Preparation ,Characterization and Thermal Properties of New Aromatic Polyester Based on Di ,Tri and Tetra Hydroxyl Monomers with 4,4Azo Di Benzoic Acid  and Adipic Acid

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Abstract
A series of new aromatic Polyesters were  synthesized by polycondensation of various aromatic monomers di ,tri and tetra diols (BCAHPMNM , BHPMB, BHPPB , BPBHP) with Acids(4,4Azo Di benzoic acid  and Adipic acid) using  dibutyltine dilaurate as Cataly st .all polyesters(P.E1-P.E4)containing pyridine heterocyclic group, aliphatic methylene linkage, and Azo group. The yield of Polyesters varies from 60-85%. All of these new aromatic polyester show very good solubility in common organic solvents , such as (pyridine , CHCl3 , CH2Cl2 , NaOH , H2SO4, HNO3, Acetone , Benzene , DMF , DMSO , THF) without need for heating. Thermal analysis of polyesters by thermo gravimetric analysis (TGA) reveals that these Aromatic polyesters possess thermal stability. Monomers and polyesters characterized by FT-IRand 1HNMRspectrosticopy.

Keywords: Aromatic polyester , 4,4Azo Di benzoic acid  , Adipic acid, dibutyltine dilaurate.

1-INTRODUCTION
Polyesters are a class of polymers that can be formulated to be hard or soft, brittle or flexible depending on the structure and can therefore be used in various applications. These applications include fibers (e. g. polyethylene terephthalate (PET)) (1-3), coatings (4-5), plasticizers (6), adhesives, polyurethane based resins, films, laminates, industrial construction, insulation, molding compounds, etc.(5-6). Polyesters are used for these applications because of their low cost (2-3, 5, 7), ease of process (7), and ease of combination with reinforcements (2-3, 7). In addition, these polymers cure rapidly without any volatile products (2-3, 7-8), have excellent dimensional stability, have high impact resistance, transparency and good weathering resistance (5). The development of process able high-performance polymers with increased thermal stability, heat resistance and good mechanical properties has become an important problem (9). Aromatic polyesters have excellent physical and mechanical properties and are among the most important commercially available polymers with widespread application, especially in the food and beverage packaging. However, due to their strong resistance to bacterial or fungal attack they usually remain unaltered under the environment conditions, and result in a considerable waste stream (10-13). Aromatic polyesters are certainly one of the most successful classes of high-temperature polymers. However these polymers encounter processing difficulty due to their insuffibility and poor solubility in organic solvents Therefore, more researches have been focused on maintaining considerable thermally stable and improving their solubility(13-15). In the present work, four aromatic polyesters (P.E1- P.E4) have been synthesized in high yields from the polyc ondensation between (di ,Tri and Tetra) hydroxyl monomers containing methylene unit,Azo group and pyridine hero cyclic ring with 4,4 Azo di benzoic acid and Adipic acid using dibutyltine dilaurate as Catalyst, and qualitative structure analysis of the polymers and monomers have been carried out by the using of FT-IR, and 1HNMR spectroscopy, thermal stability were systematically investigated .

2- EXPERIMENTAL
2.1  Materials
Dichloromethane,Hexane, all from (BIOSOLVE); Hydrochloric acid ,Salicylaldehyde, Benzaldehyde, all from (HI media); Diethyl ether from (IGCC / England); phenol, P-aminophenol,4-Chlorobenzaldehyde,Glacial acetic acid, Piperidine, PyridineTerephthaldehyde, Dimethyl Sulphoxide (DMSO), P-hydroxyacetophenone, Terohthaldehyde, Tetrahydrofurane (THF), all from(MERCK); Absolute Ethanol fromd (Scharlab S.L); N,N-Dimethylformamide (DMF)from (ALDRICH).

