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Column chromatography for separation and fractionation of flavor-active esters on hydrophobic resins and simulation of breakthrough behavior

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Abstract

For simulating an adsorption/elution step for separation and recovery of flavor-active esters in beer in the presence of ethanol at various temperatures, and validating the predicted breakthrough behavior, equilibrium data on concentration of each ester is required. This work evaluates the application of frontal analysis method (FA) for prediction of breakthrough behavior for adsorption of ethyl acetate, and determination of equilibrium concentrations and binding capacity for competitive adsorption of four major flavor-active esters in beer (i.e. ethyl acetate, isopentyl acetate, ethyl 4-methylpentanoate, and ethyl hexanoate), together with improvement of the obtained results, through fraction collection, and offline analysis, on columns packed with hydrophobic resins, Amberlite XAD16N and Sepabeads SP20SS. Single-component adsorption of ethyl acetate reveals a shorter breakthrough time, and higher slope of breakthrough curve for adsorption on SP20SS, due to smaller particle size, (50-100 μm), and enhanced mass transfer characteristics of this resin. Competitive frontal analysis tests, neatly demonstrate that increase in temperature is not favorable for adsorption but aids the elution step, 63-100% recovery of flavors at 333.15 K in comparison to 40-80% recovery at 298.15 K. Lower binding capacity of esters and shorter adsorption/elution cycle time is achieved at higher ethanol concentration and cyclic operation simulated under non-isothermal condition, exhibit higher accuracy between predicted and experimental breakthrough curves for XAD16N. A cyclic operation is simulated, for a larger scale column, for two scenarios, separation of ethyl acetate and complete separation of all flavor-active esters in the mixture. For more detailed prediction of breakthrough behavior, the influence of other components present in process streams needs to be investigated on competitive adsorption of esters.

Keywords: Flavor-active esters, Adsorption, Frontal Analysis, Fractionation, Simulation

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