MORPHOLOGICAL STUDY OF SILVER NANO PARTICLES  
BY USING Titron-X-100 [SURFACTANT]

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ABSTRACT: 
The present work aims at synthesizing silver Nanoparticles experimentally on laboratory scale by micro-emulsion technique. Silver nano particles were prepared by reducing silver ions in the hexagonal phase formed by TitronX-100 in aqueous solution. A TitronX-100 molecule helps to reduce the silver ions into silver atoms. The hexagonal phase hindered the growth and aggregation of the particles. DLS data gave narrow size distribution ranging from 20 nm to 200 nm and its morphological study has been confirmed by TEM. At the initial stage of reaction, the silver particles prepared in the hexagonal phase exhibited a size of 20 nm. As the reaction proceeded, the particles grew up to above 200nm as determined by Transmission Electron Microscopy (TEM). With the TEM, it was confirmed that surfactant aggregates, which have flexible structures, could not absolutely prevent particle from growing and aggregating.

KEY WORDS: Silver nano particles, Titron-X-100, DLS, TEM

1. INTRODUCTION TO NANOTECHNOLOGY:  
Nanotechnology is the study and design of systems at Nanometer scale \( 0.000000001 \) meter the scale of atoms and molecules. Nanomaterials are different than other materials. Every chemical element has characteristics, definite properties; like color, hardness, elasticity, conductivity, melting temperature etc. Nanoparticles which are less than 100nm in diameter are much more active than large particles because, a given weight of nanoparticles, have much higher surface area than the same weight of larger particles. It is this enormous increase in the surface area of finely divided nanoparticles that can change relatively inert substances into highly reactive one. The material can then melt faster, absorb more or simply become explosive.

1.1 Silver Nanoparticles: - Amongst all noble metals silver Nanoparticles is one of the Nanomaterials, which exhibit unusual optical, electronic & chemical properties that depend on size & shape. Silver exhibits antimicrobial or bactericidal properties of which it has been used in a number of textiles, plastics and ceramics.

1.2 Scope and Future for Nanotechnology:-
Considering the scope and future of silver Nanoparticles ,the present work aims in synthesizing silver Nanoparticles and to study its morphology, using sophisticated analytical instruments.

2. EXPERIMENTAL DETAILS
2.1 Materials
Analytical grade of Silver nitrate, Sodium borohydride, Cyclohexane, N-hexane, and TitronX-100 were procured from reputed firm for the present research.

2.2 Method
2.2.1 Preparation of precursor & reducing agent 
The various experimental runs were conducted by changing amounts and concentrations of silver nitrate, solvent and surfactant as listed in the tables-1.  

2.2.2 Preparation of micro emulsion I:  
Micro emulsion-I was prepared by taking prescribed amount of solvent cyclohexane to which co-surfactant, n-Hexanol was added and kept stirred at constant speed using magnetic stirrer. Surfactant; Triton X-100; was added drop wise during the continuous stirring. The precursor solution \( \text{Ag NO}_3 \) [0.01M] was then added into the flask.

2.2.3 Preparation of micro emulsion II:  
Micro emulsion-II was prepared with a prescribed amount of Cyclohexane with constant stirring using magnetic stirrer, Triton X-100) was then poured slowly, Solution containing the reducing agent 0.1M Sodium borohydride was then mixed into the flask.

2.3 Sample Preparation
After making of microemulsion I & II, microemulsion II was poured into microemulsion I by keeping the solution stirring at constant speed and the temperature of system is maintained up to 20°C. Due to this the colour of the solution changes from the transparent to brown which indicates the formation of silver ions.

