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Synthesis, Characterization, and Optimum Reaction Conditions of Oligo-3-Aminopyridine and Its Schiff Base Oligomer

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Abstract: The products and the oxidative polycondensation reaction conditions of oligo-3-aminopyridine with H_2O_2 and NaOCl were studied. In this reaction, NaOCl was observed to be more active than H_2O_2 and optimum reaction conditions were determined. Oligo-3-aminopyridine (OAP) was synthesized from the oxidative polycondensation of 3-aminopyridine (AP) with NaOCl and H_2O_2 in an aqueous acidic medium at $25^\circ\text{--}90^\circ\text{C}$. The products were characterized by ^1H NMR, FT-IR, UV-Vis, and elemental analysis. The number-average molecular weight, weight-average molecular weight, and polydispersity index values of OAP synthesized were found to be $250, 800 \text{ g mol}^{-1}$, and 3.20, respectively, using NaOCl, and $240, 840 \text{ g mol}^{-1}$, and 3.50, respectively, using H_2O_2 . The respective values of the Schiff base were 1140, 5190 g mol^{-1} , and 4.55. At optimum reaction conditions, yields of the reaction products were 80.1% (H_2O_2) and 82.0% (NaOCl). About 90% AP was converted into OAP. TG analyses showed that OAP was stable towards thermo-oxidative decomposition. The weight loss of OAP was found to be 5, 50, and 92 at $125^\circ, 617^\circ, \text{ and } 1000^\circ\text{C}$, respectively. The weight loss of oligo-aminopyridinylazomethinephenol (OAPAP) was found to be 5, 50, and 80.50 at $175^\circ, 660^\circ, \text{ and } 1000^\circ\text{C}$, respectively. Also, a new oligomeric Schiff base was synthesized from the condensation of OAP with salicylaldehyde and their structures and properties were determined.

Keywords: Oligo-3-aminopyridine; Oxidative polycondensation; Schiff base

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INTRODUCTION

Oligophenols and their derivatives have been used in various fields because of their electronic structural properties. They have useful properties such as paramagnetism, semi-conduction, electrochemical activity, and resistance to high energy. Because of these properties, oligophenols were used to prepare inhibitors with resistance to high temperature, thermostabilizers, graphite materials, epoxy oligomer and block copolymers, adhesives, photo resists, and antistatic materials.^[1-8] The halogen and sulfur derivatives of oligophenols were used to prepare flame-resistant composite materials such as in lead collecting battery cathodes.^[9-12] Nowadays, despite these advantages, the use of bifunctional oligophenols is limited. Schiff bases derived from the salicylaldehydes are well known polydentate ligands, able to form coordination compounds in deprotonated or neutral forms. Oligo-3-aminopyridine (OAP) and its Schiff base oligomers can form polymer-metal complexes compounds with metal salts. Therefore, they can be used to remove heavy metals from industrial wastewater.

In this article, in the first part dealing with oligo-3-aminopyridine, the effects of different parameters, such as temperature, initial concentration of 3-aminopyridine (AP), NaOCl, and acid, were investigated. In the second part, the new Schiff base oligomer was synthesized from AP with salicylaldehyde and OAP, and its Schiff base oligomer was characterized using FT-IR, UV-Vis, ¹H NMR, elemental analysis, differential thermal analysis-thermogravimetric analysis (DTA-TG), and size exclusion chromatography (SEC) techniques.

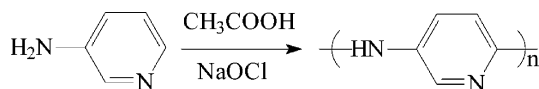
EXPERIMENTAL

Materials

3-aminopyridine (AP), salicylaldehyde (SA), 1,4-dioxane, ethanol, benzene, acetone, ethyl acetate, tetrahydrofuron (THF), dimethyl formamide (DMF), dimethyl sulfoxide (DMSO), H₂O₂ (30% solution in water), chloroform, CCl₄, and toluene were supplied from Merck Chemical Co. as chromatographic grade and were used as received. Acetic acid and sodium hypochloride (NaOCl) (34% solution in water) were supplied from Carlo Erba Co. and Paksoy Co., respectively. KOH was supplied from Analar BDH Chem. Co.

