



**COMPILED & CIRCULATED BY**  
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*Topic:*

**Nano Materials and Applications (Lab)**

1. Synthesis of Metal Nanoparticles by Chemical Route.
2. Synthesis of Semiconductor Nanoparticles.

**Nano Materials and Applications (Lab):**

**1. Synthesis of Metal Nanoparticles by Chemical Route:**

**(i) Synthesis of Silver (Ag) Nanoparticles by Chemical Route:**

**Experimental (Synthesis):**

The samples, silver nitrate ( $\text{AgNO}_3$ , analytical grade) were purchased from Sigma Aldrich Chemical Co. (U.S.A.). Triple distilled water, from Merck was used for sample preparation.

The Silver (Ag) nanoparticles (NPs) were synthesized with following a previously published protocol<sup>1</sup>. More briefly,  $\text{AgNO}_3$  (99.99%), and the reducing agent, sodium borohydride ( $\text{NaBH}_4$ , 99.9%) were used for preparation of bare Ag NPs. At first, 25 mL  $\text{AgNO}_3$  solution (10.0 mM) was added drop wise into 35 mL of sodium borohydride solution (25.0 mM) which had been chilled in an ice bath, before used. Then, the reaction mixture was stirred vigorously by magnetic stirrer in the laboratory. Thereafter, the solution turned into light yellow after the addition of 2 mL of  $\text{AgNO}_3$ , as well as the brighter yellow when all of the residual  $\text{AgNO}_3$  was added into the container. The entire addition took ~10 minutes, after which the stirring was stopped. Finally, the clear yellow colloidal silver was stable at room temperature as well as stored in a transparent vial.

**Experimental (Characterization):**

The freshly prepared clear yellow colloidal silver NPs was stable at room temperature as well as stored in a transparent vial. Fig. 1(a) shows the prepared.

The optical absorption spectra (Fig. 1(b)) of the above mentioned as prepared samples were recorded in a Shimadzu-Pharmaspec-1700 UV-VIS spectrophotometer. The UV-vis absorption spectrum of bare Ag NPs shows surface plasmon absorption peak at ~395 nm (Fig 1(b)). Bhunia et al also observed absorption peak of Ag NPs at ~390 nm.



Figure: 1(a)

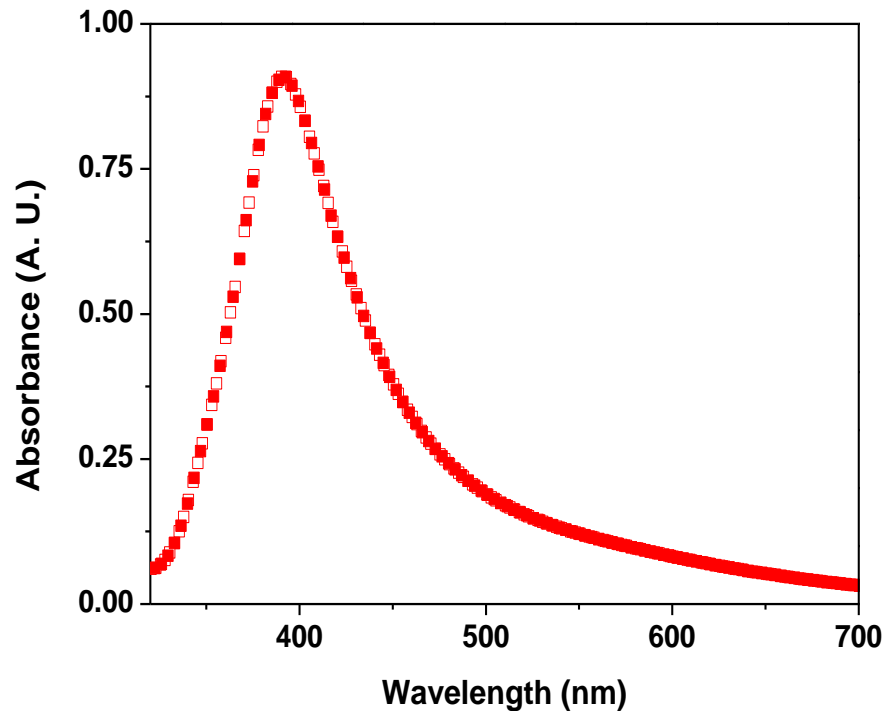


Figure: 1(b)

