



Synthesis and Characterization of Linear Thermally Stable Polyester Contain Schiff Bases



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ACOPOLYMERS are important compounds today because of their wide applications in various industrial and medical fields and in other fields. Therefore, the study of preparing many of these polymers has been studied. This research involved preparing linear polyester contain Schiff-base unit by interfacial polycondensation three type diacid chloride (Succinoyl Chloride, Adipoyl Chloride and sebacoyl Chloride) in a chloroform /water system employing phase transfer catalyst with monomer containing two hydroxyl which is derived from a 4-hydroxy aniline reacted with a 4-hydroxy benzaldehyde monomer. The monomer (EM1) and polyester (EM2, EM3, and EM4) were characterized by FTIR, ¹H NMR, ¹³C NMR and TGA analysis. The polyester–Schiff base so obtained show good thermal stability.

Keyword: Polyester, Schiff bases, Interfacial polycondensation, Phase transfer catalyst, FTIR, ¹H NMR, ¹³C NMR and TGA analysis.

Introduction

The word polymer is derived from the Greek poly and meros, meaning many and parts, respectively. Some scientists prefer to use the word macromolecule, or large molecule, instead of polymer[1]. The polymerization process is the formation of a polymer molecule, at which time the chain is constructed continuously [2, 3]. If the resulting polymer has more than one molecule in the chain, it is usually referred to as a copolymer[3]. Polymerization mechanisms involve the conversion of monomers into a large multiple of themselves. Copolymerization can occur when two or more such monomers are involved in the creation of a high molar mass compound. Polymerization can be expected if there are olefinic compounds in the feedstock. Polymers can be created by a wide variety of processing techniques that include bulk, solution, suspension, emulsion, and precipitation methods[4]. Polymerization processes typically have the characters of fast reaction rate, the high viscosity of reaction mixture, and is highly

exothermic. Therefore, properly managing heat transfer is very critical for controlling the polymerization reaction. It is also the most important factor for reactor selection and design and selecting the polymerization process and conditions [5]. Through the interaction of small molecules (Monomers) with each other to give either linear polymers is a long continuous chain or network polymers formed as a result of the chemical interaction between linear polymer chains or three-dimensional fish-net configuration polymers from build-up from monomeric resinous reactants [6]. The process of interaction is called cross-linking and is the main distinguishing element of a thermosetting material. The “thermo” implies that the cross-linking proceeds through the influence of heat energy input, although, much cross-linking occurs at room temperature (25°C, 77°F) and below. In recent years, some researchers have chosen to describe thermosets that cross-link at or below room temperature as “chemosets,” although this term has not been universally accepted. The “setting” term references the fact

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that an irreversible reaction has occurred on a macro scale [7, 34]. The polymerization process takes place in three distinct steps:

1. Chain initiation: an initiator which starts the polymerization process. The reactive initiation molecule can be a radical, cation, anion or/and organometallic complex.
2. Chain propagation: a monomer adds onto chain and each new monomer unit creates an active site for the next attachment.
3. Chain termination: the radical, cation or anion is "neutralized" stopping the chain propagation [8].

Polymeric Schiff bases, which are also called Polyazomethines, have been the subject of research for more than five decades [9]. They are synthesized by polycondensation reaction between diamine and dialdehyde or diketone [10]. They are an important class of compounds and useful complexing ligands for a number of transition metal ions and indicate paramagnetism, semiconducting and resistance to high energy [11,12]. They are used to prepare composite materials having high resistance at high temperatures, thermostabilized, photoresistors, flame resistance materials, and components of electrochemical cells. The Schiff base polymers demonstrate antimicrobial activity against bacteria, yeast, and fungi. Thus these can be used for the purification of industrial contaminants from heavy metals and microbiological organisms and are significant for environmental applications [13,14, 15, 28, 31]. The polymeric Schiff bases are attractive polymers, but they indicate poor solubility in common organic solvents and are difficult to liquefy for practical applications in various fields [16]. Organic polymers generally have long-term stability and good flexibility [17]. The polyester-amides are a particular class of polymers possessing a special structure due to the regular presence of ester and amide groups in the same macromolecular chain, which gives them the properties intermediate between the polyester and polyamide. Polyesteramides (PEA) seem to be very promising materials combining the favorable properties of aromatic polyesters and polyamides [18]. Ethylene propylene diene monomer rubber (EPDM), which has a small amount of a third monomer, a diene (two carbon-carbon double bonds in it). The diene monomer leaves a small amount of unsaturation in the polymer chain that can be used for sulfur cross-

linking [19]. Polymerization in a suspended solution medium [20] this polymerization is usually done in a liquid medium, which is water. Polymerization in suspended solutions has several preferences in relation to other methods, including:

- Heat can be easily regulated.
- The reaction speed is great.
- Spreading the product inside the solution with a small distance.
- High degree of polymerization.
- Not to use expensive organic solutions [27].

Studies show that the uses of unsaturated polyester in working life are varied and numerous, as the distinctive properties of this substance made it popular in many industrial applications.

Experimental

Chemical and Equipment

Succinic acid, Adipic acid, Sebacic acid, Sodium hydroxide and 4-hydroxy benzaldehyde were obtained from Aldrich Chemical Ltd., Citramide (cetyl-triethylammonium bromide) and 4-Aminophenol from Alfa Aesar. Solvents were purchased from Fluke. Succinyl chloride (SUCI), Adipoyl chloride (ADCI) and Sebacoyl chloride (SECI) were obtained from condensation reaction of their corresponding dicarboxylic acids with thionyl chloride, in the presence 1 ml of pyridine as a catalyst for 12 hours. These products were crystallized by heptane. The melting points were measured on an SMP30-Melting Point Apparatus and were uncorrected. $^1\text{H-NMR}$ spectra were recorded on nucleic magnetic resonance model ultra-shield 400MHz, Varian Co., USA, using TMS as internal reference and DMSO- d_6 as solvent. IR spectra were recorded on Shimadzu FT-IR 8400S Spectrophotometer, Germany, by using KBr discs. Q50 thermo gravimetric analyzer from TA Instruments.