Oxidative Polycondensation of AP with NaOCl

OAP was synthesized through oxidative polycondensation of 3-aminopyridine with solution in water of NaOCl (2.95 mL, 34%). The



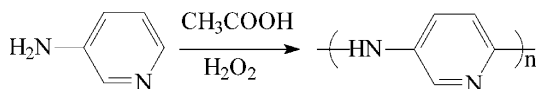
Scheme 1.

3-aminopyridine (0.6 g, 64 mmol) was dissolved in an aqueous solution of acetic acid (10%, 0.5 mL) and placed into a 50 mL three-necked round-bottom flask. It was fitted with a condenser, thermometer, stirrer, and addition funnel containing NaOCl. At 25°C, NaOCl was added dropwise over about 40 min. The reaction mixture was stirred at 25°C for 1 h (Scheme 1, Table I). The mixture was neutralized with 2.26 g KOH (10%, 40 mmol). Oligo-3-aminopyridine was then separated by extraction with ethyl acetate and precipitated. Unreacted monomer was separated from the reaction products by washing with cold CHCl_3 . The crude product was washed with water (50 mL \times 3), filtered, and dried in an oven at 105°C. Black solid, m.p.: 82°C, 82% yield.

For OAP, FT-IR (KBr; cm^{-1}): 3400 (N–H); 1338 (C–N); 1517–1573 (C–C); 3250–3100 (aromatic C–H). Analysis calculated for OAP: C, 65.21; H, 4.34; N, 30.40. Found: C, 63.62; H, 4.81; and N, 31.51. ^1H NMR (DMSO): δ ppm, 5.25 (s, 1H, NH); 7.80 (s, 1H, Ar–Ha); 8.00 (s, 1H, Ar–Ha'); 7.40 (s, 1H, Ar–Hb).

Table I. Oxidative polycondensation of amino pyridine with NaOCl in aqueous CH_3COOH

Sample	[AP] ₀ (mol/L)	[NaOCl] ₀ (mol/L)	[CH ₃ COOH] ₀ (mol/L)	Temp., K	Time, h	Conversion of AP, %
1	0.94	1.90	—	298	1	50.7
2	0.94	2.82	—	298	1	71.6
3	0.94	1.90	—	313	1	50.1
4	0.94	1.90	—	298	2	71.7
5	0.94	1.90	0.21	298	1	48.7
6	0.94	1.90	0.48	298	1	54.5
7	0.94	1.90	0.57	298	1	69.0
8	0.94	1.90	1.26	298	1	77.0
9	0.94	2.82	0.57	298	1	82.0
10	0.94	1.90	0.57	305	1	55.9
11	0.94	1.90	0.57	313	1	64.4
12	0.94	1.90	0.57	333	1	52.0
13	0.94	1.90	0.57	298	2	62.4
14	1.77	3.54	0.57	313	1	40.9
15	1.25	2.50	0.57	313	1	55.3



Scheme 2.

