Synthesis and Characterization of Novel Schiff’s Bases from Ethylenediamine Tetraacetic Acid Derivatives

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ABSTRACT

A novel Schiff’s base have been synthesized by reaction between ethylenediaminetetraacetic acid (EDTA) derivatives with (2-methoxyaniline, 4-bromoaniline, 4-amino antipyrine) are successfully prepared to obtain six Schiff’s base through the reaction of the aldehyde group with compounds containing the amine group as a catalyst glacial acetic acid in ethanol under reflux in good yield (78–92%), the prepared compounds were characterized by were synthesized and characterized by (FT-IR) and 1HNMR, 13C spectroscopy, elemental analysis. The aim of this study is to use cheap EDTA to synthesize Schiff bases, which are thought to have effective biologic and antibacterial properties.

Keywords: 2-methoxyaniline, 4-aminoantipyrine, 4-bromoaniline, Ethylenediaminetetraacetic acid, Schiff base, Synthesis.

INTRODUCTION

EDTA is considered as a pharmaceutical guide (metal complexing specialist). The corrosive, instead of any salt, is the structure most strong in expelling metals from arrangements. It might be added to attracted blood to avert coagulating and is additionally utilized in the pharmaceutical examination and for the evacuation or inactivation of undesirable particles in the arrangement. EDTA was best in avoiding the oxidation of thiol bunch in medications, for example, captopril. The edetate calcium disodium salt is principally utilized in the determination and treatment of lead harming. It is regulated as implantation containing. 1 O g in 250 or 500 mL of water over a time of 1–2 hours for 3–5 days. The edetate disodium salt is utilized to expel calcium from arrangements, and along these lines, it might be utilized as an anticoagulant. In the 1970’s it likewise turned out to be certain that EDTA as FeNa-EDTA could assume a job in battling sickliness in this world through nourishment stronghold. The FeNa-EDTA can be added to numerous nourishment items without prompting undesired taste impacts. Inorganic ingredients play a vital role in natural and organic curative procedures. Many natural mixtures used in medicine do not have a completely natural way of activity, some of which are biologically modified or modified by the digestion of mineral molecules. Many drugs have changed the toxicological and pharmaceutical properties as a compound of metals and are likely to have flexible C = N (Amin) bases containing mixtures with a wide range of natural movement and metal consolidation in plaque-type showing a level of antimicrobial and antimicrobial agents and antimicrobial mitigation measures. Chef rules are an important category of organic compounds. The Schiff base is the compound that contains the azomethine package (HC = N-). It is the result of the accumulation of ketones (or aldehydes) (aldehydes and ketones) with essential amines, first announced by Hugo Schiff in 1864, and largely based on Schiff’s base, largely under acid, base catalysis or with warmth. Schiff’s regular base is crystalline solids, which are weakly essential, but there are likely to be some insoluble salts in the structure with solid acids. The Schiff is used as an intermediary for the integration of amino acids or as links to the preparation of mineral rocks that are made in various structures. Scented aldehydes, especially with an application framework and structurally fixed chefs, as these aliphatic aldehydes are shaken and polymerized immediately. Legends form the base of aldehydes rather than carbon ketones. Its rules are fully adaptable and varied. There is a wide range of Schiff base mixtures and their perceived behavior on the basis that these mixtures have a completely adaptable and different structure. Schiff rules are mostly a two-tiered, triangular or tetrahedral shale bond and are stable structures with metal particles. Its structural and physical properties in different areas, for example, preparatory uses, identifiable evidence, assurance and confirmation of aldehydes or ketones, and the refinement of mixtures of carbonic or ammonium amino acids or the formation of such mixtures in consideration of puzzling or sensitive responses taken into account by different workers It is also known that Schiff
standards have a wide range of pharmacological activities, for example, antifungal drugs, antiviral drugs, antimicrobial, cytotoxic. Schiff’s base is used more in studies in the light of their applications, parasitic and natural, antimicrobial, antifungal, and antioxidants, the Schiff scale has been used extensively to date, in addition to countless applications of non-coated, antifungal anti-cancer activity. The objective of this study Synthesis and characterization of Schiff bases from EDTA, which is thought to have effective biological aries.