Synthesis of EMI compounds [29, 32]

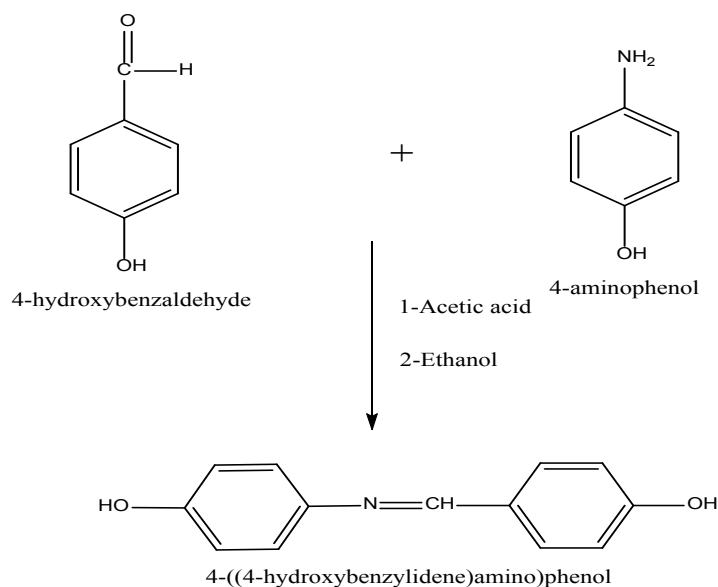
4-[4-hydroxybenzylidene) amino] phenol was synthesized by an equimolar mixture of 5 mM of 4-hydroxybenzaldehyde in 10 ml ethanol (absolute) and 5 mM of 4-aminophenol dissolved in 10 ml ethanol were transferred into a 100 ml round bottom flask equipped with condenser and a magnetic stir bar. Then two drops of glacial acetic acid was added as shown in Scheme I. The reaction mixture was refluxed with continuous stirring

for 4 hour under nitrogen atmosphere, after this step cool the mixture to room temperature and leave it a crash ice bath. The precipitated product was collected by filtration, washed with ethanol and dried in a vacuum oven at 60°C for 2 h. A yellow product was obtained in 96% yield with a melting point 212°C, the physical and chemicals properties show in Table (1 & 2) and figure 9.

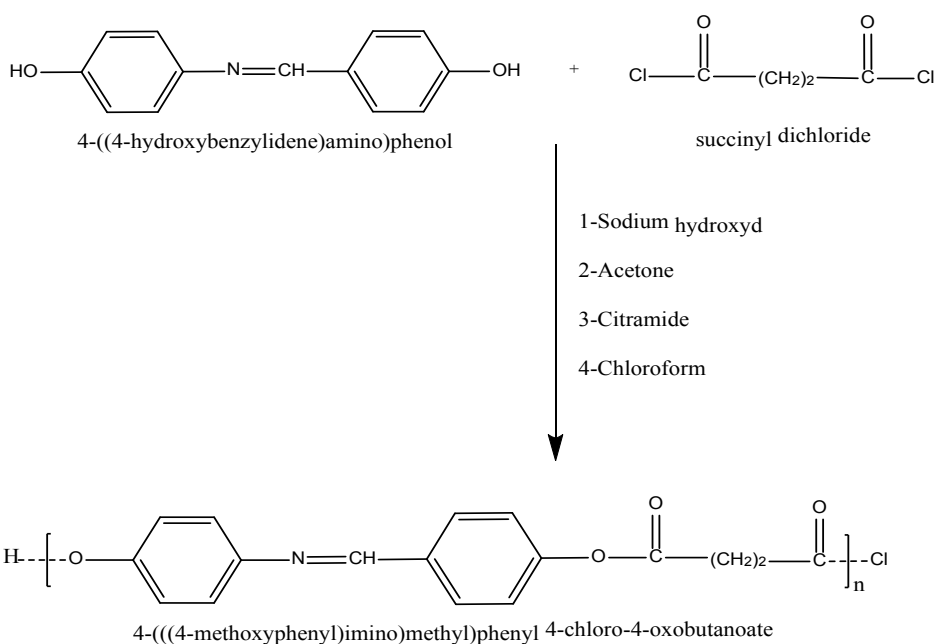
Synthesis of EM2 compounds [21, 32]

Succinyl chloride (SUCI) and 4-[4-hydroxybenzylidene) amino] phenol was polymerized

using interfacial polycondensation with chloroform – water interphase system and citramide as phase transfer catalyst. In a round bottom flask mixed 25 mM of sodium hydroxide dissolved in 10 ml of water with 12.5 mM of 4-[4-hydroxybenzylidene) amino] phenol, 25 mg citramide (phase transfer catalyst) dissolved in 10 ml of water was added. The organic phase was prepared by dissolved 12.5 mM (SUCI) in 35 ml of chloroform, the organic phase was added to water phase rapidly mixing in room temperature



Scheme I



Scheme II

with stirrer after 5 min. add (35 ml) from acetone as shown in Scheme II. The polymer was separated by filtration and washed with acetone and distilled water to removed un-reacted starting material and second product such as sodium chloride. The product was dried in a vacuum oven at 60°C for 1 h. the physical and chemicals properties show in Table (1 & 2) and Figure 9.

Synthesis of EM3 compounds [22, 32]

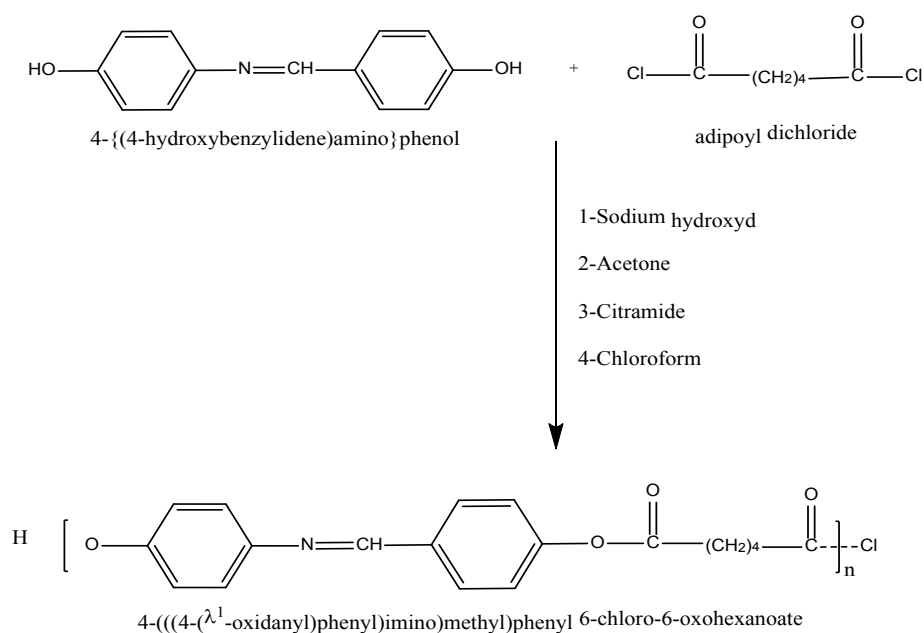
Adipoyl chloride (ADCI) and 4-[4-hydroxy benzylidene) amino] phenol was polymerized using interfacial polycondensation with chloroform – water interphase system and citramide as phase transfer catalyst. In a round bottom flask mixed 25 mM of sodium hydroxide dissolved in 10 ml of water with 12.5 mM of 4-[4-hydroxy benzylidene) amino] phenol, 25 mg citramide (phase transfer catalyst) dissolved in 10 ml of water was added. The organic phase was prepared by dissolved 12.5 mM (ADCI) in 35 ml of chloroform, the organic phase was added to water phase rapidly mixing in room temperature with stirrer after 5 min. add (35 ml) from acetone as shown in Scheme III. The polymer was separated by filtration and washed with acetone and distilled water to removed un-reacted starting material and second product such as sodium chloride. The product was dried in a vacuum oven at 60°C for 1 h. the physical and chemicals properties show in Table (1 & 2) and Figure 9.

