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Okayama University research: A rapid flow process that can convert droplets into multilayer polymeric microcapsules

(Okayama, 7 March) **In a study reported in the journal of *ACS Applied Polymer Materials* researchers at Okayama University develop a simple method to create multilayer microcapsules that can be used for controlled release of substances.**

Microcapsules are attractive vehicles for the delivery of pharmaceuticals and biologically active species. Tuning the membrane structure of microcapsules is crucial to control the release behaviour of the substances. However, it is challenging to design the structure of microcapsules in a simple fashion. Now, a research trio led by Research Associate Professor WATANABE Takaichi and YASUHARA Yuka (graduate students), Professor Ono Tsutomu at Okayama University has successfully created microcapsules containing multiple layers of polymers with a simple strategy.

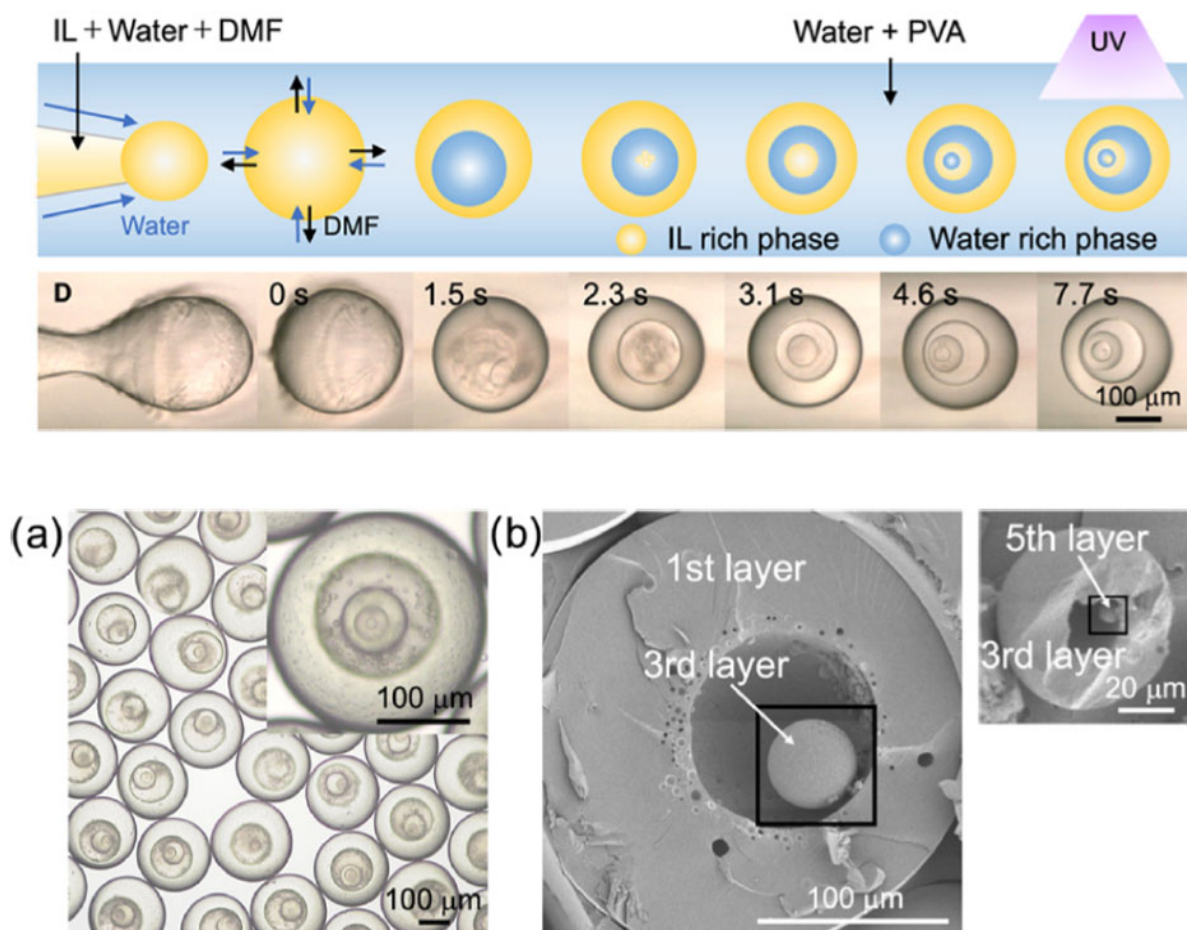
The team leveraged a process known as liquid–liquid phase separation (LLPS) for their study. LLPS works on the premise that immiscible phases in a mixture will form separate layers (much like oil droplets on water). Thus, when a combination of three different liquids—water, a water-resistant ionic liquid (IL) monomer ([C4vim][Tf2N]), and a cosolvent between the two (DMF)—were mixed, they separated out to form droplets with the IL molecules on the outside. These droplets were subsequently dispersed through a thin glass tube into an aqueous solution. The droplets then showed up to five alternating sublayers of water and IL within them. A higher proportion of water and DMF resulted in a greater number of layers within the droplets. The researchers discovered that once the initial droplets were formed, DMF molecules escaped outside into the aqueous solution resulting in the formation of multiple sublayers. Higher compositions of DMF and water also led to faster formation of sublayers due to a better separation of the phases. They also found that the size of initial monolayer droplets did not affect formation of subsequent internal layers.