**EXPERIMENTAL**

All chemicals used in the present study are of analytical grade purchased from Sigma, Aldrich, and Merck chemical co. All the solvents were used after distillation. TLC was run on the silica-coated aluminum sheets (silica gel 60 F254, E Merck, Germany) and visualized in UV light. IR spectra were recorded on the FT-IR Perkin Elmer spectrum BX spectrophotometer. H NMR spectra were obtained by using Bruker H NMR instrument 300 MHz

**Synthesis of EDTA derivatives of tetrakis(4-formylphenyl) 2, 2', 2'', 2'''- [ethane-1, 2-diylbis(azanetriyl)] tetraacetate (F1)**

To solution of EDTA (0.0245 mole, 7.17gm) were disintergrated in dry N, N-dimethylformamide (DMF) 250mL substituted 4hydroxybenzaldehyde (0.098mole, 12gm) and in a 500mL round-bottomed flask. The blend was mixed for 10 hours. At 98-100°C in a framework shielded from air dampness and oxygen. The little amount of the hasten (EDTA) kept was expelled by filtration. The item encouraged at ice shower temperature was recrystallized from 10 parts of DMF-dioxane (1:2, v/v) to give 4.2 g of unadulterated item. scheme-1: Preparation of (f1).

F1: IR (K Br cm⁻¹): 3155(CH) as (aromatic), 2960 (Alph.-C H), 1706 (C = O), 1554 (C = C), 1196 (C-O), H NMR( CDCl3/ DMSO-d6, ppm): The aromatic rings give a group of multi signals at 6.82–7.92, (1H, CHO), 9.59, (-C- CH3) groups 3.10–3.19, (2H, CH2CH2), 2, 67.

**C-NMR spectrum of compound (F1) showed signals at (122-136ppm) due to aromatic carbons and at (165-170)ppm due to C = O.

**Synthesis of EDTA derivatives of tetrakis(4-formyl-2-methoxyphenyl) 2, 2', 2'', 2'''-(ethane-1, 2-diylbis(azanetriyl)) tetraacetate (F2):**

To solution of EDTA (0.0525 mole, 15.366gm) were dissolved in dry N, N-dimethylformamide (250 mL) substituted vanillin (0.2103 mole, 32 gram) and in a 500mL round-bottomed flask. The blend was mixed for 10 hours. At 98-100°C in a framework shielded from air dampness and oxygen. The little amount of the hasten (EDTA) kept was expelled by filtration. The item encouraged at ice shower temperature was recrystallized from 10 parts of DMF-dioxane (1:2, v/v) to give 3.6 g of unadulterated item. scheme-1: Preparation of (f2).

F2: IR (K Br cm⁻¹): 3071 (C –H), 1692 (C=O)), 1569 (C=C), 1122 (C-O-C), 3165(CH) as (aromatic), 2970 (Alph. –CH).

**C-NMR ( CDCl3/ DMSO-d6, ppm): 4.41(3H, -OCH3), 7.10 (1H, Ar-H), 3.28(2H, CH2CH2), 9.5(1H, CHO).**

**Scheme 1:** Synthesis of Schiff’s bases M1 and M2. A=vanillin, B=4 amino antipyrine, C=4-hydroxybenzaldehyde, D=4-amino antipyrine
Spectrum of compound (5) showed signals at (126-133 ppm) due to aromatic carbons and at 143 ppm due to C=N, showed signal at 55.3 ppm due to CH$_3$ group, and at (165-192) ppm due to C=O.

