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AUTHORS: FEYZA KOLCU

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Synthesis, Characterization and Optimum Reaction Conditions of Oligo-N-Salicylidenephenylhydrazone via Oxidative Polymerization

Feyza Kolcu

Çanakkale Onsekiz Mart University, Lapseki Vocational School, Department of Chemistry and Chemical Processing Technologies, Çanakkale, Turkey

Abstract: The oxidative polycondensation conditions and optimum parameters of N-salicylidenephenylhydrazone (SPH) were determined using air, H_2O_2 and NaOCl as oxidants at a temperature range between 50 °C and 95 °C in an aqueous alkaline medium. The molecular structures of the obtained monomer and oligomer were confirmed by FT-IR, UV-Vis, ^{1}H-NMR and elemental analyses. The molecular weight distributions of oligo-N-salicylidenephenylhydrazone were determined (OSPH) by SEC measurement. Thermal behavior (TG-DTA) of OSPH was examined using thermogravimetric techniques. The conversion of N-salicylidenephenylhydrazone into its oligomeric form was performed using air, H_2O_2 and NaOCl as oxidants in an aqueous alkaline medium. According to SEC analysis, the number-average molecular weight (M_n), weight-average molecular weight (M_w) and polydispersity index (PDI) values of OSPH obtained using NaOCl oxidant were found to be 1436 g mol^{-1}, 1631 g mol^{-1} and 1.14, respectively. The conversion yield of N-salicylidenephenylhydrazone into oligo-N-salicylidenephenylhydrazone was 100% at optimum reaction conditions such as $[SPH]_0 = [KOH]_0 = [H_2O_2]_0 = 0.06$ mol/L and at 90 °C for 10 h. Also, according to TG-DTA analysis, oligo-N-salicylidenephenylhydrazone was shown to be thermally stable and resistant to thermo-oxidative decomposition. The weight loss of OSPH was found to be 20, 50 and 92.56% at 275°, 597° and 1000 °C, respectively.

Keywords: oligo-N-salicylidenephenylhydrazone, oxidative polycondensation, reaction conditions, thermo-oxidative decomposition.

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***Corresponding author.** E mail: feyzakolcu@comu.edu.tr, Fax: +90-286-522 61 01

INTRODUCTION

The oligo-phenols and their Schiff base derivatives including –OH and –CH=N groups have been used in various fields. They have useful properties such as paramagnetism, semi conductivity, employed in electrochemical cells and they resist to high energy (1-4). Polymer-metal complex compounds can be prepared with the reaction of metal salts and polymers containing electron donor groups such as –OH and –CH=N (5). Antimicrobial properties of oligophenols with Schiff base substitute and their oligomer-metal complex compounds were investigated by Kaya *et al* (6,7). Oligophenols may be used in cleaning poisonous heavy metals in the industrial wastewaters. Also, Schiff base compounds have been used to determine the transition metals in some natural food samples (8). Therefore, the syntheses of oligomer and oligomer-metal complexes are very important for analytical, environmental and food chemistry.

All azomethine polymers show a remarkable thermal stability. Non-conjugated polymers are white or cream colored and have thermal stabilities at about 300 °C in N_2 and 250 °C in air atmosphere. The color of aromatic azomethine polymers changes from yellow-orange to red-brown or black products, with thermal stabilities up to 500-550 °C in N_2 and 430-480 °C in air atmosphere. They are also resistant to radiation and their stability is found to be independent of dose rate and nature of the ionizing radiation (9).

In this study, we have investigated the effects of different parameters such as temperature, initial concentrations of NaOCl, H_2O_2 and KOH for the polymerization of N-salicylidenephenylhydrazone. The molecular structures of the obtained monomer and oligomer were confirmed by using FT-IR, UV-Vis, ^{1}H-NMR, elemental analyses. Additionally, OSPH was characterized by TG-DTA, SEC, and solubility tests.

MATERIALS AND METHODS

Materials

Phenylhydrazine, salicylaldehyde (SA), 1,4-dioxane, ethanol, methanol, benzene, acetone, ethyl acetate, n-heptane, tetrahydrofuran (THF), dimethylsulfoxide (DMSO), N,N′-dimethylformamide (DMF), H_2SO_4, toluene, pyridine, n-hexane, hydrochloric acid (HCl) (37%) and H_2O_2 (35% solution in water) were supplied from Merck Chemical Co. (Germany) and they were used as received. Sodium hypochlorite (NaOCl), (30% solution in water) was supplied from Paksoy Chem. Co. (Turkey).

Preparation of N-salicylidenephenylhydrazone (SPH)

N-salicylidenephenylhydrazone (SPH) was prepared by the condensation of salicylaldehyde (0.025 mol) and phenylhydrazine (0.025 mol) in 25-mL methanol under reflux for 3 h (Scheme 1). The precipitated N-salicylidenephenylhydrazone (SPH) was filtered and recrystallized from methanol and dried in a vacuum desiccator (m.p.: 125 °C, yield, 90%).

