Poly(ethylene succinamide)

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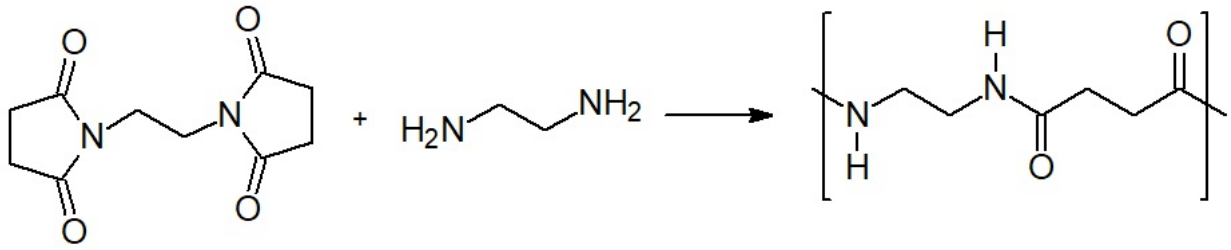

1. Procedure

Ethylenediamine (0.3 ml, Note 1) is placed in a weighed test tube of about 1.3 cm diameter. The weight of ethylenediamine is exactly determined by reweighing the test tube. After addition of an equimolar quantity of N,N′-ethylene-disuccinimide (Note 2), the test tube is swept with nitrogen, sealed, and immersed in an oil bath at 200°. After 5 h, the test tube is removed from the bath and allowed to cool to room temperature. After breaking the tube, the product is removed, ground in a mortar, and washed with water on a glass filter. The product and 120 ml of water are placed in a 150 ml flask and boiled for 1 h. The polymer is separated by hot filtration and dried under vacuum at 60° for 48 h; yd 0.81 g (63%). Anal. Calc.: C, 50.7%, N, 19.7%, H, 7.1%. Found: C, 49.4%, N, 20.1%, H, 7.3%.

2. Characterization

The polymer is soluble in formic acid, sulfuric acid, and trifluoroacetic acid, and insoluble in common organic solvents. The polymer does not show a clear melting point when heated in a sealed capillary under a nitrogen atmosphere, but decomposes at above 305°. For viscosity measurement the polymer (50 mg) is dissolved in 20 ml of formic acid. The flow time is measured at 35° in an Ubbelohde viscometer giving a flow time for water at 30° of 90 sec. The polymer has η_{sp}/c=0.21 dl/g. The infrared spectrum of the polymer measured as a KBr pellet shows the characteristic peaks of a secondary amide at 3310, 1642 and 1557 cm^{-1}. The shoulder at 1700 cm^{-1} is assignable to the succinimide group of the chain end.

3. Notes

1. Commercial anhydrous ethylenediamine (99.8%) is used. The checkers were unable to obtain commercial material of this purity, and they distilled 98% ethylenediamine from sodium under nitrogen.
2. N,N′-Ethylenedisuccinimide is prepared according to the method reported by Mason.[3] Crude material is purified by recrystallization from water. The melting point is 251-253°. Anal. Calc.: C, 53.6%; N, 12.5%. Found: C, 53.6%; N, 12.5%.

4. Methods of Preparation

This preparation is based on the paper by Kagiya, Izu, Matsuda and Fukui's,[4] although a similar method has been reported by others to prepare a high molecular weight polyamide from bisglutarimide and diamine.[5] These methods have been used for various polymers that contain succinamide units, —NHCO$(CH_2)_2$CONH—. The copolyamides prepared by these methods contains a crystalline portion of sequences having four amide linkages.

5. References

1. *Department of Hydrocarbon Chemistry, Faculty of Engineering, Kyoto University, Kyoto, Japan.*
2. *The Goodyear Tire and Rubber Company, Akron, OH 44316.*
3. Mason, A. T. *J. Chem. Soc.* **1889**, *55*, 10.
4. Kagiya, V. T.; Izu, M.; Matsuda, T.; Fukui, K. *J. Polym. Sci., Part A1*, **1967**, *5*, 15.
5. Sambeth, J.; Grundschober, F. Presented at the International Symposium on Macromolecular Chemistry, Tokyo, 1966.