**Synthesis of Schiff base derivatives of bis(4-((E)-(1, 5-dimethyl-3-oxo-2-phenyl-2, 3-dihydro-1H-pyrazol-4-yl) imino)methyl)phenyl) 2, 2’-((2-((4-((Z)-(1, 5-dimethyl-3-oxo-2-phenyl-2, 3-dihydro-1H-pyrazol-4-yl) imino)methyl)phenoxy)-2-oxoethyl)(2-((4-((E)-(1, 5-dimethyl-3-oxo-2-phenyl-2, 3-dihydro-1H-pyrazol-4-yl) imino)methyl)phenoxy)-2-oxoethyl)amino)ethyl)azanediyl) diacetate (M1)**

To a solution of F1 (0.002 moles, 2 gm) in absolute ethanol (25 mL), substituted 4-amino antipyrine (0.00966 moles, 1.9642 gram) and in a 500 mL round-bottomed flask a few drops of glacial acetic acid were included, and the blend is refluxed for around 10 hours. The response blend is cooled and filled 500 mL of super cold water, and the hasten got were sifted washed with ethanol and dried and were recrystallized from THF. TLC (chloroform: petroleum ether), (1:1), scheme-1: Preparation of (m1).

**Synthesis of Schiff base derivatives of bis(4-((Z)-(1, 5-dimethyl-3-oxo-2-phenyl-2, 3-dihydro-1H-pyrazol-4-yl) imino)methyl)phenyl) 2, 2’-ethane-1, 2-diylbis((2-((4-((E)-(1, 5-dimethyl-3-oxo-2-phenyl-2, 3-dihydro-1H-pyrazol-4-yl) imino)methyl)-2-methoxyphenoxy)-2-oxoethyl)(2-(4-((E)-(1, 5-dimethyl-3-oxo-2-phenyl-2, 3-dihydro-1H-pyrazol-4-yl) imino)methyl)phenoxy)-2-oxoethyl)amino)ethyl)azanediyl) diacetate (M2)**

To a solution of F2 (0.00988 moles, 7 gram) in supreme ethanol (25 ml), substituted 4-amino antipyrine (0.0112 mol, 8.0354 gram) and in a 500 mL round-bottomed flask a few glacial acetic acids were included, and the blend is refluxed for around 10 hours. The response blend is cooled and filled 500 mL of super cold water, and the hasten got were sifted washed with ethanol and dried and were recrystallized from THF. TLC (chloroform: petroleum ether), (1:1), scheme-1: Preparation of (m2).

**Synthesis of Schiff base derivatives of bis(4-((4-bromophenyl imino)methyl)phenyl) 2, 2’-ethane-1, 2-diylbis((2-((4-((E)-(4-bromophenyl imino)methyl)-2-methoxyphenoxy)-2-oxoethyl)(2-(4-((E)-(4-bromophenyl imino)methyl)phenoxy)-2-oxoethyl)amino)ethyl)azanediyl) diacetate (M3)**

To a solution of F1 (0.002822 moles, 0.4854 gram) in supreme ethanol (25 mL), substituted 4-bromoaniline (0.011288 mol, 1.941 gram) and in a 500 mL round-bottomed flask a few glacial acetic acids were included, and the blend is refluxed for around 8 hours. The response blend is cooled and filled 500 mL of super cold water, and the hasten got were sifted washed with ethanol and dried and were recrystallized from THF. TLC (chloroform: petroleum ether), (1:1), scheme-2: Preparation of (m3).
Synthesis of Schiff base derivatives of (bis(4-((Z)\((4\text{bromophenyl})\text{imino})\text{methyl})-2\text{methoxyphenyl})-2\text{ethane1, 2diylbis}(2((4((\text{ethylenediamine tetraacetic acid derivates})-2\text{methoxyphenoxy}-2\text{oxoethyl})azanediyl))diacetate}(M4):

To solution of F2 (0.00024132 mole, 0.4151 gram) in supreme ethanol (25ml), substituted 4-bromoaniline (0.0009652 mol, 1.6604 gram) and in a 500mL round-bottomed flask a few drops glacial acetic acid were included and the blend is refluxed for around 8 hours. The response blend is cooled and filled 500mL of super cold water and the hasten got were sifted washed with ethanol and dried and were recrystallized from THF. TLC(chloroform: petroleum ether), (1:1), scheme-2: Preparation of m4.