Anal. calcd. for SPH: C, 73.59; H, 5.66; N, 13.21. Found: C, 73.35; H, 5.52; N, 13.12. UV-Vis (λ_{max}): 220, 236, 280 and 340 nm. FT-IR (KBr, cm^{-1}): ν (O-H) 3470 s, ν (C-H aryl) 3030 m, ν (C=N) 1615 s, ν (N-H) 3300 s, ν (C=C) 1580, 1455, 1450 s, ν (Ar-O) 1280 s. ^{1}H-NMR (DMSO): δ ppm, 10.15 (s, 1H, O$\underline{H}$); 8.30 (s, 1H, C$\underline{H}$=N); 5.60 (s, 1H, N$\underline{H}$); 7.48 (d, 1H, Ar-Ha), 6.85 (t, 1H, Ar-Hb), 7.25 (t, 1H, Ar-Hc), 6.70 (d, 1H, Ar-Hd) 7.00 (d, 2H, Ar-Hee′) 7.15 (t, 2H, Ar-Hff′) 6.70 (t, 1H, Ar-Hg).

Synthesis of Oligo-N-salicylidenephenylhydrazone with air

SPH (4.24 g, 0.02 mol) was dissolved in an aqueous solution of KOH (10%) (1.12 g, 0.02 mol) and placed into a 100 mL three-necked round-bottomed flask. It was fitted with a condenser, thermometer, stirrer and a glass tube was placed over condenser for air to pass through. The reaction mixture was heated at various temperatures and times. Air was passed through an aqueous solution of KOH (20%) before being sent to the reaction tube to eliminate CO_2 in the air. It was cooled to room temperature and then, 0.02 mol of HCl (37%) was added to the reaction mixture. Unreacted monomer was separated from the reaction products by washing with n-heptane. The mixture was filtered and washed with hot water for separating it from mineral salts and dried in the oven at 110 °C

Anal. calcd. for OSPH : C, 74.29; H, 4.76; N, 13.33. Found: C, 73.75; H, 4.62; N, 13.00. UV-Vis (λ_{max}): 221, 241, 291 and 346 nm. FT-IR (KBr, cm^{-1}): ν (O-H) 3500 s, ν (C-H aryl) 3030 m, ν (C=N) 1600 s, ν (N-H) 3280 s, ν (C=C) 1500, 1470 s, ν (Ar-O) 1270 s. ^{1}H-NMR (DMSO): δ ppm, 10.40 (s, 1H, O$\underline{H}$); 9.20 (s, 1H, C$\underline{H}$=N); 6.55 (s, 1H, N$\underline{H}$); 7.30 (s, 1H, Ar-Ha), 6.80 (s, 1H, Ar-Hc), 6.50 (d, 2H, Ar-Hdd′) 7.00 (t, 2H, Ar-Hee′) 6.70 (t, 1H, Ar-Hf).

Synthesis of Oligo-N-salicylidenephenylhydrazone with NaOCl

OSPH was synthesized through oxidative polycondensation of SPH using NaOCl (30%) as the oxidant. SPH (4.24 g, 0.02 mol) was placed into a 50-mL three-necked round-bottomed flask and dissolved in an aqueous solution of KOH (10%, 1.12 g, 0.02 mol). The flask was connected to a condenser, a thermometer, a magnetic stirrer, and an additional funnel containing NaOCl. After heating at 60 °C, NaOCl (30%) was added drop wise within 20 min. The reaction mixture was heated at various temperatures and times. The mixture was neutralized by HCl (37%, 0.02 mol) at room temperature. Unreacted monomer was separated from the reaction products by washing with cold n-heptane. The mixture was filtered and washed with hot water for separating from mineral salts and dried in the oven at 110 °C.

Synthesis of Oligo-N-salicylidenephenylhydrazone with H_2O_2

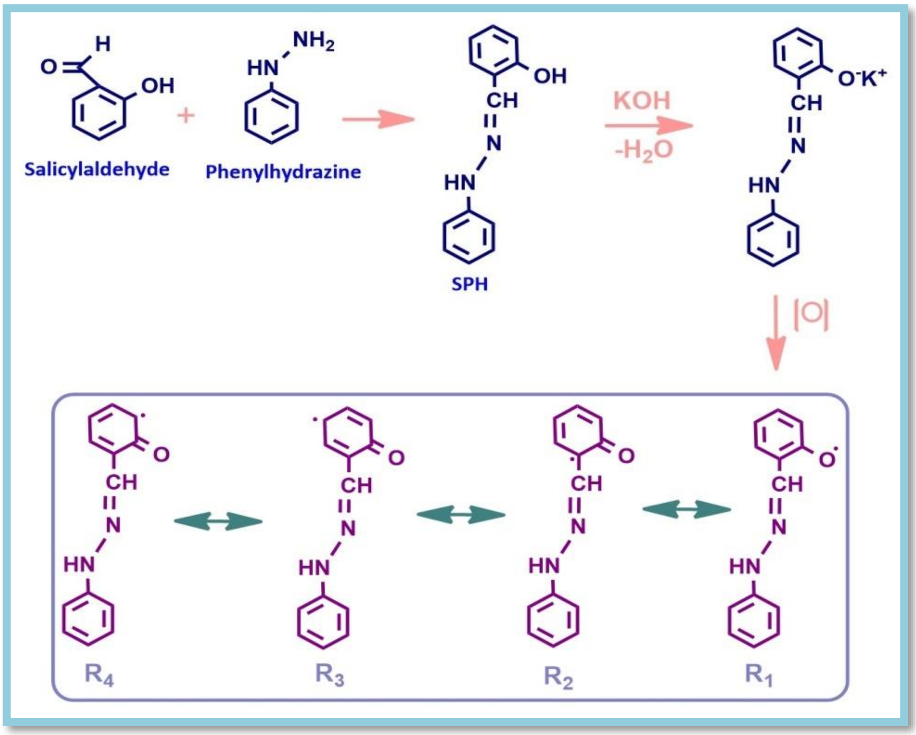
OSPH was synthesized through oxidative polycondensation in an aqueous alkaline medium using H_2O_2 (35%) as the oxidant. Polymerization was performed in a 50 mL three-necked round-bottomed flask connected to a condenser, a thermometer and a magnetic stirrer, and an addition to funnel containing H_2O_2. SPH (4.24 g, 0.02 mol) was dissolved in an aqueous solution of KOH (10%, 1.12 g, 0.02 mol). After heating at 40 °C, H_2O_2 was added drop wise within 20 min. The reaction mixture was heated at various temperature and time. The mixture was neutralized by HCl (37%, 0.02 mol) at room temperature. Unreacted monomer was separated from the reaction products using n-heptane. The mixture was filtered and washed with hot water for separating from mineral salts and dried in the oven at 110 °C.