Synthesis of EM4 compounds [23, 24, 32]

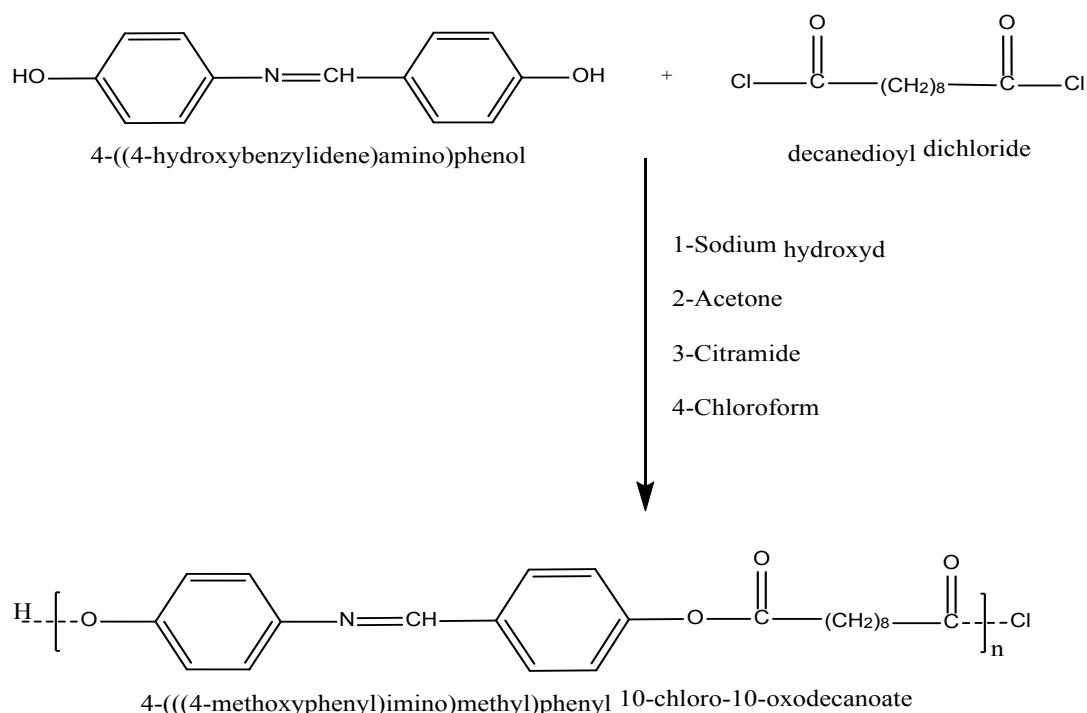
Sebacoyl chloride (SECI) and 4-[4-hydroxy benzylidene) amino] phenol was polymerized using interfacial polycondensation with chloroform – water interphase system and citramide as phase transfer catalyst. In a round bottom flask mixed 25 mM of sodium hydroxide dissolved in 10 ml of water with 12.5 mM of 4-[4-hydroxy benzylidene) amino] phenol, 25 mg citramide (phase transfer catalyst) dissolved in 10 ml of water was added. The organic phase was prepared by dissolved 12.5 mM (SECI) in 35 ml of chloroform, the organic phase was added to water phase rapidly mixing in room temperature with stirrer after 5 min. add (35 ml) from acetone as shown in Scheme IV. The polymer was separated by filtration and washed with acetone and distilled water to removed un-reacted starting material and second product such as sodium chloride. The product was dried in a vacuum oven at 60°C for 1 h. the physical and chemicals properties show in table (1 & 2) and figure 9. The general steps for synthesis compounds summarized In Scheme V.

Result and Discussion

In this paper the synthesis of some substituted polymer from 4-hydroxy benzaldehyde with 4-hydroxy aniline in abs. ethanol with glacial acetic acid to give (4-hydroxy benzylidene) aminophenol (EM1) the product name is Schiff base the reaction mechanism [33] show as follow in Scheme VI.