Synthesis of Schiff base derivatives of (bis(4-((E)\((1, 5\text{dimethyl-3-oxo-2-phenyl-2, 3-dihydro-1H-pyrazol-4-yi})\text{imino})\text{methyl})-2\text{methoxyphenyl})-2, 2'-(2-((2(4-(((1, 5\text{dimethyl-3-oxo-2-phenyl-2, 3-dihydro-1H-pyrazol-4-yi})\text{imino})\text{methyl})-2\text{methoxyphenoxy}-2\text{oxoethyl})(2-(4-((E)-(1, 5\text{dimethyl-3-oxo-2-phenyl-2, 3-dihydro-1H-pyrazol-4-yi})\text{imino})\text{methyl})-2\text{methoxyphenoxy)-2-oxoethyl})azanediyl)diacetate}(M5):

To solution of F1 (0.00014 mole, 1.0gram) in supreme ethanol (25mL), substituted 2-methoxyaniline (0.00056 mol, 0.69 gram) and in a 500-mL round-bottomed flask a few drops of glacial acetic acid were included and the blend is refluxed for around 10 hours. The response blend is cooled and filled 500ml of super cold water and the hasten got were sifted washed with ethanol and dried and were recrystallized from THF. TLC(chloroform: petroleum ether), (1:1), scheme-3: Preparation of m5.

Synthesis of Schiff base derivatives of bis(4-((E)-(1, 5\text{dimethyl-3-oxo-2-phenyl-2, 3-dihydro-1H-pyrazol-4-yi})\text{imino})\text{methyl}-2\text{methoxyphenyl})-2, 2'-(2-((2-(4-(((1, 5\text{dimethyl-3-oxo-2-phenyl-2, 3-dihydro-1H-pyrazol-4-yi})\text{imino})\text{methyl})-2\text{methoxyphenoxy}-2\text{oxoethyl})(2-(4-((E)-(1, 5\text{dimethyl-3-oxo-2-phenyl-2, 3-dihydro-1H-pyrazol-4-yi})\text{imino})\text{methyl})-2\text{methoxyphenoxy)-2-Oxo ethylamino)ethyl}azanediyl)diacetate}(M6):

To solution of F2 (0.00012 mole, 1.0gram) in supreme ethanol (25ml), substituted 2-methoxyaniline (0.00048 mol, 0.59 gram) and in a 500-mL round-bottomed flask a few drops of glacial acetic acid corrosive were included and the blend is refluxed for around 10 hours. The response blend is cooled and filled 500mL of super cold water and the hasten got were sifted washed with ethanol and dried and were recrystallized from THF. TLC(chloroform: petroleum ether), (1:1), scheme-4: Preparation of m6.

Scheme 3: Synthesis of Schiff’s bases M5 and M6. A=vanillin, B=2-methoxybenzenaminium, C=4-hydroxybenzaldehyde, D=2-methoxyaniline
refluxed for around 8 hours. The response blend is cooled and filled 500ml of super cold water and the hasten got were sifted washed with ethanol and dried and were recrystallized from THF. TLC(chloroform: petroleum ether), scheme-3: Preparation of (m6).

**RESULTS AND DISCUSSION**

New six Schiff bases were synthesized from the reaction EDTA derivatives with (2-methoxyaniline, 4-bromoaniline, 4-amino antipyrine) aldehydes and ketones, shown in scheme (1, 2, 3) Some of these Schiff bases posses' good Physical properties and the % Yield percentage of the prepared Schiff bases were in the range {55-95} % see (Table:1). Such compounds were characterized by different physicochemical techniques like melting point, elemental analysis, and 1H NMR, 13C-NMR spectroscopy show in (Table 1, 2). Compound (M1) showed 1H NMR CDCl3/ DMSO-d6, ppm): 2.35 (3H, -CH3). The signal of N-CH3 was observed at 4.41 ppm (s, 3H). The signals of aryl-H were seen at 7.39–6.83 ppm (3H ), 3.28(2H, CH2CH2), at 9.48, and 8.27due to the azomethine. 13C-NMR spectrum of compound (M1) showed signals at (122-138ppm) due to aromatic carbons and at 163ppm due to C = N. Show spectrum of compound (M2) 1H NMR CDC13/ DMSO-d6, ppm): 2.32(3H, -CH3), The signal of N-CH3 was observed at 4.43ppm (3H). The signals of aryl-H were seen at 7.41–6.81 ppm (3H ), 3.30(2H, CH2CH2), at 9.44, and 8.22due to the azomethine. The signal of carbonyl-OCH3 was observed at 1.32 ppm (3H). 13 C-NMR spectrum of compound (M2) showed signal at 43-55 ppm due to CH3 group, signals at (122-131 ppm) due to aromatic carbons and signal at 163 ppm due to C = N. Compound (M3) showed 1H NMR CDC13/ DMSO-d6, ppm): 2.35 (3H, -CH3). The signal of N-CH3 was observed at 4.41 ppm (3H). The signals of aryl-H were seen at 7.51–6.84 ppm (3H ), 3.41 (2H, CH2CH2), at 9.39, and 8.27due to the azomethine. The 13C-NMR spectrum of