Characterization Techniques

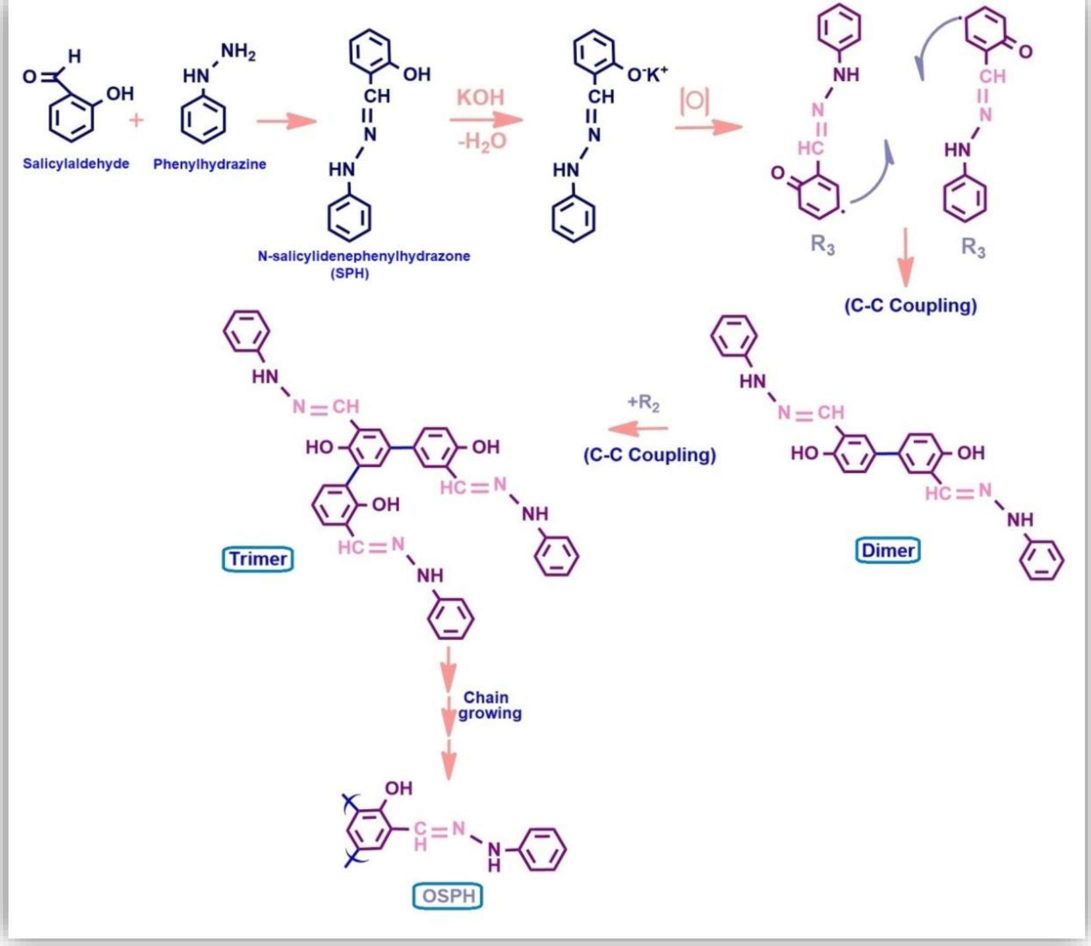
FT-IR and UV-Vis spectra were recorded by Mattson FT-IR 8010 and UV-160 (Shimadzu), respectively. The FT-IR spectra were recorded using KBr disc (4000-400 cm^{-1}). UV-Vis spectra of SPH and OSPH were determined using THF. Elemental analysis was carried out by a Carlo Erba 1106. OSPH was characterized by ^{1}H-NMR spectra (Bruker AC FT-NMR 400 MHz spectrometer) and the spectra were recorded at 25 °C using deuterated DMSO as solvent. Tetramethylsilane (TMS) was used as internal standard. Thermal data were obtained by STA 409C NETZSCH-Geratebau GmbH Thermal Analysis. TG-DTA measurements were performed between 20 and 1000 °C (in air, rate 10°C/min). SEC analyses were used to investigate both size and polydispersity of the synthesized polymers that is, the ability to be able to find the molecular range distribution of the polymer molecules (PDI), as well as the number-average molecular weight (M_n) and the average molecular weight (M_w), by using a mixture of polystyrene standards (Polymer Laboratories; the peak molecular weights, Mp, between 162 and 19880 g mol^{-1}) for calibration of the instrument SEC (Shimadzu 10AVp series HPLC-SEC system), and a SGX (100 Å and 7.7 nm diameter loading material) 3.3 mm i.d. x 300 mm column. DMF as eluent at a flow rate of 0.4 mL/min was used in SEC analyses. A refractive index detector (at 25°C) was used to analyze the product.