2.2  Instruments
FTIR 8400S, Fourier Transform infrared spectrophotometer, SHIMADZU, Japan),(Oven ,Trip International Corp. Italy),(Measuring the degree of fusion (Melting Points)Stuart smp30 melting point apparatus ) ,( Hot plate stir ,BibbyStrlindt. UK), ( Measurement of 1HNMR: recorded NMR spectra using a type of Bruker, Ultra, Shiel 300MHZ,Switzear, Land and using (DMSO-d6) as solvent at the University's Educational teacher -Tehran Iran),(Thermogravimetry analysis (TGA) were performed on a Polymer laboratories co England, Model PL-TG at Iran polymer & petrochemical institute, using a heating rate of 10°C/min in Argon atmosphere within the temperature range of 25–800°C).
2.3 monomer Synthesis

2.3.1 Bis(3-chloro aniline 4-hydroxy phenyl)methyl 4-nitro phenyl Methan (M1):
A mixture of P-Nitro acetophenone (2.72gm(16mmol), 2-Chloro phenol (4gm(42mmol), HCl catalyst (37.5gm) and thioglycolic acid(0.5gm) protomer were placed in a three necked –round bottom flask equipped with the acondenser, mechanical stirrer and thermometer and it was kept in athermostate bath at 60°C for 6hs .After a definite period of time, the reaction mixture was transferred to cold water to quench the reaction. Then, the product was washed, dried (16). the yield was (85%) of yellow crystals, m.p= (187-190°C),Figure (1) Structure of monomer (M1).

2.3.2Synthesis1,4-(bis(4- hydroxyphenyl)methyl)Benzene(M2):
To a 0.1M solution of H2SO4 in methanol (60% : 40%), phenol( 2gm,21mmol ) and salicylaldehyde(1.5gm,12mmol) were added and the mixture was refluxed at 120°C for 10 hs with the constant stirring . Upon completion of the reaction, the solvent was removed under vacuum(17).the yield was (71%) of brown crystals, m.p= (80°C) crystals.

2.3.3 Synthesis of 4(4-(2,4 bis(4-hydroxy phenyl pyridine(2,4-bis(4-hydroxy phenyl pyridine  (M3)
In a(150ml) round-bottomed flask equipped with a reflux condenser, a mixture of Terephthalaldehyde (1.34 gm, 10mmol ), P-hydroxy acetophenone(6.62gm,48mmol), ammonium acetate(7.5gm), and glacial acetic acid(20ml)was refluxed at 140-142°C for 2hs. Upon cooling,crysals separated ,which were filtered and washed first with acetic acid (50%) and then with cold ethanol. These product crystals were recrystallized from absolute Ethanol, and then dried at 60°C under vacuum(18,19) the yield was (77%) of white crystals, m.p= (140°C), Figure(3) Structure of monomer (M3).

2.3.4 Synthesis of 6-(4-Bromo phenyl) 2,4 bis (4-hydroxy phenyl) pyridine (M4):
This monomer synthesis by same way that (M3) is formed . but by using metrials (4-bromo benzaldehyde) (1.41gm,7mmol) and (4-hydroxy acetophenone) (1.31 gm,24mmol ) where yield (80%), m.p= (101°C) and color is yellow. Figure (4) structure of monomer (M4).

2.3.4 Preparation of 4,4Azodibenzoic acid
4,4Azodibenzoic acid was prepared according to atypical procedure, shown in scheme(1) (Thirteen grams of 4-nitrobenzoic acid (79mmol) was heated in a solution of 50 gm of sodium hydroxide in 250mL of water at 50°C. A
Solution of 100gm of glucose in 150 mL water was added slowly at this temperature with occasional shaking. The reaction mixture was then cooled to ambient temperature and aerated for 8h with vigorous stirring until orange- colored Crystals were formed.

\[
\text{O}_2\text{N}\text{CO}_2\text{H} \xrightarrow{\text{Glucose} \ \text{NaOH}} \text{HO}_2\text{C}-\text{N}=\text{N}-\text{CO}_2\text{H}
\]

Scheme(1) Preparation of 4,4Azodibenzoic acid

The FTIR spectra of (4,4Azodibenzoic acid) as shown in (Figur5) which indicates absorption bands of C=O Aromatic at 1589 cm-1, C-H Aromatic at 3109 cm-1, OH at 3425 cm-1, C-H aliphatic at 2839 cm-1, C=O at 1951 cm-1, and C-O at 1103 cm-1, and C-N at 1311 cm-1.

