Experimental report

26/10/2015

Proposal:	**9-12-346**	**Council:**	4/2014
Title:	Dynamics of CTAB in hybrid CTAB-Hydroxyapatite System		
Research area:	Biology		

This proposal is a new proposal

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Samples: $Ca_{10}(PO_4)_6(OH)_2 + C_{16}H_{33}N(CH_3)_3Br$

Instrument	Requested days	Allocated days	From	To
IN5	3	0		
IN6	4	3	12/11/2014	15/11/2014

Abstract:

Hydroxyapatite (HAp), $Ca_{10}(PO_4)_6(OH)_2$ is a form of bioceramics material. It is chemically similar to bones and hard tissues found in humans and biocompatible. HAp is widely used in medicine and dentistry as a material for metallic implant coatings and bone cavity fillings. Recently, synthesis of mesoporous HAp using cationic surfactant (e.g. cetyltrimethylammonium bromide or CTAB) as the template is achieved using a supramolecular templating technique. The micelles act as nucleating points for the growth of HAp crystals. During the thermal incubation stage, CTABHAp complexes are produced and they coalesce to form a stable three-dimensional rod-like structure. This composite provides an ideal system to study the confined micellar system and a great opportunity to understand the role of organic molecules and its internal dynamics in bio-mineralisation process. The aim of the proposed experiment is to carry out a systematic quasi-elastic neutron scattering study on CTAB-HAp material investigating the dynamics of CTAB monomers in confined geometry produced by organic matrix as a function of temperature and compare the results vis-a-vis CTAB micellar systems.

Dynamics of CTAB in Hybrid CTAB-Hydroxyapatite System

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Hydroxyapatite (HAp) with molecular formula $Ca_{10}(PO_4)_6OH_2$ is a biocompatible material [1] and has been extensively studied and applied in variety of fields due to its similarity with inorganic mineral constituent of human bone and teeth. Its properties depend intimately on nano scale structure, which is dictated specifically by collagen template. Recently, surfactant assemblies have been employed as templates for the synthesis of nanostructured hydroxyapatite particles. Our system of interest here is hydroxyapatite nanoparticles and rod like micelles made of cetyl-trimethyl ammoniumbromide (CTAB) composite. Earlier, we studied the dynamics of different micellar and surfactant systems using neutron scattering techniques [2]. However, in micellar system apart from the internal dynamics of the surfactant, whole micellar motion also contributes to QENS data. HAp-CTAB composite system provides us the opportunity to study internal dynamics of CTAB surfactant alone since these surfactants are trapped in HAp composite. Dynamics of CTAB in HAp-CTAB composite system is studied using Quasielastic Neutron Scattering (QENS) technique. Data from pure hydroxyapatite prepared in the absence of any template under similar experimental conditions are also measured to estimate the contribution of HAp alone. Neutron scattering experiments were carried out using the IN6 spectrometer at Institut Laue Langevin, France. This instrument has an energy resolution (full width at half maxima) of 80 μeV at the incident neutron wavelength of 5.12 Å. Quasielastic data for both HAp-CTAB composite as well as pure HAp are recorded in the wave vector transfer (Q) range of 0.4–1.8 $Å^{-1}$ using in the temperature range 300–355 K.

QENS data obtained from the composite HAp-CTAB system showed presence of quasielastic broadening over the temperature range 300–355 K. Fig. 1 shows the typical QENS spectra along with resolution function of the instrument, measured using standard vanadium sample. Contribution from pure HAp is subtracted from the QENS spectra from HAp-CTAB composite system. Significant quasielastic (QE) broadening over the instrumental resolution is evident at all the measured temperatures. To proceed with the data analysis, it is customary to separate the contribution from different kinds of motions (vibrational, diffusive, etc.) according to their time scales. Within diffusive motions, there could be different motions such as segmental, torsional etc. of the CTAB monomers. It is found that QENS data could be described by a combination of an elastic line and two Lorentzian functions suggesting that there exists two time scales in the dynamics of CTAB monomers.

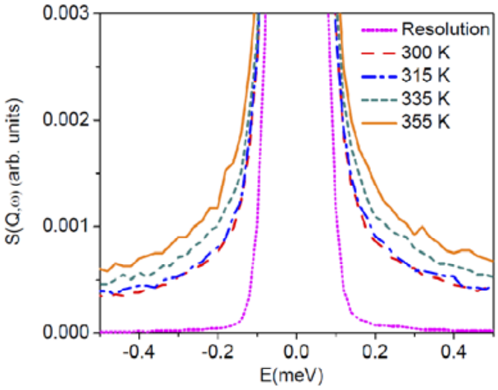

Fig. 1 Typical QENS spectra of CTAB monomers in HAp-CTAB composite at Q =1.6 Å^{-1} for different temperatures. Resolution function is also shown. All data are peak normalized with respect to resolution function.

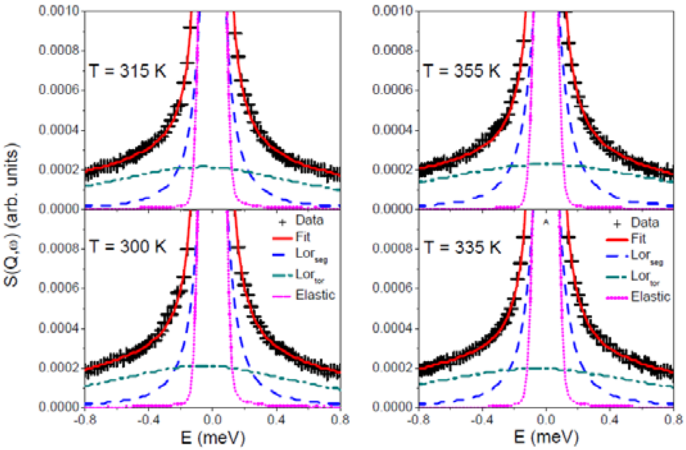

Fig. 2 Typical fits of QENS spectra of CTAB monomers at Q=1.8 Å^{-1} for different temperatures.

The fast dynamics should be the one associated with the conformational or torsional motion of the alkyl chain, as reported in the NMR and MD simulation studies [3]. The slow one is the segmental dynamics of the alkyl chain as suggested by our earlier studies on CTAB in micellar solutions [2]. Torsional motion is found to be faster than that of the segmental motion and can be modeled as 2-fold jump diffusion. Segmental motion can be modeled as diffusion of hydrogen atoms inside a sphere [4]. Both the motion showed temperature dependence in terms of either residence time or diffusivity.

REFERENCES

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