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Synthesis of Transition Metal (Ag.) Nanoparticles in a Continuous Flow Spiral Micro Reactor

S. Sharada*[a], T. Bala Narsaiah[a] and Shirish Sonawane[b]

[b]*National Institute of Technology, Warangal, Telangana, India- 506004*
[*a,a]*Jawaharlal Nehru Technological University Anantapur, College of Engineering, India- 515002*

Abstract—*The work describes about the synthesis of Transition metal silver nanoparticles was done from silver nitrate in a continuous flow microreactor. For the formation of nanoparticles Sodium borohydride, as a reducing agent and surfactant sodium dodecyl sulphate was used . Reduction reaction was carried out using silver nitrate and sodium borohydride. The experiment was done with different molar concentrations and varying surfactant loadings. The obtaines samples were characterized by UV absorption at definite intervals of time. Results show that use of 0.02 g/ml SDS with 1 ml/min (0.001 M) $AgNO_3$ and 3 ml/min (0.003 M) sodium borohydride flow rate shows minimum particle size of 4.8 nm. The flow pattern was determined by calculating the Reynolds number .The amount of time that the reaction is heated and cooled was calculated from the volume of the reactor and the flow rate which was achieved by calculating the residence time.*

Keywords: *Micro reactor, Silver nanoparticles, Reduction reaction, Particle size, Residence time.*

1. INTRODUCTION:

Microreactors which is a new concept for chemical synthesis and technological feasibility**[1]**. Control of crystal structure is very important as it is influenced by the physical and chemical properties of nanoparticles. The use of microfluidic reactors represents a bottom-up approach. By the novel method, hence the particle size, size distribution and physical as well as biological properties can be maintained under low temperature conditions. Hence from various microreactors, segmented flow or droplet-based approach proved the best due to the encapsulation of nanoparticles**[2]** .The reaction is suitable for continuous flow reactors because of the moderate temperature for the formation of silver nanoparticles. The produced silver nanoparticles observed was narrow size distribution. Temperature profiles of the reactant fluids obtained represents the formation of silver nanoparticles in the microreactor**[3].** synthesis of ZnO nanoparticles was done by using Zinc Nitrate $Zn(No_3)_2$ and Ammonium Carbonate $(NH_4)_2CO_3$**[4].** Experimental parameters were optimized to obtain narrow size distributions, which were at average two times narrower than those obtained in a conventional synthesis**[5]** .The optical properties of the colloidal product solutions depend both on the mixing order of the reactant solutions and on the over-all flow rates. The quality of colloidal solutions of gold/silver nanoparticles can be monitored by theoptical absorption**[6].** The continuous flow synthesis of silver nanoparticles was carried out in a stainless steel helical coil and also in a spiral polymericminichannel reactor**[7].**Low investment costs of a micro reactor plant, higher production amount, lower labour costs efficient heat control and small reactor volume makes for sustainable production of silver nanoparticles**[8]**.Increased mixing and higher Ba2+ ion concentrations in the reactor yields smaller particles in the product**[9].** Lab-on-a-chip technology allows for rapid, cost-effective, and environmentally friendly prototyping that will accelerate the rational development and production of nanostructures[**10**].The continuous formation of particles in a microfluidic system allows for dynamic control of flow and mixing parameters[**11**]. The microreactor synthesis is very fast and improves the monodispersity with excellent reproducibility[**12**]. At short reaction times, growth and aggregation are observed, while at longer times the particles are completely stable[**13**]. The order of addition as well as the concentration ratio of the reactants did also influence the particle size distribution [**14**]. A stable interface between two insoluble currents in a microchannel reactor has been obtained by selecting the solvents and adjusting the flow rate **[15].** The silver nanoparticle is one of the inorganic nano materials which is a good antimicrobial agents.[**16**]. The silver nanoparticle is one of the inorganic nano materials which is a good antimicrobial agents**[17].**

2. EXPERIMENTAL

2.1. Materials

Silver nitrate ($AgNO_3$, 99%) of analytical grade, Analytical grade sodium borohydride (99%, $NaBH_4$) and Sodium dodecyl sulphate (99% purity) was procured from SLScientific Chemicals, Anantapur. Millipore deionised water was used for preparation of all solutions used in the preparation for the experimental procedure.

