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3,251,799
LACTAM POLYMERIZATION, HETEROCYCLIC PROMOTERS THEREFOR, AND PRODUCTS THEREOF

Edward W. Pietrusza, Morris Township, Morris County, and Rudolph Pinter and Jack R. Pedersen, Morristown, N.J., assignors to Allied Chemical Corporation, New York, N.Y., a corporation of New York No Drawing. Filed Sept. 11, 1962, Ser. No. 222,959 19 Claims. (Cl. 260—37)

This invention relates to process, catalysts, catalyst promoters, and products of lactam polymerization wherein the lactam ring is saturated and in addition to at least one imide group, said lactam ring consists of at least 5 additional atoms, all of which additional ring atoms are carbon atoms. This application is a continuation-in-part of our copending U.S. application Ser. No. 146,449, filed Oct. 20, 1961. Copending U.S. applications Ser. No. 185,537, filed Apr. 6, 1962; and Ser. No. 188,960, filed Apr. 20, 1962, are directed to related subject matter.

The lactams polymerized in accordance with this invention are particularly lactams as above defined bearing on the ring only hydrogen atoms, hydrocarbon subtitutents, and/or alkylene bridges between 2 lactam rings; but other substituents can also be present when they do not decompose the catalysts used or otherwise prevent

the desired polymerization. More particularly this invention relates to anionic 30 polymerization of said lactams in presence of a co-catalyst or promoter whereby the polymerization can be effected at temperatures below the softening point of the result-ing polymer, and in bulk, i.e., in absence of inert diluents. The rapidity of polymerization possible by use of our invention at temperatures below the polymer softening point, together with absence of volatile by-products, allows production of shaped articles, e.g. molded and extruded articles, directly from liquid monomer by methods such as rotational casting similar to those utilized 40 with vinyl plastisols; by extrusion or injection methods; etc. Moreover by virtue of this same rapid conversion of monomer to solid polymer, a wide variety of addi-tives can readily be incorporated in our polymers, such as fillers, pigments, reinforcing materials, plasticizers, heat and light stabilizers, blowing agents, etc., which can be dissolved or suspended in the monomer or impregnated with the monomer (woven or unwoven glass

Conditions and metal or metal compound catalysts suitable for anionic polymerization are broadly known, including conditions suitable when a promoter or co-catalyst is present, e.g., in accordance with U.S. Patents 3,015,652, 3,017,391, 3,017,392, 3,018,273, etc. Like conditions and the same metal or metal compound catalysts are applicable in our polymerization processes in accordance with this invention. In particular the temperatures used in our polymerizations are in the range from about 100° C. to about 250° C. and the metal or metal compound catalysts which we use are an alkali

fibers, etc.), the monomer being then polymerized by

our process.

group of the lactam.

The lactam moiety of our promoter preferably is saturated and has at least 7 ring members, all ring members other than the imido groups being methylene groups; such as for example the residue after removal of imide hydrogen from e-caprolactam, omega-enatholactam, omega-caprylolactam, omega-laurolactam, a dilactam such as isopropylidene dicaprolactam, etc., especially e-caprolactam.

The heterocyclic substituent attached in our promoter to the imido group of said lactam moiety can be saturated, unsaturated, or aromatic and can contain one or more further hetero atoms in the ring in addition to nitrogen therein. As above stated, at least one nitrogen atom in this heterocyclic substituent is doubly bonded to a ring carbon atom, and said ring carbon atom is tautomerically linked to the imido group of the lactam moiety. The term "tautomerically" linked has reference to the so-called "tautomeric effect"-see, for example, Hammett's Physical Organic Chemistry (McGraw-Hill, 1940) page 195; by this term we mean that the carbon atom, doubly bonded to nitrogen in the heterocyclic ring, is attached directly to the imido group of the lactam, or is attached thereto by a chain of alternating singly and doubly bonded atoms in the heterocyclic ring, the chain consisting of an even number of such atoms whereby the said carbon doubly bonded to ring nitrogen is conjugated with the imido group.

The heterocyclic ring can have a wide variety of substituents-even including substituents replaceable by the lactam anion of the lactam-metal salt serving as catalyst in the polymerization and substituents containing active hydrogen-provided sufficient metallic catalyst is used in the polymerization to provide adequate catalyst after any reaction of the catalyst with substituents in the promoter. These substituents can be halo, alkoxy, and other replaceable substituents; alkyl, aryl, alkaryl, arylalkyl and other hydrocarbyl substituents; and hydroxyalkyl, amino, amino-alkyl, etc., to the extent these do not eliminate the active catalyst. Amino groups tend to act as terminators in the polymerization. Since our promoters become the starting point of the molecular chains in our polymers, the substituents in the promoters can be chosen for effects, e.g., dyeing, stabilizing against aging, etc., which they impart to the polymer product.

A simple compound illustrative of the promoters of our invention is N-(2-pyridyl)-e-caprolactam which can be represented as a resonance hybrid of the formulas below (and others);

In this compound the lactam moiety is e-caprolactam, and the heterocyclic substituent is the 2-pyridyl radical. It will be observed that this pyridyl ring in the first formula contains one nitrogen atom doubly bonded to a ring car-

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