Scheme III



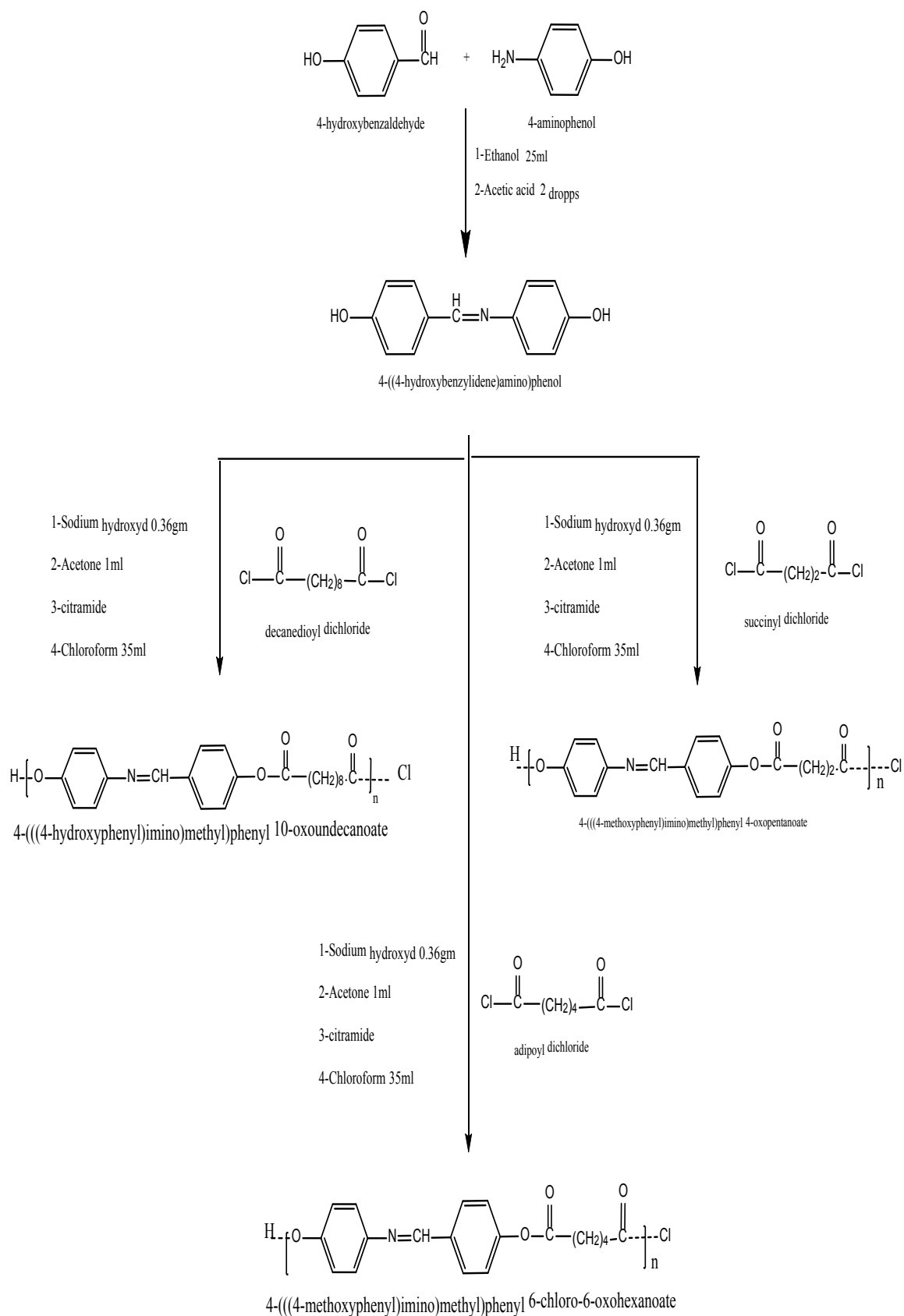
Scheme IV

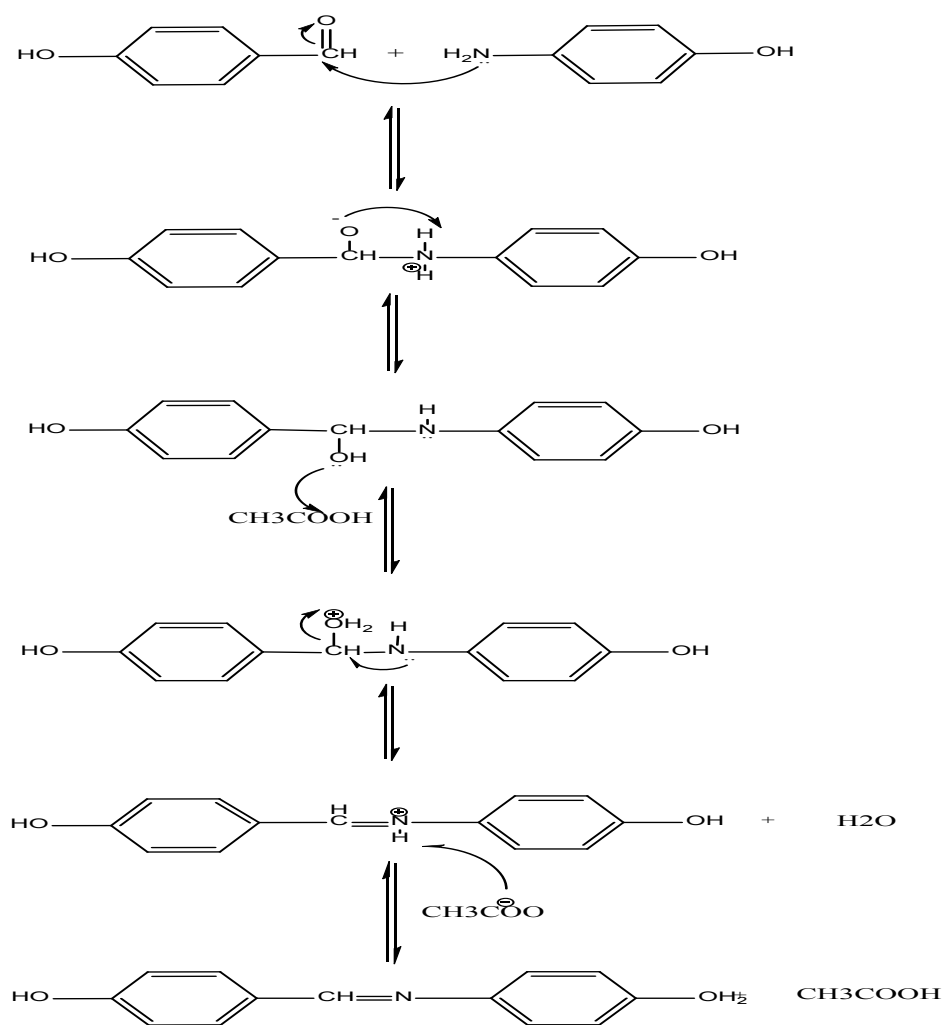
TABLE 1.physical properties and Melting Point

No. of Compound	M.P. °C	Time of reaction(min)	Color	Yield %
EM1	214-215	240	Yellow	83
EM2	200-202	5	Brown	78
EM3	189-191	5	Pale Brown	86
EM4	205-206	5	Brown	84

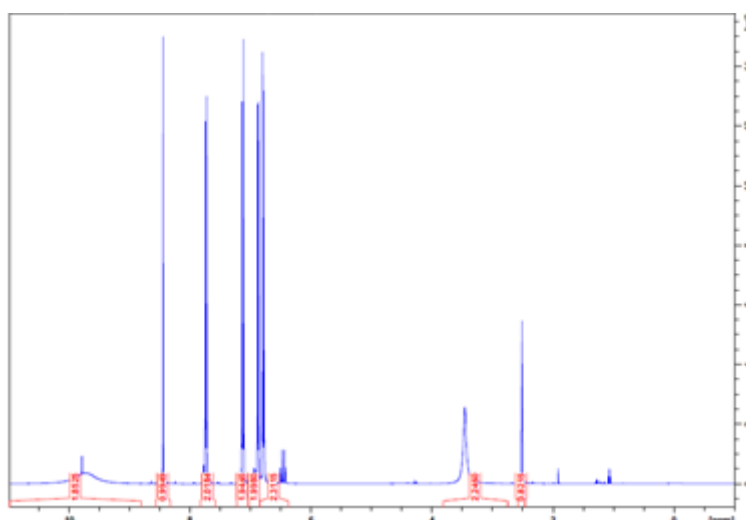
The Infrared spectra of compound (EM1) showed absorption wide broadcasts at 3300-3500 cm^{-1} for OH and absorption at 3001 cm^{-1} for C-H aromatic and absorption at 2823-8373 cm^{-1} for C-H aliphatic and absorption stretch C=N strong pack at 1641 cm^{-1} . the ^1H NMR compound (EM1) show a singlet at δ 2.55 for benzedine proton, triplet at δ 6.46 for C2&C6 aminophenol ring and double doublet δ 6.75 for C3&C5 aminophenol ring, doublet at δ 7.15 for C3&C5 benzedinephenol ring, doublet at δ 7.74 for C2&C6 benzedinephenol ring and two singlet pike at δ 8.45 & 9.77 for OH phenolic proton. These interpretations match the literature[25] the ^1H NMR & ^{13}C NMR properties show in table (3&4). Figure (1&2) show the ^1H NMR & ^{13}C NMR spectrum for compound (EM1).

