

Experimental report

24/01/2024

Proposal: 9-12-671

Council: 10/2022

Title: Binding of perfluoroalkyl substances (PFAS) at interfaces

Research area: Soft condensed matter

This proposal is a new proposal

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Samples: Si

sapphire crystal

Moringa seed proteins

perfluorotanoic acid

Instrument	Requested days	Allocated days	From	To
D17	2	2	15/06/2023	16/06/2023

Abstract:

The presence of perfluoroalkyl substances (PFAS) in the environment is of on-going major concern and attracting considerable public attention as risks to health. These are known as 'forever' pollutants as breakdown is difficult although some routes to this process are now being developed. Knowledge of the interaction of these materials with relevant materials is an essential step to understanding the transport and eventual location of PFASs in the environment as well as their eventual remediation. To complement other work that has investigated various mineral surfaces and lipid layers, the present study will investigate binding to a protein layer. The protein chosen has been proposed as a binding agent for 'filter' materials. Results of neutron reflection measurements giving quantitative information about amounts of materials in layers and structure will guide further work both at a fundamental level to understand mediation of interactions and in practical development of treatment processes.

Experimental results:

Characterization of the sapphire surface was carried out to check that the surface was clean and to determine the interface roughness before further studies as this is important in subsequent modelling of data. The roughness of the sapphire surface was found to be 3 Å. The aim of the experiment was to study binding of sodium perfluorooctanoate to a sapphire surface in the presence of *Moringa oleifera* seed protein. A single day was allocated to demonstrate the viability of the proposed experiment. The sapphire surface was then exposed to 0.2 wt. % *Moringa oleifera* seed protein. As seen in figure 1a) the signal from the protein layer was substantially unchanged after rinsing with further (20 ml) D₂O indicating that the *Moringa oleifera* seed protein adsorbed irreversibly to the sapphire surface. This allows further characterization of the layer with H₂O as solvent as shown in Figure 1 b). A simple model using an exponential density profile of a hydrated protein layer with a decay length of 58 ± 1 Å and a very thin layer (2 ± 1 Å) with a uniform protein concentration of volume fraction of about 55% close to the alumina surface. The amount of protein on the surface was 4.2 ± 0.5 mg m⁻². These results agree with those of previous studies of this protein interacting with sapphire¹.

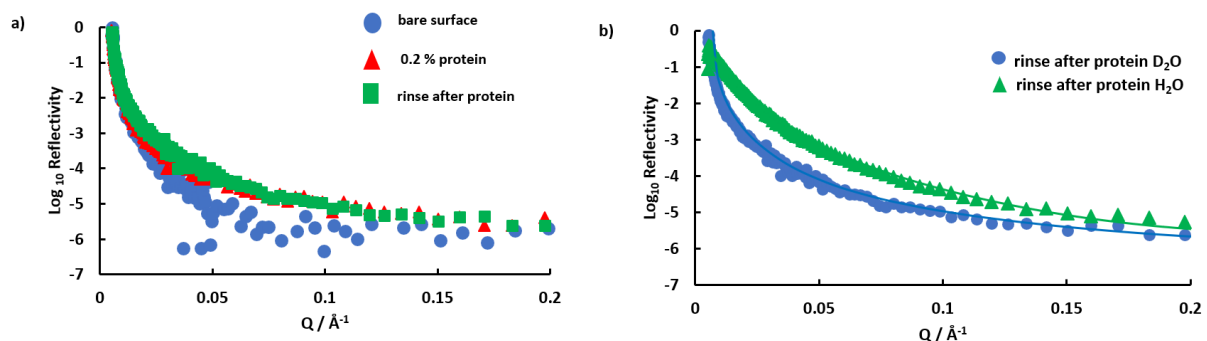


Figure 1. a) Sapphire surface with 0.2 % *Moringa oleifera* protein in D₂O b) characterisation of 0.2 % *Moringa oleifera* protein in D₂O and H₂O. The continuous lines are the fits of the model described in the text.

The sapphire surface with the adsorbed protein was exposed to 9 mmol dm⁻³ and 18 mmol dm⁻³ sodium perfluorooctanoate in H₂O. Sodium perfluorooctanoate interacts with the protein and adsorption increased as the concentration increased. The sapphire surface with the adsorbed protein was exposed again to 18 mmol dm⁻³ of the sodium perfluorooctanoate in two contrasts (H₂O and D₂O) and an effect was seen. As seen in figure 2b), a preliminary model fit was made and we established that the surface layer was no longer an exponential density profile, instead a thick uniform compact layer comprised of the protein, sodium perfluorooctanoate and water was formed. The sodium perfluorooctanoate possibly occupies the place of displaced protein or water. Therefore, the composition and structure of this layer needs to be studied further using different contrasts of water (D₂O and H₂O) with both surfactant solutions and pure water as we need to establish whether protein has been displaced after the addition of sodium perfluorooctanoate and with subsequent rinsing.

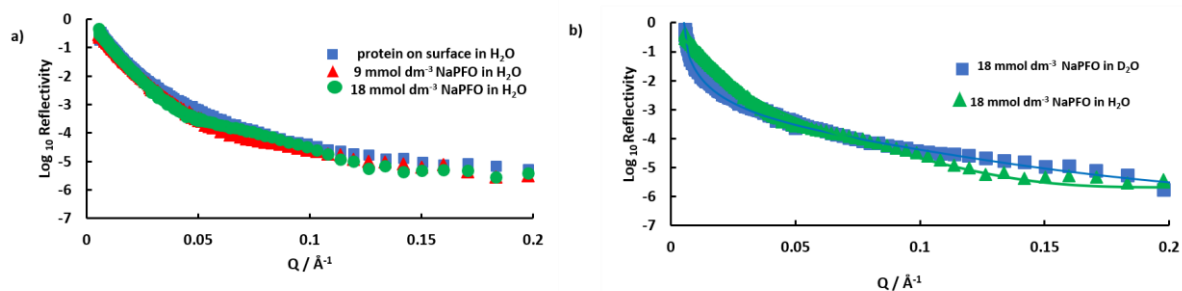


Figure 2 a) sodium perfluorooctanoate bound on *Moringa oleifera* protein in H_2O b) 0.018 mmol dm^{-3} sodium perfluorooctanoate simultaneous fits in D_2O and H_2O .

There were considerable instrumental problems during the experiment, half of the detector failed early in the experiment that lead to some loss of measuring time. Subsequent measurements only recorded about half of the reflected beam. In consequence, only very limited measurements could be made with adequate statistics.

Despite the difficulties, we were able to demonstrate that the experiment is viable but more measurement time is needed to complete the original planned experiment and provide full information about the interactions at the surface.

Reference:

1. [Kwaambwa, H.M et al., J Colloid Interf.Sci.,448, 2015,339-346.](#)