ISIS@MACH ITALIA

Science and Technology Facilities Council
ISIS Neutron and Muon Source

Experiment Proposal

Experiment number GP2023041

Principal investigator	Dr Gavino Bassu, Università degli studi di Firenze, ITALY
Co-investigator (*)	Dr Monica Tonelli, University of Florence, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Polymer-coated silica capsules with enhanced retention properties: structural and stability investigation.
MRF Instrument	**Confocal Microscope 1**
Access Route	Direct Access
Science Areas	Chemistry
Sponsored Grant	None
Grant Title	-
Start Date	-
Similar Submission?	-
Industrial Links	-

Days requested: 3
Previous GP Number: No
DOI: -
Sponsor: -
Grant Number: -
Finish Date: -

Non-Technical Abstract

Silica capsules are widely used as carriers for water-insoluble cargo reaching an increasing interest in many fields of technological applications. Thanks to their versatility, the well-known solid-stabilized “Pickering emulsions” has drawn significant research interest. This approach involves solid particles that accumulate at the interface between immiscible liquids, stabilizing the droplets from coalescence and functioning as a scaffold for the capsule’s shell formation. We propose an approach to improve and modulate the shell diffusion by using polymer-coating. The reinforced microcapsules can be prepared by hydrogen-bonds or charge interaction between the silica shell and polymer. Due to the negative charge that characterizes silica capsule surface at neutral pH it is possible to reinforce the surface with a positively charged polymer such as (pDADMAC) or alginate/ chitosan derivates

Publications

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IM@IT E-platform: No

Instruments	
Access Route	
Science Areas	
Sponsored Grant	
Grant Title	
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Previous RB Number:
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Sample record sheet

Principal contact Dr Monica Tonelli, University of Florence, ITALY
MRF Instrument **Confocal Microscope 1** **Days Requested:** 3
Special requirements:

SAMPLE

Material	SiO2 microcapsules	-	-
Formula	microcapsules of SiO2; PDADMAC: (C8H16ClN)n; Alginate: (C6H8O6)n ; Chitosan: C56H103N9O39	-	-
Forms	Solid		
Volume	1 cc		
Weight	1 mg		
Container or substrate	Lab-Tek Chambers with N.1 borosilicate glass coverslips	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	288,15 - 298,15 K	-	-
Pressure Range	1013,25 - N.A. mbar	-	-
Magnetic field range	N.A. - N.A. T	-	-
Standard equipment	None	-	-
Special equipment	No special equipment are requested	-	-

SAFETY

Prep lab needed	Yes	-	-
Sample Prep Hazards	No other hazards are associated with the sample	-	-
Special equip. reqs	No otherspecial equipment are requirements	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	No other hazards are associated with the experiment	-	-
Equipment Hazards	-	-	-
Biological hazards	No Biological hazards are associated with the sample	-	-
Radioactive Hazards	No Radioactive hazards are associated with the sample	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed of by instrument scientist	-	-

Polymer-coated silica capsules with enhanced retention properties: structural and stability investigation.

1. Background and Context

Silica capsules are widely used as carriers for water-insoluble cargo reaching an increasing interest in many fields of technological applications, including drug delivery, food industry and more. Thanks to their versatility, the well-known solid-stabilized “Pickering emulsions” has drawn significant research interest[1]. Pickering approach involves solid particles those accumulate to the interface between immiscible liquids, stabilizing the droplets from coalescence and functioning as a scaffold for the capsule’s shell formation. The stability of the emulsion is strictly related to the solid particles’ nature as well as the resulting morphology and structural properties of the so synthetized shell. Due to its abundance and facile customization, silica (SiO_2) is one of the most studied Pickering emulsifiers. Despite extensive studies on the stabilization and shell-formation mechanism, the structural morphology and permeation control of silica microcapsules’ shell is not completely understood. The application of SiO_2 nanoparticles with different size, ranging from 5 to 20 nm in diameter, as different building-blocks to constitute the microcapsule' shell, can strongly modify the nanoscale topology and consequently the mechanical strength and permeation properties of the resulting capsules. Characteristic pores size and connectivity determine mechanical strength and permeation properties, which are usually enhanced by successive mineralization steps that require a consolidating material to obtain the desired features[2]. The final properties of the so-obtained microcapsule are usually not satisfactory in terms of mechanical resistance and cargo leaching. Here we propose a facile approach to improve and modulate the shell diffusion by using polymer-coating. The reinforced microcapsules can be prepared by hydrogen-bonds or charge interaction between the silica shell and polymer. In particular, due to the negative charge that characterize the silica capsule surface at neutral pH it is possible to easily reinforce the surface with positive charged polymer as diallyldimethylammonium (pDADMAC) or alginate/chitosan derivates. By fine tuning of the capsule coverage a significant enhancement of stability was expected to be observed.