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<th>Comp. NO.</th>
<th>Compound structure</th>
<th>Elemental analysis,% Found/(calc.) C</th>
<th>Melting Points 0C</th>
<th>yield %</th>
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<td>Dark yellow</td>
<td>286</td>
</tr>
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</table>
Table 2: FTIR spectra data, 1H-NMR, and 13C-NMR spectral data for some of the new Schiff bases prepared compounds.

<table>
<thead>
<tr>
<th>mpd</th>
<th>FTIR spectra data</th>
<th>1H-NMR spectra data</th>
<th>13C-NMR spectra data</th>
</tr>
</thead>
<tbody>
<tr>
<td>M1</td>
<td>3157 cm⁻¹ (CH) as (aromatic), 2965 cm⁻¹ (aliphatic−CH), 1710 cm⁻¹ (C = O), 1560 cm⁻¹ (C=C), 1193 cm⁻¹ (C-O), 1598 cm⁻¹ (CH=N).</td>
<td>2.41 ppm (3H, -CH3), The signal of N-CH3 = 4.41ppm (3H).The signals of aryl-H = 7.51–6.84 ppm (3H), 3.41(2H, CH2CH2), at 9.39 and 8.27 due to the azomethine.</td>
<td>Signals at (122-138ppm) due to aromatic carbons and at 163ppm due to C=N.</td>
</tr>
<tr>
<td>M2</td>
<td>3161 cm⁻¹ (CH) as (aromatic), 2968 cm⁻¹ (aliphatic−CH), 1713 cm⁻¹ (C=O), 1559 cm⁻¹ (C=C), 1191 cm⁻¹ (C-O), 1561 cm⁻¹ (CH=N).</td>
<td>2.32 ppm (3H, -CH3), The signal of N-CH3 = 4.43ppm (s, 3H).The signals of aryl-H = 7.41–6.81 ppm (3H), 3.30(2H, CH2CH2), at 9.44 and 8.22 due carbonyl-OCH3 = 1.32 ppm.</td>
<td>43-55ppm due to CH3 group, signals at (122-131ppm) due to aromatic carbons and signal at 163ppm due to C=N.</td>
</tr>
<tr>
<td>M3</td>
<td>3168 cm⁻¹ (CH) as (aromatic), 2971 cm⁻¹ (aliphatic−CH), 1711 cm⁻¹ (C=O), 1559 cm⁻¹ (C=C), 1191 cm⁻¹ (C-O), 1561 cm⁻¹ (CH=N).</td>
<td>2.41 ppm (3H, -CH3), The signal of N-CH3 = 4.41ppm (3H).The signals of aryl-H = 7.53–6.90 ppm (3H), 3.29(2H, CH2CH2), at 9.43 and 8.32 due to the azomethine.</td>
<td>showed signals at (121-129ppm) due to aromatic carbons and signals at (169ppm) due to C=O.</td>
</tr>
<tr>
<td>M4</td>
<td>3161 cm⁻¹ (CH) as (aromatic), 2967 cm⁻¹ (aliphatic−CH), 1714 cm⁻¹ (C=O), 1554 cm⁻¹ (C=C), 1197 cm⁻¹ (C-O), 1570 cm⁻¹ (CH=N).</td>
<td>2.44 ppm (3H, -CH3), The signal of N-CH3 = 4.39ppm (3H).The signals of aryl-H = 7.53–6.90 ppm (3H), 3.29(2H, CH2CH2), at 9.43 and 8.32 due to the azomethine, The signal of carbonyl-OCH3 was observed at 1.42 ppm (3H).</td>
<td>showed signal at 55.70 ppm due to CH3 group, signals at (122-139ppm) due to aromatic carbons and signals at (168ppm) due to C=O.</td>
</tr>
<tr>
<td>M5</td>
<td>3233 cm⁻¹ (CH) as (aromatic), 2965 cm⁻¹ (aliphatic−CH), 1722 cm⁻¹ (C=O), 1566 cm⁻¹ (C=C), 1207 cm⁻¹ (C-O), 1602 cm⁻¹ (CH=N).</td>
<td>2.44 ppm (3H, -CH3), the signal of N-CH3 = 4.39ppm (3H).The signals of aryl-H = 7.63–6.80ppm(3H), 3.31(2H, CH2CH2), at 9.39 and 8.42 due to the azomethine, the signal of aromatic carbons and signals at (115-140ppm) due to aromatic carbons and signal at 161ppm due to C=N, showed signal at (55-58)ppm due to CH3 group.</td>
<td>signals at (126-133ppm) due to aromatic carbons and at 143ppm due to C = N, showed signal at 30ppm due to CH3 group.</td>
</tr>
<tr>
<td>M6</td>
<td>3231 cm⁻¹ (CH) as (aromatic), 2954 cm⁻¹ (aliphatic−CH), 1733 cm⁻¹ (C=O), 1545 cm⁻¹ (C=C), 1211 cm⁻¹ (C-O), 1612 cm⁻¹ (CH=N).</td>
<td>2.41 ppm (3H, -CH3). The signal of N-CH3 was observed at 4.32ppm (3H). The signals of aryl-H were seen at 7.53–6.70 ppm (3H), 3.21(2H, CH2CH2), at 9.19, and 8.32 due to the azomethine, The signal of carbonyl-OCH3 was observed at 1.42 ppm (3H).</td>
<td>Signals at (122–139ppm) due to aromatic carbons and signals at (168ppm) due to C=O.</td>
</tr>
</tbody>
</table>

