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Chemistry

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Fig. 1

Figure 1: Fig. 1

Abstract**Full Text****Chemistry****N. M. SEIDOV, Academician of the Academy of Sciences of the Azerbaijan SSR, M. A. DALIN, S. M. KYAZIMOV****PREPARATION OF AN ETHYLENE-BUTYLENE ELASTOMER IN A LIQUID BUTYLENE MEDIUM**

In recent years, copolymers of ethylene with α -olefins, possessing the properties of plastics and elastomers, have acquired great importance. The literature contains extensive material on the synthesis of ethylene-propylene elastomer (SKEP) in the presence of organometallic catalysts, in various solvents, including a liquid propylene medium.

Fig. 1

Together with this, research in the field of copolymerization of ethylene with butene-1 is of great interest. This reaction has been studied mainly for modifying the properties of polyethylene (¹⁻³). As for the preparation of ethylene-butylene synthetic rubber (SKEB), works on this question have been published in the literature, chiefly on copolymerization in various inert solvents (⁴⁻⁷).

It is known that ethylene in polymerization reactions on organometallic catalysts has a higher reactivity than butene-1. This makes it possible to carry out the copolymerization of ethylene in a liquid butylene medium without the use of special solvents. Such a method for conducting copolymerization has been described in the literature only recently (^{8, 9}).

In the present article, the first results are presented of a study of the copolymerization reaction of ethylene with butene-1 for the purpose of obtaining elastomers.

Ethylene of a high degree of purity was used as the feedstock. Butene-1 was obtained by dehydration of *n*-butyl alcohol over an aluminum oxide catalyst poisoned with 0.25 KOH, and had a concentration of 97-98%. Copolymerization was studied in the presence of the catalytic system $[VCl_4 + (iso-C_4H_9)_2AlCl]$.

Table 1

| No. | Experimental temperature, °C | Pressure, atm | C ₂ H ₄ content in liquid phase | [η] | Copolymer yield, g/g VCl ₄ | Reaction rate, g/g VCl ₄ per hour |
|-----|------------------------------|---------------|---|-----|---------------------------------------|--|
| 1 | 30 | 6 | 5 | 1.3 | 280 | 600 |
| 2 | 30 | 6.5 | 7.5 | 1.7 | 385 | 640 |
| 3 | 30 | 8 | 10 | 2.4 | 420 | 1000 |
| 4 | 30 | 11 | 15 | 3.2 | 640 | 2500 |
| 5 | 30 | 17 | 25 | 3.4 | 1200 | 3000 |
| 6 | +0 | 4 | 10 | 2.9 | 800 | 2400 |
| 7 | +60 | 10.5 | 10 | 1.9 | 150 | 300 |

The catalyst (VCl₄) and cocatalyst (iso-C₄H₉)₂AlCl were used in the form of a 5-7% solution in purified benzene. Copolymerization was carried out in an apparatus with an autoclave capacity of 2 l. One of the principal factors influ-

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Fig. 2

Fig. 3

...affecting the copolymerization process is the composition of the liquid phase. By changing the composition of the liquid phase, a copolymer with different butylene content in the copolymer chain can be obtained.

The results of the experiments show that, as the ethylene content in the liquid phase increases, the reaction rate and the yield of copolymers increase (Table 1). As can be seen from the data in this table, with increasing temperature the yield and molecular weight of the copolymer decrease.

By determining the ethylene and butylene content in the copolymer chain from the IR spectrum, it was possible to establish the dependence of the copolymer composition on the ratio of ethylene to butylene in the liquid phase (Fig. 1).

With increasing butylene content in the copolymer, the crystallinity of the copolymer decreases.

Figures 2 and 3 show X-ray diffraction patterns of two samples of ethylene-butylene copolymer containing 10 and 33.5% butylene in the copolymer chain. The first sample (Fig. 2) contains 36% crystalline phase, while the second is amorphous. The latter dissolves completely in boiling *n*-heptane.

On the basis of the copolymers obtained, rubber compounds were prepared and vulcanized with dicumyl peroxide. It was observed that, with increasing butylene content in the copolymers, their processability improves; moreover, they mill well and mix with ingredients. Vulcanization was carried out at 160° for 60 min. The data obtained are shown in Table 2. IR spectra of the ethylene-butylene copolymer are shown in Fig. 4.

Fig. 4

Table 2

| Properties | Butylene content in the copolymer chain, mol. % | Butylene content in the copolymer chain, mol. % | Butylene content in the copolymer chain, mol. % |
|---|---|---|---|
| Tensile strength, kg/cm ² | 33.5 220 | 18 165 | 14 135 |
| Relative elongation, % | 530 | 650 | 600 |
| Residual elongation, % | 12 | 130 | 140 |
| Rebound elasticity, % | 51 | 42 | 40 |
| TIR hardness | 62 | 78 | 58 |

As can be seen from the data in Table 2, the vulcanizate of the copolymer containing 33.5 mol.% butylene in the copolymer chain possesses high physico-mechanical properties.

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