Synthesis and characterization of polyamides as thermally stable polymers

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In recent years the innovation of novel polyamides as thermally stable polymers play a vital role in many areas, among that the most important factor of thermally stable polymers for different industry. Because of that, these advances are interested in improvement of heat and thermal stability and safety. Day to day in this globalization, thermal stable polymers requires a long shelf life, along with monitoring the safety and quality based upon international standards. In this chapter it incubates, thermally stable polymers its stability, long life, mechanical and thermal properties for different applications.

Keywords: Thermally stable polymers; polyamides; DSC and TGA

1. Introduction

Wholly aromatic polyamides are characterized as high temperature resistant materials with a favorable balance of other physical and chemical properties [1]. However, difficulties are encountered in processing these polymers into articles due to their high melting and/or glass transition temperatures and/or their limited solubility in common organic solvents result in difficult processability and restrict their applications [2]. In recent years, the need for novel polyamides with improved specific properties such as high thermal stability and good mechanical properties, has strongly affected the research and production of composite and hybrid (organic/inorganic) materials [3-8]. However, the inherent problems of wholly aromatic polyamides have been dealt with by many research efforts during the last decades by copolymerization[9,10], introduction of bulky alkyl or aryl substituents to reduce the hydrogen bonding at the amide linkage, flexible linkage[10,11], asymmetric/symmetric bulky units in the aromatic rings, pendant phenyl group into the polyamide backbone[12,13], and etc. The ordering and varying of backbone in synthetic polyamides have had profound effect on their end properties [8]. Aromatic polyamides are well known as high performance engineering thermoplastics and have been widely used in various industries because of their good thermal stability, chemical resistance, and mechanical properties [14]. However, their poor solubility in many organic solvents and also high softening or melting temperatures caused by their rigid structures and high stiffness of the polymer backbone or high crystallinity lead their process ability difficult. With the ever increasing demand for new processable engineering plastics, attempts have been oriented to the introduction of substituents having various chemical structures onto the benzene rings of monomers, the use of monomers containing several benzene rings separated by flexible bridges, or the use of heterocyclic monomers [15-19]. Another approach to increasing the processability is the introduction of a bulky pendant group [20] or a polar, rigid and zigzag group (heterocyclic monomer) into the main chains of polyamide [21-23]. Among various heterocyclic units which have been introduced into the main chain of aromatic polyamides, the phenylquinoxaline rings proved to be very beneficial [24, 25]. Such modifications into the aromatic backbone of polymers cause a decrease in the crystallinity and improve solubility in organic solvents and therefore better capability to be processed from solutions, while maintaining a high thermal resistance [26, 27]. Preparation of polyamides based on quinoxaline ring and its related derivatives has been under investigation by many researchers. Akutsu et al. has prepared aromatic polyamides containing quinoxaline ring by transformation of a-diketone moieties in the main chain [28] and by polycondensation of 4,4’-(2,3-quinoxalinediyl)dibenzoyle chloride and 4,4’-(2,3-benzo[g]quinoxalinediyl)benzoic acid with aromatic diamines [29-32]. They have also reported the preparation of polyamides and polyimides from 2,3-bis(4-aminophenyl)quinoxalines and aromatic dicarboxylic acids and tetracarboxylic dianhydrides [33, 34]. Hedrick et al [35] reported preparation of polyimide copolymers containing imide-aryl ether phenylquinoxaline by polycondensation of aryl ether phenyl-quinoxaline diamines with dianhydrides. There are reports of preparation of copolymers containing phenylquinoxaline rings in the main chain [36, 37]. Jenekhe et al [38] reported preparation of copolymers of 9,9-dioctylfluorene and 2,3-bis(p-phenylene)quinoxaline. They used 2,3-Bis(4-bromophenyl)quinoxaline as a monomer which has been prepared by the reaction of o-phenylene diamine with 4,4-dibromobenzil. Direct polycondensation methods have been developed by several investigators and especially by Higashi [39, 40]. In direct polycondensation, preparation and separation of acid chlorides are not necessary and the reactions proceed as one pot reaction using the free carboxylic acid, diamines and compounds such as CaCl₂, LiCl, triphenylphosphite, phosphorusoxychioride, etc. [41]. Polymers with a system of conjugated C=C—C—C— and —C=N— bonds in their main chain have been drawing the attention of researchers due to their importance in many aspects [42-51]. Among these polymers, Schiff base polymers, which are characterized by the presence of HC=NC linkages, are of considerable interest and have generally been produced by the polycondensation of diamines with various dicarboxyl compounds [46-48, 52-56]. They have thermal
Polyamides find many industrial and textile applications due to their high mechanical strength and durability. Many polyamides, the most versatile class of engineering polymers, display a wide range of properties. Aliphatic polyamides have good solubility at room temperatures and better solubility. Among various methods for polyamidation reaction, direct polycondensation is one of the most popular due to its simplicity and efficiency. Direct polycondensation methods involve the base-catalyzed condensation of diamines with dicarboxylic acids or ketones. They have thermal stability similar to that of polyamides and have been used as solid stationary phases for gas chromatography. High-performance polyamides exhibit excellent balance of thermal and mechanical properties, which make them useful as high performance materials with exceptional properties for advanced technologies. However, the application of these polyamides is often hampered by problems in their fabrication. For instance, the poor solubility and high softening or melting temperatures caused by the high crystallinity and high stiffness of the polymer backbone lead to difficult processability of the mentioned aromatic polyamides. Much effort has been made to create structurally modified aromatic polymers having better solubility and processability with retention of their high thermal stability. It is well-known that the solubility of polyamides is often improved when flexible bonds [69], meta-linkages [70, 71], non-coplanar units [72], linkages of (ester-amide) and (amide-ester-imide) [73, 74], and large pendant groups or polar substituents [75–79] are included into the polymer backbone. If these groups are carefully chosen, they are likely to increase solubility without affecting thermal and mechanical properties to any great extent. These modifications work by breaking the chain symmetry and regularity and by destroying hydrogen bonds and generally lead to lower transition temperatures and better solubility. Among various methods for polyamidation reaction, direct polycondensation methods have been developed by several investigators and especially by Yamazaki and Higashi [80–83].