Compound (3) showed signals at (121-129ppm) due to aromatic carbons and signals at (169ppm) due to C = O. Compound (M4) showed ¹H NMR CDCl3/ DMSO-d6, ppm): 2.44(3H, -CH3), The signal of N-CH3 was observed at 4.39ppm (3H). The signals of aryl-H were seen at 7.53–6.90 ppm (3H), 3.29(2H, CH2CH2), at 9.43, and 8.32 due to the azomethine, The signal of carbonyl-OCH3 was observed at 1.32 ppm (3H). The ¹³C-NMR spectrum of compound (4) showed a signal at 55.70 ppm due to the CH3 group signals at (122–139ppm) due to aromatic carbons and signals at (168ppm) due to C = O. Compound (5) showed ¹H NMR CDCl3/ DMSO-d6, ppm): 2.44(3H, -CH3), the signal of N-CH3 was observed at 4.39ppm (3H). The signals of aryl-H were seen at 7.63–6.80 ppm (3H), 3.31(2H, CH2CH2), at 9.39, and 8.42 due to the azomethine, the signal of carbonyl-OCH3 was observed at 1.42 ppm (3H). ¹³C-NMR spectrum of compound 5 showed signals at (115–140ppm) due to aromatic carbons and at 161ppm due to C=N, showed signal at 30ppm due to CH3 group.
carbons and at 161ppm due to C = N, showed signal at (55–58) ppm due to CH3 group. Compound 6 showed 1H NMR CDCl3/ DMSO-d6, ppm): 2.41(3H, -CH3), The signal of N-CH3 was observed at 4.32ppm (3H). The signals of aryl-H were seen at 7.53–6.70 ppm (3H ), 3.21(2H, CH2CH2), at 9.19, and 8.32 due to the azomethine, The signal of carbonyl-OCH3 was observed at 1.44 ppm (3H). The 13C-NMR spectrum of compound 5 showed signals at (126–133ppm) due to aromatic carbons and at 143ppm due to C = N, showed signal at 30ppm due to the CH3 group. All these results are shown in Table 2.

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REFERENCES