![FTIR spectra of (4,4Azodibenzoic acid)](image)

2.3.4 Synthesis of polyesters [P.E1-P.E4].

Dihydroxy monomer and Di acid, as shown in (Table 1) were mixed at 120°C under constant stirring. After the dissolution of the acid in Dihydroxy monomer, the dibutyltine dilurate catalyst (0.15 wt.%) was added and the temperature was increased up to 160°C and was kept at this temperature for 1 hour. The materials were dried under vacuum at 50°C giving rise to the P.E series. The materials were obtained as powders.

1) Synthesis of polyesters [P.E1-P.E4].

<table>
<thead>
<tr>
<th>polyester</th>
<th>Monomers</th>
<th>diacid</th>
<th>Dihydroxy</th>
<th>diacid gm/mmol</th>
<th>Dihydroxy gm / mmol</th>
<th>Yield (%wt)</th>
<th>Color</th>
</tr>
</thead>
<tbody>
<tr>
<td>P.E1</td>
<td>Adipic acid</td>
<td>M1</td>
<td>1.2gm</td>
<td>1 gm</td>
<td>77%</td>
<td>white</td>
<td></td>
</tr>
<tr>
<td>P.E2</td>
<td>4,4Azidibenzoic acid</td>
<td>M2</td>
<td>2.4gm</td>
<td>1.3gm</td>
<td>85%</td>
<td>Orange</td>
<td></td>
</tr>
<tr>
<td>P.E3</td>
<td>4,4Azidibenzoic acid</td>
<td>M3</td>
<td>2.4 gm</td>
<td>1.1gm</td>
<td>78%</td>
<td>Orange</td>
<td></td>
</tr>
<tr>
<td>P.E4</td>
<td>4,4Azidibenzoic acid</td>
<td>M4</td>
<td>1.2 gm</td>
<td>1 gm</td>
<td>75%</td>
<td>Yellow</td>
<td></td>
</tr>
</tbody>
</table>
Results and Discussion 3-
Synthesis of monomers: 3.1.  
3.1.3 Synthesis and characterization  
Bis(3-chloro aniline 4-hydroxy phenyl)methyl 4-nitro phenyl) methan (M1):  
This monomer was synthesized by the condensation of one molecule of P-nitro acetophenone with two molecules of 2-Chloro phenol in the presence of acid catalyst, and suitable promoter at 60 °C for 6 h. This monomer was characterized by FTIR.

Characterization of [M1]  
FT-IR spectrum:-  
The FTIR spectra of (M4) as shown in Figur6) which indicates absorption band of C=C Aromatic at 1697 cm⁻¹, C-H Aromatic at 3109 cm⁻¹, OH at 3425 cm⁻¹, C-H aliphatic at 2923 cm⁻¹, and NO₂ at 1527 cm⁻¹, and C=O at 1951 cm⁻¹, and C-O at 1164 cm⁻¹, C-Cl at 748 cm⁻¹, and C=N at 1257 cm⁻¹.
3.1.2 Synthesis and Characterization of 1,4-(bis(4-hydroxyphenyl)methyl)Benzene (M2):
This monomer was synthesized by the condensation of one molecule of salicylaldehyde with two molecules of phenol in the presence of H$_2$SO$_4$ and methanol as catalysts at 120$^\circ$C for 10 hrs. This monomer was characterized by FTIR.

![Scheme 3 Synthesis of M2](image)

**Characterization of [M2]**
**FT-IR spectrum:**
The FTIR spectra of (M2) as shown in Figure 7 which indicates absorption band of C=C Aromatic at 1666 cm$^{-1}$, C-H Aromatic at 3062 cm$^{-1}$, OH at 3379 cm$^{-1}$, C-H aliphatic at 2862 cm$^{-1}$, and C-N at 1230 cm$^{-1}$, and C=N at 1550 cm$^{-1}$, and C-O at 1095 cm$^{-1}$.

![Figure 8 FTIR spectra of M2](image)

3.1.3 Synthesis and Characterization of 4(4-(2,4-Bis(4-hydroxy phenyl pyridin)2,4-Bis hydroxyl phenyl pyridin (M3):
This monomer was synthesized by the condensation of one molecule of Terphthaldehyde with four molecules of 4-hydroxyl aceto phenone in the presence of ammonium acetate and glacial acetic acid at 140-142$^\circ$C for 2 hrs. This monomer was characterized by FTIR characterized by FTIR.