Oxidative Polycondensation of AP with H₂O₂ in an Aqueous Acidic Medium

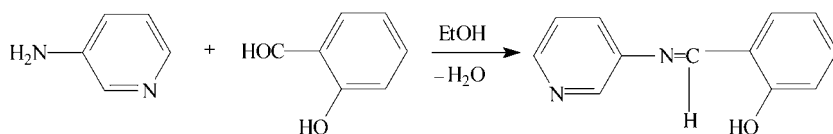
AP (0.6 g, 64 mmol) was dissolved in an aqueous solution of acetic acid (10%, 0.5 mL) and placed into a 50 mL three-necked round-bottom flask. It was fitted with a condenser, thermometer, stirrer, and addition funnel containing H₂O₂. At 25°C, H₂O₂ (2.95 mL, 30%) was added dropwise over about 40 min. The reaction mixture was stirred at 70°C for 1 h (Scheme 2, Table II). Then 2.26 g of KOH (10%, 40 mmol) was added to the reaction mixture. Oligo-3-aminopyridine was separated by extraction with ethyl acetate and precipitated. The crude product was washed with water (50 mL × 3), filtered, and dried in an oven at 75°C. Black solid, m.p.: 82°C, 80% yield.

Syntheses of Schiff Bases of AP

AP (0.94 g, 10 mmol) and salicylaldehyde (1.05 g, 10 mmol) were dissolved in ethanol (25 mL) and placed into a 50 mL two-necked

Table II. Oxidative polycondensation of amino pyridine with H₂O₂ in aqueous CH₃COOH

Sample	[AP] ₀ (mol/L)	[H ₂ O ₂] ₀ (mol/L)	[CH ₃ COOH] ₀ (mol/L)	Temp., K	Time, h	Conversion of AP, %
1	0.94	1.90	—	298	1	—
2	0.94	1.90	—	298	2	—
3	0.94	1.90	—	313	1	—
4	0.94	1.90	—	343	1	6.6
5	0.94	2.82	—	343	2	8.5
6	0.94	1.90	0.78	313	2	—
7	0.94	1.90	0.78	343	4	72.9
8	0.94	1.90	0.57	343	1	69.6
9	0.94	1.90	0.57	343	2	67.6
10	0.94	1.90	0.57	343	4	41.2
11	0.94	2.82	0.57	343	2	72.8
12	2.82	2.82	1.07	343	2	57.8
13	0.94	1.90	0.57	353	1	73.2
14	0.94	1.90	0.57	363	1	80.1



Scheme 3.

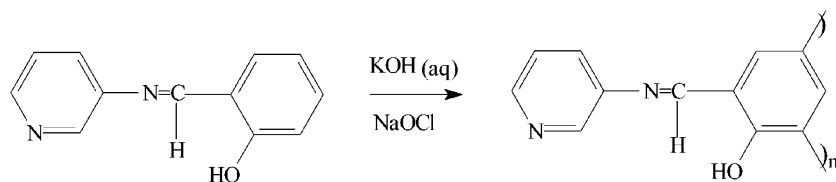
round-bottom flask (Scheme 3). It was fitted with a condenser and thermometer. The mixture was stirred magnetically on a water bath at 70°C for 2 h. Then, ethanol and unreacted salicylaldehyde were distilled from the condensation product, which was dried in an oven at 105°C (98%, yield).

For APAP, FT-IR (KBr; cm⁻¹): 1338 (C–N); 1517–1573 (C–C); 3250–3100 (aromatic C–H); 1620 (CH=N); 3418 (Ar–OH); 3060 (olefinic –CH). Analysis calculated for APAP: C, 73.46; H, 4.08; N, 14.30. Found: C, 70.77; H, 5.81; and N, 12.64.

Synthesis of Schiff Base Oligomer of 3-Aminopyridine with Salicylaldehyde in NaOCl^[6]

Oligo-aminopyridinylazomethinephenol (OAPAP) was synthesized through oxidative polycondensation of APAP in an aqueous solution of NaOCl (34%). The APAP (0.3 g, 1.5 mmol) was dissolved in an aqueous solution of KOH (10%, 0.089 g, 1.5 mmol) and placed into a 50 mL three-necked round-bottom flask. It was fitted with a condenser, thermometer, stirrer, and addition funnel containing NaOCl. After heating at 60°C for 30 min, NaOCl was added dropwise over about 20 min. The reaction mixture was heated at 70°C for 5 h. The mixture was neutralized with 0.13 mL HCl (37%) at room temperature (Scheme 4). The mixture was filtered and washed with hot water to separate it from mineral salts, and then dried in an oven at 110°C.

