Off-line reaction monitoring of the oxidation of alkenes in water at low concentrations using DROP COATING DEPOSITION RAMAN Spectroscopy

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The application of drop coating deposition Raman (DCDR) spectroscopy to the field of reaction progress monitoring is addressed in this contribution. Although, DCDR spectroscopy has seen recent application in the study of biological fluids [1], its application in other areas has not yet been explored. Here we apply the technique to the catalysed oxidation of alkenes to epoxides in aqueous solutions [2] at concentrations < 10 mM. The effect of surface characteristics, background interferences, homogeneity of distribution of analytes, drying time, as well as instrumental limits of detection and calibration are discussed. We demonstrate that reproducible spectra can be obtained routinely, with relatively little variance, with short acquisition times and samples volumes of 2-10 µl and as little as 1 µg of analyte. The utility of the technique compared with online reaction monitoring by 1H NMR and Raman spectroscopy is demonstrated in the excellent correlation between data obtained off and on-line.[3]

Figure 1. Raman spectra (λexc = 785 nm) of vinylbenzoic acid (1) and the epoxide product (2) obtained on a treated surface at low concentration (sample mass ca. 3 µg)