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Combining LED-driven photochemistry and Rose Bengal-anchored polymer colloids as an efficient strategy for carrying out photooxygenation process

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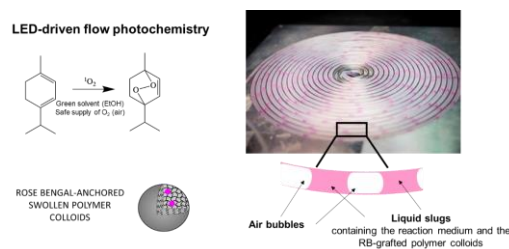
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Organic photochemistry is a key synthetic pathway for sustainable chemistry.¹ In particular, photooxygenation reactions are very attractive:² (i) they involve only singlet oxygen, a powerful selective oxidant, produced by safe photosensitization of oxygen in the visible range at ambient temperature, and (ii) they are very efficient for preparing oxetanes, hydroperoxides and endoperoxides. Their industrial implementation is limited to a few examples in the flavour, fragrance or pharmaceutical industries, for which batch reactors equipped with energy-demanding mercury light sources are generally used.³ Continuous-flow microstructured technologies, combined with LEDs, are promising alternatives to batch photochemical processing.⁴ Most of the works considers a sensitizer solubilized in the reaction medium, while the concept of solid-supported sensitizers offers many advantages: (i) the separation of the sensitizer from the other products is then easier, and (ii) it is a strategy for transposing poorly soluble sensitizers into green solvents while simultaneously enhancing their photostability.

In this context, the present work studies the reactivity of poly(N-vinylcaprolactam-co-vinyl acetate-co-vinylbenzyl Rose Bengal) microgels as heterogeneous photosensitizers in a continuous-flow process. Photoactive colloids were synthesized by miniemulsion copolymerization of vinyl acetate, N-vinyl caprolactam, divinyl adipate crosslinker and vinyl benzyl Rose Bengal monomer.⁵

Experiments were carried out in a LED-driven spiral-shaped microreactor⁶ in which “slurry” Taylor flows were generated, allowing accurate control of irradiation (emission spectrum and intensity), light absorption (tunable absorbance) and gas-liquid flow conditions (volumetric and stoichiometric ratio). The benchmark photooxygenation of α -terpinene was implemented in ethanol to provide a green solvent using air as a safe supply of oxygen.

The swollen RB-grafted colloids in ethanol were demonstrated to be efficient in converting α -terpinene into ascaridole with high conversion and high selectivity, and in short residence times (few minutes).⁷ They exhibited a reactivity similar to that of the free RB, even with a lower quantum yield. Their photo-reactivity was stable over 8 months of storage with good reproducibility. Finally, they proved to be reusable during several cycles and to decrease the RB photobleaching levels compared to free RB.



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