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Synthesis of microcapsules containing diisocyanate as a healing agent

There are several ways to produce self-healing coatings, and incorporation of microcapsules containing a healing agent into the coatings' formulations is one of the most studied approaches. Using microcapsules system for self-healing, the active agent released from the ruptured microcapsules can repair the damage and continue the role of the protective layer [1]. The double-capsule system based on the encapsulation of two reactive healing agents is easy to operate and achieve (Fig. 1). The healing system such as epoxy-amine, epoxy-thiol, isocyanate-thiol and isocyanate-amine had shown good self-healing and corrosion protection features in coating applications [2].

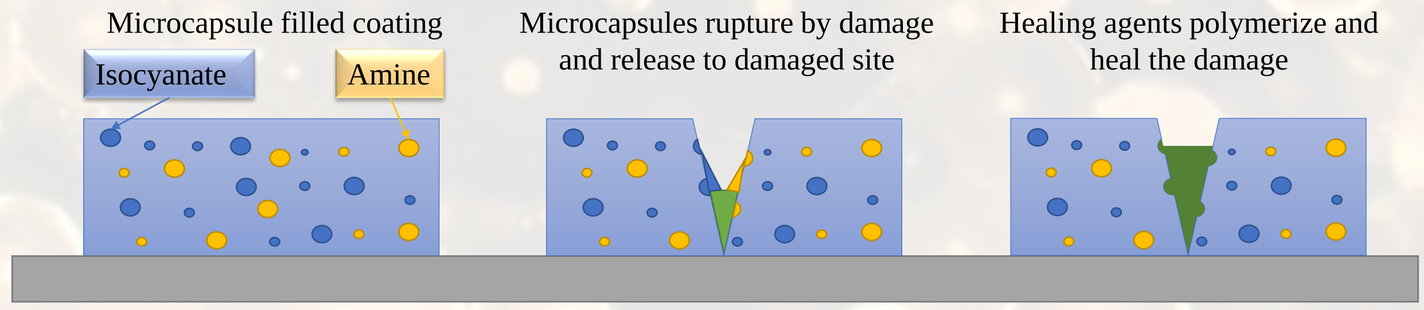

Fig. 1. Microcapsules based self-healing

In this work, encapsulation of isophorone diisocyanate as a healing agent was studied. Microcapsules with isophorone diisocyanate core and single or double-layered shell were synthesized using *in situ* polymerization in an oil-in-water emulsion. The single-layered shells of the microcapsules were formed by poly(melamine-formaldehyde) (MF), poly(urea-formaldehyde) (PUF) or polyurea (PU) (Fig. 2, 4).

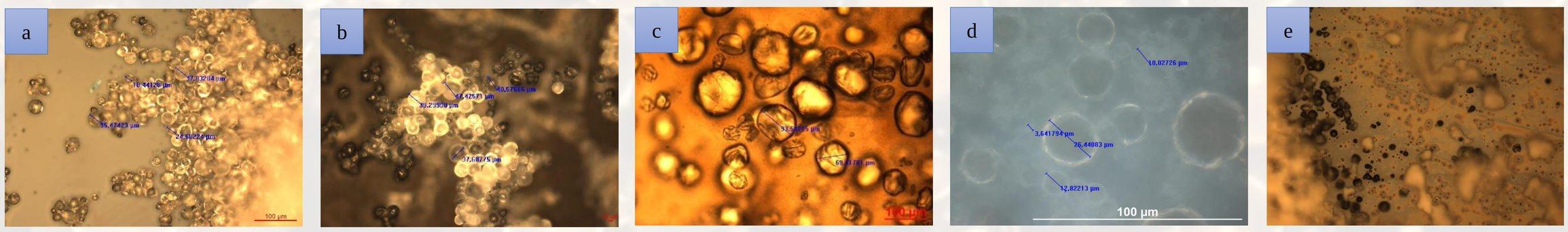

Fig. 2. IPDI capsules with PU shell (1 layer) (a) , PU-PUF shell (2 layers) (b) , PMF shell (c) , PUF shell (d) , damaged capsules (e)

Microcapsules with double-layered shells were synthesized by forming polyurea/poly(urea-formaldehyde) envelopes (Fig. 2, 3). The microcapsules were prepared in high yields (45–78 %). The effect of the reaction time, type and concentration of stabilizers on size and properties of the microcapsules was evaluated. Size distribution and surface structure of the microcapsules were evaluated by optical microscopy. Using gumarabic as a stabilizer, stable microcapsules with single layered shells and size in the range of 10–100 µm were obtained. Using poly(ethylene-maleic anhydride) copolymer as a stabilizer, double layered poly(urea-formaldehyde)/polyurethane microcapsules with the size of 30–60 µm were prepared. Thermal properties of the microcapsules were evaluated by TGA and DSC analysis. Successful encapsulation of isophorone diisocyanate was confirmed by FTIR spectra.

