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DOCTOR OF ENGINEERING SCIENCES

of **Vincent Pepermans**

The public defense will take place on **Friday, 5th March 2021 at 5pm.**

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DETAILED STUDY AND ENHANCEMENT OF PEAK SHAPES IN LIQUID AND SUPERCRITICAL FLUID CHROMATOGRAPHY

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Abstract of the PhD research

Liquid chromatography is an extremely powerful analytical separation technique. This holds especially true when two or more chromatographic separation modes are combined. However, such combinations often raise issues of dilution (lowering the sensitivity with which the analytes can be detected) and solvent incompatibility (causing analyte peak deformation and concomitant deterioration of the separation power). In the present work, we have used a combination of experimental and modelling work to study these problems in detail. We explored the possibilities to counter dilution by refocusing the separated peaks before entering the detector and investigated the possibility to eliminate dilution in the transfer line towards the column by directly screwing the column in the injector port. To study the problem of solvent mismatch, detailed simulations of the evolution of the peak shape during its transition through the column under the influence of a solvent gradient were made. The problem of injection mismatch is also prominent in Supercritical Fluid Chromatography (SFC), where it is impossible to use the same injection solvent as the mobile phase solvent. This often leads to peak deformations because of the difference in elution strength at the start of the column.

In addition, we also addressed the problem of collecting appropriate physicochemical data to carry out the numerical modelling simulations. One such problem occurs in SFC, an interesting complementary technique to other chromatographic modes, where little knowledge exists on the actual diffusion rates of analytes in the commonly used mobile phases containing significant fractions of organic modifiers. For this purpose, a specific measurement set-up and method has been developed that overcomes the impeding problems (system constant under high pressure, long capillary needed at constant temperature, solvent incompatibility between injected sample plug and mobile phase solvent). Using this set-up, molecular diffusion coefficients have been accurately determined for a set of relevant pharmaceutical products, producing results that are within physical expectations.