Compound (EM2) propertied by react the 4-(4-hydroxybenzylidene) with Adipoyl Chloride in acetone, citramide sodium hydroxide and chloroform to give polymer compound. The Infrared spectra of compound (EM2) showed absorption wide broadcasts at 3066 cm^{-1} for C-H aromatic and absorption at 2945-2872 cm^{-1} for C-H aliphatic and absorption stretch C=O pack at 1753 cm^{-1} and absorption stretch C=N strong pack at 1682 cm^{-1} and last stretch C-O-C two pack at 1199, 1124 cm^{-1} . the ^1H NMR compound (EM2) show disappearance of OH phenolic signal and appearance of multiple signals of (CH_2) . the ^1H NMR & ^{13}C NMR properties show in table (3&4). Figure (3 &4) show the ^1H NMR & ^{13}C NMR spectrum for compound (EM2).

**Scheme V**



Scheme VI

Fig.1. ^1H NMR spectrum of EM1 compound

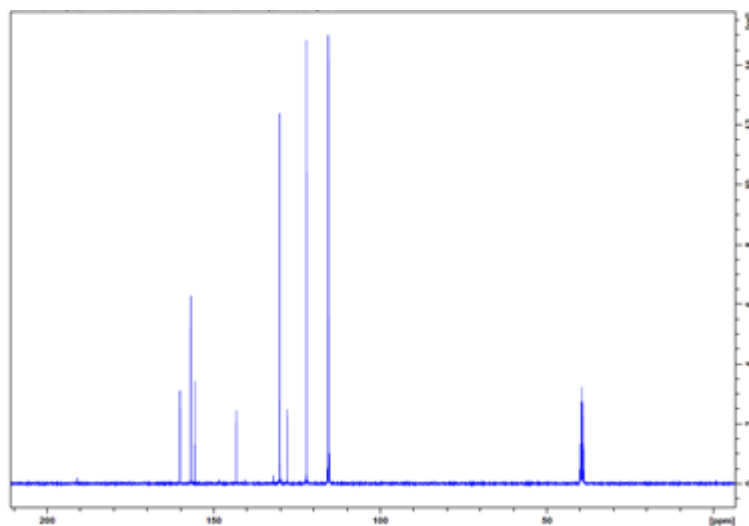


Fig.2. ¹³C NMR spectrum of EM1 compound

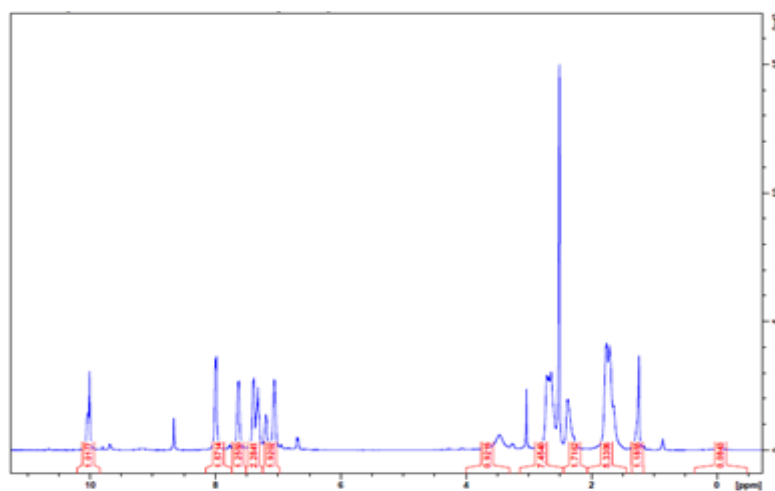


Fig.3. ¹H NMR spectrum of EM2 compound

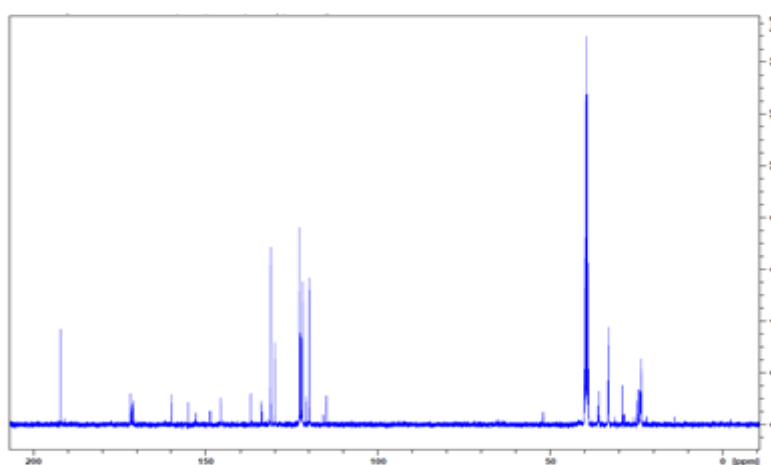


Fig.4. ¹³C NMR spectrum of EM2 compound

Compound (EM3) prepared by reacting 4-(4-hydroxybenzylidene) with Succinoyl Chloride in acetone, citramide sodium hydroxide and chloroform to give polymer compound. The Infrared spectra of compound (EM3) showed absorption wide broadcasts at 3068 cm^{-1} for C-H aromatic and absorption at $2929\text{--}2854\text{ cm}^{-1}$ for C-H aliphatic and absorption stretch C=O peak at 1751 cm^{-1} and absorption stretch C=N strong peak at 1684 cm^{-1} and last stretch C-O-C two peak at $1197, 1132\text{ cm}^{-1}$. The ^1H NMR compound (EM3) show disappearance of OH phenolic signal and appearance of multiple signals of (CH_2) . The ^1H

NMR & ^{13}C NMR properties show in table (3&4). Figure (5 &6) show the ^1H NMR & ^{13}C NMR spectrum for compound (EM3).