RESULTS AND DISCUSSION

Possible four main coupling modes for the polymerization of N-salicylidenephenylhydrazone (SPH) and the general synthetic route for the synthesis of oligo-N-salicylidenephenylhydrazone (OSPH) and were identified and given in Schemes 1 and 2 respectively.



Scheme 1. Synthetic route of N-saliciylidenehydrazone (SPH) and possible resonance and coupling modes of SPH.



Scheme 2. Synthetic pathway of OSPH.

Specifying the optimal reaction conditions and solubility of OSPH

Oxidative polymerization of SPH was observed to be affected to different reaction parameters. To specify the optimum polycondensation conditions, polymerization of SPH was carried out using various oxidants such as NaOCl, H_2O_2 and air in aqueous KOH solution by tuning the temperature in the range between 40 and 95 °C at constant SPH, KOH and oxidant concentration (Figure 1(a)). Additionally, the polymerization was carried out at different reaction time to assign the optimum reaction time (Figure 1(b)).

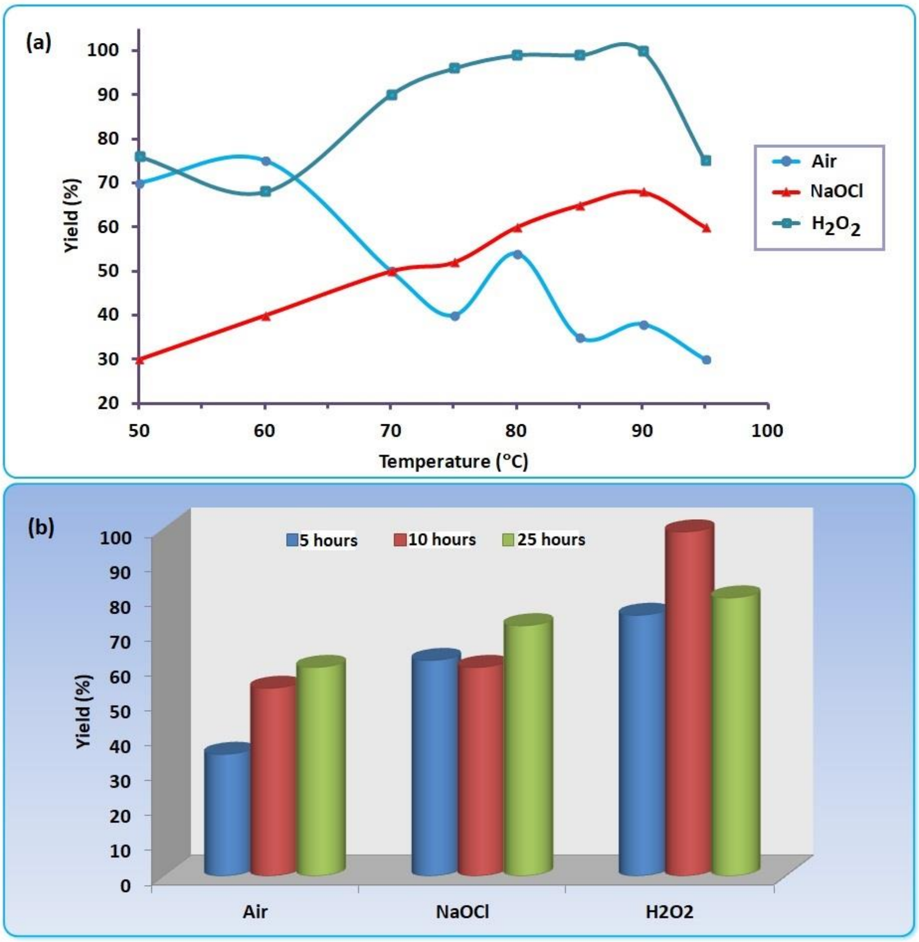


Figure 1. Reaction yields of OSPH at various temperature (a) and time (b) (Conditions: [SPH]= 0.06 mol L^{-1}, [KOH]= 0.06 mol L^{-1}, [NaOCl]= 0.06 mol L^{-1}, [H_2O_2]= 0.06 mol L^{-1} and air= 8.5 L h^{-1}).

The oxidative polycondensation conditions of SPH with air as oxidant in aqueous alkaline medium are given in Table 1. The oxidative polycondensation of SPH was not observed in THF and acetic acid medium at various temperature and time. SPH was soluble in aqueous KOH but no reaction was observed when it was heated for 3 h at 80 °C. However, the oxidative polycondensation of SPH took place in an aqueous alkaline solution and the color turned into brown by adding oxidants such as NaOCl, H_2O_2 and air. With $[SPH]_0 = [KOH]_0 = 0.06$ mol/L, the yield of OSPH was 75% by air oxidant for 5h at 50 °C (Table 1). While the yield of OSPH was 80% for 5 h at 60 °C, the polymerization yield was found to be 35% for 5 h at 80°C. The reason may be the disruption of the polymer into monomers at high temperatures.