Polyamides, the most versatile class of engineering polymers, display a wide range of properties. Aliphatic polyamides find many industrial and textile applications due to their high mechanical strength and durability. Many studies on polyamides have been reported previously [84-89]. Aromatic polyamides (aramids) are being used in industry because of their outstanding properties. However, poor solubility in common organic solvents and high melting temperatures are the limiting factors for the processing of these materials. A lot of attempts have been made to solubilize these polymers in order to prepare their composites using different techniques [90-94]. Aliphatic–aromatic polyamides (glass clear nylons) offer a wide range of properties including transparency, thermal stability, good barrier, and solvent resistant properties. These commercial polyamides have been reinforced with various ceramic phases [95-98]. There are numerous references to polyamides from aliphatic diamines and aromatic diacids and a far lesser number to polyamides from aromatic diamines and aliphatic diacids [99-107]. Probably the reason that aliphatic–aromatic polyamides have been studied in greater detail than the aromatic–aliphatic is that many of the former group can be made by melt and plasticized melt methods [101, 102, 108] or by standard interfacial procedures [104, 106, 109]. The aromatic–aliphatic polyamides, on the other hand are difficult to prepare by interfacial and solution methods [99, 110] and when prepared by melt methods, frequently are discolored and may have branched or network structures. In the other hand, polymers with a system of conjugated -C=C- and -C=N- bonds in their main chain have been drawing the attention of researchers because of their importance in many areas [111–115]. Among these polymers, polyamides, which are characterized by the presence of HC=N linkages, are of considerable interest and are produced by the polycondensation of diamines with various dicarbonyl compounds. They have thermal stability similar to that of polyamides and have been used as solid stationary phases for gas chromatography [116]. The high thermal stability of fully aromatic polyamides is due to the great number of aromatic rings and the extent of electronic conjugation over the entire molecule, which lead to polymers with high conducting properties. To lower the transition temperatures and to improve the solubility, several methods are used, such as copolymerization and the introduction of bulky alkyl, alkoxy, or aryl groups into the aromatic rings or into the polymer backbone [115, 117].

