixed wavelength UV-Vis absorption at their respective  $\lambda_{\max}$  to study their growth with time. GNPs formed by the reaction of 0.5 mM HAuCl<sub>4</sub> and 0.5 mM Trp in 1 mM SDS had very low final absorbance as shown in Figure 2(a), which made them unsuitable for study of mechanism of growth of nanoparticles. The same case was with the GNPs formed by the reaction of 0.5 mM HAuCl<sub>4</sub> with 1 mM Trp in presence of 1 mM SDS as shown in Figure 2(b). Again GNPs formed by the reaction of 1 mM HAuCl<sub>4</sub> with 0.5 mM Trp in presence of 1 mM SDS were rendered unsuitable since the particles precipitated within the time of study. Only the GNP synthesis by the reaction of 1 mM HAuCl<sub>4</sub> and 1 mM Trp in presence of 1 mM SDS was the most suitable

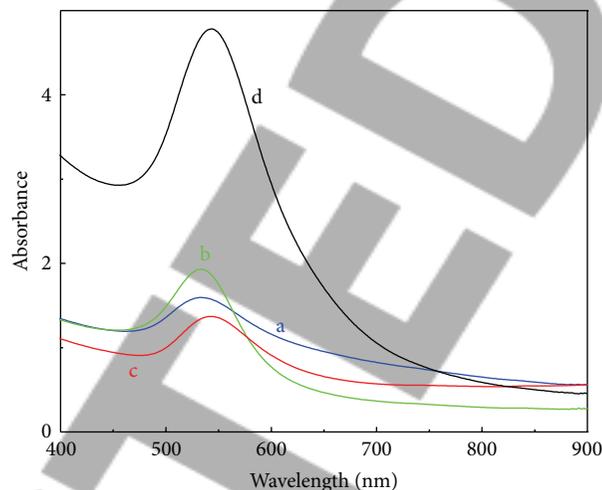


FIGURE 2: UV-Vis spectrum of gold nanoparticles in 1 mM SDS solution formed by the reaction of 0.5 mM HAuCl<sub>4</sub> with (a) 0.5 mM Trp and (b) 1 mM Trp and that of 1 mM HAuCl<sub>4</sub> with (c) 0.5 mM Trp and (d) 1 mM Trp.

candidate for fixed wavelength UV-Vis absorption study. UV-Vis absorption with time of the above solution was recorded at 543 nm as shown in Figure 5.

The absorbance versus time plot (Figure 5) is divided into five regions, namely, A, B, C, D, and E. At the end of region A, GNPs first start appearing 509 nm. In region B, the peak remains in the range from 509 to 512 nm. A drastic peak shift from 512 nm to 543 nm is observed in region C within a short span of 22 minutes. In region D, the peak remains almost at the same position (543 to 545 nm). In region E, a slight shift of peak is seen from 543 nm to 541 nm. Then onwards the peak remains at 541 nm. This phenomenon is also observed in case reaction of 0.5 mM HAuCl<sub>4</sub> with 0.5 mM Trp as well as 1 mM Trp, but because of low final absorbance and taking the sensitivity of the UV-Vis measurement into consideration, these reactions were not studied to determine the mechanism of reaction. The UV-Vis spectrum of the above solution at different times is also recorded as shown in Figure 6.

In region A (Figure 5), it seems that smaller GNPs are forming by the reduction of HAuCl<sub>4</sub> by Trp. In region B (Figure 5), the reduction process seems to have achieved saturation. In region C (Figure 5), the drastic shift can be attributed to bimolecular agglomeration and autocatalytic agglomeration as there is an observable shift in  $\lambda_{\max}$  value. In region D (Figure 5), the almost linear and saturation type of growth at a fixed  $\lambda_{\max}$  can be attributed to predominance of autocatalytic agglomeration mechanism. In region E (Figure 5), the small shift in  $\lambda_{\max}$  may be attributed to adsorption of surfactant molecules on the GNPs. In the above reaction, bimodal size distribution is observed during the course of the reaction. This reaction can be said to follow a bimodal mechanism.

Finney et al. [33] and Sabir et al. [34] have observed similar mechanisms to occur during synthesis of GNPs by different routes. A proposed scheme of bimodal mechanism is shown in Scheme 1.