### (ii) Synthesis of Gold (Au) Nanoparticles by Chemical Route:

#### Experimental (Synthesis):

Here we need Hydrochloroauric acid tri hydrate ( $\text{HAuCl}_4 \cdot 3\text{H}_2\text{O}$ , 99%), tri sodium citrate ( $\text{C}_6\text{H}_5\text{Na}_3\text{O}_7 \cdot 2\text{H}_2\text{O}$ , >99%, TSC) as well as NaOH. Deionized water from Merck was used as solvent. Colloidal gold (Au) nanoparticles (NPs) suspension is prepared by citrate reduction method introduced by Turkevich at reaction temperature  $80^\circ\text{C}$  by previously published protocol<sup>3</sup>. Generally, 25 mg  $\text{HAuCl}_4 \cdot 3\text{H}_2\text{O}$  is taken in a 50 ml of de-ionised water in a conical flask kept in the water bath. To avoid any loss of the solution by evaporation, the flask is fitted with a vertical condenser. At first, a light yellow transparent solution was obtained. Then, the solution is heated up to  $80^\circ\text{C}$  by increasing the temperature of the bath. 1.5 mL tri-sodium citrate dihydrate solution, containing 50 mg of trisodium citrate per mL is added to the heated Au (III) chloride solution under vigorous stirring through the side neck of the flask that was kept closed otherwise. Within several minutes the colour is changed first to yellowish black to black as well as finally to deep wine red. This confirmed the synthesis of Au NPs.

#### Experimental (Characterization):



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The freshly prepared wine red colloidal gold NPs was stable at room temperature as well as stored in a transparent vial. Fig. 2(a) shows the prepared.

The optical absorption spectra (Fig. 2(b)) of the above mentioned as prepared samples were recorded in a Shimadzu-Pharmaspec-1700 UV-VIS spectrophotometer.

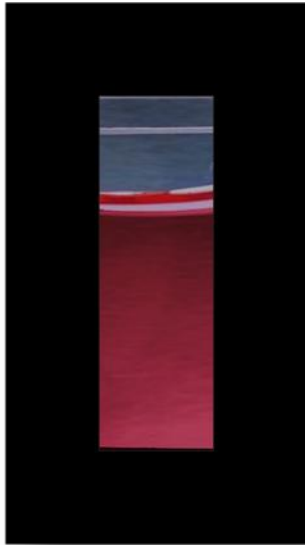


Figure: 2(a)

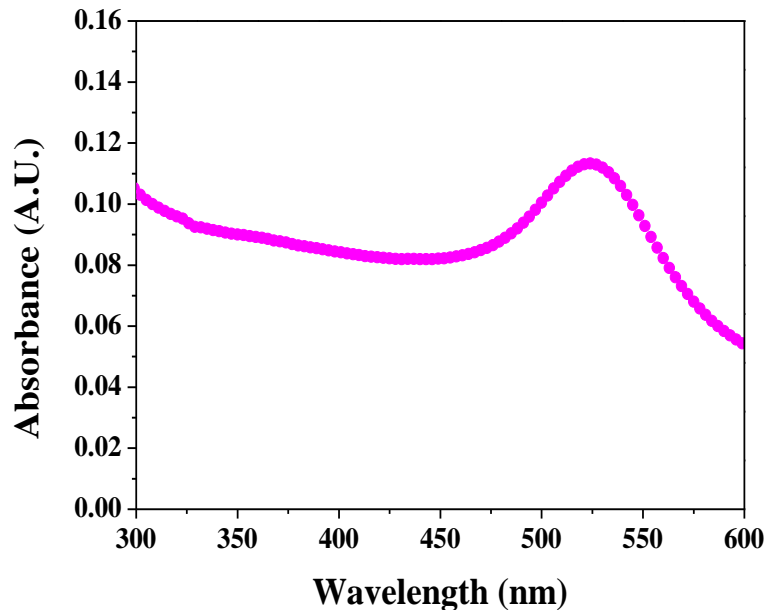


Figure: 2(b)

The UV-vis absorption spectrum of colloidal Au NPs shows surface plasmon absorption peak at  $\sim 530$  nm (Fig 2(b)). Aich et al also observed absorption peak of Au NPs at  $\sim 524$  nm.

