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Abstract - Dissertation entitled

"EPOXIDATION OF ALLYLIC COMPOUNDS OVER THE Ti-SBA-15 CATALYST"

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In the first step of this work, a method of the synthesis of the mesoporous titanium silicate Ti-SBA-15 catalyst with a different molar ratio of Si/Ti was developed. The obtained material had the structure of a SBA-15 type and had appropriate physicochemical properties. The developed method of the synthesis of this mesoporous material was very repeatable. The full characteristic of the obtained Ti-SBA-15 catalyst (with a different molar ratio of Si/Ti) was performed using various instrumental methods: XRD, UV-Vis, IR, SEM, X-ray microanalysis and sorption studies.

In the next stage of this studies, it was chosen the sample of the Ti-SBA-15 catalyst (which was characterized by the most favorable physicochemical properties and having the appropriate structure) for the studies on the influence of technological parameters on the course of the epoxidation at the atmospheric pressure the following allylic compounds: allyl alcohol, methallyl alcohol, crotyl alcohol and 1-butene-3-ol. During the examinations the influence of such technological parameters was in detail studied: temperature, the molar ratio of allylic compound to hydrogen peroxide, the solvent concentration, the type of solvent - polar protic solvent (methanol and ethanol) and aprotic (acetone and acetonitrile), the Ti-SBA-15 catalyst content, the reaction time and the intensity of stirring. The process was also carried out without addition of any solvents. The main functions describing the process were: the selectivity of transformation to epoxy compounds in relation to allylic compound consumed, the conversion of allylic compound, the conversion of hydrogen peroxide and the selectivity of transformation to organic compounds in relation to hydrogen peroxide consumed. A special attention was paid to the determination such process parameters at which the epoxy compound can be obtained with the highest selectivity and simultaneously at the high conversion of the organic raw material. All products obtained during the epoxidation of allylic compounds were analyzed with help of the mass spectrometry coupled with the gas chromatography (GC-MS), whereas the quantitative

composition of the post-reaction mixtures were established after analyses with the following methods: the gas chromatography, the iodometric titration method (unreacted hydrogen peroxide), and the potentiometric method – establishing of glycerol (only for the epoxidation of allyl alcohol).

In the next stage of the studies, it was conducted the optimization of the process of allyl alcohol epoxidation over the Ti-SBA-15 catalyst (in an aqueous medium and with addition of the polar protic solvent - methanol) using the mathematical method of experiments planning (according to the rotatable-uniform design). The process which underwent in the aqueous medium was optimized using four variables: temperature, the molar ratio of allyl alcohol to hydrogen peroxide, the Ti-SBA-15 catalyst content and the reaction time. Whereas the process which underwent in the presence of methanol as the solvent was optimized using four independent factors (listed above), plus the fifth - the solvent concentration.

The next stage of the studies relied on examinations the possibility of the separation of the epoxy compound (glycidol) from the post-reaction mixtures using the thin film distillation method. As a result of the performed distillations the epoxy compound was obtained with the high purity - about 95.8%.

Next, the regeneration of the titanium silicate Ti-SBA-15 catalyst after the process of epoxidation of allyl alcohol with 30 wt% hydrogen peroxide in methanol as solvent was carried out. The studies were carried out at the most favorable conditions established previously for this process. The studies showed that the Ti-SBA-15 catalyst was active catalyst in V stages of examinations (in 30 steps of AA epoxidation).

Moreover, for comparison also the regeneration of the „older" titanium silicate catalyst - Ti-MCM-41 - was carried out. The comparison showed that the Ti-MCM-41 catalyst is characterized by a much poorer durability than the Ti-SBA-15 catalyst.

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