PhD thesis abstract

Epoxidation of canola oil in the presence of heterogeneous catalysts

mgr inż. Kornelia Malarczyk-Matusiak

supervisor: prof. dr hab. inż. Eugeniusz Milchert
auxiliary supervisor: dr inż. hab. Robert Pełech

Research on the process of vegetable oils epoxidation has been conducted for several decades. In industrial applications the epoxidation processes are carried out using carboxylic peracids. Those are synthesized in separate reactors or formed *in situ* in a reaction of formic or acetic acid with hydrogen peroxide, in the presence of a mineral acid (usually sulfuric acid). However, the use of mineral acids prevents obtaining high selectivity of transformation of ethylenic unsaturation to oxirane rings, as it intensifies side reactions of the opening and isomerisation of the epoxide ring. These disadvantages can be reduced by carrying out processes in the presence of heterogeneous catalysts.

This dissertation includes research on the epoxidation of canola oil in the presence of titanium-silicalite materials and acidic ion exchange resins.

The literature review covers the issue of epoxidation of vegetable oils using various methods. It also contains characteristics of vegetable oils: structure, properties, market, production and applications.

The experimental part is composed of four main stages. The first involves chromatographic analysis of canola oil used in the research.

In the second stage, the usefulness of various methods and catalysts in the epoxidation of canola oil was tested. The investigation results of epoxidation with hydrogen peroxide and *tert*-butyl hydroperoxide in the presence of titanium-silicalite materials (TS-1, Ti-MWW, Ti-MCM-41) and the results of epoxidation with performic and peracetic acid, obtained *in situ* in the presence of acidic ion exchange resins (Amberlite IR-120, Amberlyst 15, Dowex 50WX2) were presented. The values describing the course of processes were: conversion of ethylenic unsaturation, selectivity of transformation of ethylenic unsaturation to oxirane rings and yield of epoxidized canola oil. Particular attention was paid to the determination of the process parameters at which epoxidized canola oil was obtained with the highest selectivity at favourable values of the other process evaluation values. The product obtained in the epoxidation process was characterized by the determination of iodine and epoxide number. As a result of the studies, the method of using the peracetic acid and Dowex 50WX2

was considered the most advantageous method of conducting epoxidation. This system was chosen for further research.

Using the method of one variable the effect of technological parameters such as: temperature, molar ratio of hydrogen peroxide to unsaturated bond, molar ratio of acetic acid to unsaturated bond, amount of catalyst, time and stirring speed on the course of the epoxidation process was examined.

The main aspect of this dissertation was to optimize the epoxidation process of canola oil, based on the mathematical method of experimental design. This optimization allowed to obtain a mathematical dependence describing the influence of process conditions: temperature, molar ratio of hydrogen peroxide to unsaturated bond, molar ratio of acetic acid to unsaturated bond and reaction time on conversion of ethylenic unsaturation, selectivity of transformation of ethylenic unsaturation to oxirane rings and yield of epoxidized canola oil. The maximum values of the examined functions and the optimal technological parameters were established.

Kornelia Malarczyk - Matusiak
10.12.2018 r.