2. History of thermally stable polyamides

2.1 Polyamides with heterocyclic rings

Akutsu has done a lot of things heterocyclic groups such as quinoxaline used in the preparation of polyamides [118-124]. This kind of polyamides is acceptable solubility and heat stability.
2.2 Polyamides with ether and silicon groups in the main chain

Bruma has entered the investigation into the ether and silicon groups which hopes to bridge their existing silicon in the main chain polymers can be achieved easily in polar solvents (Aprotic) and they have high thermal stability so that decomposition temperature higher than their glass transition temperature 450 °C and around 300 °C [125].

2.3 Aromatic-Cycloaliphatic Polyamides

Which have phenyl groups az pendant groups and were synthesized by Park and colleagues. The polyamides, Yamazaki method in which the phenyl phosphite catalysts are used more and with direct condensation polymerization has been prepared. They are soluble in polar solvents very good. [126].

2.4 Biodegradable Polyamides

This kind of polyamide due to the biodegradable organic compounds known to have good mechanical strength behavior and they were synthesized by Tani Hara and his colleagues. The molecular weight of polymers that are prepared by condensation polymerization method in water at a temperature of 10-20 °C, is about 200,000 [127].
2.5 Electro-active Polyamides

Chau and colleagues have synthesized a polyamide are electrically active and because metal can having electrical properties, optical and magnetic interestingly show. These polymers have Conjugated bond in the main chain and can explain the mechanism of electrical conductivity in polymers [128].

2.6 Schiff-base polyamides

Figures 1, 2, 3, 4 with N2O2 or polyamides from diamins that has a metal structure are Schiff-base polyamides and can be used in the electronics industry [129-135].
In this method, the diamine synthesis, which have the ability to bind to metals. In this mode you can use inorganic compounds, metals such as copper, nickel, zinc, iron and cobalt into the diamine compound that gives new properties to prepared polyamide [136-140].

3. Experimental

3.1 Polyamides from copper-benzilbisthiosemicarbazone complex [141]

3.1.1 Preparation of monomers

3.1.1.1 Benzilbisthiosemicarbazone LH₆[142]

The thiosemicarbazide (3.64 g, 40.30 mmol) was dissolved in 40ml of methanol, 40ml of 2N HCl and 1ml of conc. HCl and then added to a suspension of benzil (4.24 g, 20.20 mmol) in 50ml of methanol and a few drops of conc. HCl. The mixture was stirred for 6 h at room temperature. The yellow solid was filtered off, washed with methanol and dried in a vacuum oven at 70 °C for 2 h. A yellow solid product was obtained in a 75% yield which starts to melt and decompose at 240 °C.

IR(cm⁻¹): 3420, 3250 and 3150 (NH , NH₂), 1610 (CN), 1585 (NH₂), 848 (CS). ¹HNMR (CDCl₃): δ/ppm 8.8(2H, s), 7.6(6H, m), 7.4(4H, m), 6.6(4H, s).

3.1.1.2 Benzil bis(thiosemicarbazonato) copper(II) CuLH₄

Using a standard method [15], copper (II) acetate monohydrate (144 mg, 0.722 mmol) was added to a solution of LH₆ (257 mg, 0.722 mmol) in ethanol (15 ml). The mixture was heated under reflux for 5 h, then cooled and the resulting solid collected, washed with ether and dried in vacuum oven at 70 °C to give CuLH₄ as a dark brown powder in a 48% yield. IR(cm⁻¹): 3419, 3241(NH₂), 1626, 1600, 1569(CN), 3419, 3343, 3150(CH), and 449, 428(N-M),(S-M). ¹HNMR (DMSO-d₆): δ/ppm 7.5(4H NH₂, s), 7.1-7.3(10H Ph, m).

3.1.2 Preparation of polyamides

3.1.2.1 Poly(Benzil bis(thiosemicarbazone) copper)terephthalate (PBCT)

A typical procedure for the preparation of polyamides is given in scheme 2. A two-necked flask equipped with a dropping funnel and gas inlet tube was charged with a mixture of CuLH₄ (0.836 g, 2 mmol), 20 ml dimethylacetamide (DMAc) and triethylamine (0.8ml). Diacidchloride (DC) (0.406g, 2 mmol) dissolved in 20 ml DMAc was added dropwise to the stirred solution at 0°C under N₂. The mixture was subsequently stirred at ambient temperature for 5 h under N₂, and then it was poured into cold water. The reddish solid product was separated by filtration and washed with NaHCO₃ solution. Then the red solid product dried in vacuum oven at 70°C. A purified sample was obtained by soxhlet
from methanol for 24h. IR (cm⁻¹) 3270 (NH), 1680 (CO). ¹HNMR (DMSO-d₆): δ (ppm) 9.52 (NH amid, 2H), 9-9.3 (CH arom, 14H).

