C8-123

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September 11,1989

AID-CDR Project C8-123
DEVELOPMENT OF NOVEL PRODUCTS FROM GUAYULE RUBBER

PROGRESS REPORT FOR THE PERIOD 12/88-6/89

In this report we will outline our scientific activities during the period from December 1988 to June 1989. The personnel involved in the project during this period consisted of the P.I., one graduate student (Ms. Gabriela Marchuk), and a senior technician Ms. Y. Yegen.

Our activities for the above-mentioned period involved the following:

1.Development of the necessary calibration curves for quantitative analysis of ketones and aldehydes by FTIR. This enables us to follow by IR spectroscopy the first stage of the Polyisoprene cleavage and to quantify its kinetics. It was detected that the absorption of the formed ketones and aldehydes are at the same wavelength. As result, it was necessary to generate a combined concentration - absorption curve taking into account the different proportionality constants of the two groups.

2.Development of a viscosity-molecular weight-extent of cleavage calibration curve for the keto-aldehyde terminated PIP molecules.

3.Development of an analytical procedure for the determination of diol concentration in PIP. This procedure is important for the determination of extent of conversion of ketones and aldehydes into diols- the main product of the cleavage step. The FTIR was found unsatisfactory for the quantitative analysis of diol conversion due to the very broad OH peak in the IR spectrum. Several chemical methods have been tested and a titration based method has been selected as the most appropriate for our system.

4.Kinetic study of the first stage in the cleavage procedure. The conversion at this stage translated into the amount of cleavage or average length of cleaved chain has been determined by viscometric and spectroscopic methods. The temperature was held constant at 30 degrees C and concentration of reactants was also held constant with only the reaction time being modified. The determination of the kinetic equation from the kinetic data is presently under investigation.

5.Molecular weight distribution of products of the cleavage reaction is being currently studied by GPC. These data will supplement already available kinetic data described above to give a complete picture of the kinetics of the first cleavage step.

6.Determination of the optimal conditions for the second step in the diolization of PIP. The products of the chain cleavage are subsequently converted into diols. The series of steps involved in this process has been studied and the set of conditions necessary to achieve close to 100% conversion of the keto-aldehydes has been determined. In addition, the original procedure as described in the literature has been modified enabling us to simplify the process and to obtain complete conversion in only 1 hour of reaction. This achievement is very important to the development of a practical process for generation of commercial reactive PIP.

7.Qualitative study of possible reaction schemes for PIP-PDMS copolymerization. Three different routs for the copolymerization of the PIP diols with PDMS have been tested. Reaction with chlorosilane and with silane terminated PDMS has been found to yield desirable chemical changes in reasonable extent of time. The third route involving ethoxysiloxane was unsuccessful.

After some delays in beginning their research due to technical difficulties, our Mexican collaborators have started working on the project. At this point four Mexican researchers are involved: Dr. Mario Gutierrez, Dr. Gabriel Cojon, and two students a M.S. student and one undergraduate. According to the research program they have started the study of the diolization process and the development of analytical techniques necessary for the kinetic studies. Utilizing the experience gained by the Israeli team they will be able to complete the initial stages in relatively short time and proceed with the subsequent stages of the program.

To summarize, at this point we have enlarged considerably our knowledge on the diolization step enabling us to control it to the point of obtaining the desired products by control of the reaction kinetics. We are making progress on the second step which will enable us to produce the silicone-PIP products which are the main goal of this project.

Sincerely yours,

Moshe Gottlieb
Associate Professor
Principal Investigator