For OAPAP, FT-IR (KBr; cm⁻¹): 1338 (C–N); 1517–1573 (C–C); 3250–3100 (aromatic C–H); 1620 (CH=N); 3418 (Ar–OH); 3060



Scheme 4.

(olefinic –CH). Analysis calculated for OAPAP: C, 73.46; H, 4.08; N, 14.30. Found: C, 70.77; H, 5.81; and N, 12.64. ^1H NMR (DMSO): δ ppm, 9.80 (s, 1H, OH); 9.30 (s, 1H, CH=N); 7.80 (s, 1H, Ar–Ha); 7.50 (s, 1H, Ar–Hb); 8.07 (d, 1H, pridynyl-Hc); 8.01 (d, 1H, pridynyl-He); 8.01 (d, 1H, pridynyl-Hc'); 7.68 (t, 1H, pridynyl-Hd).

Characterization Techniques

IR and UV-Vis spectra were measured by Shimadzu FT-IR 8300 and UV-160 spectrometers, respectively. Elemental analysis was carried out with a Carlo Erba 1106. The FT-IR spectra were recorded using a KBr disc (4000–400 cm^{-1}). UV-Vis spectra of OAP and OAPAP oligomer were determined by using THF. OAP and its Schiff base oligomer characterized by were ^1H NMR spectra (Bruker DPX FT-NMR spectrometer operating at 400 MHz) recorded at 25°C using deuterated DMSO as solvent. TMS was used as internal standard. Thermal data were obtained using an STA 409C NETZSCH-Geratebau GmbH Thermal Analysis. The thermal and thermogravimetric measurements were made between 20° and 1000°C (in air, rate 10°C/min). The number-average molecular weight (M_n), weight-average molecular weight (M_w), and polydispersity index (PDI) were determined by SEC (Kovo Co.). For SEC, columns used were a SGX (100 Å, 7 μm), 3.3 mm i.d. \times 150 mm; eluent: DMF (0.2 mL/min), and polystyrene standards. Polystyrene standards were supplied by Polymer Laboratories Co. (Germany) and their M_w values were 162, 580, 925, 1260, 2350, 3370, 4920, 8450, 12970, and 19880 g mol^{-1} . A refract metric detector (at 25°C) was used to analyze the product.

RESULTS AND DISCUSSION

Investigation of Synthesis Conditions of OAP

The conditions for oxidative polycondensation reaction of AP with NaOCl solution in aqueous acidic and neutral medium are given in Table I. The oxidative polycondensation reaction of AP immediately formed in an aqueous solution, which turned brown by the addition of NaOCl or H_2O_2 . When $[\text{AP}]_0 = 0.94 \text{ mol/L}$ and $[\text{CH}_3\text{COOH}]_0 = 1.26 \text{ mol/L}$, conversion of AP oligomer by $[\text{NaOCl}]_0 = 1.90 \text{ mol/L}$ at 25°C for 1 h was 77.0% (see Table I).

As seen in Table I, the yield of the oligomer depended on the initial concentrations of AP and NaOCl. The maximum yield (82.0%) of OAP formed at the concentrations of AP (0.94 mol/L), NaOCl (2.82 mol/L), and CH_3COOH (0.57 mol/L) at 25°C for 1 h. The yield of OAP increased from 50.7 to 71.6%, when the concentration of NaOCl was increased

Table III. Oxidative polycondensation of aminopyridineazomethynephenol with NaOCl in aqueous KOH

