Using Ion Beam Analysis to Study the Surfactant Distribution in Cross-Linked Colloidal Polymer Films

T.R. Palmer1, K. Greiner2, M. Duewel2, R.W. Smith3, H.M. van der Kooij1, J. Sprakel3, J.L. Keddie1

1 University of Surrey, Guildford, UK, 2 Synthomer, Marl, Germany, 3 Surrey Ion Beam Centre, Guildford, UK, 4 Wageningen University, Wageningen, The Netherlands

1. Introduction
Pressure-Sensitive Adhesives (PSAs) adhere to almost any surface following the application of light pressure. Surfactant accumulation at the surface of PSAs is known to cause detrimental effects on their properties [1], hence the ability to control migration of surfactant during the drying stage is of great interest. In this work, we have studied the effect of particle deformation on the migration of surfactant and its final distribution within colloidal polymer films. Particle deformation has been controlled via cross-linking of the polymer chains within particles at differing amounts, from 0 mol% cross-linking up to a maximum of 35 mol% cross-linking. Poly (Butyl Acrylate) films synthesized via emulsion polymerization with SDS surfactant and EGDMA cross-linker [2] have been studied using a combination of Rutherford Backscattering Spectrometry (RBS) and Atomic Force Microscopy (AFM) in order to establish a picture of the surfactant accumulation both on the top surface of films, as well as depth profiles in the top several hundred nanometers of the films.

2. Film Formation & Cross-Linking

Polymer dispersed in water

Surfactants

Particle deformation

Coalescence

Pressure Sensitive Adhesive (PSA) Film

Cross-Linking

Cross-links are bonds which join two polymer chains together. The effect of the cross-linking density is being studied.

3. Rutherford Backscattering Spectrometry

Helium RBS provides depth profiles of hydrogen and deuterium in the top several hundred nanometers of a film.

The film surface is bombarded with a He+ ion beam.

The beam may collide with Na or S causing it the incident He+ to be backscattered towards the detector.

The mass of the elements in the film determines the energies of the backscattered He+ ions.

Technique tells us what is in the sample, how much of it there is, and where it is.

Example Spectrum

4. Results: RBS

- Inside the red region of interest, we can see spectra for films with low (5%), medium (20%) and high (35%) levels of polymer cross-linking.
- What we see is that for only the green, intermediate level of cross-linking are there significant amounts of Na & S, indicative of surfactant at the surface.
- These results suggest that surfactant is only accumulating at the surface of films with intermediate levels of cross-linking.

5. Results: AFM

In order to verify the results of the RBS, we decided to perform Atomic Force Microscopy (AFM) on the top surfaces of the films. This qualitative technique enabled us to visualise the top surface, and identify any potential surfactant on the surface. This technique does not provide depth profiling.

- The results of the AFM appear to nicely confirm those from the RBS, agreeing that surfactant is only seen on the surface of films with an intermediate level of polymer cross-linking.

6. Interpretation

The result of the RBS & AFM agree well, however explaining why surfactant only accumulates at the surface of certain films still needs to be done. The following chart an initial attempt at explaining the trends in accumulation of surfactant.

7. Interpretation cont.

- Low Cross-Linking: Soft particles that readily film form and act as a barrier to the surface.
- Intermediate Cross-Linking: Surfactant is trapped between deforming particles near the surface.
- High Cross-Linking: Channels between particles stay open, surfactant travels to the bottom of the film whilst in the water phase.

8. Summary & Conclusions

- RBS & AFM has been used to study surfactant migration in cross-linked colloidal polymer films, specifically PSAs.
- Surfactant accumulates at the surface for films with intermediate levels of cross-linking, however surfactant did not enrich the surfaces for low and high levels of cross-linking.
- Keeping surfactant away from the surface is desired for applications such as PSAs, therefore avoiding this intermediate cross-linking level would be suggested [1].

References


Acknowledgements

TRP would like to thank Violeta Douková & Malin Schulz for their technical assistance in the lab. Thanks go to Hanne van der Kooij & Joris Sprakel from Wageningen for providing the pBuA dispersions. Funding for the PhD studentship from Synthomer & EPSRC is gratefully acknowledged. Access to the Surrey Ion Beam Centre was provided through EPSRC PhD student grant G660.