Other polyamides except PBCP, were also prepared by the same procedure using different acid chlorides such as:

(i) Isophthaloyl chloride (IC) for preparation of Poly(Benzil bis(thiosemicarbazonato) copper) isophthalate (PBCI); IR (cm⁻¹) 3270 (NH), 1680 (CO), and ¹HNMR (DMSO-d₆): δ (ppm) 9.44 (NH amid, 2H), 9-9.3 (CH arom, 14H).

(ii) Sebacoyl chloride (SC) for preparation of Poly(Benzil bis(thiosemicarbazonato) copper) sebacoylate (PBCS); IR (cm⁻¹) 3270 (NH), 3150 (CH), and 1680 (CO). ¹HNMR (DMSO-d₆): δ (ppm) 9.4 (NH amid, 2H), 8.1-8.6 (CH arom, 10H), 1.46-2.2 (CH₂ aliph, 16H).

(iii) Adipoyl chloride (AC) for preparation of Poly(Benzil bis(thiosemicarbazonato) copper) adipoylate (PBCA), IR (cm⁻¹) 3260 (NH), 3150 (CH), and 1680 (CO). ¹HNMR (DMSO-d₆): δ (ppm) 9.63 (NH amid, 2H), 8.1-8.7 (CH arom, 10H), 1.3-2.2 (CH₂ aliph, 8H).

Pyridine 2,6-dicarboxylic acid for preparation of Poly(Benzil bis(thiosemicarbazonato) copper) pyridindicarboxylate (PBCP) was used in direct polycodensation with CuL₄ in the mixture of NMP-pyridine (30/6 v/v) as solvent at 110 °C for different period of times: 4, 8, and 15 hours. The inherent viscosity of the polyamide was measured in H₂SO₄ were: 0.5, 0.75, and 0.78 respectively. IR (cm⁻¹) 3300 (NH), 1670 (CO), and 1570 (CN).

![Scheme 2](image)

**Scheme 2** The typical procedure for the preparation of polyamides.

### 3.2 Polyamides from Quinoxaline Based Diamine [143]

#### 3.2.1 Preparation of monomers (scheme 3)

The reaction steps for the synthesis of compounds 4, 5-diphenyglyoxalione (I), 4, 4'-dinitrobenzil (II), and 2, 3-bis-(p-nitrophenyl)-quinoxaline (III) were shown in scheme 1. Compounds (I) and (II) were synthesized according to the procedures given in the literature [144]. The yields for the compounds (I) and (II) were 71.5% and 21% with the melting points of 330 °C and 213 °C, respectively. Compound (III) was synthesized according to the procedure given in the literature [145] and the yield for this compound was 77% with the melting point of 201 °C.

#### 3.2.2. Synthesis of 2, 3-bis-p-aminophenylquinoxaline (IV):

A solution of 4.46 g (12 mmol) of dinitro compound (III) and 0.4 g of Pd/C in 200 ml of ethanol in a flask equipped with dropping funnel, reflux condenser and thermometer was heated for 2 hours and a solution of 10 ml hydrazine hydrate 85% in 20 ml ethanol were added drop wise in 1 hour. The reaction was refluxed for 2 hours and the hot solution was filtered. The precipitate was formed during cooling the filtrate which was filtered and dried in a vacuum oven. The powder was obtained in a yield of 85% with melting at about 265 °C.
3.2.3 Synthesis of polyamides

Synthesis of polyamides was shown in scheme 2 and a typical procedure for the preparation of polyamides is given below:

A two-necked flask equipped with a magnetic stirrer, a condenser, and a nitrogen inlet/outlet tube was charged with 0.624 g (2 mmol) diamine (IV) and 2 mmol dicarboxylic acid such as terephthalic acid, isophthalic acid, pyridine-2,6-dicarboxylic acid, 2-phenyl-1H-imidazole-4,5-dicarboxylic acid, adipic acid, or sebacic acid in a mixture of 30 ml NMP and 6 ml pyridine containing 1.3 ml triphenylphosphite, 0.6 g LiCl, and 1.8 g CaCl₂. The reaction mixture was heated at 110 °C for 10 hours under N₂. The reaction solution was poured into methanol; the precipitate was filtered off and washed with refluxing methanol for 24 h. The solid product was dried in a vacuum oven at 100 °C.
3.3 Polyamides from Bis(thiosemicarbazone)acenaphthenequinone diamine [146]