Next, the multilayer droplets were exposed to ultraviolet light in order to create solid poly(ionic liquid) (PIL) microcapsules. When examined under a microscope, the droplets turned from transparent to opaque suggesting a transition to the capsular form. What's more, the structures of all internal layers remained intact. Since the microcapsules were engineered using water-resistant (hydrophobic) PIL, the team also investigated the possibility of swapping these out for water loving (hydrophilic) chemical groups on the PIL layers. Using a technique called anion exchange they replaced all [Tf2N] molecules on the surface with hydrophilic ones. When the microcapsules were then immersed in aqueous solvents, they swelled up and became permeable to the solvents indicating the procedure was successful.

This study showed a novel method of obtaining multilayer solid systems for PIL use. “The sequential phase separation system observed in the ternary emulsion droplets can pave the way for the design of PIL-based colloidal materials with thermodynamically nonequilibrium structures, thereby extending their application in functional materials,” concludes the trio.

Background

PILs: Ionic liquids are bulky salts with low melting points rendering them a gooey liquid consistency at room temperature. PILs are groups of ionic liquid molecules chained together. These have superlative physicochemical properties and desirable safety profiles making them ideal for use in industrial reactions. A major challenge with PILs, however, is formulating them into solid systems such as films and fibers that can be used in industrial applications. The solid microcapsules generated in this study as are a path forward in this direction.



Figure

Top. Dynamics of single droplets transforming to form alternating multilayer PIL/water droplets as DMF escapes into the outer solution.

Bottom. Microscopy images showing the opacity of solid PIL microcapsules after ultraviolet light treatment (a) and the intact structure of all internal PIL/water layers within them (b).

Reference

Takaichi Watanabe, Yuka Yasuhara, Tsutomu Ono. Multilayer Poly(ionic liquid) Microcapsules Prepared by Sequential Phase Separation and Subsequent Photopolymerization in Ternary Emulsion Droplets. *ACS Appl. Polym. Mater.* 2022, 4, 1, 348–356.

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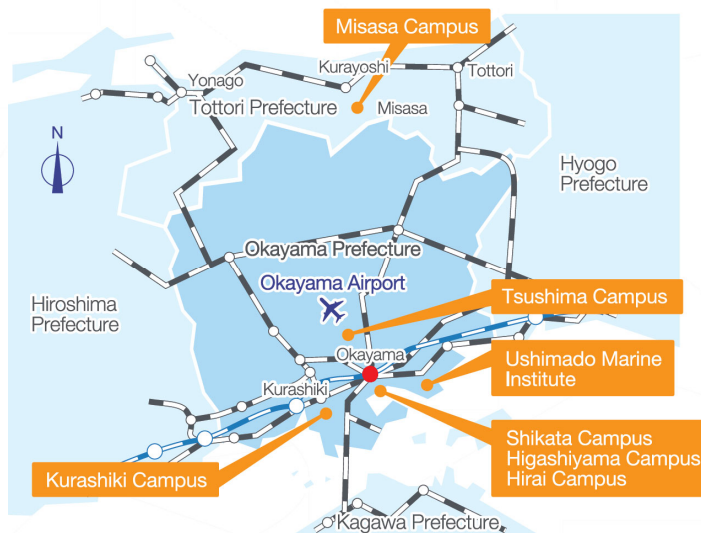
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Okayama University is located in the heart of Japan approximately 3 hours west of Tokyo by Shinkansen.

Website: http://www.okayama-u.ac.jp/index_e.html



Japan (日本)



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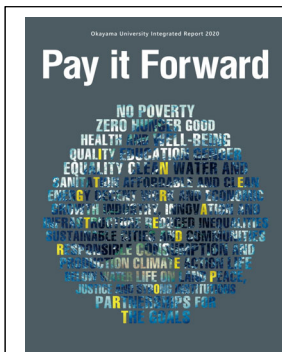
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