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Chromatography at high viscosity

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Costs associated to chromatographic separation hinder implementation in purification processes in a wide range of industries. On one hand, large food process streams containing valuable complex molecules in low concentration are not fully utilized; processing and auxiliary costs could be reduced if process stream size is reduced. On the other hand, concentrated viscous streams are often diluted prior to chromatographic processing. The added water needs to be removed later on in energy intensive processes. Both situations have sparked interest of researchers and industry in high viscosity chromatography. Minimizing process streams is advantageous for the size of equipment, but leads to higher viscosities which will decrease mass transfer and increase pressure drop. The influence of feed viscosity on separation performance has largely been ignored in literature and practice. The objective of this research was to investigate separation performance as a function of viscosity for food type streams. Benefits due to decreased stream volume and disadvantages due to increased viscosity were evaluated, aiming to find maximum productivity (gproduct/m^3resin hour). Separation performance was evaluated for a range of tracers in a preparative lab scale system using a size exclusion resin for different viscosities. Viscosity was increased using sucrose. For comparison either linear velocity or pressure drop over the column bed were kept constant. Mass transfer models were applied to account for observed effects on column efficiency and peak shape due to viscosity. These models were used to predict the influence of viscosity on productivity. The results are especially relevant for industries other than pharmaceuticals, where main driver for processes development is cost reduction.

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Determination of ochratoxin A in food samples by liquid chromatography/electrospray ionization triple quadrupole-MS-MS spectrometry

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Ochratoxin A (OTA) is the most important compound in a group of structurally related mycotoxins. It is a potent carcinogen in rats and possibly associated with human kidney diseases in certain countries. OTA is a moderately stable molecule that will survive to some extent most food processing (e.g. boiling, baking, roasting or fermentation) and may thus occur in consumer products. Therefore, food chemistry branch, Northeast Regional Laboratory of FDA is monitoring OTA in a number of food products. We have established a method by HPLC-ESI-MS-MS for the analysis of OTA. By engaging SRM (selective reaction monitor) mode mass spectrometry for four OTA most distinguish and strong ions m/z 404, 386, 358 and 239, not only has the specific identification power against the method by HPLC-fluorescence detector, but also this method can reach the sensitivity of 1 parts per billion concentration (ppb) with injection volume 8 µl equivalent to 8 pg on column for quantification analysis of OTA.

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