Sample	[APAP] ₀ (mol/L)	[KOH] ₀ (mol/L)	[NaOCl] ₀ (mol/L)	Temp. K	Time, h	Conversion of APAP, %
1	0.85	0.85	0.32	343	5	48.8
2	0.85	0.85	0.32	343	10	57.0
3	0.85	0.85	0.32	363	15	42.3
4	0.85	0.85	0.64	343	15	65.0
5	0.85	1.90	0.32	343	15	55.0
3	0.85	0.85	0.32	343	15	42.3
6	0.85	0.85	0.96	343	15	70.5
7	0.85	0.85	0.32	368	15	40.0

from 1.90 to 2.82 mol/L under neutral condition (Table I). Also, the yield of oligomer was affected by increasing the concentration of acetic acid. AP is known for its relatively higher activity than phenols during oxidative polycondensation. As seen in Tables I and II, the yields of the reaction products were 80.1% (H₂O₂) and 82.0% (NaOCl). For the oligomer of amino pyridine, NaOCl was a better oxidant than H₂O₂.

Conditions of oxidative polycondensation reaction of AP in 30% H₂O₂ aqueous solution are given in Table II. The conversion of AP was 80.1% at optimum conditions such as [AP]₀ = 0.94 mol/L, [CH₃COOH]₀ = 0.57 mol/L, [NaOCl]₀ = 1.90 mol/L, at 90°C for 1 h.

Product of APAP solution was 57% at the NaOCl medium after 10 h at 70°C. As seen in Table III, products increased with increasing temperature. The yield of the second fraction (3.7%) and the third fraction (2.8%) was lower than that of the first fraction (36.3%) at the same conditions. When the molar amount of NaOCl increased, total conversion to oligomer APAP was 70.5%. When the temperature increased from 343 to 363 K, conversion of APAP was found to be 42.0%. This decrease could have resulted from the degradation of the azomethine bond in the Schiff base oligomer.

Solubility

The oxidative polycondensation product of AP with NaOCl and H₂O₂ in aqueous acidic medium was a black solid powder. OAP was soluble in H₂SO₄, THF, DMF, DMSO, and 1,4-dioxane. However, OAP was poorly soluble in aqueous NaOH, toluene, and chlorinated solvents, such as CHCl₃ and CCl₄. The oxidative polycondensation product of APAP with NaOCl in aqueous alkaline medium was a dark black solid powder.

Table IV. M_n , M_w , polydispersity index (PDI), and peak area % of OAP (NaOCl), OAP (H_2O_2), and its Schiff base oligomer (OAPAP), as determined by SEC

Oligomers	Fraction I				Fraction II				Fraction III						
	M_n	M_w	PDI	Peak area, %	M_n	M_w	PDI	Peak area, %	M_n	M_w	PDI	Peak area, %			
OAP (NaOCl)	250	800	3.20	4070	4800	1.18	10	430	540	1.25	15	210	220	1.05	75
OAP (H_2O_2)	240	840	3.50	2220	4910	2.21	12	210	230	1.10	88	—	—	—	—
OAPAP (NaOCl)	1140	5190	4.55	5885	7380	1.25	67	430	660	1.53	23	—	—	—	—

OAPAP was soluble in H_2SO_4 , DMF, and DMSO. However, OAPAP was poorly soluble in aqueous NaOH, THF, 1, 4-dioxane, and toluene. OAPAP was insoluble in n-hexane, CHCl_3 , and CCl_4 .

Structure of OAP and Its Schiff Base Oligomer

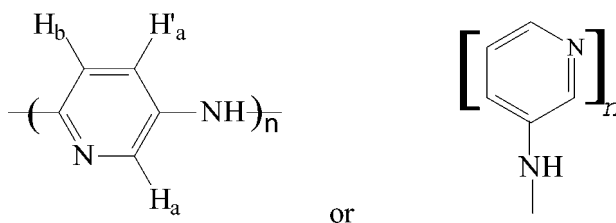
According to molecular weight distribution, M_n , M_w , and polydispersity index (PDI) values of OAP (as oxidant, NaOCl) were found to be 250, 800, and 3.20, respectively. The M_n , M_w , and PDI values of OAP (as oxidant, H_2O_2) were found to be 240, 840, and 3.50, respectively. The M_n , M_w , and PDI values of OAPAP were found to be 1140, 5190, and 4.55, respectively. The molecular weight distribution (MWD) of OAP for both NaOCl and H_2O_2 oxidants and for OAPAP was multimodal; the data are summarized in Table IV.