Table 1. The optimum oxidative polycondensation conditions of SPH in the aqueous alkaline medium with air oxidant.

Run	$[SPH]_0$ (mol/L)	$[KOH]_0$ (mol/L)	Air (L/h)	Temp. (°C)	Time (h)	Yield of SPH (%)
1	0.06	0.06	8.5	50	5	75
2	0.06	0.06	8.5	60	5	80
3	0.06	0.06	8.5	80	25	60
4	0.06	0.06	8.5	80	3	20
5	0.06	0.12	8.5	80	5	35
6	0.06	0.06	8.5	70	10	50
7	0.06	0.06	8.5	75	10	40
8	0.06	0.06	8.5	80	10	54
9	0.06	0.06	8.5	85	10	35
10	0.06	0.06	8.5	90	10	38
11	0.06	0.06	8.5	95	10	30

As seen in Table 1, the yield of oligomer depends on temperature and time variables. For air as the oxidant, the maximum yield of OSPH was observed as 80% at optimum conditions, such as $[SPH]_0 = [KOH]_0 = 0.06$ mol/L for 5 h at 60 °C (Table 1).

The oxidative polycondensation conditions of SPH with 30% NaOCl solution in aqueous alkaline medium are given in Table 2. The yield of OSPH was 77% at optimum conditions such as $[SPH]_0 = [KOH]_0 = [NaOCl]_0 = 0.06$ mol/L for 3 h at 80 °C. The yield of OSPH was 73% at the reaction conditions such as $[SPH]_0 = [KOH]_0 = 0.06$ mol/L and $[NaOCl]_0 = 0.12$ mol/L for 10 h at 90 °C. The various conditions of OSPH are given for NaOCl in Table 2.

Table 2. The optimum oxidative polycondensation conditions of SPH in the aqueous alkaline medium with NaOCl as the oxidant.

Run	**$[SPH]_0$ (mol/L)**	**$[KOH]_0$ (mol/L)**	**$[NaOCl]_0$, (mol/L)**	**Temp. (°C)**	**Time (h)**	**Yield of SPH (%)**
1	0.06	0.06	0.06	70	10	50
2	0.06	0.06	0.06	70	5	58
3	0.06	0.06	0.06	80	10	60
4	0.06	0.06	0.06	80	5	62
5	0.06	0.06	0.06	90	10	68
6	0.06	0.06	0.06	90	5	65
7	0.06	0.06	0.06	95	10	60
8	0.06	0.06	0.06	95	5	55
9	0.06	0.06	0.06	70	2	40
10	0.06	0.06	0.06	80	3	77
11	0.06	0.06	0.06	90	25	73
12	0.06	0.06	0.12	90	10	73
13	0.06	0.12	0.06	90	10	68

The oxidative polycondensation conditions of SPH with 35% H_2O_2 solution in aqueous alkaline medium are given in Table 3. The yield of OSPH was 100% at optimum conditions such as $[SPH]_0$ = $[KOH]_0$ = $[H_2O_2]_0$ = 0.06 mol/L for 10 h at 90 °C. The polymerization of SPH was 64% at the reaction conditions such as $[SPH]_0$ = $[KOH]_0$ = 0.06 mol/L and $[NaOCl]_0$ = 0.12 mol/L for 10 h at 95 °C. While the polymerization of SPH was 100% and 75% for 10 h at 90 °C and for 10 h at 95 °C, respectively. The reason may be the disruption of the polymer into monomers at high temperatures.

Table 3. The optimum oxidative polycondensation conditions of SPH in the aqueous alkaline medium with H_2O_2 as the oxidant.

Run	[SPH]$_0$ (mol/L)	[KOH]$_0$ (mol/L)	[H_2O_2]$_0$ (mol/L)	Temp. (°C)	Time (h)	Yield of SPH (%)
1	0.06	0.06	0.06	50	10	76
2	0.06	0.06	0.06	60	10	68
3	0.06	0.06	0.06	70	10	90
4	0.06	0.06	0.06	75	10	96
5	0.06	0.06	0.06	80	10	99
6	0.06	0.06	0.06	85	10	99
7	0.06	0.06	0.06	90	10	100
8	0.06	0.06	0.06	95	10	75
9	0.06	0.06	0.06	95	2	60
10	0.06	0.06	0.06	95	25	46
11	0.06	0.06	0.12	95	10	64
12	0.06	0.06	0.06	75	5	70

Since the growing macromolecular chain come out of solution during the polycondensation at low polymerization degrees, high molecular weight polymers can not be obtained. Oxidatively polymerized products of OSPH synthesized by air, H_2O_2 and NaOCl in aqueous alkaline medium were brown solid powders. OSPH was soluble in common organic solvents such as $CHCl_3$, DMSO, concentrated H_2SO_4 and aqueous KOH, DMF, benzene, toluene, acetone, THF, ethyl acetate, 1,4-dioxane, ethanol, methanol and pyridine, but it was insoluble in n-hexane and n-heptane.