![Scheme 4 synthesis of M3](image)
Characterization of [M3]

FT-IR spectrum :-
The FTIR spectra of (M1) as shown in Figur8) which indicates absorption band of C=C Aromatic at 1697 cm-1, C-H Aromatic at 3062 cm-1, OH at 3250 cm-1, C-H aliphatic at 2985 cm-1, and C-N at 1280 cm-1, and C=N at 1573 cm-1, and C-O at 1134 cm-1.

![Figure 9: FTIR spectra of (M3)](image)

3.1.4 Synthesis and Characterization of 6-(4-bromo phenyl)2,4 bis(4-hydroxy phenyl) pyridine (M4)

This monomer was synthesized by the condensation of one molecule of 4- Bromo benzaldehyde with two molecules of 4- hydroxyl aceto phenone in the presence of ammonium acetate and glacial acetic acid at 140 142° C for 2 hrs. This monomer was characterized by FTIR.

![Scheme 5 synthesis of M4](image)

Characterization of [M3]

FT-IR spectrum :-
The FTIR spectra of (M4) as shown in Figur9) which indicates absorption band of C=C Aromatic at 1650 cm-1, C-H Aromatic at 3170 cm-1, OH at 3300 cm-1, C-H aliphatic at 2893 cm-1, and C-N at 1265 cm-1, and C=N at 1512 cm-1, and C-O at 1172 cm-1, C-Br at 810 cm-1.

![Figure 10: FTIR spectra of (M4)](image)

1H-NMR spectrum of (M4), is shown in Figure() assigns the following chemical shifts; δ (2.477) ppm for DMSO, δ (4.877) ppm (s,1H) for OH group, δ(3.392) ppm (s,1H) for H2O, δ (6.678-8.055) ppm (14H) for Ar-H group(23).

In this section, used polycondensation method for synthesis of a series of new polyester from the direct polycondensation reaction Adipic acid with new aromatic di hydroxyl monomer [M1] and new polyester from the direct polycondensation reaction of 4,4Azo di benzoic acid with new aromatic di, Tri and Tetra hydroxyl monomers[M2,M3,M4] respectively, by using the dibutyltin dilaurate Gatalyst(0.15 wt.%). These polymers have a soft segment such as methylene unit, pyridine heterocyclic ring and Azo group in main chain for improving solubility in organic solvents. These polyesters were characterized by FT-IR and 1H-NMR spectra.

Characterization of polyesters [P.E1-P.E4]
FT-IR spectrum of P.E1
The FT-IR spectrum of (P.E1) as shown in (Figur11) which indicates absorption band at (3109 cm$^{-1}$) to (aromatic-CH2str), (2923 cm$^{-1}$) to (aliphatic-CH str), (1944 cm$^{-1}$) to (C=O str, ester), (1596 cm$^{-1}$) to (C=C), (1450 cm$^{-1}$) to (N=N) (1225 cm$^{-1}$) to (C=N), (1180 cm$^{-1}$) to (C-O str), (1226 cm$^{-1}$) to (C-N) (856 cm$^{-1}$) to (C-Cl).

$\text{Figure 12 : FTIR spectra of (P.E1)}$

$^1$H-NMR spectrum of (M4), is shown in Figure(12) assigns the following chemical shifts; $\delta$ (2.484) ppm for DMSO, $\delta$ (5.190) ppm (1H) for CH, $\delta$(3.394) ppm (1H) for CH2, $\delta$ (7.426-8.833) ppm (10H) for Ar-H group.

$\text{Figure 13 : }^1\text{H NMR spectra of P.E1}$

FT-IR spectrum of P.E2
The FT-IR spectrum of (P.E2) as shown in (Figur13) which indicates absorption band at (3070 cm$^{-1}$) to (aromatic-CH2str), (2962 cm$^{-1}$) to (aliphatic-CH str), (1944 cm$^{-1}$) to (C=O str, ester), (1789 cm$^{-1}$) to (C=O str), (1697 cm$^{-1}$) to (C=C), (1465 cm$^{-1}$) to (N=N) (1225 cm$^{-1}$) to (C=N), (1010 cm$^{-1}$) to (C-O str), (1296 cm$^{-1}$) to (C-N).