The UV-Vis spectra of AP and its oligomer have highly intensive K, less intensive B, and less intensive R bands observed at 247, 270, and 306 nm, respectively. The difference in the visible region (400–740 nm) between AP and its oligomer is a shift of the B band. The mesomeric and inductive effects of the NH_2 group on the monomer and oligomer are structural similarity. The UV-Vis spectra of oligomer synthesized from AP in the H_2O_2 and NaOCl media were similar.

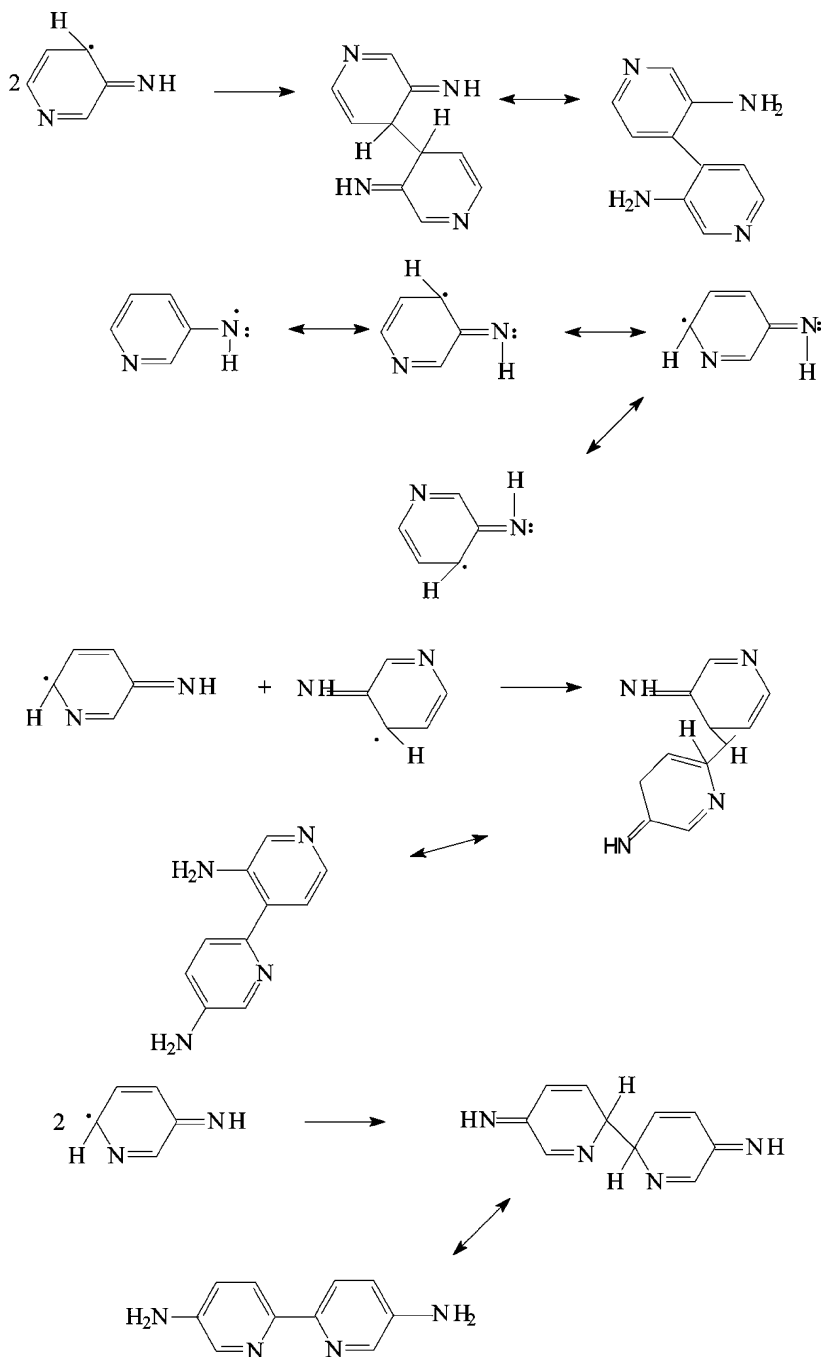
When the FT-IR spectra of AP and its oligomers are compared, both have similar bands. In the oligomer, the vibration band of the NH groups gave intense and wide bands at 3400 cm^{-1} . Weak vibration bands of the aromatic C–H and C–N groups were observed at $3250\text{--}3100\text{ cm}^{-1}$ and 1338 cm^{-1} , respectively.