Characterization of SPH and OSPH

Based on the SEC chromatograms of OSPH, the calculated number-average molecular weight (M_n), weight average molecular weight (M_w), and polydispersity index (PDI) values were organized in Table 4. According to SEC analyses, M_n, M_w and PDI values of OSPH were found to be 1436 g mol^{-1}, 1631 g mol^{-1} and 1.14, respectively, for NaOCl oxidant. Total values delineated that M_n, M_w and PDI values of OSPH were found to be 850 g mol^{-1}, 1155 g mol^{-1} and 1.36, respectively, for air as the oxidant. At the molecular weight distribution of OSPH, two fractions were observed with 93% of OSPH for the low molecular weight (for the first fraction: M_n, 1418 g mol^{-1}; M_w, 1650 g mol^{-1}; PDI, 1.16), but 7% of its weight for the high molecular weight (for the second fraction: M_n, 37632 g mol^{-1}; M_w, 40217 g mol^{-1}; PDI, 1.07). M_n, M_w and PDI values of OSPH were found to be 825 g mol^{-1}, 1660 g mol^{-1} and 2.01, respectively, for H_2O_2 oxidant. Two peaks were observed with 92% of OSPH for the low molecular weight (for the first fraction: M_n, 1635 g mol^{-1}; M_w, 2157 g mol^{-1}; PDI, 1.32), but 8 % of its weight for the high molecular weight (for the second fraction: M_n, 8736 g mol^{-1}; M_w, 10123 g mol^{-1}; PDI, 1.16).

Table 4. SEC analyses data of the synthesized compounds.

	Total			Fraction I				Fraction II			
Compounds	**M_n**	**M_w**	**PDI**	**M_n**	**M_w**	**PDI**	**%**	**M_n**	**M_w**	**PDI**	**%**
OSPH (air)	850	1155	1.36	1418	1650	1.16	93	37632	40217	1.07	7
OSPH(NaOCl) (na(NaOCl)	1436	1631	1.144	1436	1631	1.144	100	-	-	-	-
OSPH (H_2O_2)	825	1660	2.01	1635	2157	1.32	92	8736	10123	1.16	8

The electronic spectra of SPH and OSPH were recorded in THF. UV-Vis spectrum of SPH was observed with the bands at 220, 240, 287, 315 and 342 nm. K band belongs to –OH and azomethine groups of SPH observed at 240 and 315 nm, respectively. B and R bands for SPH were observed at 287 and 342 nm, respectively. R band for -CH=N group of SPH was observed at 342 nm. UV-Vis spectrum of OSPH was observed with the same bands at 221, 241, 291 and 346 nm. K bands for –OH and azomethine groups of OSPH were observed at 241 and 291 nm, respectively. R band for -CH=N group of OSPH was seen at 346 nm.

FT-IR spectrum of SPH (Figure 2) shows the characteristic peaks of the functional groups for SPH. Phenolic O-H stretching and secondary N-H stretching frequencies were observed at 3470 cm^{-1} and 3300 cm^{-1}, respectively. The peak at 2980 cm^{-1} was attributed to the aromatic –C-H stretching frequency. The peak for imine (-HC=N-) group was observed at 1615 cm^{-1}. Aromatic -C=C stretching and the sharp peak attributed to the N-H bending in the molecular structure of SPH were observed at 1580 cm^{-1} and 1380 cm^{-1}, respectively. The results confirm the structure of Schiff base containing an imine (–HC=N-) bond in the structure of SPH.

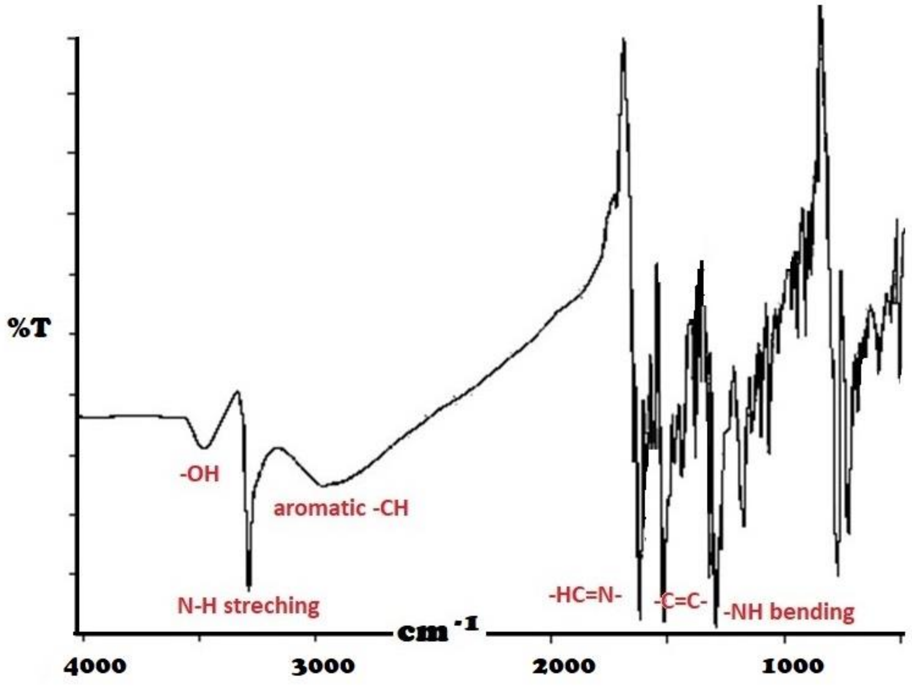


Figure 2. FT-IR spectrum of N-salicylidenehydrazone (SPH).

