

Experimental report

08/02/2018

Proposal: INTER-352

Council: 4/2016

Title: Internal time on IN11

Research area:

This proposal is a new proposal

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Local contacts: Orsolya CZAKKEL

Samples: Heavy water D2O
Graphene oxide C2O
poly(N-isopropyl acrylamide) (C6H11NO)_n
polymer gel composites

Instrument	Requested days	Allocated days	From	To
IN11	10	10	25/11/2016	05/12/2016

Abstract:

Experimental report – INTER-352

Experiment dates: 25/11/2016-05/12/2016

Experiment team: Barbara Berke, Orsolya Czakkel

Local contact: Orsolya Czakkel

INTRODUCTION

A scheduled experiment was cancelled, we used this time to perform part of the experimental program described in proposal 9-12-445.

The aim of the experiment was to study the internal dynamics of carbon nanoparticle (CNP) filled soft gel systems based on poly(N-isopropyl acrylamide) (PNIPA). Such polymer gels exhibit a thermoresponsive behavior in which the matrix swells or deswells according to external stimuli. Our previous results showed that while the nature of the volume phase transition (VPT) is conserved the temperature response of the systems is strongly affected by incorporating CNPs.

EXPERIMENT

We investigated the dynamical behaviour of the nanocomposites above their VPT ($\sim 34^\circ\text{C}$). We used the IN11A configuration with the small-angle sample environment and the double echo configuration, with $8\text{ \AA}$ wavelength incident neutron beam. A Lauda thermostat bath was used for precise temperature control of the samples.

Measurements were made at 4 different Q values in the range of $0.04\text{--}0.137\text{ \AA}^{-1}$.

We have continued the measurements of INTER-329 at 40°C with pure PNIPA, 20 mg/g NIPA graphene oxide (GO) and a carbon nanotube (CNT) containing samples. As it was mentioned in our previous experiment report, the scattering signal of the collapsed gel is weaker, especially at higher q values, so longer acquisition times were needed.

To gain more information about the ratio of the incoherent and coherent scattering we measured the polarization at a wider angle-range.

RESULTS

As it was seen during the first test as well, the internal dynamics of the gels at 40°C are different from that of the swollen gels below the VPT.

We previously proposed that there might be fast dynamics inside the gel systems, but during these measurements we excluded this possibility.

The measured samples and the preliminary fits are shown on Figure 1. For all samples the curves obtained could have been fitted to a single exponential curve with a fixed baseline similarly to previous experiments (see experimental report 9-12-374). In all cases the relaxation rates (Γ) were proportional to q^2 . From a linear fit to Γ vs q^2 the diffusion constant was calculated (D_{diff}), yielding the hydrodynamic correlation length (ζ_H) of the systems. ζ_H values above the VPTT were found to be significantly higher than those of below the VPTT.

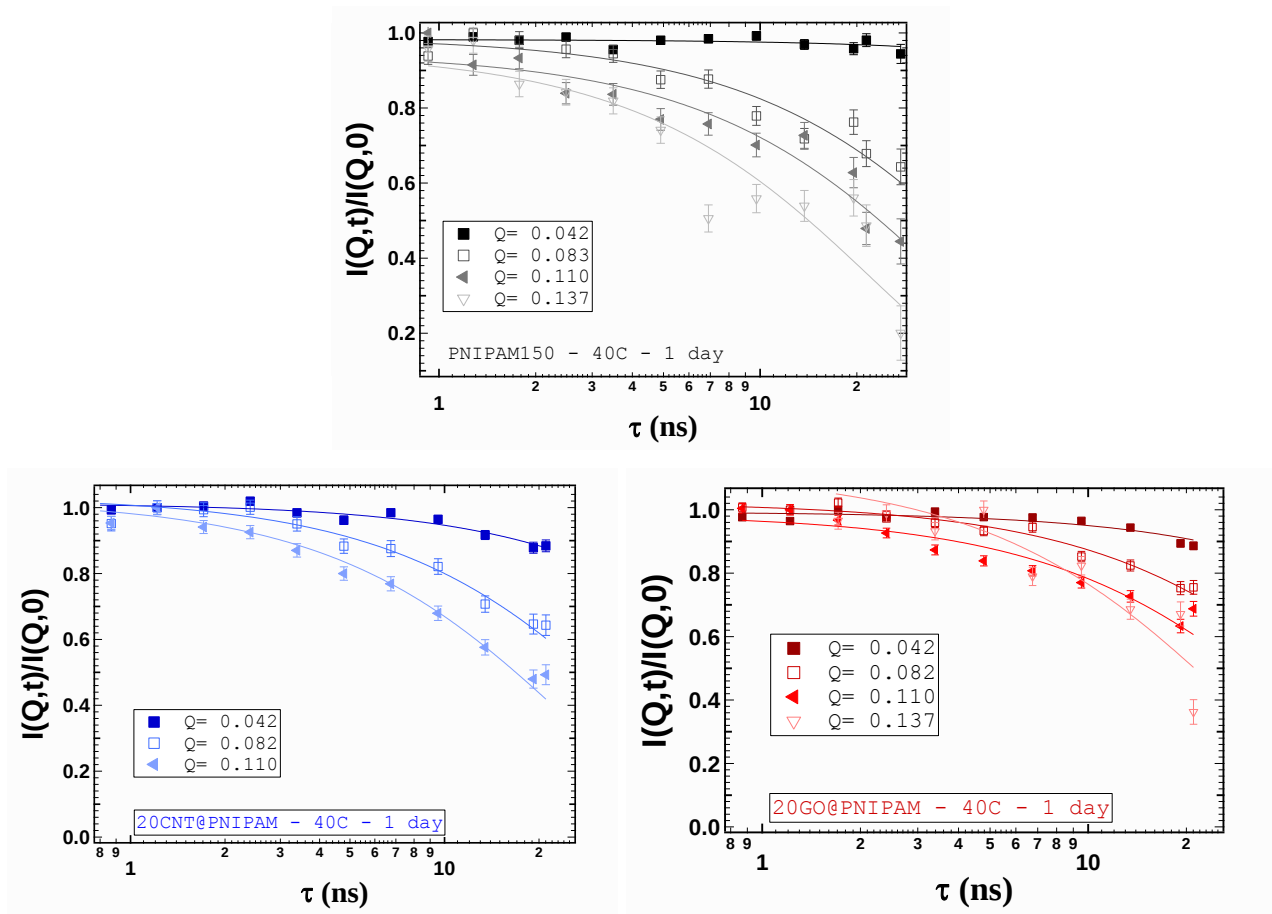


Figure 1: Corrected ISFs of a pure PNIPAM150 gel, CNT and GO-containing composite with preliminary fits on the coherent dynamics