

# A NOVEL METHOD FOR THE PREPARATION OF SILVER HALIDE NANOPARTICLES FROM THEIR BULK SOLID USING MICROEMULSIONS

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Nanoparticles of silver halides are widely used to study the quantum confinement effects in indirect band gap semiconductors [1]. These particles are typically prepared by carrying out a precipitation reaction in the water pools of w/o microemulsions, or reverse micelles, by mixing two identical microemulsions containing aqueous solutions of AgNO<sub>3</sub> and KBr, or other halides [2]. The mixing of two microemulsions technique is the most common technique used to prepare precipitates of nanoparticles employing microemulsions. Precipitation reactions are known to have rapid kinetics and are limited by the opening of the surfactant layer upon collision of the reverse micelles carrying the reactants [3]. Slow opening of the surfactant layer results in simultaneous nucleation and aggregation, which in turn, results in the formation of larger particles with wider size distribution. In addition to the above limitation, microemulsion techniques generally suffer from low reactant solubilization capacity [4].

In the current work, we present a novel method for the preparation of silver bromide precipitate of nanoparticles that is aimed at minimizing the impact of the surfactant surface layer opening and the reactant solubility limits. The same method was successfully applied by our group for the preparation of AgCl precipitates of nanoparticles [4]. The new method utilizes the interaction between a cationic surfactant counterion, a halide anion, and the solid powder of the silver halide, to form soluble higher silver halides. These higher halides eventually migrate to the water pools of the reverse micelles, where they precipitate as nanoparticles of silver halides. The present work considers the formation of AgBr precipitate of nanoparticles in CTAB/n-butanol/water/isooctane microemulsions. The effect of the following variables on nanoparticle uptake and the particle size distribution was investigated: 1) operation variables; including rate of mixing and temperature, and 2) microemulsion variables; including CTAB and n-butanol concentrations, and water to surfactant mole ratio, R.

## **Measurements**

The molar concentration of the colloidal AgBr nanoparticles, the nanoparticle uptake, was determined by measuring the concentration of silver in the microemulsion using AA spectroscopy. The nanoparticle size distribution was determined from the analysis of TEM photographs. The UV-spectroscopy confirmed the existence of colloidal nanoparticles and provided a rough estimate on the nanoparticle uptake and the particle size distribution.

## **Results and Discussion**

The nanoparticle uptake increased as the surfactant concentration, the temperature and the rate of mixing increased. All these factors speeded up the reaction between the surfactant counterion and the AgBr powder and contributed towards increasing the concentration of the AgBr monomers in the water pools to values higher than the minimum nucleation concentration. The probability of particle aggregation upon collision of the nanoparticle-loaded reverse micelles increased. Hence, an increase in the particle size was associated with increasing these variables. Figs. 1 and 2 show the effect of CTAB concentration on the nanoparticle uptake, and the particle size distribution. On the other hand, the nanoparticle uptake was found to decrease and the particle size distribution to increase as the concentration of n-butanol or the water to surfactant mole ratio increased. This decrease in the nanoparticle uptake is attributed to particle aggregation following the decrease in the rigidity of the surfactant protective layer associated with increased values of these variables. Particle aggregation is evident in the TEM photographs

pertaining to high values of  $R$  and  $n$ -butanol concentration. Figs. 3 and 4 show the variation in the nanoparticle uptake and the particle size distribution upon increasing  $R$ .

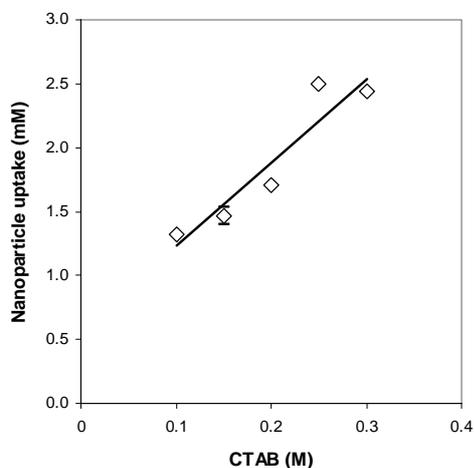
### Conclusions

A novel method for the preparation of AgBr precipitate of nanoparticles from solid AgBr powder was developed. In this method appreciable amounts of colloidal AgBr nanoparticles could be stabilized. In addition, the method offered control over the particle size by manipulating the operation and/or the microemulsion variables.

