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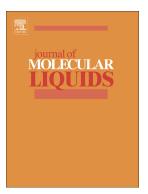
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Surfactant-free microemulsions with cleavable constituents

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Abstract

Surfactant-free microemulsions (SFME) comprising a cleavable hydrotrope/hydrophobic component were investigated regarding potential applications for green, microemulsion-mediated extraction processes. To this purpose, dynamic light scattering (DLS) experiments were performed for ternary systems consisting of water/(2,2-dimethyl-1,3-dioxolan-4-yl)methanol (Solketal)/oleic acid and water/ethanol/triethyl citrate (TEC) in order to detect mesoscopic structured compositions. Further, kinetic measurements were performed for the acid-catalyzed hydrolysis of Solketal (cleavable hydrotrope) and base-catalyzed hydrolysis of TEC (cleavable hydrophobic component) at different temperatures/different pH of the aqueous pseudo-phase. As proof of concept, α -tocopherol and vanillic acid were dissolved in the ternary mixtures as extractable surrogates in order to simulate an extraction and separation process. It was found that well-structured SFME can be obtained for both systems, which basically allows microemulsion-mediated extraction. Moderate changes of temperature and/or pH, leads to rapid hydrolysis of Solketal and TEC within a few hours. Finally, it could be shown that upon hydrolysis of Solketal/TEC, separation of the used surrogate solutes from the hydrolysed products is facilitated and quantitative separation could be achieved.

1. Introduction

Surfactant-mediated extraction is a versatile tool for the extraction of natural compounds and metal ions for analytical and industrial processes [1–3]. In this technique, profit is often taken of the formation of supramolecularly structured solutions (e.g. micelles or microemulsions) to extract and dissolve organic/ionic compounds [4]. First, the formation of a micellar solution/microemulsion takes place (direct/reverse), leading to a compartmentation of hydrophilic and hydrophobic pseudo-phases. Upon extraction, the desired products can be dissolved in the corresponding pseudo-phase, depending on the nature of the extracted compounds. Surfactant-mediated extraction comprises plenty of advantages compared to conventional solvent extraction: low amounts of surfactants are needed, compared to the amount of organic solvents used in conventional solvent extraction, the solvent properties can be tuned using different surfactants and the resulting mixtures are usually non-volatile and non-flammable [3,4]. Yet, subsequent to the extraction process, the extracted compounds have to be separated from the surfactant solution, which can be a very delicate issue. Several techniques are known to do so (e.g. the use of thermo-, salt-, pH-sensitive or photo-switchable surfactants)[4,5].

Aside of solely changing the environmental conditions of the surfactant, also the use of chemical processing methods are used comprising *e.g.* the use of cleavable surfactants [6–15]. Such molecules are amphiphiles with a weak linkage commonly, but not always, between the hydrophobic tail and the polar headgroup [11]. Prerequisite for their application is that the surfactant is stable under given conditions for the intended process. Afterwards, the surfactant properties get destroyed upon controlled cleavage of the surfactant at the designated labile bonds inducing phase separation. The mechanism by which cleavage occurs can be of different origin, including for instance acid/alkaline hydrolysis [15], UV/visible light irradiation [10,13], heat decomposition [9], oxidation [10] or enzyme-catalyzed degradation [11]. Using such methods can be desirable, especially regarding environmental

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