Extraction from vegetable oil by sorption assisted by CO₂ : critical analysis of results

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In a global context of recovery of components from oily matrices, we investigated herein a process of sorption assisted by CO_2 in which a vegetable oil was injected in a flow of CO_2 that fed a tubular vessel filled with aluminium oxide as sorbent¹. The sorbent was selected among other materials for its sorption capacity towards the free fatty acids (FFA) that were the components to remove from the oil, namely a walnut oil at 6% of FFA content. In standard conditions, CO₂ and Oil flows were of 11g/min and 0.85 ml/min which means a CO₂/oil ratio of 19 g/g or a composition of 99.6 mol% in CO₂. Oil and sorbent were kept at 50°C and the pressure was of 11 MPa. The breakthrough curve of the oil was monitored by regularly collecting fractions that were analyzed for their FFA content so the breakthrough curve of the FFA was built as well (curve reported in the graphical abstract). 20 g of oil were deacidified at FFA content below 1 %, and as more oil was injected, the sorbent got enriched in FFA, and ultimately, saturated so the collected oil had the same FFA content as the injected oil. Unfortunately, when pure oil was processed, i.e. with no CO_2 , the breakthrough curve of FFA was about the same as the one obtained in presence of CO₂. From literature survey for CO₂-oil phase equilibria and its modeling (J-N. Jaubert, France²) it was assumed that the injected oil and CO_2 was not completely miscible. To solve that aspect, the oil was diluted with propanol 2 but again, the FFA breakthrough with or without CO₂ were almost alike. Another cause is the probable development of a two phases flow when the oil merged with the CO₂ in the mixing capillary, a segregation that was kept during the progress within the sorbent bed. At the end of the sorption step, a de-oiling protocol allowed the recovery of two by-products: an oily concentrate of FFA and a free-flowing beige sorbent due the sorption of coloring compounds.



¹ Financial support : Ministère de l'agriculture, de l'agroalimentaire et de la forêt (Projet DEACOL 2017 – 2019)

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