$\text{Figure 14 : FTIR spectra of (P.E2)}$

$^1$H-NMR spectrum of (M4), is shown in Figure(14) assigns the following chemical shifts; $\delta$ (2.474) ppm for
DMSO, δ (4.152) ppm (1H) for CH, δ (7.486-8.137) ppm (27H) for Ar-H group(23).

**Figure 15**: $^1$H-NMR spectra of P.E2

FT-IR spectrum of P.E3

The FT-IR spectrum of (P.E3) as shown in (Figure 15) which indicates absorption band at 3070 cm$^{-1}$ to (aromatic-CH$_2$ str), (2923 cm$^{-1}$) to (aliphatic-CH str), (1944 cm$^{-1}$) to (C=O str,ester), (1712 cm$^{-1}$) to (C=O str), (1573 cm$^{-1}$) to (C=C), (1427 cm$^{-1}$) to (N=N), (1288 cm$^{-1}$) to (C=N), (1221 cm$^{-1}$) to (C-O str)(22).

**Figure 16**: FTIR spectra of (P.E3)

$^1$H-NMR spectrum of (P.E3), is shown in Figure(16) as signs the following chemical shifts; δ (2.483) ppm for DMSO, δ (6.660-8.144) ppm (40H) for Ar-H group (23).

**Figure 17**: $^1$HNMR spectra of P.E3

FT-IR spectrum of P.E4

The FT-IR spectrum of (P.E4) as shown in (Figur17) which indicates absorption band at (3150 cm$^{-1}$) to (aromatic-CH$_2$ str), (2954 cm$^{-1}$) to (aliphatic-CH str), (1951 cm$^{-1}$) to (C=O str,ester), (1704 cm$^{-1}$) to (C=O str), (1589 cm$^{-1}$) to (C=C), (1458 cm$^{-1}$) to (N=N), (1226 cm$^{-1}$) to (C-O str)(22).
Figure 18 : FTIR spectra of (P.E4)

1H-NMR spectrum of (P.E4), is shown in Figure(18) assigns the following chemical shifts; δ (2.470) ppm for DMSO, δ(3.352) ppm (s,1H) for H2O, δ (6.859 - 8.099) ppm (22H) for Ar-H group(23).

Figure 19' HNMR spectra of P.E4

Solubility of polyesters:- 3.3
Solubility of polyesters P.E1-P.E4 was qualitatively tested in organic solvents and the results are summarized in (Table 1). The method that attempt to enhance their process abilities and solubilities were either by introducing bulky groups, flexible linkages, or molecular asymmetry into the polymer backbones. In this work, the attachment of bulky pendant groups in polymer backbone not only could provide an enhanced solubility because of decreased packing density and and crystallinity, but also could impart an increase in Tg by restricting the segmental mobility. One of the major objectives of this work was producing polyesters with improved solubility. The Solubility was investigated as (0.01gm) of polymeric sample in (2ml) of solvent. All of the newly synthesized polyesters have good soluble in common polar and dipolar aprotic solvents without need for heating.
Table (1) Solubility of polyesters:

<table>
<thead>
<tr>
<th>Solvent</th>
<th>Polyester</th>
<th>P.E1</th>
<th>P.E2</th>
<th>P.E3</th>
<th>P.E4</th>
</tr>
</thead>
<tbody>
<tr>
<td>pyridin</td>
<td>+ -</td>
<td>++</td>
<td>++</td>
<td>++</td>
<td>- +</td>
</tr>
<tr>
<td>CHCl3</td>
<td>+ -</td>
<td>++</td>
<td>++</td>
<td>++</td>
<td>- +</td>
</tr>
<tr>
<td>CH2Cl2</td>
<td>++</td>
<td>++</td>
<td>- -</td>
<td>++</td>
<td>- +</td>
</tr>
<tr>
<td>NaOH</td>
<td>+ -</td>
<td>++</td>
<td>++</td>
<td>++</td>
<td>- +</td>
</tr>
<tr>
<td>H2SO4</td>
<td>+ -</td>
<td>++</td>
<td>++</td>
<td>++</td>
<td>- +</td>
</tr>
<tr>
<td>HNO3</td>
<td>+ -</td>
<td>++</td>
<td>++</td>
<td>++</td>
<td>- +</td>
</tr>
<tr>
<td>Acetone</td>
<td>+ -</td>
<td>++</td>
<td>+ +</td>
<td>++</td>
<td>- +</td>
</tr>
<tr>
<td>DMF</td>
<td>++</td>
<td>+ +</td>
<td>+ +</td>
<td>+ +</td>
<td>+ +</td>
</tr>
<tr>
<td>DMSO</td>
<td>+ + + +</td>
<td>- +</td>
<td>- +</td>
<td>+ +</td>
<td>+ +</td>
</tr>
<tr>
<td>THF</td>
<td>- +</td>
<td>++</td>
<td>+ +</td>
<td>+ +</td>
<td>+ +</td>
</tr>
</tbody>
</table>