Compound (EM4) prepared by reacting 4-(4-hydroxybenzylidene) with Sebacoyl Chloride in acetone, citramide sodium hydroxide and chloroform to give polymer compound. The Infrared spectra of compound (EM4) showed absorption wide broadcasts at 3050 cm^{-1} for C-H aromatic and absorption at $2928\text{--}2850\text{ cm}^{-1}$ for C-H aliphatic and absorption stretch C=O peak at 1747 cm^{-1} and absorption stretch C=N strong peak at 1685 cm^{-1} and last stretch C-O-C two peak at

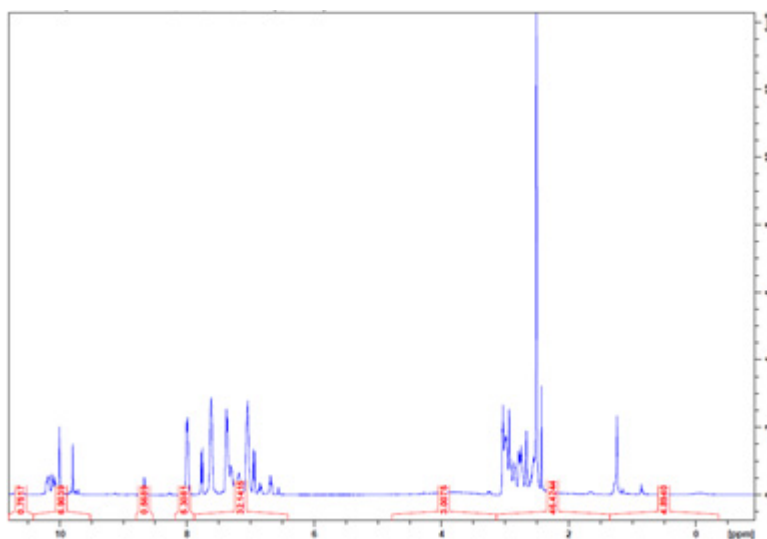


Fig.5. ^1H NMR spectrum of EM3 compound

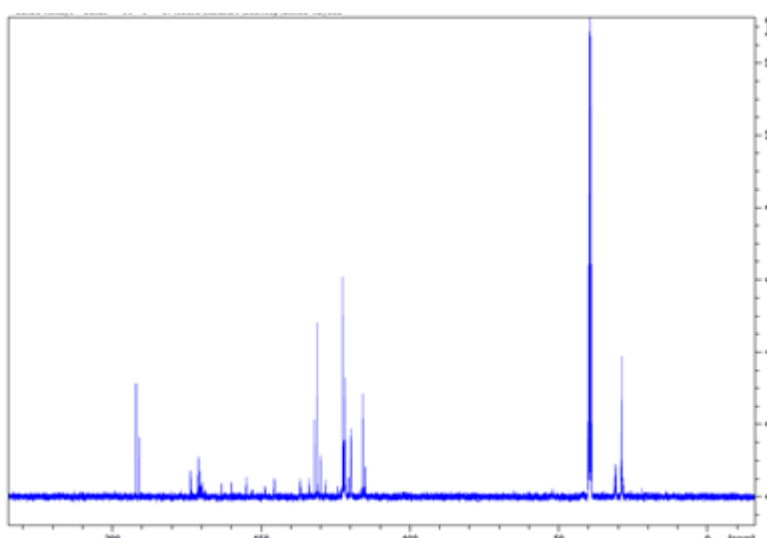


Fig.6. ^{13}C NMR spectrum of EM3 compound

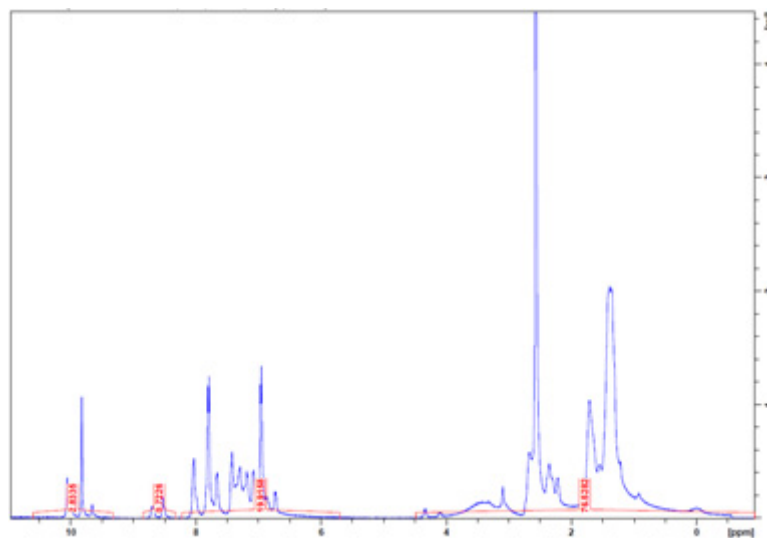
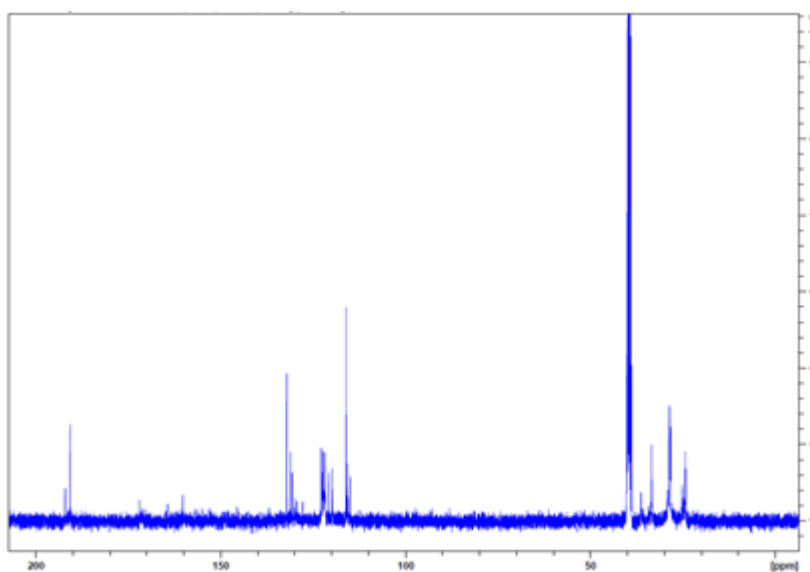
Fig.7. ^1H NMR spectrum of EM4 compoundFig.8. ^{13}C NMR spectrum of EM4 compound

TABLE 2. The IR spectra for Compounds (EM1, EM2, EM3, and EM4)

No. of compound	H-O ν	$\nu\text{C-H}_{\text{Ar}}$	$\nu\text{C-H}_{\text{Alph}}$	O=C ν	N=C ν	C—O ν
EM1	3300	3178	2893	—	1510	—
EM3	—	3068	2910	1751	1685	1132
EM2	—	3100	2945	1751	1683	1122
EM4	—	3000	2850	1747	1685	1203

1203, 1136 cm^{-1} . The ^1H NMR compound (EM4) show disappearance of OH phenolic signal and appearance of multiple signals of (CH_2) [35]. The ^1H NMR & ^{13}C NMR properties show in table (3&4). Figure (7 & 8) show the ^1H NMR & ^{13}C NMR spectrum for compound (EM4).

