Study of the possibility of synthesis of poly(copperphenylsiloxanes) by the interaction of polyphenylsiloxane with acetylacetonate copper under conditions of mechanochemical activation

© Alevtina A. Kapustina,⁺ Nikolay P. Shapkin,*

Evgenia A. Merkulova, and Vitaly V. Libanov

Department of General, Inorganic and Elementoorganic Chemistry. Far Eastern Federal University. Oktyabrskay St., 27. Vladivostok, 690090. Russia. Phone: +7(914) 32-25-291. E-mail: kapustina.aa@dvfu.ru

*Supervising author; ⁺Corresponding author Keywords: polycopperphenylsiloxanes, polyphenylsiloxane, mechanochemical activation.

Abstract

The interaction of polyphenylsiloxane (PPS) with copper acetylacetonate under the conditions of mechanochemical activation in a planetary boll mill "Pulverisette 6" was used. The steel balls, 0.8 cm in diameter, were used as a packing. The ratio of the mass of the nozzle to the mass of the payload was 1.8. The reaction mixtures are separated into soluble and insoluble fractions by extraction with toluene at room temperature. Soluble in toluene policopperphenylsiloxanes (PCPS) with a copper content of 3.2% to 3.5% were obtained. It is shown that the Si/Cu ratio obtained in the PCPS differs from the preset (1:1) and is 9.47 after 3 minutes of activation and 9.72 at 5 minutes of activation. The composition of the obtained products was studied by the methods of elemental, X-ray phase analysis, gel-permeation chromatography, IR spectroscopy. A comparison was made of the mass fractions and composition of soluble PCPS obtained in a planetary boll mill and an activator of the vibration type. It is shown that the mass fractions of soluble fractions are practically independent of the type of activator. The content of copper in soluble PCPS obtained in a more energy-intensive planetary mill is higher than in polymers synthesized in a vibration type activator. The relative molecular weights of the soluble fractions are greater than or equal to 5000. The polymer character of the soluble fractions is confirmed by X-ray diffraction analysis. Two wide halos, characteristic of amorphous polymeric substances, were observed on the diffractograms. The assumption is made of the presence in the structure of soluble PCPS bridged acetylacetonate groups that bind copper atoms in the side chains.

It is shown that insoluble fractions are unreacted copper acetylacetonate with a small amount of polyphenylsilsesquioxane impurities.

References

- [1] A.A. Kapustina, N.P. Shapkin, N.I. Gavrilova, M.Yu. Kalugina, V.I. Bessonova. Synthesis of polycuproorganosiloxanes with mechanochemical activation. Russian Journal of General Chemistry. 2000. Vol.70. Iss.2. P.241-243.
- [2] N.P. Shapkin, A.A. Kapustina, S.V. Gardionov, I.G. Khalchenko. Interaction of Polyphenylsiloxane with Magnesium Acetylacetonate. Russian Journal of General Chemistry. 2015. Vol.85. P.1487-1490. DOI: 10.1134/S1070363215060225
- [3] J.F. Brown, Jr.L.H. Vogt, P.J. Presscott. J. Amer. Chem. Soc. 1964. Vol.86.P.1120-1125.