# UNDERSTANDING THE GROWTH MECHANISM AND ASSEMBLY OF ANISOTROPIC NANOSTRUCTURES WITHIN MICROEMULSIONS

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by


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# CERTIFICATE

This is to certify that the thesis entitled, **“Understanding the growth mechanism and assembly of anisotropic nanostructures within microemulsions”**, being submitted by **Mrs. Soma Sharma**, to the Indian Institute of Technology Delhi for the award of the degree of **Doctor of Philosophy** in Chemistry, is a record of bonafide research work carried out by her. Mrs. Soma Sharma has worked under my guidance and supervision, and has fulfilled the requirements for the submission of this thesis, which to my knowledge has reached the requisite standard.

The results contained in this dissertation have not been submitted in part or full, to any other university or institute for award of any degree or diploma.

Date:

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## Abstract

Nanostructures with controlled size/shape can be designed by following microemulsion route. Theoretical approaches and simulations have provided a wealth of information on the structural and dynamic properties of the nascent MEDs and exchange taking place during reaction which are difficult to be explored experimentally. In this thesis we aimed to understand the nanostructure growth-kinetics of ME-based reactions and the mechanism of how starting reactants react and nucleate to grow into nanostructures by using different spectroscopic, scattering and microscopy techniques.

**Chapter 1**, (Introduction) discusses the background of the thesis with a brief overview of the state of knowledge existing in the area of growth kinetics of microemulsion-based reactions.

**Chapter 2**, deals with the synthesis of aligned $Co_3O_4$ nanoparticles which yield a range of anisotropic nanostructures with controlled aspect ratio (1:5 to 1:13) from cobalt oxalate under controlled kinetic parameters and the temperature of decomposition. $Co_3O_4$ nanorods were found to be efficient electrocatalysts for oxygen evolution reactions (OER) and nanorods consist of particles of size 8-10 nm show very high current density of 104 mA/cm$^2$.

In **Chapter 3**, we aimed to understand the nanostructure growth-kinetics by studying the formation of iron oxalate nanorods inside the polar core of water-in-oil MEDs. FCS, DLS and TEM, has been employed to monitor the nanostructure growth. We obtain transient dimer lifetime (28 μs) and the droplet fusion rates on each day as the reaction proceeds. Three distinct growth periods has been observed in the entire growth process

**Chapter 4**, focuses on the understanding of the structural evolution of CTAB MEDs and the product zinc oxalate (ZO) synthesized in it by using SAXS, DLS and TEM. Our aim was to

correlate the size and shape of MEDs and that of resulting product. Importantly, once the reaction is triggered, in a very short nucleation dominant period MEDs become cylindrical with aspect ratio ~ 4:1 in which nanostructures grow anisotropically and achieve a critical size of 55 nm (elongated nanoparticles), beyond which a transition from elongated nanostructures to small rods is observed.

**Chapter 5**, deals with the determination of size and shape of different surfactant aggregates by varying $W_0$, oil phase and surfactant and their effect in controlling the size, morphology and aspect ratio of nickel oxalate nanostructures synthesized in them. The role of the cationic surfactant is critical for obtaining the anisotropic nanostructures while neutral surfactants favor the formation of spherical and cube-like morphology due to isotropic growth of particles as compared to cationic surfactants. This growth process can be viewed as a morphologically templated nucleation process and the droplets act as shaping vesicles for the formation of metal oxalate nanostructures.

In **Chapter 6**, We have studied Förster Resonance Energy Transfer (FRET) using CdS Quantum dots as donor and Sulforhodamine B (SRB) dye as acceptor inside RMs. In the results, an efficient FRET is observed (62% efficiency) within the confined environment of reverse micelle droplets which is higher than that in bulk phase (18%). This type of FRET study between a donor and an acceptor in a confined media provides a new vision to explore biological processes.

**Chapter 7**, outline the major results of our studies on understanding the evolution of nanostructures and their growth in RMs by following the kinetic steps from the time of mixing of reactants to their nucleation through intermicellar exchange to their growth into nanostructures.

Here, we will conclude the thesis and will discuss the future scopes of this work.

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