ENCLOSURE (B) 34

STUDIES ON HIGH FREQUENCY INSULATING MATERIALS

by

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SUMMARY

In order to obtain isobutylene polymers as insulating materials, studies on the polymerization of isobutylene at low temperature in the presence of aluminum chloride were carried out. Isobutylene was prepared by the dehydration of tertiary butyl alcohol in the presence of anhydrous oxalic acid followed by precise distillation. Liquefied isobutylene was polymerized in liquid ethylene at (-)150°C in the presence of an ethylchloride solution of aluminum chloride and a vigorous reaction took place immediately, forming a polymer of 170,000 average molecular weight.

I. INTRODUCTION

A. History of Project

In April 1944, polyisobutylene for use as a high frequency insulating material was requested by the Second Naval Technical Depot.

According to the published literature the author studied the polymerization of isobutylene at (-)150°C in the presence of an ethyl chloride solution of aluminum chloride was carried out and a polymer, having an average molecular weight of about 170,000 was obtained in September 1944.

B. Key Personnel Working on Project

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II. DETAILED DESCRIPTION

An attempt was made to prepare an isobutylene polymer at low temperature in accordance with U. S. Patents. In the patent, BF_3 had been used mainly, but in our experiments aluminum chloride was used, since the supply of BF_3 was scarce in Japan. As a diluent, liquid ethylene was used to avoid the rapid rise in temperature due to the violent polymerization of isobutylene, and aluminum chloride was dissolved in ethyl chloride in order to make the reaction uniform and as a protection from the humidity. Isobutylene was prepared by the dehydration of tertiary butyl alcohol in the presence of anhydrous oxalic acid. After redistilling by the Podbilniak distillation apparatus, the isobutylene was stored in a 20 liter glass bottle. Ethylene was prepared by the dehydration of ethyl alcohol in the presence of an alumina catalyst and stored in a pressure bomb. Ethyl chloride was obtained in the market. A 30 gram sample of isobutylene was taken in a 1000cc beaker and diluted with 35 grams of liquid ethylene and cooled to (-)170°C with liquid air. Ethyl chloride solution (50cc) containing 1.5% $AlCl_3$ was diluted with 39 grams of liquid ethylene and cooled to (-)140°C. The solution of catalyst was poured into the isobutylene solution. At (-)165°C, reaction did not occur so the beaker was removed from the liquid air vessel, and at -145°C reaction took place. The solution was agitated with a resistance thermometer and at -100°C, a small quantity of ethyl alcohol was added and reaction was stopped. The white spongy material, (26 grams), which was floating on the surface was gathered and was dissolved in toluene. After purification by a precipitation method, the product was obtained. The average molecular weight was determined to be 170,000 by the viscosity method in normal heptane solution.

At the reaction temperature of (-)170°C, cooled by solid carbon dioxide, a polymer of 12,000 molecular weight was prepared, and at (-)100°C, cooled by

liquid ethylene, a polymer of 31,000 molecular weight was prepared.

When isobutylene, prepared from the dehydration of isobutyl alcohol by alumina catalyst at 350°C, was used as a starting material, a polymer of 80,000 molecular weight was obtained.

It was possible to obtain a high isobutylene polymer of 170,000 molecular weight, using aluminum chloride as a catalyst.

In order to develop to a commercial scale, further experiments would be necessary in order to find economical methods.