The band of azomethine (C=N) group was observed at 1620 cm^{-1} from OAPAP that was synthesized from the oxidative polycondensation reaction of APAP with NaOCl. The ^1H NMR and FT-IR results showed the formation of oligomeric macromolecules from AP by the polymerization on $-\text{NH}-$ and C_6 or $-\text{NH}-$ and C_4 positions (Scheme 5).

Monomer and dimer interconversion combinations of radical units are proposed as shown in Scheme 6.



Scheme 5.



Scheme 6.

Structure of the Schiff Base Derivatives of OAP

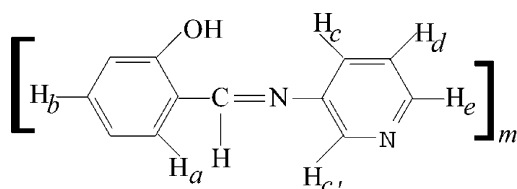
The condensation products of OAP with salicylaldehyde, that is, oligo-aminopyridinylazomethinephenol (OAPAP), were all dark black powders insoluble in ethanol. OAPAP (T_m : 250°C) has a higher softening temperatures (T_g) than OAP. This oligomer is melted at 82°C, like OAP.

Polymeric Schiff bases can be prepared only from the condensation of salicylaldehyde with electron-acceptor substituents. The K band of Schiff base oligomers gave two peaks, 208 and 222 nm, because of conjugation between the benzene ring and nonbonding electrons of nitrogen in the azomethine group. B and R bands of CH=N groups were observed at 269 and 339 nm, respectively and CH=N absorption bands were observed in 1620 cm^{-1} . The stretching frequency at 2745 cm^{-1} shows the presence of O-H...N intramolecular hydrogen bond, which is in agreement with reported values.^[12]

To identify the structures of OAP and its Schiff base oligomer (Schemes 2 and 7), ^1H NMR spectra were recorded in DMSO- d_6 . The FT-IR spectral data of the OAP and its Schiff bases oligomer confirm the results of the ^1H NMR spectra. The OH signal in the Schiff base derivative (OAPAP) appeared at a much lower field because of intramolecular H-bonding (Scheme 7). This case is also supported by the FT-IR bands at 2745–2768 cm^{-1} .

Thermal Analyses of OAP and Its Schiff Base Oligomer

TGA and DTA of OAP and its Schiff base oligomer were measured under an air atmosphere in the temperature ranges of 20°–1000°C to investigate thermal stability. Figures 1 and 2 show the TG and DTA tracings for the OAP and its Schiff base oligomer, respectively. OAP and its Schiff base oligomer started to degradation between 110° and 128°C. The TG data indicate that the OAP and its Schiff base oligomer decomposed at high temperatures: 5% and 50% of mass OAP was lost at 125° and 617°C, respectively, and 5% and 50% of mass OAPAP was lost at 175°



Scheme 7.