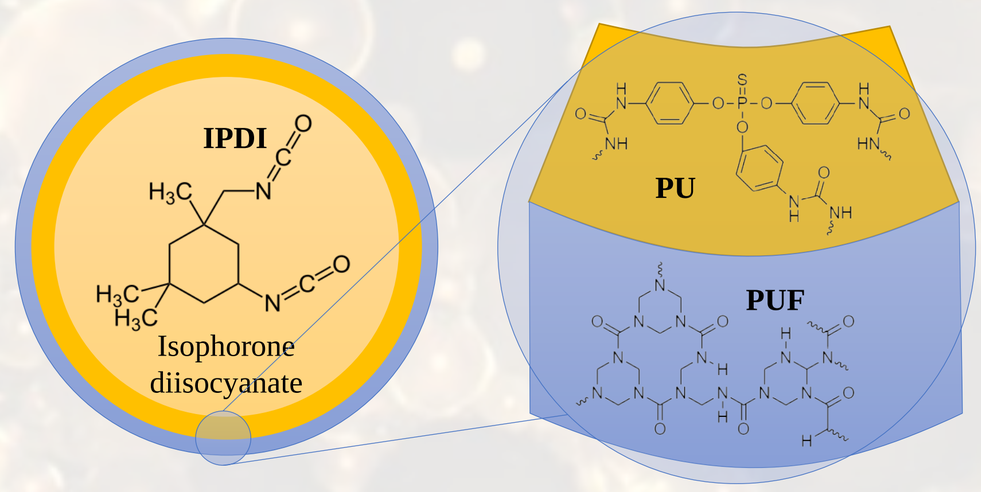

Fig. 3. Structure of IPDI-core microcapsules with double-layered shell

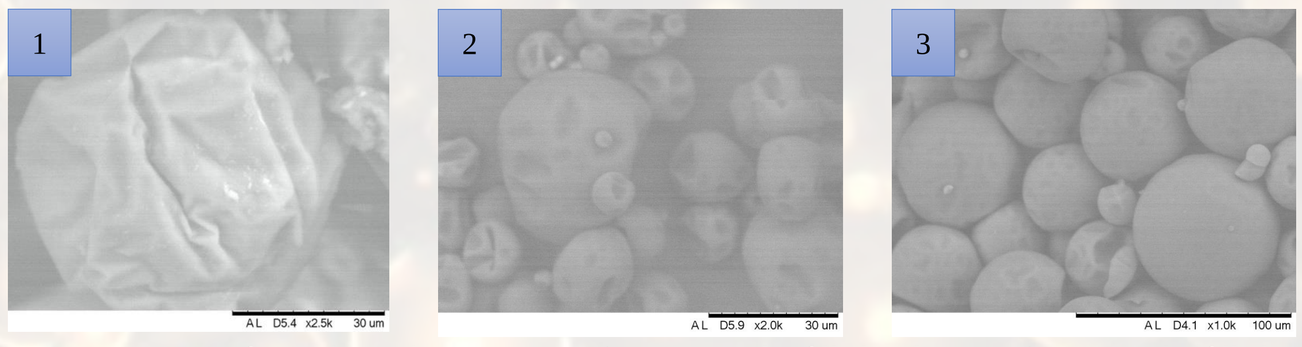

Fig. 4. SEM images of capsules with isocyanates: 1) PMF capsules filled with IPDI; 2) PU capsules filled with IPDI 3) PUF capsules filled with TDI/ethyl acetate.

