Synthesis New of Epoxy Ethers on Basis of Cyclic Dioximes


Institute of Chemical Problems named after acad. M.F. Nagiyev of Azerbaijan National Academy of Sciences, Baku, H.Cavid-29 elia_58 @ mail.ru

In spite of great variety of epoxy compounds, the synthesis of new epoxyethers with complex of useful properties is of not only theoretical, but also practical interest, so far as they are successfully used as stabilizers, plastifiers, emulsifiers, bactericides, foaming agents foam plastics, lubricants and facilities for dyeing. Earlier we have carried out investigation on the synthesis of epoxy ethers of alicyclic raw by interaction of alcohols, acids, oximes, amins and aminacids with epichlorhydrine by following transformations of the obtained chlorohydrins into epoxy ethers [1-4]. The work was devoted to the synthesis of epoxyethers on the basis of epichlorhydrine and cyclic dioximes. The cyclohexanediondioxime – 1,2 (nioxime) and cycloheptanediondioxime -1,2 (heptoxime) were taken as initial oximes. Condensation reactions of ECH with cyclic oximes were carried out with the presence of catalyst BF3\(\cdot\)O(C2H5)2 by scheme:

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\begin{align*}
\text{CH}_2\text{O} + \text{Cl} - \text{CH}_2 - \text{CH} - \text{CH}_2 & \xrightarrow{\text{BF}_3\cdot\text{O(C}_2\text{H}_5)_2} \text{C} = \text{N-O-CH}_2\text{-CH(OH)-CH}_2\text{Cl} \\
\text{C} = \text{N-OH} & \xrightarrow{\text{KOH}} \text{C} = \text{N-O-CH}_2\text{-CH-CH}_2 \\
\text{C} = \text{N-O-CH}_2\text{-CH-CH}_2 & \text{C} = \text{N-O-CH}_2\text{-CH-CH}_2
\end{align*}
\]

where, \(n = 4;5\)

Reactions were carried out at medium of sulphuric ether at catalysis by boron fluoride etherate 0,01% on correlation to epichlorohydrine. Duration of reaction is 1-2 hour. In the second stage of reaction the obtained chlorohydrins of oximes were transformed to epoxyethers by dehydrochlorination under the action of caustic alkalis potassium. Regulating varying process of initial components it is possible to obtain mono- and diepoxy ethers. Preliminary experiments, which carried out with oxims show, that the atom of hydrogen in them is more mobile and inclined to quitting, than in alcohols. That is caused by structural peculiarities of oxims. In consequence of presence of double bond and atom of nitrogen in structure, the displacement of electron density from atom of hydrogen of hydroxyl group takes place, which accelerates joining of epichlorohydrine. However, it must be noted, that at high temperatures and under the influence of selected catalyst the oxims are subjected to decomposition and Beckman regrouping. While choosing optimum conditions 30-35°C reaction temperature was selected.

Composition and structure of synthesized compounds were established by NMR- and IR-spectroscopy, but purity of epoxyethers by GLC analysis. Synthesized epoxide compounds can be wed as synthetic fibres in textile industry.

REFERENCES