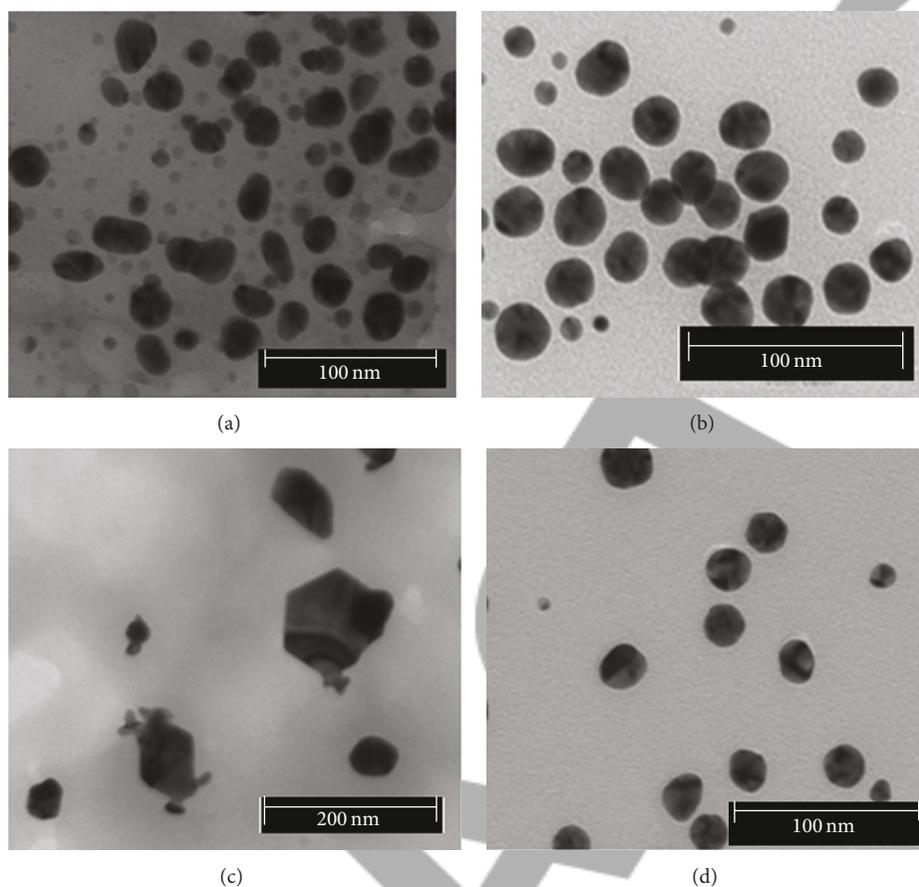


FIGURE 3: Transmission electron microscope image of gold nanoparticles in 1 mM SDS solution formed by the reaction of 0.5 mM  $\text{H}_2\text{AuCl}_4$  with (a) 0.5 mM Trp and (b) 1 mM Trp and that of 1 mM  $\text{H}_2\text{AuCl}_4$  with (c) 0.5 mM Trp and (d) 1 mM Trp.

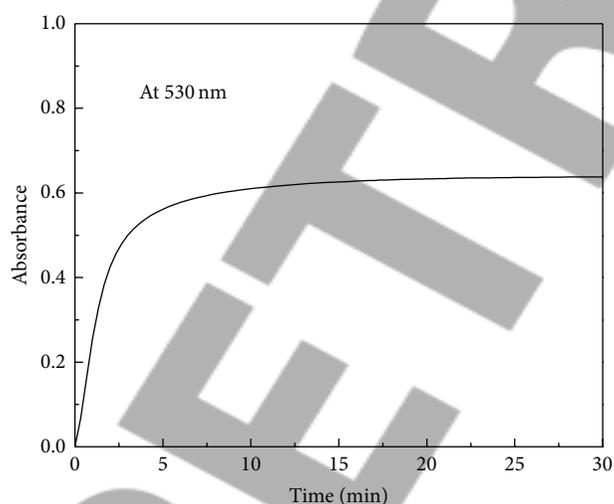


FIGURE 4: UV-Vis spectrum recorded at 530 nm with time (in minutes) for the reaction of 0.5 mM Trp with 1 mM  $\text{H}_2\text{AuCl}_4$  in absence of any stabilizer.