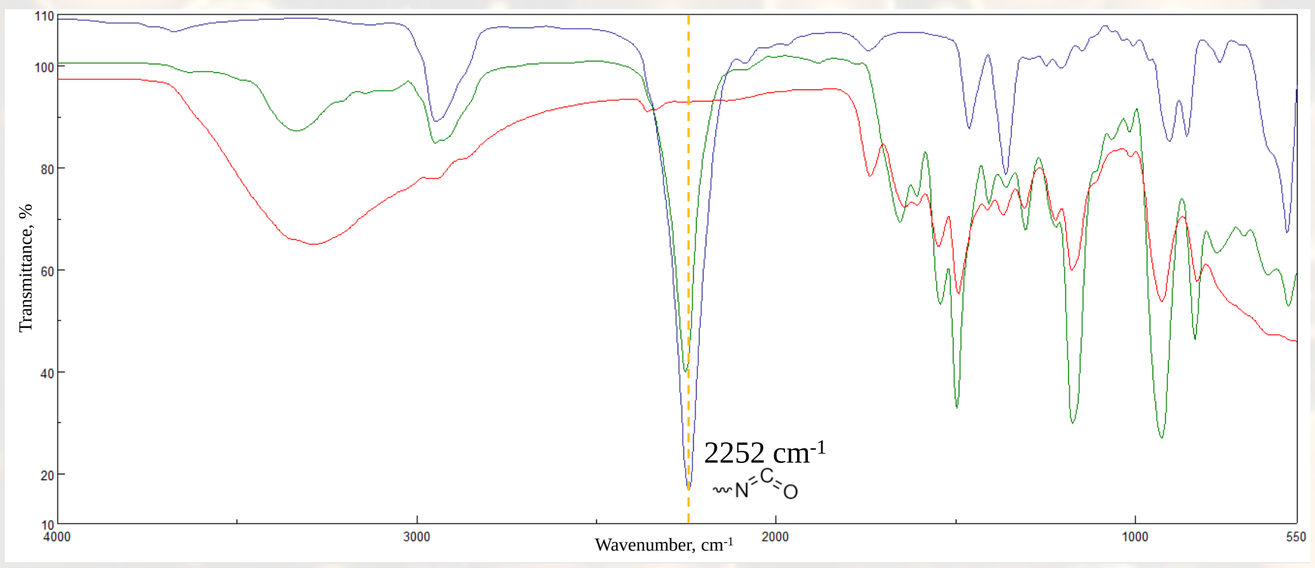

Fig. 5. FTIR spectra of microcapsules, healing agent and shell material. RED – spectra of PU shell polymer; BLUE – pure IPDI healing agent; GREEN – PU microcapsules filled with IPDI.

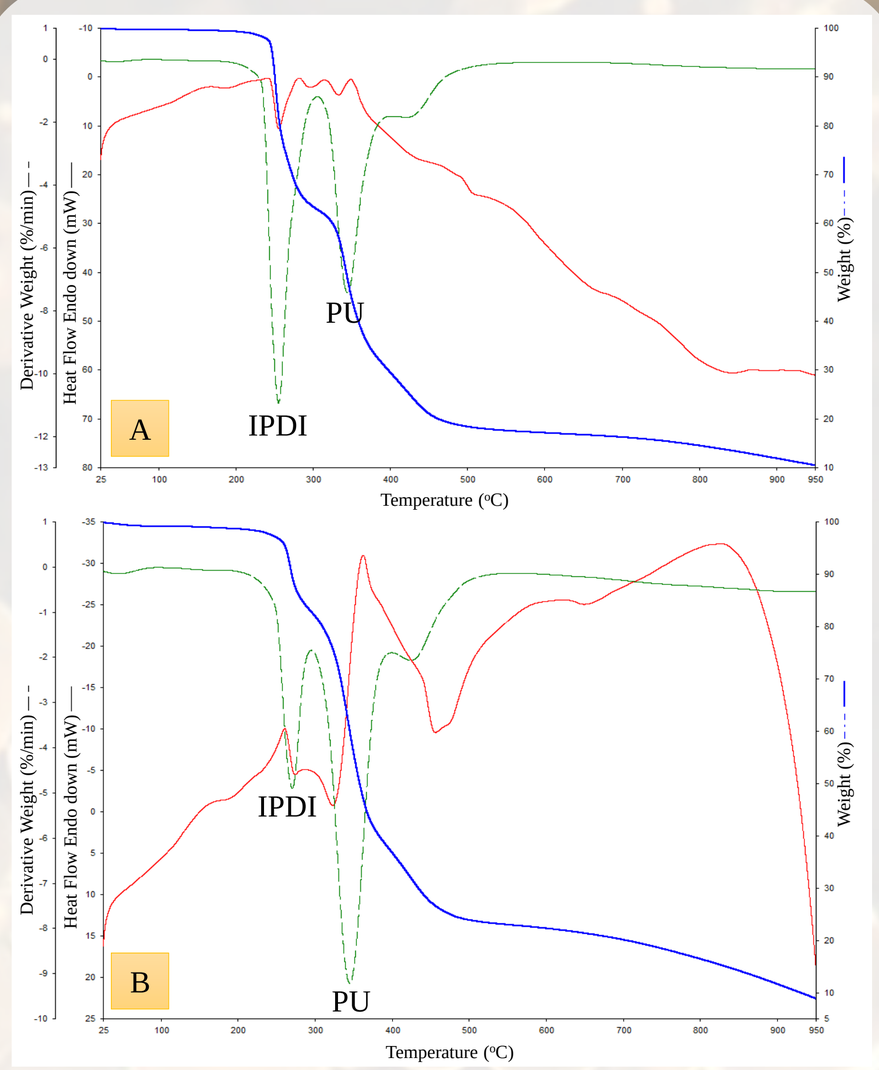

Fig. 6. DSC and TGA curves of IPDI filled capsules with PU single layer shell. IPDI:PU ratio: A) 3:1; B) 2:1

References

1. D. G. Bekas, K. Tsirka, D. Baltzis, A. S. Paipetis. Composites Part B **87** (2016) 92-119.
2. M. Guo, Y. He, J. Wang, X. Zhang, W. Li. J. Appl. Polym. Sci.**137**(2019) 48478.

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