2. Proposed experiment

Tunable mechanical and diffusive properties are strictly related to the pore size, morphology, and the structural topology of the silica shell. In order to explore the structural feature resulting from the different building-blocks and information of the polymeric-coating interaction on the silica surface the U-\SAXS (Xenocs XEUSS 3.0) instrument is requested to investigate microcapsules obtained from solid emulsifier with 5 and 20 nm diameter, respectively M_5 and M_20. The pure silica microcapsules will be used as reference for the U-\SAXS experiments of the coated samples providing information about the polymer-silica interaction with significant differences in characteristic fractal dimensions. These results will be also compared to the microcapsules coated by pDADMAC, alginate and chitosan derivates (M_D, M_A, and M_C respectively) at different surface coverage. According to the Kline et al. model[3], the fractal-like aggregates features were extrapolated by the fitting of the scattering profiles. To obtain the complete overview on the morphological and structural features and the diffusion properties of the pristine microcapsule and the polymeric-coated sample, reaching the micron-scale, the Confocal Microscope 1: Leica TCS SP8 (CLSM) are requested. Thanks to the precise 3D analysis, this instrument allows the structural investigation at the micron-scale required to elucidate the changes induced by the different precursors giving information on the shell thickness, defects, and relative distribution of inhomogeneities. As observed in preliminary experiments, volumetric stacks of capsules labelled with selective fluorescent markers allows the determination of the desired features.

3. Summary of previous experimental proposals or characterisation

Preliminary tests have shown the feasibility of studying microcapsules prepared with Pickering emulsions stabilized with 5 nm silica nanoparticles, which were characterized by scanning electron microscopy as prepared and following a conventional method of strengthening using salt-based consolidation material. The experimental evidence reported by the micrographies in the fig.1-A confirms the presence of reinforced silica shell after mineralization procedure; moreover, the micron-scale morphology were explored by confocal microscopy (CLSM) were confirmed by confocal microscopy studies. The selective labeling of the investigated systems allowed the identification of microcapsules permeable and impermeable to the chosen fluorescent probes (rhodamine B isothiocyanate (RBTC) and fluorescein isothiocyanate (FITC)); the selective marking by the two probes allowed the high-resolution imaging of the silica shell and the coating material as well as local defects present in the structure. Preliminary studies on the stability, in terms of permeability of the capsules, confirmed a consistent improvement in the retention properties after the reinforcement process, see fig.1-C.

Despite these experiments allowed the analysis of the mineralization effects at the micron-scale, highlighting permeable from impermeable capsules, and the distribution of the mineralized material, several efforts are required to completely understand the nanoscale topology.

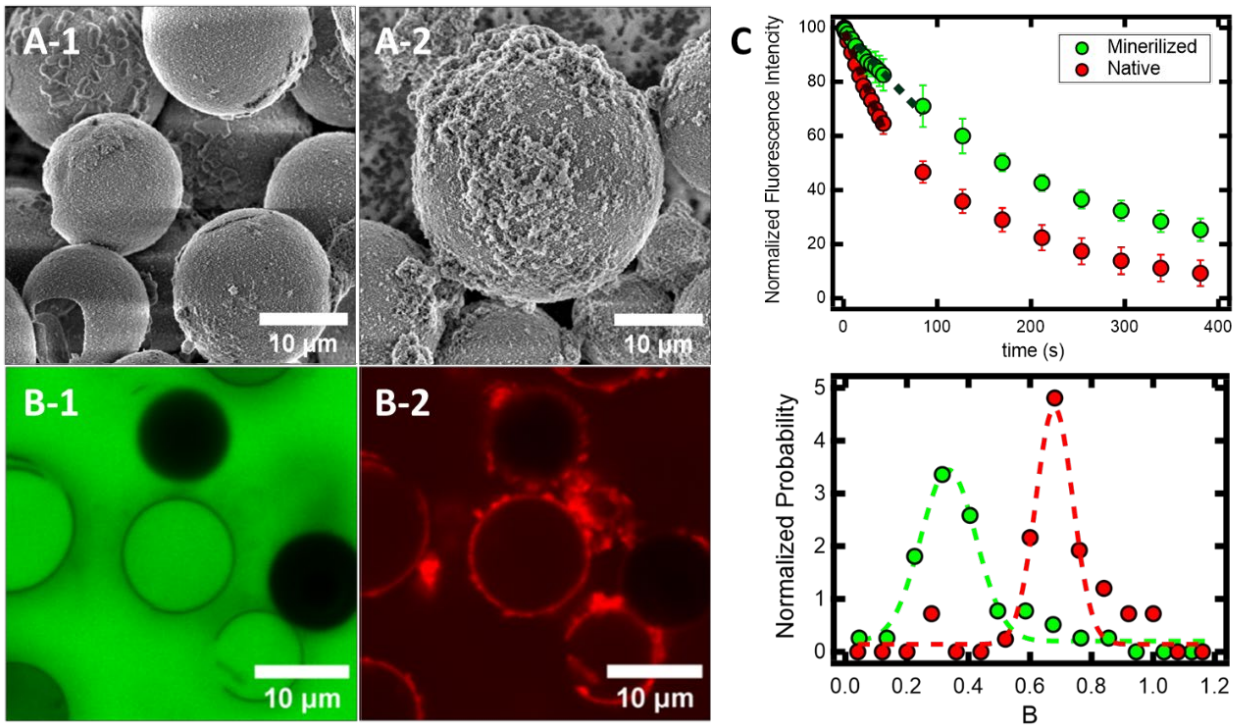

Figure 1. Scanning electron micrographies of the native(A-1) and mineralized microcapsules(A-2); Confocal images of microcapsules labelled by FITC (B-1, green channel) and RBTC (B-2, red channel); time-resolved fluorescence decay and the extrapolated decay coefficient B of native and mineralized capsules (C)