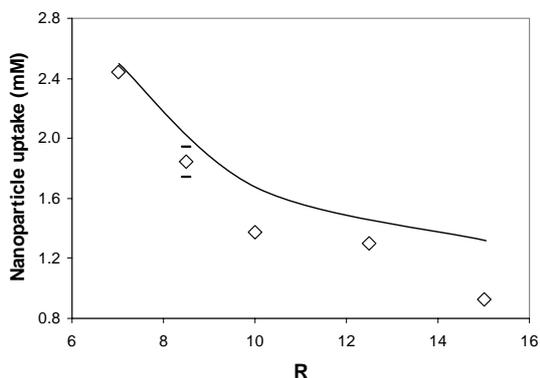
### References

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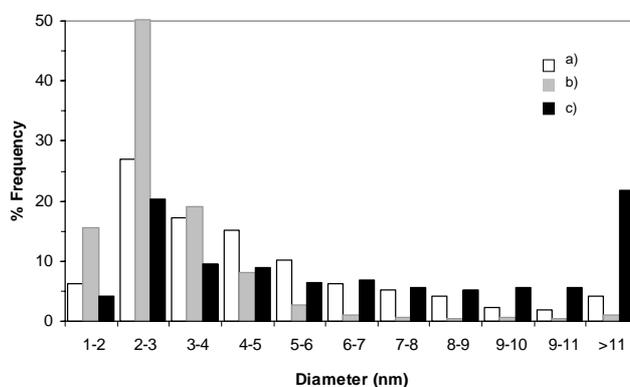
### Figures



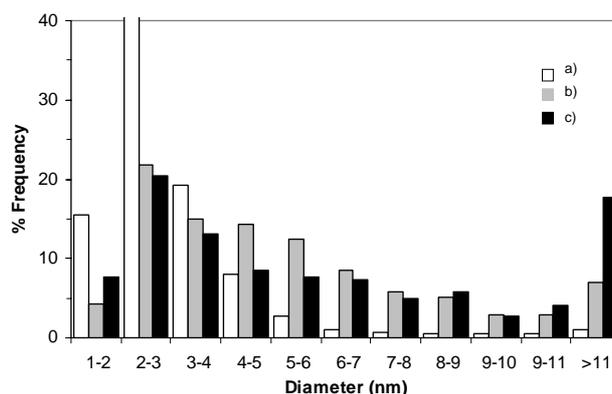
**Figure 1.** Nanoparticle uptake at different CTAB concentration when mixing AgBr powder for 6 h at 300 rpm and 25°C with CTAB microemulsion: 1.6 M Butanol,  $R=7$



**Figure 3.** Nanoparticle uptake at different water to surfactant mole ratio,  $R$ , obtained when mixing AgBr powder for 6 h at 300 rpm and 25°C with CTAB microemulsion: 0.2 M CTAB, 1.6 M Butanol.



**Figure 2.** Particle size distribution histograms obtained when mixing AgBr powder for 6 h at 300 rpm and 25°C with CTAB microemulsion: (a) 0.1 M; (b) 0.2 M; (c) 0.3 M CTAB, 1.6 M Butanol,  $R=7$



**Figure 4.-** Particle size distribution histograms obtained when mixing AgBr powder for 6 h at 300 rpm and 25°C with CTAB microemulsion: 0.2 M CTAB, 1.6 M Butanol, (a)  $R=7$ ; (b)  $R=10$ ; (c)  $R=15$ .