

21. On the Properties of Polyvinylacetals Containing Sulphuric Acid.

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The polyvinylacetals containing combined sulphuric acid were obtained by the acetalization of polyvinylalcohol using sulphuric acid as catalyst, and the combined acid in these products was also known to decrease the stability of the acetals. By the utilization of these unstabilities we could manufacture the insoluble or less-swelling films from these acetals by the baking treatment at relatively low temperature or for considerably short time.

Polyvinylacetals were prepared from polyvinylacetate (its degree of polymerization ca. 4,000) by the direct method using conc. HCl as solvent and H₂SO₄ as catalyst. The compositions of the products were as follows; degree of acetalization ca. 60%, contents of acetyl radicals ca. 3%, and sulphuric acid combined ca. 0-1.5%. All products of polyvinylacetoacetal before baking were easily soluble in methanol, ethanol, butanol or acetone. After baking at 80°C, 110°C and 130°C respectively, the products became insoluble in these solvents, and also the films obtained by the baking had the same luster as unbaked one.

Also with polyvinyl formacetal we could recognize that the swelling properties toward such a solvent as furfural, transformer-oil or dichlorethane were largely improved, but toward benzene, ethanol or water were not affected.

In conclusion, the polyvinylformal, having high degree of formalization, no acetyl groups and 0.2-0.5% combined sulphuric acid was found to be suitable for the wire insulation-coating from the standpoint of the oil-resistance.

22. Studies on the Reactivity of Vinyl Compounds. (IV)

Reactions of Vinyl Ethyl Ether and Vinyl Acetate with Carbazole.

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a. Reaction of Vinyl Ethyl Ether with Carbazole.

Carbazole was suspended in such solvent as ether, benzene and xylene in a reaction vessel cooled with ice-water, and some drops of BF₃-etherate were added. To this mixture, with stirring, a little more than equimolecular quantity of vinyl ethyl ether diluted with solvent was added little by little. A rapid and exothermic reaction occurred. In progress of the reaction, the mixture became transparent, yellow and fluorescent. After all the carbazole was consumed, potassium carbonate was added to stop the reaction. On fractionation yellow sticky product was obtained with good yield (B.P. 178-180°C/7mm, m. p. 65-66, mol. weight (obs.) 240). On cooling and agitating it solidified to a white rhombic crystal, when cold it