2.2. Synthesis of silver nanoparticles in a Continuous flow microreator

Synthesis of silver nanoparticles were carried out in a continuous flow copper spiral microreactor. Microreactor was fabricated using linear low density polyethylenetube (LLDPE) having 173 cm length and 1.2 mm diameter. Two syringes mounted on syringe pumps were connected to the spiral shape microreactor making Y shape geometry and synthesis of silver nanoparticles were carried out in it. As shown in **fig. 1.** below

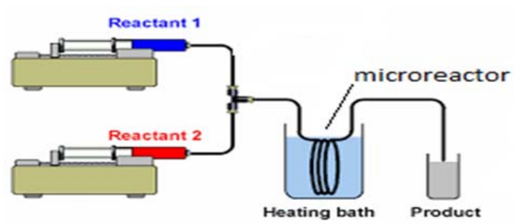

Fig. 1: MICRO REACTOR

Silver nitrate solution of various molar concentration was prepared from 0.001M to 0.005M was prepared by dissolving 0.0084g of $AgNO_3$ into 20ml of deionized water and Sodium borohydride (0.001M) was prepared by dissolving 0.001g of $NaBH_4$ into 60ml of deionized water in a conical flasks. In both solutions Sodium dodecyl sulphate (SDS) as a surfactant is added so as to cover the surface of particles and to remain in the stable form without aggregation. In both the solutions 0.02g of SDS was added. Initially, these solutions were cooled to 10°C for 20 min in an ice bath. Both solutions were filled in two separate syringes and were mounted on syringe pump. The flow rate of $AgNO_3$ solution was set at 1 mL/min and 3 ml/min for $NaBH_4$solution . The reaction was carried out for 20 minutes at a temperature at 60^0C. The volume of $NaBH_4$ used was in excess in order to reduce the ionic silver and to stabilize the silver nanoparticles. Both precursor and reducing agent were passed through spiral microreactor as shown in **Fig. 1**.and the product was collected in sample bottles .Initially, faint yellow color of solution appeared and then clear yellow solution was formed when final product was obtained .By varying the molar concentrations of silver nitrate and sodium borohydride from 0.001M-0.005M, different experiments were conducted with varying surfactant loading .

2.3. Characterization

The obtained samples were characterized by UV absorption at definite time of intervals using UV spectrophotometer. From UV absorption spectra maximum wavelength value was found to be 420 nm for the formation of silver nanoparticles. Various molar concentrations from 0.001-0.005M were checked for absorbance in UV-spectroscopy.

3. RESULTS AND DISCUSSION

The **Fig. 2.** Showing the absorption peaks of 0.001-0.005M molar concentrations. It indicates that an increase in SDS loading results in increase in the number of nuclei formation which in turn leads to reduction in the particle size of silver nanoparticles, which avoids the agglomeration of silver nanoparticles which results into smaller nanoparticle size.

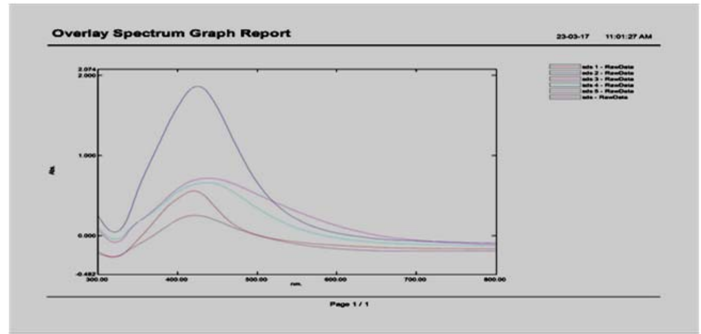

Fig. 2: Spectroscopy graph

4. CONCLUSION

Synthesis of silver nanoparticles in microreactor was done by using sodium borohydride and silver nitrate. $AgNO_3$ flow rate was 1ml/min and $NaBH_4$ was 3 ml/min shows minimum particle size of 4.8 nm with an absorbance value of 0.55- 1.85. It was observed that with simple experimental setup, nanoparticles with controlled size can be synthesized continuously using microreactor. It indicates that an increase in SDS loading results in increase in the number of nuclei formation which in turn leads to reduction in the particle size of silver nanoparticles. The flow was laminar with a Reynolds number 23.6483. The analysis of residence time was calculated and found to be 0.49 min. The Colloidal silver nanoparticles find applications in different areas such as catalysis, biological tags, gene detection, targeted drug delivery system, antimicrobial additives, conducting inks.

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