Study on Synthesis of High Molecular Poly(m-phenylene terephthal amide) preparation and properties

Fei Fei Han, Min gyeong Lee, Jae Wang Ko, Tea Won Son

[1]Department of Textile Engineering, Yeungnam University, Gyeongsan, Korea
School of Textiles, Yeungnam University, Gyeongsan, Korea

1. Introduction

Aramid are well known for their high strength and excellent heat-resistant properties. Wholly aramids can be divided into two types, para-aramid and meta-aramid. In this paper a new kind of aramid polymer was synthesized. Its name is poly (m-phenylene terephthalamide), shortened form is PMTA. In the study, a series of synthetic experiments were done, aimed to find the optimum condition of polymerization. The inherent viscosities of the polymers were checked by Ubbelohde viscometer, also the molecular weight of the polymers were calculated. The polymer properties were checked by X-ray, EA, FTIR, NMR spectroscopic techniques, TGA and DSC.

2. Experiment

2.1 Material

1,3-phenylenediamine(MPD): the product of Aldrich Chemical co.
terephthaloyl chloride (TPC):the product of Aldrich Chemical co.
N-methylpyrrolidone (NMP): the product of DC Chemical co. Ltd.

2.2 Purification of MPD and TPC

MPD and TPC were purified by vacuum sublimation to essentially white solids at 80℃ and 110℃ respectively.

2.3 Co-solvent making

NMP and $CaCl_2$ with different concentrations (1%, 3%, 5%, 7%, 9%) were made by stirring at 80℃ for 2hours under nitrogen. After making, the co-solvent was kept dried over molecular sieves at room temperature until used.

2.4 Synthesis

Solid (PMTA) concentrations were chosen at 5%, 10%, 12.5%, 15%, 17.5%, 20%, and 25%. MPD was placed in a round-bottomed 3 necked flask. Then NMP (including CaCl2) was added. The flask was swept out with nitrogen. After stirring for 15minutes at 80℃, it was placed to ice-water bath to cool the solution until 5℃. TPC was divided into two parts-30% and 70%. First, added the 30% part into the flask, mixed for 20minutes at 5℃. Then replaced the flask into water-bath at 21℃. Put the 70% part into the flask, stirring, and the temperature was continue to increased from 21℃ to 54℃.

2.5 Film forming

Put solution onto the glass plate, used film maker to form a film with the thickness of 100um. Then put glass plate into water for 1 hour, after that put them into the oven for drying for 12 hours at 50℃.

2.6 Powder making

Put polymer and distilled water into blender, agitated it adequately. Then the mixture was filtrated and dried in oven for 12hours at 50℃.

2.7 Inherent viscosity determination

The inherent viscosities of polymers were checked by Ubbelohde viscometer at 30℃.

2.8 Analysis and measurement

Chemical structure was checked by X-ray Diffraction, Elemental Analysis (EA), Fourier Transform Infrared Spectroscopy (FT-IR) Analysis and Nuclear Magnetic Resonance (NMR) Spectroscopic Techniques. The thermal properties were checked by Thermogravimetric Analysis (TGA) and Differential Scanning Calorimery (DSC).

3. Results and discussion

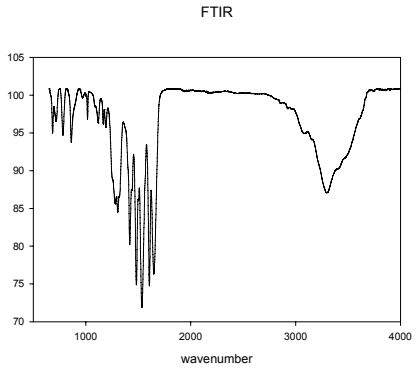

Fig. 1. FT IR spectra of PMTA.

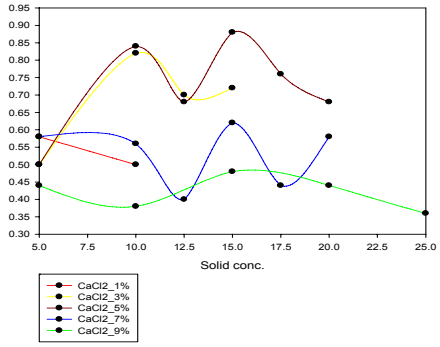

Fig. 2. Inherent viscosity of polymers.

Fig.1 was the FT-IR spectra of PMTA. Fig. 2 shows that, the one with the highest inherent viscosity(0.92) was on this polymerization condition: solid concentration 15% with $CaCl_2$ concentration 5% in co-solvent. And that is the optimum condition of polymerization.

4. Conclusion

Among the series experiments, the optimum polymerization condition was: solid concentration 15% with $CaCl_2$ concentration 5% in co-solvent. It had highest inherent viscosity also can be used to make a good film.

References

1. 洪性一,金炳哲; “Ultra-High Modulus Aramid Fibres”; 1982.
2. “Aramids”; Wikipedia.
3. Y.P.KHANNA; “Aromatic Polyamides. I. Synthesis and Characterization of Some Aromatic Polyamides and Their Model Diamides”;1981.
4. Y.P.KHANNA and E.M. PEARCE; “Aromatic Polyamides. II. Thermal Degradation of Some Aromatic Polyamides and Their Model Diamides”;1981.