Full Soluble.+++ Soluble at room temperature ++ Partially Soluble .+-

3.4 Thermal analysis of polyester

Thermo gravimetric Analysis (TGA) involves determining changes in mass as a function of temperature. It is commonly used to research degradation temperatures, absorbed content of materials, levels of inorganic and organic parts contained in a material and analyses solvent residues. It employs a sensitive electronic balance from which the sample is suspended in a furnace controlled by a temperature programmer (22). The thermal properties of four samples of these polyester were investigated by means of thermo gravimetric analysis (TGA) in Argon atmosphere at heating rate of 10°C/min. The results such as $T_i$, $T_{op1}$, $T_{op2}$, $T_f$, $T_{50%}$, % Residue at 800°C, and char yields at 500°C are summarized in (Table 2). The temperatures of 50% weight loss of (P.E1-P.E4) as a standard indication for thermal stability of polymers were all from 248 to 761°C. The char yields of (P1-P4) at 500°C are 11% until 83% in Argon atmosphere, which indicate they could meet high temperature resistant requirements as some special materials in modern aerospace. Weight residue of (P.E1-P.E4) at 800°C are the highest than Aromatic polyester of this series, (7% until 50%).

Table (2): Thermal behavior data of Aromatic polyester:

<table>
<thead>
<tr>
<th>Aromatic polyester</th>
<th>DT/°C</th>
<th>$T_i$</th>
<th>$T_{op1}$</th>
<th>$T_{op2}$</th>
<th>$T_f$</th>
<th>$T_{50%}$</th>
<th>Residue at 8°C800</th>
<th>Char % at 500°C</th>
</tr>
</thead>
<tbody>
<tr>
<td>P.E1</td>
<td>152</td>
<td>380</td>
<td>----</td>
<td>800 &gt;</td>
<td>248</td>
<td>7%</td>
<td>11%</td>
<td></td>
</tr>
<tr>
<td>P.E2</td>
<td>382</td>
<td>444</td>
<td>575</td>
<td>800 &gt;</td>
<td>546</td>
<td>42%</td>
<td>58%</td>
<td></td>
</tr>
<tr>
<td>P.E3</td>
<td>311</td>
<td>429</td>
<td>620</td>
<td>800 &gt;</td>
<td>761</td>
<td>50%</td>
<td>83%</td>
<td></td>
</tr>
<tr>
<td>P.E4</td>
<td>222</td>
<td>302</td>
<td>429</td>
<td>800 &gt;</td>
<td>492</td>
<td>18%</td>
<td>48%</td>
<td></td>
</tr>
</tbody>
</table>

DT : Decomposition temperature.

$T_i$: Initial decomposition temperature.

$T_{op}$: Optimum decomposition temperature.

$T_f$: Final decomposition temperature.

$T_{50%}$: Temperature of 50% weight loss, obtained from TGA.

Char % at 500°C: Residual weight percentage at 500°C in Argon by TGA.
Figur 20 TGA curve of P.E1

Figur 21 TGA curve of P.E2

Figur 22 TGA curve of P.E3
Figur 23 TGA curve of P.E4

Conclusions
In this work several new polyesters (PE1-PE4) have been synthesized by the solution polycondensation reaction of the monomer with four different of aromatic diols in hexane presence of dibutyltine dilaurate as catalyst. These new polyester are soluble in various organic solvents and have good thermal stability. These properties can make these polyesters attractive for practical applications such as process able high- performance engineering plastics.

REFERENCES
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