Quantitative monitoring of biphasic reactions using flow systems by Raman spectroscopy

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Biphasic reactions offer tremendous opportunities in industrial chemical syntheses due to the ease at which the phases and hence reagents can be separated, thereby avoiding energy intensive separation processes such as distillations. [1] A contemporary challenge presented in the implementation of biphasic reaction conditions is to determine and control the chemistry occurring in each phase, in order to improve manufacturing processes. Inline HPLC techniques for monitoring biphasic reaction monitoring have already been developed and demonstrate the potential benefits of inline monitoring in controlling and optimizing reactions. [2] In this contribution, our recent work in developing a fully automated system for monitoring changes in the composition of two immiscible phases by Raman spectroscopy using a flow sampling system is presented. This Raman/Flow system can acquire and distinguish Raman spectra of the phases present in an emulsified reaction mixture (Figure 1) and offers a key advantage in the real-time information it can deliver. The final goal is to obtain reliable real-time kinetic data on two phase reaction mixtures in which substrates in the organic phase undergo oxidation.

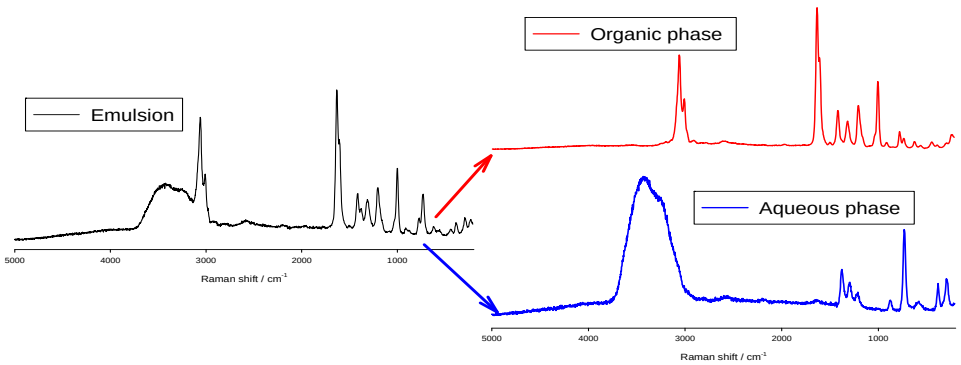

Figure 1: Separation of the Raman spectra in a styrene/H_2O_2 (3.4 % aq.) emulsion. (λ_{exc} = 532 nm (Cobolt Lasers, 200 mW at sample), 2 accumulations of 30 ms exposure using an iDus416-CCD (Andor), Shamrock163 spectrograph (Andor) coupled to a Raman probe (Avantes).

1. C. W. Kohlpaintner, R. W. Fischer, B. Cornils; Appl. Catal A-Gen 221 (2001) 219
2. W. A. Schafer, S. Hobbs, J. Rehm, D. A. Rakestraw, C. Orella, M. McLaughlin, Z. Ge, C. J. Welch; Org. Process Res. Dev. 11 (2007) 870