In the FT-IR spectrum of OSPH (Figure 3), the stretching frequencies of -OH and –CH=N groups were observed at 3500 and 1600 cm^{-1}, respectively. The characteristic peaks clearly show the binding of functional groups of the synthesized products (10,11).

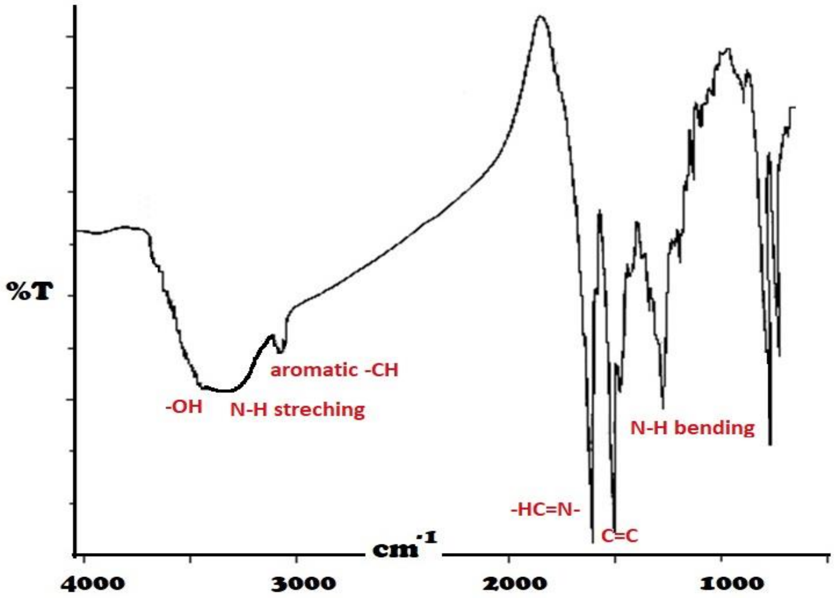


Figure 3. FT-IR spectrum of oligo-N-Salicylidenehydrazone (OSPH).

According to the spectral results and SEC analyses, OSPH's molecular structure is given as in Scheme 3. In order to identify the structures of Schiff base (SPH) and its oligomer (OSPH), ^{1}H-NMR spectra were recorded in DMSO-d$_6$. At the ^{1}H-NMR spectrum of SPH, the characteristic peaks of the functional groups were observed. Phenyl-O$\underline{H}$ group at 10.15 ppm, azomethine (-C$\underline{H}$=N) group at 8.30 ppm and secondary amine (–N$\underline{H}$) group at 5.60 ppm. The ^{1}H-NMR and FT-IR results showed the formation of oligomeric macromolecules from SPH unit by the oxidative polymerization through the carbons which are *ortho* and *para* positioned to hydroxyl group. At the ^{1}H-NMR spectrum of OSPH, the characteristic peaks of the functional groups were observed. Phenyl-O$\underline{H}$ group at 10.40 ppm, azomethine (-C$\underline{H}$=N) group at 9.20 ppm and secondary amine (–N$\underline{H}$) group at 6.55 ppm (Scheme 3). The FT-IR spectral data and the results of ^{1}H-NMR spectra of OSPH confirm the formation of the synthesized molecules. The spectral results are given below the synthesis part of the compounds.

