Synthesis and Analysis of poly (1, 2, 4-oxadiazole)

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Abstract: Polyamide hydrazide is synthesized from IPC and BABH polyamide hydrazide is analysed by X-ray diffraction to show crystalline in polymer. Thermal properties by TGA. Synthesis of poly (1,2,4 triazole)s from orthophosphoric acid P₂O₅, polyhydrazide and aniline analysis of poly(1,2,4-triazole)s by IR spectrum absorption bands of triazole ring breathing vibrations with disappearance of hydrazide bands. Synthesis of poly (1,2,4 oxadiazole)s from poly (amide hydrazide ) and PoCl₃. Conversion of hydrazide group into 1,2,4 oxadiazole ring is monitored by FTIR.

Keywords: Polyhydrazide, Polyamide hydrazide, Polyoxadiazoles, Poly (1,2,4-Oxadiazole)s.

I. Introduction

Preparation of Polyhydrazide: A preferred technique for high molecular weight polyhydrazides containing the \(-R\text{CONHNHCOR}_2\) group is low temperature solution polycondensation under anhydrous conditions. A diacid chloride is allowed to react with the dihydrazides in amide solvent that functions as both acid accepter and solvent for the polyhydrazide.

Application of Polyhydrazides:
1. Polyhydrazide fibers having high tensile properties are useful as reinforcing elements in plastics and rubbers.
2. Zinc chelates of polyhydrazides are flame retardant and practically nonsoftening.
3. Phosphorus containing fibrous polyhydrazide is flame retardant.
4. Polyhydrazide yarns are used as belts for absorbing energy during automobile and air craft accidents.
5. Polyhydrazides are useful for immobilization of proteins. Acrylic hydrazide resins which are soluble are useful as protective and decorative coatings.
6. Poly(1,3,4-oxadiazole) copolymer prepared from 5-tert-butyl isophthalic acid and hydrazine sulphate were useful for fibers, films coatings, laminates and molded objects.
7. Material for filtering gases and liquid at temperature up to 350°C can be produced from polyoxadiazole fibers.

POLYAMIDE-HYDRAZIDES

Preparation of Polyamide hydrazide: polyamide-hydrazide is usually prepared from dicarboxylic acid chlorides and aminohydrazides such as p-aminobenzhydrazide by the low temperature solution polycondensation method, salts such LiCl may be added to aid the solubility of polymers. When p-aminobenzhydridne is used, the polymer obtained may be highly or sequentially ordered, depending upon the mode of addition of the acid chloride. This difference is trackable to the different reactivities of the amino and hydrazide groups towards the acid chloride.

Properties of Polyamide hydrazide:
Most of the polymers in the class are soluble in dimethyl sulphoxide which is used for spinning. Light scattering, Sedimentation, Rheological studies on X-500 class. Polyamide hydrazide show that they are semi-flexible polymers and undergo phase transition induced by the shear in H₂SO₄ from isotropic to the anisotropic state.

Application of Polyamide hydrazide:
1. Excellent mechanical properties.
2. Polyamidehydrazide containing group 1A, 2A, 2B, 3B, 5B metals are fire resistant.

Polyoxadiazoles: Polymer containing oxadiazole units are known to have excellent thermal and hydrolytic stability, in addition to their capacity to form fibre, film and membranes.

Preparation of Poly (1,2,4-Oxadiazole) : It can be prepared by thermal cyclodehydration of linear poly (ortho-acyl oxime) at 150°C to 300°C (o-acyl amidoxime) can be obtained by treating amino oximes with dicarboxylic acid derivatives in suitable solvents at low temperature in presence of acid acceptors or 1,3-dipolar cyclodehydration of nitrile oxide to nitriles.

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Properties of poly(1,2,4-oxadiazole) : The properties of poly(1,2,4-oxadiazole) have been investigated. The all aromatic poly(1,2,4-oxadiazole) are soluble only in strong acids such as trifluoroacetic acid, sulphuric acid and slow extremely good hydrolytic stability in both acidic and basic medium. They have good resistance to UV catalyzed oxidative attack and have lower melting temperature than their precursors namely poly (o-acylamidoxime). When suitably compounding these polymers may be used as adhesives, caulking compounds, channel sealants and fuel tank liniers.

Synthesis of poly(1,2,4-oxadiazole) : Into a 100mL round bottom flask equipped with a magnetic stirrer, a calcium chloride guard tube was placed polyamide hydrazide (PAHI-3) 0.1 gm, POCl₃ (99%) 10mL and the reaction mixture was refluxed with stirring for 5hrs in oil bath at 80°C. Then the whole reaction mixture was poured in 200mL distilled water with stirring. White precipitate of poly(1,2,4-oxadiazole) was obtained. The precipitated polymer was filtered, washed with water, methanol and dried under vacuum at 80°C for 2hrs. Yield 0.9g (94.83%).

Application of Polym (1,2,4-Oxadiazole)
1. Some of the aromatic polyhydrazides and polyoxadiazole are known to form liquid crystals from which high strength and high modulus fibres could be spun.
2. Mansato consider commercialization of the x-500 class of fibres based on aromatic polyamide hydrazide and phenylene oxadiazole N-methyl hydrazide co-polymer fibres.

II. Conclusion
Preformed hydrazide and methylene linkage containing aromatic diamine, viz, bis (4-amino benzyl) hydrazide (BABH), was successfully synthesized. A series of poly (amide hydrazide)s and copolyamide hydrazide)s and the related polyamide were synthesized from BABH or ODA with IPC by low temperature solution polymerization method. Polymers had inherent viscosities in the range of 0.18-0.93 dL/g indicating formation of medium to high molecular weight polymers. XRD analysis of polymers showed partially crystalline nature possibly due to high level of hydrogen bonding in hydrazide units incorporated. The polymers having higher mol % of BABH.

References