Diffusion as a Means to Determine the Variability of Adsorption Properties on HiSiV3000 Single Pellets

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The use of kinetic and equilibrium information extracted from mg-scale measurements to scale up adsorption processes is often considered questionable, due to the lack of homogeneity on commercial adsorbents from the same batch. This inhomogeneity is caused mainly by the large equipment and the inappropriate treatment of materials during manufacturing [1]. Hence, we developed a methodology for the quantification of variability and reproducibility through the measurement of the effective diffusivity of individual commercial pellets.

In this analysis, the Zero Length Column technique [2] measurements are carried out on individual commercial extruded pellets from UOP, namely HiSiV3000 (silicalite). ZLC response with ppm-level of n-Pentane as a probe molecule are used for the characterization of the effective diffusivity following the recommended experimental protocol[3]. This allows the unequivocal determination of the effective macropore diffusivity and the Henry law constant through a campaign of experiments at different flowrates, partial and full equilibration of the sample over a temperature range of 150-200˚C.

To determine intra-batch variability, a number of randomly chosen pellets are examined under identical conditions with the ZLC. By applying a simple statistical analysis such as the moving average, the stabilization of the diffusion time constant for each additional individual pellet can be monitored. When the moving average was determined to be stable, the iterative procedure was completed and the total number of pellets needed was found to be 9.

To demonstrate the validity that the values extracted above are representative of the performance for a column with a 100 times larger mass, a breakthrough column experiment under the same conditions was carried out. The breakthrough results were matched perfectly and this approach allowed also to determine the maximum deviation from the average possible, confirming that for materials of good quality the model predictions are satisfactory even in the worst possible case, i.e. the single pellet that is furthest from the average.

This study examines for the first time the intra-batch variability among a batch of pellets with the use of ZLC technique. This sequence can be applied as an indication of homogeneity for each batch during the quality control of manufactured adsorbents. It is also a further verification that the data from the ZLC technique can be used to predict accurately the behavior of a scaled-up process through a proper model implementation [4].

References

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