## 2. Synthesis of Semiconductor Nanoparticles:

### (i) Synthesis of Zinc Oxide (ZnO) Nanoparticles by Chemical Route:

ZnO NPs were synthesized using a simple wet chemical method as reported elsewhere<sup>3</sup> with minor modification. The reagents used for fabricating ZnO NPs were of analytical grade (MERCK, 99.99% pure). Triple distilled water, from Merck was used for sample preparation. Under constant stirring 0.7 M Zinc Nitrate solution NaOH solution (1M) was added drop-wise for 5 min and the stirring was continued further for 40 min. A white precipitate was deposited at the bottom of the flask. The precipitate was filtered and washed 2-3 times with Millipore water for removal of any residual salts and dried in a furnace. The so prepared ZnO NPs were dispersed in Millipore water using ultrasonication for 20 minutes.



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### Experimental (Characterization):

The freshly prepared wine red colloidal gold NPs was stable at room temperature as well as stored in a transparent vial. Fig. 3(a) shows the prepared.

The optical absorption spectra (Fig. 3(b)) of the above mentioned as prepared samples were recorded in a Shimadzu-Pharmaspec-1700 UV-VIS spectrophotometer



Figure: 3(a)

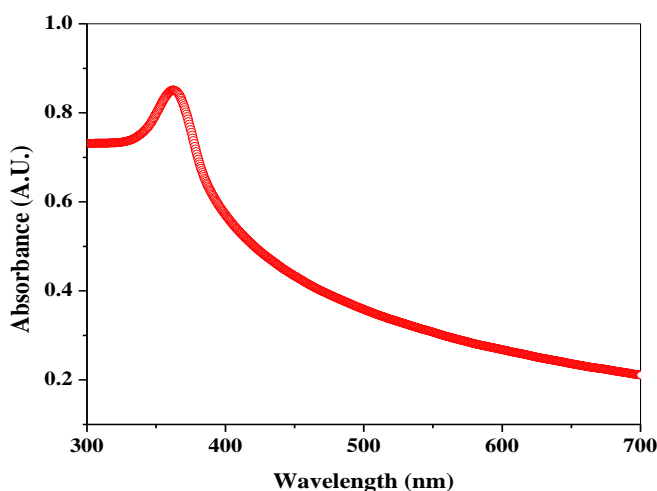


Figure: 3(b)

The UV-vis absorption spectrum of bare ZnO NPs shows surface plasmon absorption peak at  $\sim 360$  nm (Fig 3(b)). Samanta et al also observed absorption peak of ZnO NPs at  $\sim 360$  nm

### References:

1. AK Bhunia, PK Samanta, D Aich, S Saha, T Kamilya, *Journal of Physics D: Applied Physics* 48 (23), 235305.
2. D Aich, S Saha, RN Mondal, T Kamilya, *NANO, Nano, Vol. 15, No. 01, 2050008* (2020).
3. AK Bhunia, PK Samanta, S Saha, T Kamilya, *Applied Physics Letters* 103 (14), 143701.

### Link to Audio visual Lectures (e-Lectures) on this topic given by Distinguish Professors of Indian & Foreign Universities:

- I. <https://www.youtube.com/watch?v=L-GwpAJ9MJ0>
- II. <https://www.youtube.com/watch?v=H78KYT4pQhg>
- III. <https://www.youtube.com/watch?v=urmi99jQSZY>
- IV. <https://www.youtube.com/watch?v=1vr4OKIGwPA>
- V. <https://www.youtube.com/watch?v=1ttlw0HRYYo>