Table 1: Composition of samples ‘S’ series:

<table>
<thead>
<tr>
<th>Sample composition code</th>
<th>Sample composition code</th>
<th>Microemulsion I</th>
<th>Microemulsion II</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Cyclohexane</td>
<td>Triton X-100</td>
<td>AgNO3</td>
</tr>
<tr>
<td>S1</td>
<td>35gm</td>
<td>7.5gm</td>
<td>6gm</td>
</tr>
<tr>
<td>S2</td>
<td>35gm</td>
<td>7.0gm</td>
<td>7gm</td>
</tr>
<tr>
<td>S3</td>
<td>35gm</td>
<td>6.0gm</td>
<td>8gm</td>
</tr>
<tr>
<td>S4</td>
<td>35gm</td>
<td>7.5gm</td>
<td>6gm</td>
</tr>
<tr>
<td>S5</td>
<td>35gm</td>
<td>7.0gm</td>
<td>7gm</td>
</tr>
<tr>
<td>S6</td>
<td>35gm</td>
<td>6.0gm</td>
<td>8gm</td>
</tr>
</tbody>
</table>

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The PDI indicates the quality of dispersion from the values lower than 0.1 are suitable for measurement and are supposed to be good quality colloidal suspensions, whereas the values close to one are poor quality samples, in which droplet sizes are not in the colloidal range or they exhibit a very high droplet size.

The observations/results obtained by analyzing on DLS are illustrated in observation table

Table 2: Observation for ‘S’ series on DLS:

<table>
<thead>
<tr>
<th>Sample Composition Code</th>
<th>Effective Dia. nm</th>
<th>Polydispersity</th>
<th>Average count Rate</th>
<th>Base Line Index</th>
</tr>
</thead>
<tbody>
<tr>
<td>S 1</td>
<td>163.5</td>
<td>0.123</td>
<td>355.4</td>
<td>10.0</td>
</tr>
<tr>
<td>S 2</td>
<td>146.8</td>
<td>0.217</td>
<td>374.8</td>
<td>8.0</td>
</tr>
<tr>
<td>S 3</td>
<td>193.8</td>
<td>0.005</td>
<td>414.9</td>
<td>10.0</td>
</tr>
<tr>
<td>S 4</td>
<td>261.2</td>
<td>0.234</td>
<td>419.7</td>
<td>6.3</td>
</tr>
<tr>
<td>S 5</td>
<td>149.1</td>
<td>0.005</td>
<td>409.9</td>
<td>4.0</td>
</tr>
<tr>
<td>S 6</td>
<td>181.1</td>
<td>0.011</td>
<td>518.4</td>
<td>4.5</td>
</tr>
</tbody>
</table>

Analysis by TEM:
The TEM patterns were obtained by employing “PHILLIPS 200 TEM” operated at 200kv voltage. Samples were prepared for TEM analysis by placing a drop of the solution on a carbon coated copper grid and then drying under electric bulb for half hour. Average particle size

Table 3: - TEM Observation for S series

<table>
<thead>
<tr>
<th>Sample Composition Code (Image)</th>
<th>Average particle size of silver (nm)</th>
<th>Morphology</th>
</tr>
</thead>
<tbody>
<tr>
<td>S 1</td>
<td>200</td>
<td>Spherical</td>
</tr>
<tr>
<td>S 2</td>
<td>20</td>
<td>Spherical</td>
</tr>
<tr>
<td>S 5</td>
<td>100-500</td>
<td>Spherical</td>
</tr>
</tbody>
</table>

Fig 1: S1

Fig 2: S1

Fig 3: S2

Fig 4: S5

COMMENTS ON THE RESULT

The size and morphology of silver particles was confirmed by TEM analysis. The figures 1, 2, 3, 4 show photographs of TEM analysis of silver particles synthesized by two step micro emulsion (RM) for S series. According to table 1 of composition of sample S series, the amount of n-Hexanol was reduced in these series of experiments, the concentration of AgNO₃ was 0.02 M, and NaBH₄ 0.2M for S1, S2, S3 and for S4, S5, S6 the concentration of AgNO₃ was 0.03M and that of NaBH₄ was 0.3M. As can be seen from the observation table, the increase in the concentration has a detrimental effect in synthesizing Ag Nanoparticles. As the concentration was increased there was increase in particle size figure NO 1, 2, 3, 4 shows Nanoparticle size as 163nm for S1, as 146nm for S2, 261nm for S4 & 149 nm for S5. For S1 the average size ranges from 62nm to 66nm that are spherical in shape.

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