3.3.1 Preparation of monomers

3.3.1.1 (2H)-acenaphtylenilidenbisthiosemicarbazone LH₆

The thiosemicarbazide (3.64 g, 40.30 mmol) was dissolved in 40 ml of ethanol, 40 ml of 2N HCl and 1 ml of conc. HCl and then added to a suspension ofacenaphthochinone (3.64 g, 20.20 mmol) in 50 ml of ethanol and a few drops of conc. HCl. The mixture was stirred for 10 h under reflux condition. After cooling the reaction, the yellow solid was filtered off, washed with ethanol and dried in a vacuum oven at 70 °C for 2 h. A yellow solid product was obtained in a 70% yield which starts to melt and decompose at 250 °C.

IR (KBr) (ν max Cm⁻¹): 3440 (NH), 3250 (NH₂), 3050 (C-H aromatic ring), 1598 (CN), 1479 (C=C Ar), 823 (CS).

1H NMR (400, DMSO, d₆, TMS) δ ppm: 4.2 (1H, S, NH), 5.28 (1H, S, SH), 5.75 (2H, S, NH₂), 7.8-7.9 (3H, m, CH Ar), 8.0 (2H, t, CH₂), 8.38 (1H, d, CH₂), 12.93 (1H, S, NH), 13.38 (1H, S, NH). 13C NMR (400, DMSO, d₆, TMS) δ ppm: 118.13 (C₅Ar), 122.91 (C₅Ar), 127.28 (C₆Ar), 129.08 (C₆Ar), 133.39 (C₆Ar), 140.18 (C₆Ar), 155.07 (C=N), 165.94 (C=S). Anal. Calcd. For [C₁₄H₁₂N₆S₂]: C, 51.22; H, 3.65; N, 25.6. Found: C, 50.87; H, 3.87; N, 26.23.

Scheme 5 Structure of ligand.

3.3.2 Preparation of polyamides

3.3.2.1 General procedure

A typical procedure for the preparation of polyamides is given in scheme 2. A two-necked flask equipped with a dropping funnel and gas inlet tube was charged with a mixture of LH₆ (0.712 g, 2 mmol), 20 ml dimethylformamide (DMF) and triethylamine (0.8 ml). Diacid chloride (DC) (2 mmol) dissolved in 20 ml DMF was added dropwise to the stirred solution at 0°C under N₂. The mixture was subsequently stirred at ambient temperature for 3 h under N₂, and then it was poured into cold water. The yellow solid product was separated by filtration and washed with NaHCO₃ solution. Then the red solid product dried in vacuum oven at 70 °C. A purified sample was obtained by suxelated from methanol for 24 h.

Scheme 6 The typical procedure for the preparation of polyamides.
3.3.2.2 Poly[1(2H)-acenaphtyleniliden bis(thiosemicarbazono)terephthalate] (PA1)
Yield 92%, mp 262°C, IR (KBr) (υ max Cm⁻¹): 3423 (N-H), 3300 (N-H), 3000 (C-H aromatic), 1684 (C=O), 1630 (C=N), 1596 (C=Cα), 8.1, 8.89 (4H, m, CH₉), 8.31, 8.38 (1H, m, CH₉), 8.41 (1H, CH₉). IR (KBr) (υ max Cm⁻¹): 3423 (N-H), 3300 (N-H), 3000 (C-H aromatic), 1684 (C=O), 1630 (C=N), 1596 (C=Cα), 8.1, 8.89 (4H, m, CH₉), 8.31, 8.38 (1H, m, CH₉), 8.41 (1H, CH₉). Anal. Cald. for [C₂₂H₁₄N₆O₂]: C, 57.64; H, 3.05; N, 18.34. Found: C, 56.89; H, 3.25; N, 18.67.