First two steps, namely, nucleation and autocatalytic growth, are difficult to characterize by optical method as magnitude of  $\sigma_{\text{abs}}$  is very low for small GNPs (here B

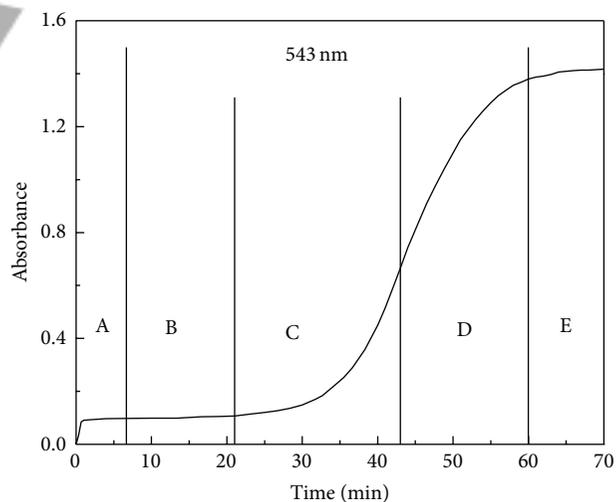
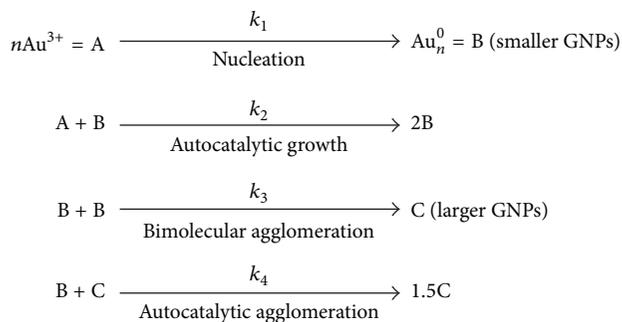


FIGURE 5: UV-Vis spectrum recorded at 543 nm with time (in minutes) for the reaction of 1 mM Trp with 1 mM  $\text{H}_2\text{AuCl}_4$  in 1 mM SDS solution.

is assumed to be small GNPs). Here it is assumed that, before the commencement of bimolecular agglomeration and autocatalytic agglomeration step, the first two elementary



SCHEME 1: Schematic representation of bimodal mechanism of formation of gold nanoparticles.

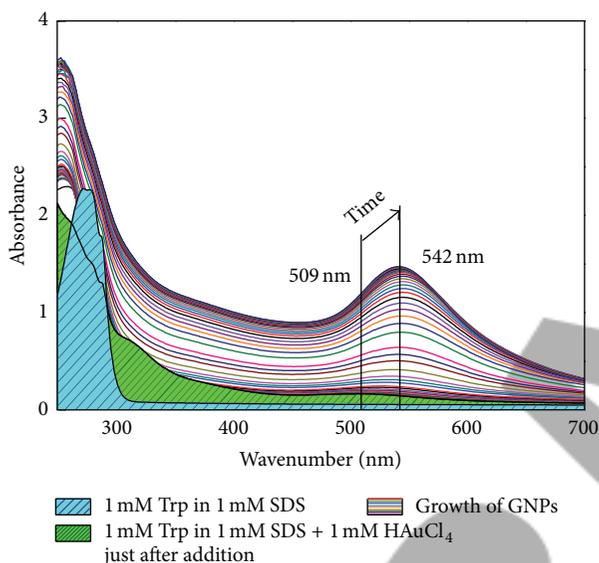


FIGURE 6: UV-Vis spectrums recorded during the reaction of 1 mM Trp with 1 mM HAuCl<sub>4</sub> in 1 mM SDS solution.

reactions involving reduction of Au<sup>3+</sup> ions, are complete. But as the size of the GNPs falls within the range of 5–20 nm, optical measurements become quite reliable.

It can be seen that the variation in wavelength in region B is very less. It reflects that though bimolecular agglomeration has started but the rate is not that high. The rate law for the last two steps, namely, bimolecular agglomeration and autocatalytic agglomeration, may be given as follows:

$$\begin{aligned}
 -\frac{d[\text{B}]}{dt} &= 2k_3[\text{B}]^2 + k_4[\text{B}][\text{C}] \\
 \therefore 2[\text{C}] &= [\text{B}]_0 - [\text{B}], \quad \text{we get} \\
 \frac{d[\text{C}]}{dt} &= k_3[\text{B}]^2 + \frac{1}{2}k_4[\text{B}][\text{C}].
 \end{aligned} \quad (2)$$

In region B, as the number density of larger GNPs, that is [C], is very low, (2) reduces to  $d[\text{C}]/dt = k_3[\text{B}]^2$ , and solving it we get

$$[\text{C}] = \frac{[\text{B}]_0}{2} \left\{ 1 - \frac{1}{2k_3[\text{B}]_0 t + 1} \right\}. \quad (3)$$

Thus with increasing time, number density of C increases. Again as absorbance is directly proportional to the number density, absorbance also increases.

