Treatment of Viscous Solutions

Simple Extraction and In-line Monitoring



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Successful extraction of dye from a viscous solution using a SpinChem RBR in an EasyMax 102 Advanced Synthesis Workstation.

Heterogeneous reactions involving viscous solutions put high demands on equipment and materials. As the pressure drop over a fixed bed reactor column is proportional to the viscosity of the liquid, using such a method requires powerful pumps and durable solid phase particles. While stirred tank reactors do not face the same problem, high liquid viscosity and low particle density will in this case entail inefficient reactions as well as tedious and time-consuming filtration steps to separate the two phases during downstream processing.



Figure 1. Temperature and UV-vis probes used with a SpinChem® S2 rotating bed reactor (RBR) in an EasyMax[™] workstation..

The SpinChem® rotating bed reactor (RBR) is a device designed

for heterogeneous reactions. The solid phase is kept contained as a packed bed within a rotating cylinder, through which the liquid phase is repeatedly percolated. The very efficient mass transfer achieved, together with no filtrating steps or particle grinding, allows for efficient and reproducible reactions. In this application note, blue dye was removed from a highly viscous liquid using a SpinChem® RBR S2 in an EasyMaxTM 102 Advanced Synthesis Workstation. The solid phase used to adsorb the dye was Amberlite IRA-900 Cl⁻ ion exchange resin, and the concentration of dye in the liquid over time was monitored in real-time using an in-line UV-Vis probe.

Conditions: A SpinChem® RBR S2 containing Amberlite IRA-900 Cl⁻ (10 g), was rotated at 800 rpm in a viscous solution (150 mL) containing a blue anion dye (details cannot be disclosed) at 25 °C. In-line monitoring of the decolouration process was accomplished using a UV-vis probe at 500 nm. The viscosity of the liquid was determined to be 230 cP using a viscometer.



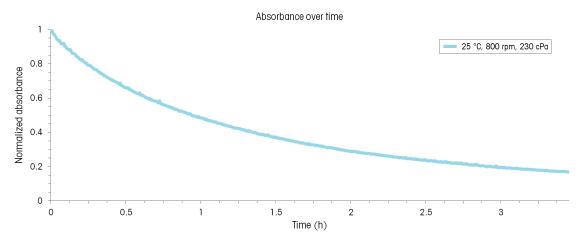


Figure 2. Adsorption of blue dye as a function of time during treatment of the viscous solution with a SpinChem® RBR S2 in an EasyMax™ 102 Advanced synthesis workstation.

Results

Initial screening of resins showed that Amberlite IRA-900 Cl⁻ proved to be suitable for the extraction of the blue dye from the viscous solution. Monitoring was abandoned after 3.5 h when the concentration of the dye reached about 10% of the initial levels (Figure 2). A completely colourless solution was obtained after approximately 24 h.

Conclusions

Monitoring of the reaction was easily recorded as no freely suspended ion exchange resin beads or resin debris interfered with the readings. This demonstrates that the RBR technology is extremely well suited for in-line monitoring. The viscosity of the solution was determined to ca 230 cP at 25°C, showing that it is possible to absorb dye even from a highly viscous solution. The absorption of the dye was very slow due to the inherent kinetics of viscous solutions. The corresponding absorption in water takes minutes rather than the hours seen here. The set-up can be used to treat solutions of even higher viscosity.



SpinChem® S2 RBREfficient rotating bed reactor for 100-500 mL reaction vessels

EasyMax[™] 102 Advanced Robust synthesis workstation for high R&D productivity

Process Analytical Technology EasyMax integrate seamlessly with real-time in situ probes



SpinChem® S2 RBR in EasyMax 102 Advanced glass vessel

Adsorption extraction in viscous media:

- Easy in-line monitoring
- No filtration and no bead attrition
- Quick and easy work-up

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For more information

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