3.3.2.3 Poly[1(2H)-acenaphtylenilidenbis(thiosemicarbazano)isophthalate] (PA2)
Yield 90%, mp 265°C, IR (KBr) (υ max Cm⁻¹): 3246 (N-H), 3153 (N-H), 1640 (C=O), 1593 (C=N), 1476 (C=O aromatic). Anal. Cald. for [C₂₂H₁₄N₆O₂]: C, 57.64; H, 3.05; N, 18.34. Found: C, 56.89; H, 3.25; N, 18.67.

3.3.2.4 Poly[1(2H)-acenaphtylenilidenbis(thiosemicarbazano)succinate] (PA3)
Yield 91%, mp 255°C, IR (KBr) (υ max Cm⁻¹): 3253 (N-H), 3175 (N-H), 2900 (C-H Aliphatic), 1717 (C=O), 1595 (C=N), 1478 (C=Cα), 8.1, 8.89 (4H, m, CH₉), 8.31, 8.38 (1H, m, CH₉), 8.41 (1H, CH₉). Anal. Cald. for [C₁₈H₁₄N₆O₂]: C, 52.68; H, 3.41; N, 20.48. Found: C, 53.23; H, 3.25; N, 20.27.

3.3.2.5 Poly [1(2H)-acenaphtylenilidenbis (thiosemicarbazono) adipate] (PA4)
Yield 94%, mp 252°C, IR (KBr) (υ max Cm⁻¹): 3257 (N-H), 3100 (N-H), 2800 (C-H Aliphatic), 1685 (C=O), 1601 (C=N), 1489 (C=Cα). Anal. Cald. for [C₂₀H₁₈N₆O₂]: C, 54.79; H, 4.11; N, 19.17. Found: C, 55.27; H, 3.87; N, 19.93.

3.3.2.6 Poly[1(2H)-acenaphthylenilidenbis (thiosemicarbazono)sebacolate] (PA5)
Yield 85%, mp 250°C, IR (KBr) (υ max Cm⁻¹): 3430 (N-H), 3248 (N-H), 2926 (C-H Aliphatic), 1685 (C=O), 1599 (C=N), 1488 (C=Cα), 8.1, 8.89 (4H, m, CH₉), 8.31, 8.38 (1H, m, CH₉), 8.41 (1H, CH₉). Anal. Cald. for [C₂₀H₂₆N₆O₂]: C, 58.29; H, 5.26; N, 17. Found: C, 57.89; H, 5.62; N, 17.41.

3.4 Polyamides from Acenaphthohydrazinomercaptotriazole diamine [147]

3.4.1 Preparation of monomers

3.4.1.1 Acenaphthohydrazinomercaptotriazoldiamine (AHTD)
The 1,2-dihydro-acenaphthylene-1,2-dione (1.82 g, 10 mmol) was dissolved in 25 ml of ethanol and 1ml of conc. HCl and then added to a suspension of 4-amino-3-hydrazino-5-mercapto-1,2,4-triazole (2.92 g, 20 mmol) in 25 ml of ethanol and 5 ml water. The mixture was stirred for 10 h at reflux temperature. The orange solid was filtered off, washed with ethanol and water and dried in a vacuum oven at 70 °C. A orange solid product was obtained in a 65% yield which starts to melt at 260 °C.
The chemical structure of diamine and its procedure is shown in Scheme 7.
3.4.2 Preparation of polyamides

3.4.2.1 Polyyacenaphthoynhydrinomercaptotriazolterephthalate (PA1)

A typical procedure for the preparation of polyamides is given in scheme 8. A 100 ml two-necked flask equipped with a dropping funnel and gas inlet tube was charged with a mixture of AHTD (0.656 g, 2 mmol), 30 ml dimethylformamide (DMF) and triethylamine (0.8ml). 2 mmol Diacidchloride (DC) (such as terephthaloyl chloride, isophthaloyl chloride, adipoyl chloride and sebacoyl chloride) dissolved in 20 ml DMF was added dropwise to the stirred solution at 0 °C under N2. The mixture was subsequently stirred at ambient temperature for 5 h under N2, and then it was poured into cold water. The orange solid product was separated by filtration and washed with NaHCO3 solution. Then the solid product dried in vacuum oven at 70 °C. An orange solid product was obtained in a 86% yield which starts to melt at 280 °C.

![Scheme 8](image)

Scheme 8 The typical procedure for the preparation of polyamides.

References