Understanding the Mechanical Strength of Microcapsules and Their Adhesion on Cellulosic Surface

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Introduction

There is a growing need to deliver "active molecules" to cellulosic surfaces, which may be realised by encapsulation. For such applications, the microcapsules should be able to be deposited on cellulosic surfaces, followed by mechanical rupture. Therefore, it is important to investigate the mechanical properties of such microcapsules and their adhesion on cellulosic surfaces in air or liquid media relevant to end-use applications in order to target the delivery of the "active molecules" effectively. In this work, melamine formaldehyde (MF) microcapsules with an oil-based industrial precursor, MF microparticles and a flat cotton cellulose film were used as a model system.

Experimental

Methods to formulate and characterise microcapsules

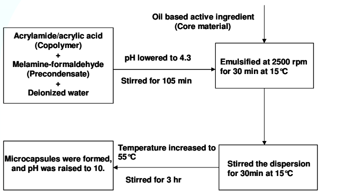

Fig.1 In-situ polymerisation method to make melamine-formaldehyde microcapsules.

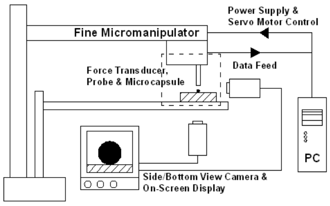

Fig.2 Schematic diagram of micromanipulation rig

Production of a thin and uniform cotton cellulose film

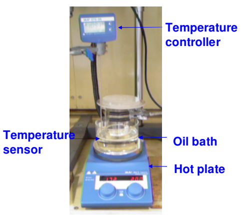

Fig.3 Equipment setup to dissolve cotton cellulose.

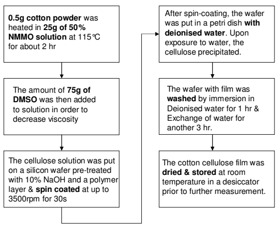

Fig.4 Procedures to generate a thin and uniform cotton cellulose film.

Measurement of adhesive force & characterisation of cotton film

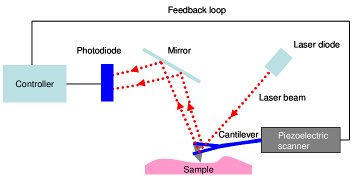

Fig.5 Schematic diagram of an Atomic Force Microscopy (AFM) system.

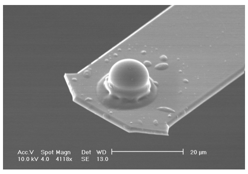

Fig.6 A SEM image showing a MF microparticle of 11.9µm in diameter was attached to a tipless cantilever.

Atomic Force Microscopy (AFM) was employed to measure the roughness of cotton film as well as the adhesive force between microcapsules/microparticles and cotton fabric/film.

Results & Discussions

Wall thickness of microcapsules & rupture force of one representative sample (D30)

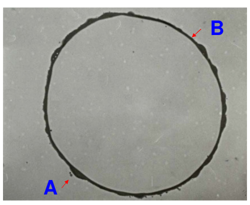

Fig.7 Transmission Electron Microscopy (TEM) image of a microcapsule.

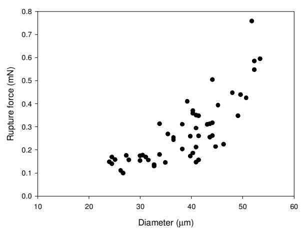

Fig.8 Rupture force versus diameter of single microcapsules for Sample D30.

In *Fig.7*, the microcapsule diameter is 27.3µm, and wall thickness at Points A & B are 875nm and 275nm, respectively. In *Fig.8*, as diameter of microcapsules increases, the rupture force of microcapsules also increases on average.

Results & Discussions

Force versus deformation data from loading and unloading experiments

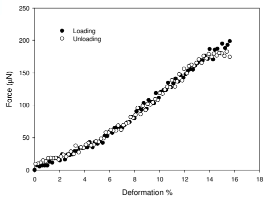

Fig. 9 A microcapsule (18.0µm) was compressed to a deformation of 15.5% and then released.