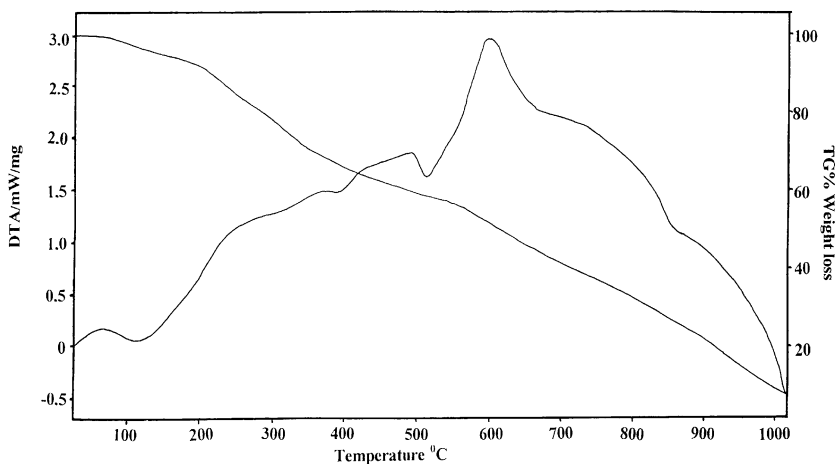


Figure 1. TG-DTA curves of OAP.

and 660°C, respectively. OAPAP demonstrated more resistance than OAP to temperature at the same condition. The respective weight loss of OAP and OAPAP was found to be 92% and 80.50% at 1000°C. The multiple endothermic and exothermic/endothermic processes of OAP and OAPAP are seen in Figures 1 and 2. According to DTA and TG analyses, OAP and OAPAP were shown to be stable to temperature and thermo-oxidative decomposition.

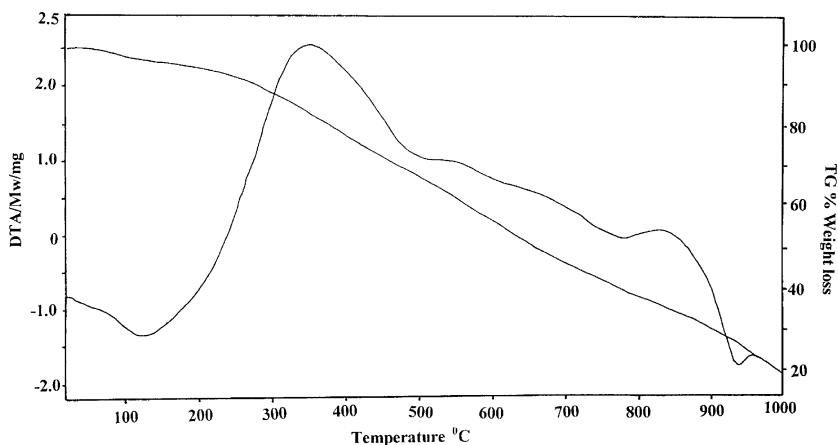


Figure 2. TG-DTA curves of OAPAP.

CONCLUSION

We determined the optimum conditions of the oxidative polycondensation reaction of AP with NaOCl and H₂O₂, and described new synthesis methods for OAP. We synthesized for the first time condensation products of OAP and its Schiff base oligomer with salicylaldehyde and determined their structure and chemical and physical properties. In this reaction, NaOCl was found to be more active than H₂O₂ and gave AP with a conversion yield of 82%. M_n, M_w, and PDI values of OAP were found to be 250, 800 g mol⁻¹, and 3.20 respectively, using NaOCl, and 240, 840 g mol⁻¹, and 3.50 respectively, using H₂O₂. M_n, M_w, and PDI values of OAPAP were found to be 1140, 5190 g mol⁻¹, and 4.55. According to TG, OAP and its Schiff base oligomer was resistant against thermo-oxidative degradation, and carbonaceous residue values of OAP and OAPAP were found to be 8.00% and 19.50%, respectively, at 1000°C.

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