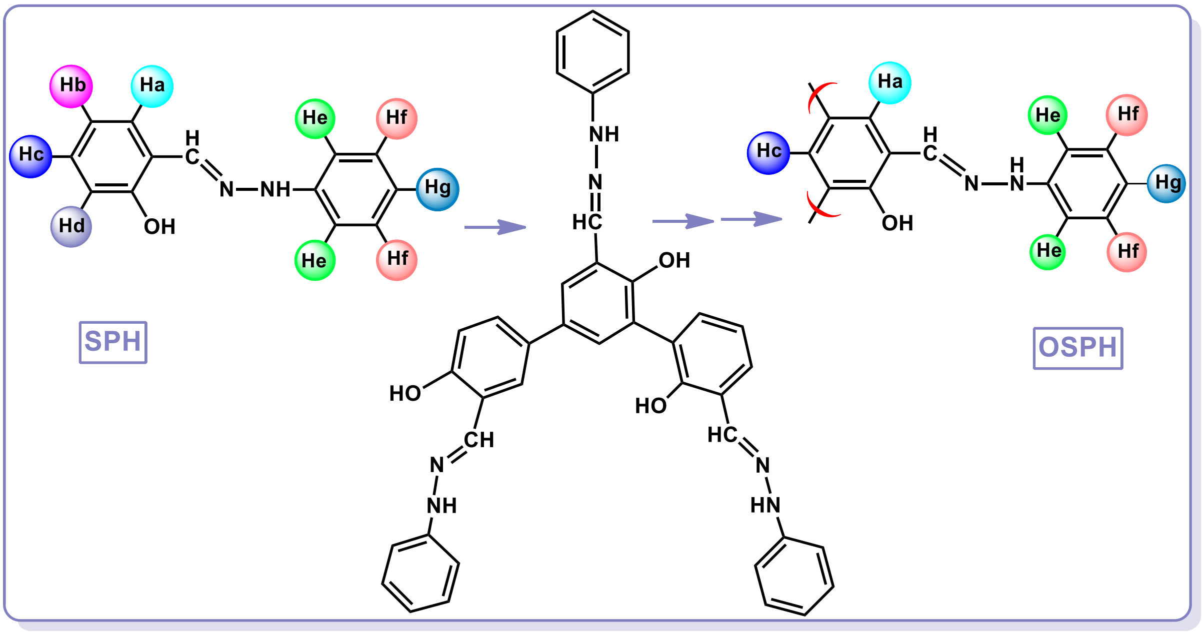


Figure 4. The structures of SPH and OSPH.

Thermal analysis of OSPH

The TG-DTA measurements of OSPH were performed under air atmosphere in the temperature range from 20 °C to 1000 °C in order to investigate its thermal stability. Figure 4 shows the TG-DTA curves for OSPH which was oxidatively polymerized of SPH in the aqueous alkaline medium by air as the oxidant. According to TG curves, OSPH started to degrade at 190 °C. OSPH thermo-oxidatively degraded in three steps. According to TG curve of OSPH, T_{max} values were observed at 506, 625 and 820 °C. 20% and 50% weight losses of OSPH were found to be at 275 °C and 597 °C, respectively. The weight loss of OSPH was 92.56% at 1000 °C. 5% weight loss in the range of 50-150 °C assigned to water removal was also seen on TG curve of OSPH. TG-DTA analyses showed that OSPH was found to be thermally stable and resistant to thermo-oxidative decomposition.

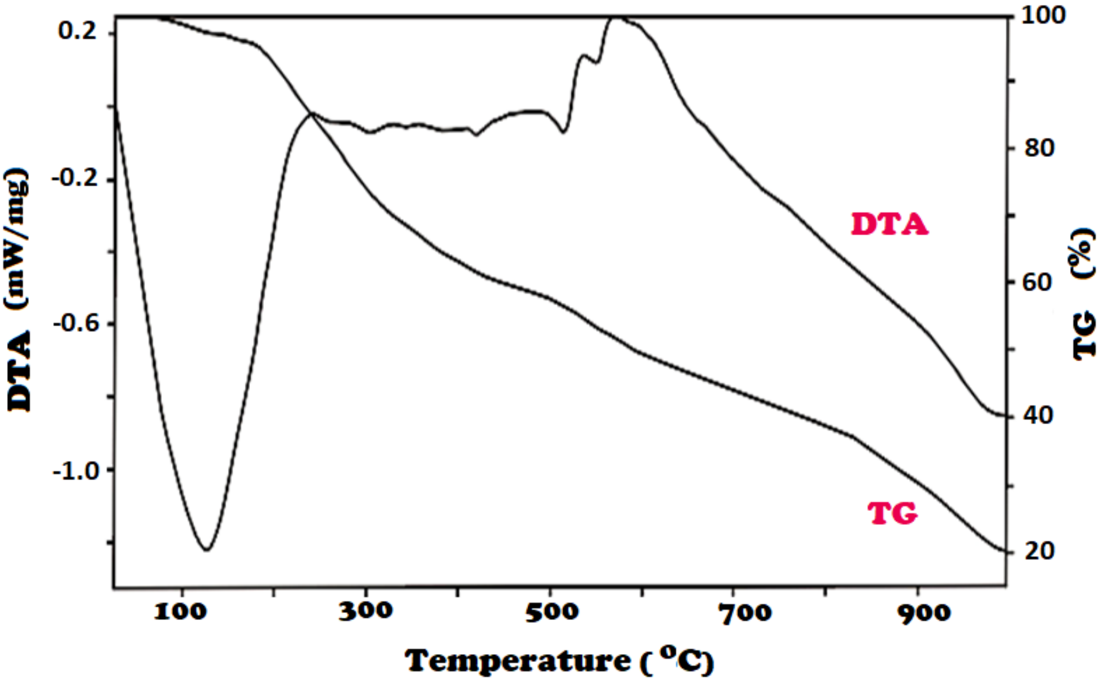


Figure 5. TG/DTA curves of oligo-N-Salicylidenehydrazone (OSPH).

CONCLUSIONS

In this study, the synthesized Schiff base (SPH) was transformed into oligo-N-salicylidenephenylhydrazone (OSPH) using air, H_2O_2 and NaOCl as oxidants in an aqueous alkaline medium. According to spectral analyses, the synthesized oligo-N-salicylidenephenylhydrazone oligomer was confirmed by bonding units through *ortho* and *para* carbons to hydroxyl group of N-salicylidenephenylhydrazone. M_n, M_w and PDI values of OSPH were found to be 1436 g mol^{-1}, 1631 g mol^{-1} and 1.14; 850 g mol^{-1}, 1155 g mol^{-1} and 1.36; 825 g mol^{-1}, 1660 g mol^{-1} and 2.01, respectively, for NaOCl, air and H_2O_2 oxidants. Based on TG analysis OSPH in the aqueous alkaline medium with air oxidant, carbonaceous residue value of OSPH was found to be 7.44% at 1000 °C.

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