

# Experimental report

10/07/2018

**Proposal:** 9-11-1838

**Council:** 4/2017

**Title:** Structure and conformation of charged thermo-responsive nanogels at air/water interface

**Research area:** Soft condensed matter

**This proposal is a new proposal**

**Main proposer:** Ali ZARBAKSH

**Experimental team:** Pengfei LIU  
Ali ZARBAKSH

**Local contacts:** Armando MAESTRO

**Samples:** N-isopropylacrylamide nanogels

Instrument	Requested days	Allocated days	From	To
FIGARO	4	3	01/06/2018	04/06/2018

## Abstract:

Soft polymeric nanoparticles possess many interesting properties. The aim of this proposal is to explore the interfacial properties of NIPAM-based thermo-responsive gels when charged monomers are introduced in the polymerisation step, resulting in overall charged particles. The work will focus on the study of the conformation and structural of charged NIPAM-based nanogels at air/water interface and examining them and the changes occurring as a function of concentration and temperature.

**1 PRINCIPAL INVESTIGATOR**

Name and institution of the Principal Investigator

Dr A Zarbakhsh  
Department of Chemistry  
Queen Mary University of London  
UNITED KINGDOM

**2 EXPERIMENT DETAILS**

Experiment: 9-11-1838

Title: Interfacial behaviour of charged nanogels at the air/water interface

Instrument: FIGARO

Dates scheduled: 1st June 2018 to 3rd June 2018

No. Days allocated: 3

Date of experimental report: 09/07/2018

**3 EXPERIMENT OBJECTIVES**

In the past we have successfully used neutron reflectivity (NR) to study the structural conformations of thermoresponsive N-isopropylacrylamide (NIPAM) based nanogels, cross-linked with methylene-bis-acrylamide (MBA), both at the air-water and hydrophobic solid (Si)-water interface. Results from neutron reflectivity and additional complementary experiments demonstrate that the adsorption process of NIPAM based nanogels onto the air-water interface can be described by lay-by-layer deposition with increasing layer thickness as the concentration of nanogels in the bulk and temperature increase in the absence of any bulk aggregation. For a well characterised hydrophobic (Si-C8) coated substrate, much thicker layers (order of magnitude thicker) than those at the air-water interface were observed.

We have also investigated the interfacial behaviour of N-n-propylacrylamide (NPAM) based nanogels at air-water interface. NPAM, the linear isomer of NIPAM, was selected on the basis that it could increase the overall hydrophobicity of the nanogels, while keeping a similar monomer structure. The preliminary results suggest a better ordered and much more compact packing for the NPAM compared with those observed of NIPAM. This indicates that the hydrophobic interactions may play a major role governing the adsorption process of these thermal responsive nanogels onto the interfaces.

In this experiment the main focus was to gain more comprehensive information on the main factors influencing the surface behaviour of nanogels, and extending these studies to examine the interfacial behaviour of NPAM-MBA nanogels, when charges are introduced by adding N-acryloyl-L-Proline (A-Pro-OH) during the polymerisation. We believe that the combination of a pH-responsive system with a thermo-responsive polymer can further alter the dynamic of the adsorption process also providing a better control over the layer deposition architecture. Hence in this experiment we explored the behaviour of these NPAM and A-Pro-OH based charged nanogels (short as NPAMP) at the air-water interface as a starting point.

#### 4 EXPERIMENT REPORT

We have investigated adsorption behaviour of NPAMP based nanogels crosslinked with 5% and 10%MBA at the air/water interface as a function of concentration (0.01, 0.05 and 0.10 mg/ml), temperatures (27, 35 and 40°C) for two aqueous sub-phase contrasts (D2O and NRW). Detailed data analysis is still ongoing.

Typical NR profiles of NPAMP nanogels with 5% crosslinker are presented as a function of temperature (Fig 1a) and concentration (Fig 1b). Distinct fringes can also be observed at higher concentration of nanogels in the bulk as well as at elevated temperature, which again suggests a well-ordered layer-by-layer and compact packing of these charged nanogels at the air-water interface. The incorporation of functional monomer A-Pro-OH into the NPAM polymeric network does not noticeably change its adsorption properties onto the surface around the isoelectric point even though the phase transition temperature increases more than 10°C.

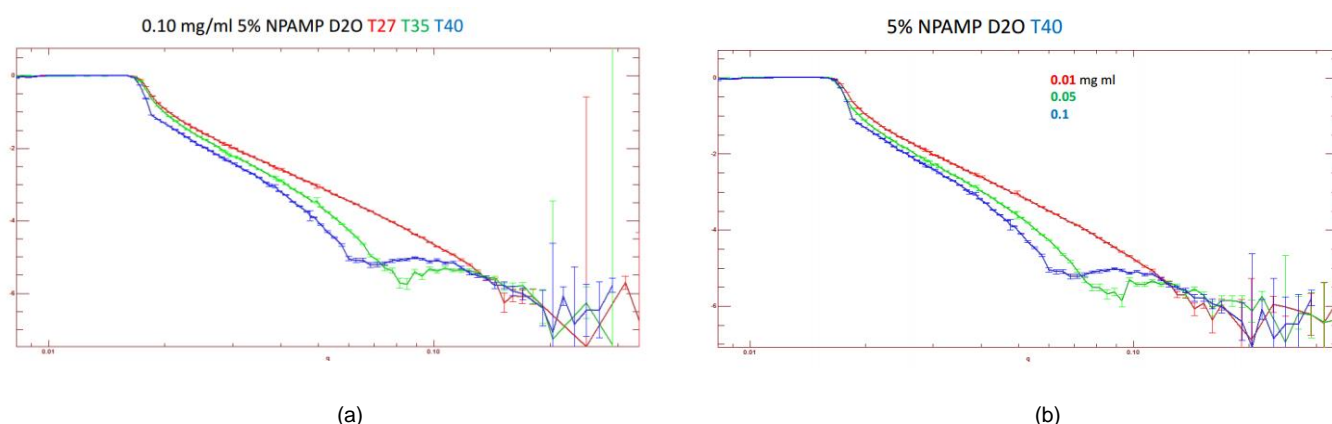


Fig. 1. NR profiles for 92.5%NPAM, 2.5%A-Pro-OH and 5%MBA nanogels at the air-water interface as a function of temperature (a) and concentration (b)

#### 5 LIKELY OUTCOMES FROM EXPERIMENT

Please indicate what the experiment is likely to lead to by putting an 'x' next to one or more of the possible outcomes below.

Likely outcome

Journal publication	X
Data for thesis	X
Follow-up experiment at ILL	-
Follow-up experiment at another facility	X
Other	X
No outcome anticipated	-

#### 6 SUGGESTIONS FOR IMPROVEMENTS TO YOUR EXPERIMENT, EQUIPMENT OR THE FACILITY

NA