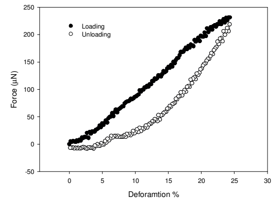

Fig. 10 A microcapsule (19.0µm) was compressed to a deformation of 24.3% and then released.

- *Fig.9* shows the microcapsule (18.0µm) exhibits elastic behaviour at 15.5% deformation, and *Fig.10* shows the microcapsule (19.0µm) exhibits plastic behaviour at 24.3% deformation.

Comparison of the mean mechanical property parameters of microcapsules between samples

Sample Name	Number Mean Diameter (µm)	Number of capsules tested	Average Rupture Force (mN)	Displacement at Rupture (µm)	Deformation at rupture (%)	Nominal stress at Rupture (Mpa)
D10	11.3 ± 0.6	55	0.1 ± 0.0	2.8 ± 0.3	25.1 ± 2.3	1.4 ± 0.2
D15	18.4 ± 1.4	50	0.2 ± 0.0	4.2 ± 0.4	24.0 ± 3.2	1.0 ± 0.2
D30	37.2 ± 2.2	61	0.4 ± 0.1	7.0 ± 1.0	19.5 ± 3.1	0.4 ± 0.2

Table 1 Mechanical properties of tested samples with different sizes

- On average, the rupture force of microcapsules increased with their diameter.
- The nominal stress at rupture of microcapsules decreased with their diameter.
- However, the deformation at rupture did not seem to change with their diameter significantly.

Roughness of cotton film and measurement of adhesive force

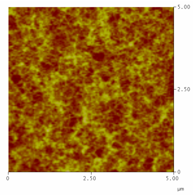

Fig.11 AFM image (tapping mode, height image) of a dry cotton cellulose film. Cellulose concentration in solution was 0.5%w/w.

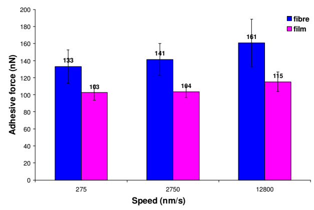

Fig.12 Adhesive force between a 34.8µm microcapsule and various locations on a cotton fabric fibre/cotton cellulose film in air at different approach speeds of the force probe.

In *Fig.11*, the root-mean-square (rms) surface roughness is 5.0nm. *Fig.12* suggests the adhesive forces between the microcapsule and cotton fabric/film in air are in the order of 100nN.

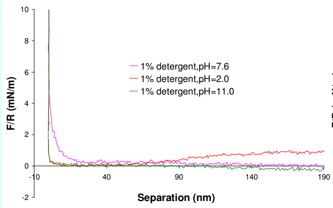

Fig.13 Interaction between a 34.8µm microcapsule attached to 7.5N/m tipless cantilever and a cotton film surface immersed in 1.0wt% detergent at different pH values (Approach curve).

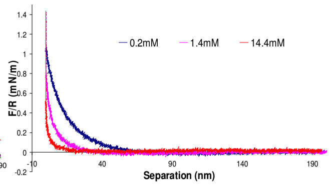

Fig.14 Interaction between a 9.2µm microparticle attached to 0.13N/m cantilever and a cotton film surface immersed in anionic surfactant sodium dodecylbenzenesulfonate solution with various concentrations (Approach curve).

In *Fig.13*, the resolution of force curve is 2nN. There was no significant adhesive force observed between the microcapsule and cotton film in detergent at various pH values using the 7.5N/m cantilever. In *Fig.14*, a more sensitive cantilever (0.13N/m) was applied. Again, there were no significant adhesive force observed from the graphs.

Conclusions & Future Work

- The **micromanipulation technique** is a powerful tool to characterise the mechanical properties of microcapsules.
- A thin and uniform cotton cellulose film was generated. The adhesive forces between the microcapsule and cotton film **in air** have been obtained, and they are in the order of 100nN.
- There were no significant adhesive forces observed between the **microcapsule** and regenerated cotton film in **detergent** solutions (Force resolution = 2nN).
- No significant adhesive force was detected between the **microparticle** and regenerated cotton film in **surfactant** solutions (Force resolution = 0.1nN).