In region C, there is a drastic increase in the wavelength as well as absorbance. This indicates that the formation of C has increased and as a result the size distribution shifts toward the higher wavelength. Now the rate equation can be given by

$$\frac{d[\text{C}]}{dt} = \frac{1}{2}k_4[\text{B}][\text{C}]. \quad (4)$$

Here the basic assumption is that formation of B has reached saturation.

In region D, trace resembles a saturation growth type of profile. In this region, the change in wavelength is very less. It may be said that, in this region, the predominant process occurring is autocatalytic agglomeration.

This bimodal mechanism of formation of GNPs is further confirmed by transmission electron microscopy by recording the image of the GNPs formed after 5 minutes of initiation of reaction, that is, in the plateau region shown in Figure 7. The TEM image shows that the particles are roughly 10 nm in size, whereas the particles formed after the completion of the reaction are about 20 nm in size

It is assumed that UV-Vis absorbance value is proportional to the concentration and approximate concentration of gold nanoparticles determined by taking the approximate size of gold nanoparticles as obtained from TEM image shown in Figures 3(d) and 7. With the above assumptions, the curve shown in Figure 5 is fitted with (3) and (4). It was found that the bimolecular aggregation step has initiated just after the onset of reduction step and the autocatalytic agglomeration has started at around 28 minutes from the onset of reaction as shown in Figure 8.

## 4. Conclusion

L-tryptophan reduced gold nanoparticles are stabilized in aqueous solution by SDS. Results revealed that SDS plays an important role in the formation mechanism of the gold nanoparticles. Moreover, the reaction kinetics of GNPs in this case is easily observable by a steady-state spectroscopic method such as UV-Vis spectroscopy unlike NaBH<sub>4</sub> where the reaction is too fast and citrate method where heating is required to initiate the reaction. The kinetic details of the formation processes of GNPs reveal the bimodal mechanism. The observation of formation of gold nanoparticles of two different sizes, spaced over a time gap of about 30 minutes during the process of synthesis, opens up the prospect of utilizing the same reaction to tune nanoparticles size. The GNPs prepared by this method are quite stable, which suggests that their usage in biological application can be safely extrapolated in future. This study guides our attention towards the potential usage of anionic surfactants in tuning the size of GNPs along with their stabilization.

## Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

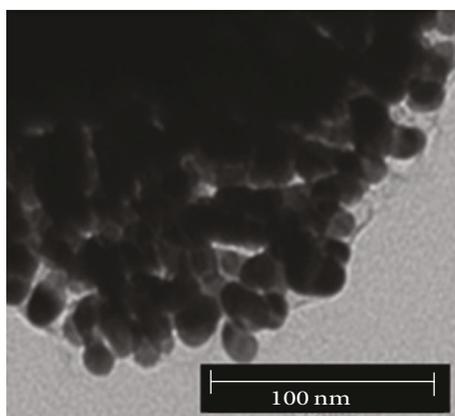


FIGURE 7: Transmission electron microscope image of GNPs formed after 5 minutes of initiation of reaction between 1 mM  $\text{HAuCl}_4$  and 1 mM Trp in presence of 1 mM SDS.

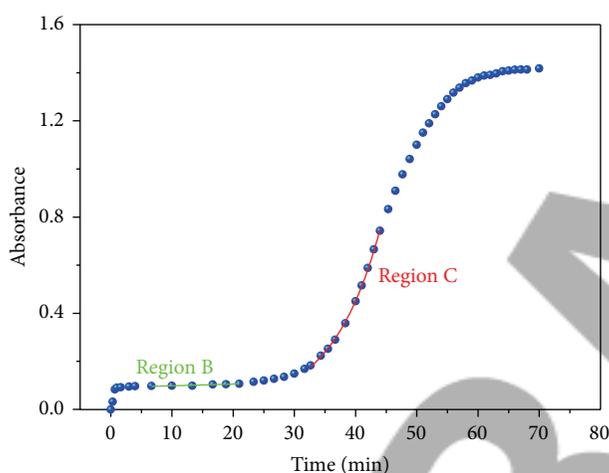


FIGURE 8: UV-Vis spectrum recorded at 543 nm with time (in minutes) for the reaction of 1 mM Trp with 1 mM  $\text{HAuCl}_4$  in 1 mM SDS solution, showing fitted curve using (3) and (4) for the regions B and C as mentioned in Figure 5.

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