Conclusion

Three polymer have been synthesized by interfacial polycondensation employing phase transfer catalyst. The polymers was prepared, characterized and thermal stability was investigated. The polymers indicate better thermal

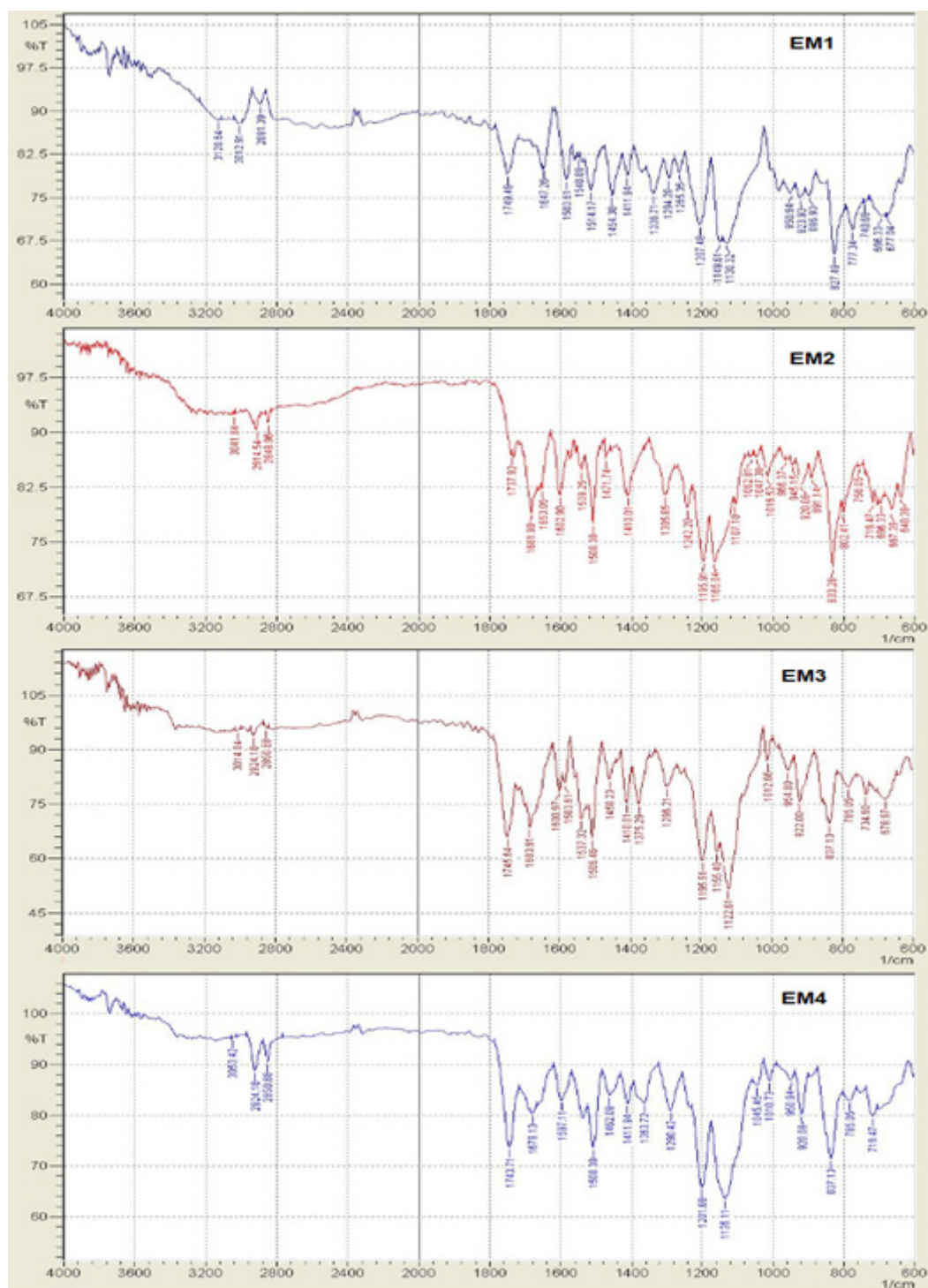


Fig.9. IR spectrum for compounds (EM1, EM2, EM3, and EM4)

TABLE 3: ¹H NMR data for compounds (EM1, EM2, EM3, and EM4)

Compound No.	¹ H NMR (ppm) – DMSO-d ₆
EM1	2.55(1H, s, Ar-CH=N), 6.46(2H, t, C2&C6 Ar-H aminophenol ring), 6.75(2H, dd, C3&C5 Ar-H aminophenol ring), 7.15(2H, d, C3&C5 Ar-H benzedinephenol ring), 7.74(2H, d, C2&C6 benzedinephenol ring), 8.45(1H, s, OH aminophenol ring) 9.77(1H, s, OH benzedinephenol ring)
EM2	1.42(4H, m, CH ₂ -CH ₂), 2.42(1H, s, Ar-CH=N), 2.78(4H, m, tow group CH ₂ -C=O), 7.16(2H, t, C2&C6 Ar-H aminophenol ring), 7.40(2H, dd, C3&C5 Ar-H aminophenol ring), 7.65(2H, d, C3&C5 Ar-H benzedinephenol ring), 7.99(2H, d, C2&C6 benzedinephenol ring)
EM3	2.52(1H, s, Ar-CH=N), 2.88(4H, m, tow group CH ₂ -C=O), 6.69(2H, t, C2&C6 Ar-H aminophenol ring), 7.08(2H, dd, C3&C5 Ar-H aminophenol ring), 7.88(2H, d, C3&C5 Ar-H benzedinephenol ring), 8.12(2H, d, C2&C6 benzedinephenol ring)
EM4	1.32-1.62(4H, p, (CH ₂ -CH ₂) ₆), 2.28(1H, s, Ar-CH=N), 2.70(4H, m, tow group CH ₂ -C=O), 6.91(2H, t, C2&C6 Ar-H aminophenol ring), 7.45(2H, dd, C3&C5 Ar-H aminophenol ring), 7.80(2H, d, C3&C5 Ar-H benzedinephenol ring), 8.01(2H, d, C2&C6 benzedinephenol ring)

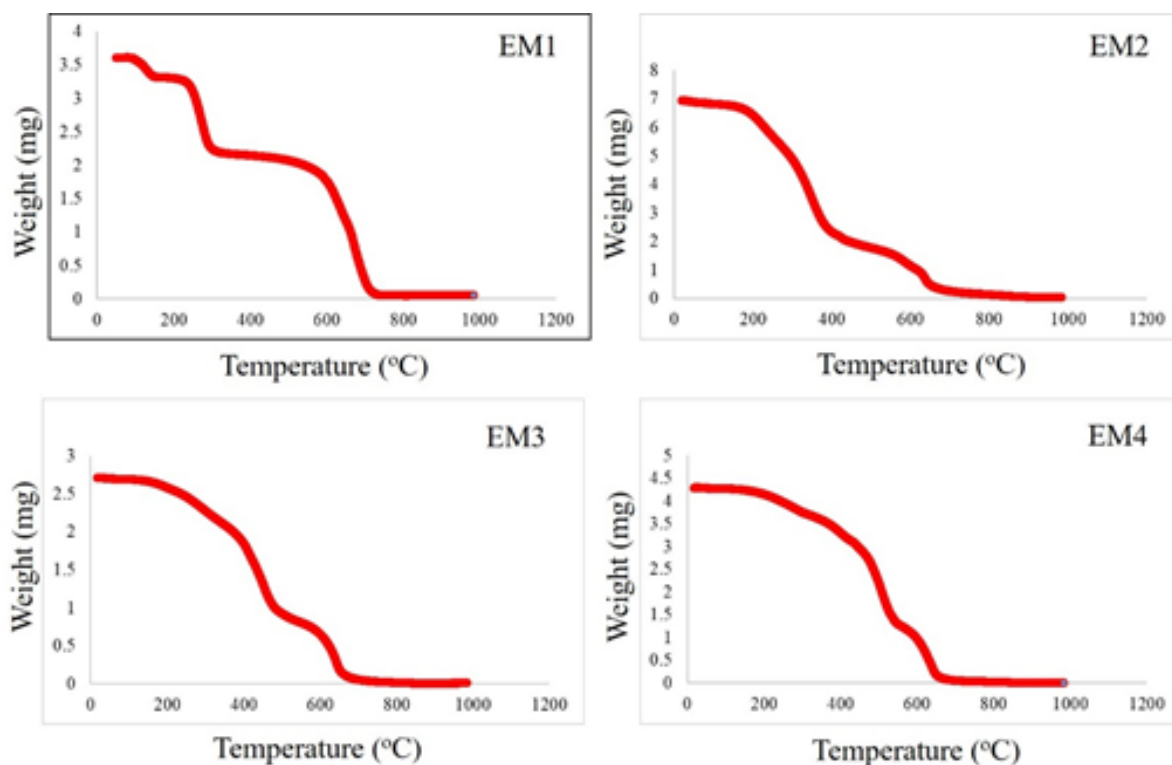
**Fig.10. Thermo gravimetric analysis (TGA) of the EM1, EM2, EM3 and EM4.**

TABLE 4: ^{13}C NMR data for compounds (EM1-EM4)

Compound No.	^{13}C NMR (ppm) – DMSO-d ₆
EM1	39.57(Ar-CH=N), 113.50(C1 aminophenol ring), 121.67(C2&C6 aminophenol ring), 130.04(C2&C6 benzedinephenol ring), 143.74(C1 benzedinephenol ring), 155.82(C3&C5 aminophenol ring), 157.01(C3&C5 benzedinephenol ring), 161.16(C4 for two carbon benzedinephenol ring and aminophenol ring)
EM2	26.77(C2 for Adipoyl chain), 28.17(C3 for Adipoyl chain), 33.65(C1 for Adipoyl chain), 36.76(C4 for Adipoyl chain), 39.02(Ar-CH=N), 114.87(C1 aminophenol ring), 120.27(C2&C6 aminophenol ring), 123.48(C2&C6 benzedinephenol ring), 132.42(C1 benzedinephenol ring), 138.02(C3&C5 aminophenol ring), 153.18(C3&C5 benzedinephenol ring), 163.60(C4 for two carbon benzedinephenol ring and aminophenol ring), 192.68(C=O for two carboxyl group)
EM3	28.98(C1 for Succinyl chain), 31.27(C2 for Succinoyl chain), 39.94(Ar-CH=N), 115.98(C1 aminophenol ring), 120.74(C2&C6 aminophenol ring), 122.86(C2&C6 benzedinephenol ring), 132.25(C1 benzedinephenol ring), 138.92(C3&C5 aminophenol ring), 154.86(C3&C5 benzedinephenol ring), 161.68(C4 for two carbon benzedinephenol ring and aminophenol ring), 190.14,192.85(C=O for two carboxyl group)
EM4	25.75(C2 for Sebacoyl chain), 26.72(C3 for Sebacoyl chain), 28.70(C4,C5,C6,C7 for Sebacoyl chain), 35.54(C1 for Sebacoyl chain), 37.86(C8 for Sebacoyl chain), 40.92(Ar-CH=N), 115.76(C1 aminophenol ring), 120.70(C2&C6 aminophenol ring), 123.88(C2&C6 benzedinephenol ring), 131.24(C1 benzedinephenol ring), 133.92(C3&C5 aminophenol ring), 158.88(C3&C5 benzedinephenol ring), 163.02(C4 for two carbon benzedinephenol ring and aminophenol ring), 191.08,192.88(C=O for two carboxyl group)

Thermogravimetric analysis can provide information about the thermal analysis and the reaction take place. Figure 10 shows the thermo gravimetric analysis of the EM1, EM2, EM3 and EM4 polymers in a nitrogen atmosphere environment heating rate was $10\text{ }^{\circ}\text{C min}^{-1}$. EM1 decomposed in three steps and lost 8.6, 41.4 and 98.5 % at temperatures of 197, 453 and $748\text{ }^{\circ}\text{C}$ respectively. The samples EM2, EM3 and EM4 show same steps of chemical transformations with associated mass loss could be appreciated. The decomposition of EM2 show a mass loss 73.54% beginning at about $180\text{--}483.05\text{ }^{\circ}\text{C}$ and finishing at $880.85\text{ }^{\circ}\text{C}$. The decomposition of EM3 show a mass loss 69.58% beginning at about $180\text{--}539.23\text{ }^{\circ}\text{C}$ and finishing at $862.91\text{ }^{\circ}\text{C}$. The decomposition of EM4 show a mass loss 71.21% beginning at about $180\text{--}563.10\text{ }^{\circ}\text{C}$ and finishing at $877.92\text{ }^{\circ}\text{C}$. The weight lost was about 99.8% at 880.85 , 862.91 and $877.92\text{ }^{\circ}\text{C}$ indicated T_{max} . As show references[24].

stability than monomer and slight difference in thermal stability is due to the difference in the number of methylene groups. The synthesized Polymeric Schiff Bases recommended for Nano-Biosynthesized as in [30] for detect its applications in medical field.

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