Preparation, characterization and biological activity of some novel Schiff bases

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Abstract
The [C-1] was synthesized by reaction of acide red-37 with 3,4,5 tribromobenzaldehyde and [c-2] was synthesized by reaction of sulfamethoxazole with 3,4,5 tribromobenzaldehyde, the compounds [c-1] and [c-2] were characterized by elemental analysis, infrared, $^1$H-NMR and mass spectroscopy. The biological activity were compared with amoxicillin as standard.

Keywords: 6-(3,4,5tribromobenzylideneamino)-5-(4-(3,4,5trimethoxybenzylidene)acrylamido)-2-sulphatophenyl)diazenyl-4-hydroxynaphthalene-2-sulphonic acid [c-1], 4-(2-chloro3,4,5trimethoxybenzylidene)amino-N-(5-methyl–isoxazol-3-yl)-benzene sulfonamide [c-2]

Introduction
Schiff bases are an important class of organic compounds [1]. They were reported by Hugo Schiff in 1864 [2]. Schiff bases are widely studied and used in the fields of organic synthesis and metal ion complex [3, 4]. For a number of reason their physiological and pharmacological activity’s [5, 6, 7]. Schiff bases are important compounds owing to their wide range of industrial applications [8]. Schiff bases have also been shown to exhibit a broad range of biological activities, including antifungal, antibacterial, antimalarial, anti-proliferative, anti-inflammatory, antiviral & antipyretic properties [9, 10].

Experimental
Instrumentation
Melting point were measured on gallenkamp Electronic melting points apparatus, the elemental analysis was performed on a perkin-Elmer 2400. Infrared spectra were recorded using potassium bromide disks on a pye Unicam SP3-300 infrared spectrophotometer. $^1$H-NMR experiments were run at 300MHz on a varian mercury vx-300 NMR spectrometer using TMS as internal standard in deuterated dimethyl sulfoxide. The mass spectra were recorded on shimadzu GCMS-Q-P 1000EX mass spectrometer at 70ev.

Synthesis of 6-(3,4,5tribromo benzylideneamino)-5-(4-(3,4,5tribromophenyl)acrylamido)-2-sulphatophenyl)diazenyl-4-hydroxynaphthalene-2-sulphonic acid [c-1]:
Amixture of (0.01 mole) of acid red-37, (0.01mol) of 3, 4,5tribromobenzaldehyde were mixed together in 200ml dry ethanol. The mixture was heated to reflux for 12hrs at which a brown residue was separated.
The reaction mixture was then cooled and the light brown residue was separated by filtration. The solid was recrystallized from ethyl alcohol to give faint brown crystals.
Synthesis 4 (2-chloro3,4,5trimethoxy-benzylidene) amino-N-(5-methyl-isoxazol-3-yl)-benzene sulfonamide(C-2)Amixture of Sulfamethoxazole (0.01mole) and 2-chloro-3,4,5trimethoxybenzaldehyde (0.01mole) and in around bottom glass (pyrill) flask (250ml) in pure ethanol (100ml) are stirred for 30minutes.
The mixture was heated to reflux for 8 hours and kept overnight the solid product was separated by filtration. The solid was recrystallized from ethanol.

Results and discussion
Spectroscopic studies of 6(3,4,5tribromo benzylideneamino)-5-(4-(3,3,4,5tribromophenyl)acrylamido)-2-sulphato phenyl)diazenyl)-4-hydroxynaphthalene-2-sulphonic acid (c-1).
The IR spectra of [c-1]table (1) exhibited a strong stretching frequency band for carbonyl group at 1671cm\(^{-1}\)and two absorption bands at 3312cm\(^{-1}\)due to (N-H)The \(^1\)H-NMR spectrum of [c-1] in deuterated DMSO showed a singlet signal at 9.53 ppm due to proton of the NH group, singlet signal at 8.65 ppm suggested the attribution of the proton of the CH=N group, as well as multiplets in the rang 6.90-7.88 ppm due to the phenyl protons and the \(^1\)HNMRSpectrum exhibit doublet signal at 8.20 ppm due to (Ar-CH =CH,J=12.3Hz). The mass spectrum of [C-1] showed the molecular ion peak at m/z465.21(89%), the base peak at 77.33(100%).

Biological activity
Measurement of antimicrobial activity using diffusion disc method: A filter paper sterilized disc (diameter80mm) saturated with measured quantity of the sample is placed on plate containing solid bacterial medium (nutrient agar broth) or fungal medium (Dox's medium) which has been heavily seeded with the spore suspension of the tested organisms after incubation, the clear zone of inhibition surrounding the sample is taken as a measure of inhibitory power of the sample [11, 12, 13, 14].
The compounds [c-1] and [c-2] were screened for their antibacterial activity against one gram positive bacteria, one gram negative bacteria and fungi candida albicans, the results of antimicrobial studies are given in table 3

<table>
<thead>
<tr>
<th>Compound number</th>
<th>IR (KBr)/CM(^{-1})</th>
<th>(^1)HNMR(\delta) (PPM)</th>
</tr>
</thead>
<tbody>
<tr>
<td>C-1</td>
<td>(\text{\text{\text{\text{v c=0 1671}}}})</td>
<td>9.53(S,NH)6.85(S,CH=N)8.20(d, CH=CH)6.90-7.88(m,Ar)</td>
</tr>
<tr>
<td>C-2</td>
<td>(\text{\text{\text{\text{2N-H 3246}}}})</td>
<td>9.10(S-NH)8.82(S,CH=N)6.71-7.78(m,Ar)6.11(S,CH=CH=CH=CH)</td>
</tr>
</tbody>
</table>

Fig 1: 6-(3,4,5tribromo benzylideneamino)-5-(4-(3,4,5tribromophenyl)acrylamido)-2-sulphotophenyl)diazenyl)-4-hydroxynaphthalene-2-sulphonic acid (c-1):

Fig 2: 4(2-chloro 3,4,5 trimethoxy benzylidene) amino-N-(5-methyl-isoxazol-3-yl) benzene sulfonamide

Table 1: Spectroscopic for {c-1} and {c-2}

\(^1\)HNMR\(\delta\) (PPM):

- \(\text{\text{\text{\text{v c=0 1671}}}}\)
- \(\text{\text{\text{\text{2N-H 3312}}}}\)
- \(\text{\text{\text{\text{2N-H 3246}}}}\)

- \(\text{\text{\text{\text{2N-H 3246}}}}\)
- \(\text{\text{\text{\text{2N-H 3312}}}}\)
- \(\text{\text{\text{\text{2N-H 3246}}}}\)

- \(\text{\text{\text{\text{2N-H 3312}}}}\)
Table 2: Physical data of the prepared compounds

<table>
<thead>
<tr>
<th>Compound</th>
<th>MP/C/colour</th>
<th>Solvent yeild %</th>
<th>MF (Mwt)</th>
<th>Elemental analysisCalcd/found</th>
</tr>
</thead>
<tbody>
<tr>
<td>c-1</td>
<td>254-256 Brown</td>
<td>Ethanol 78</td>
<td>C_{12}N_{4}S_{6}0_{6}B_{2}H_{7}</td>
<td>1129.615</td>
</tr>
<tr>
<td>c-2</td>
<td>162-164 yellow</td>
<td>Ethanol 90</td>
<td>C_{20}H_{8}S_{6}O_{6}CL</td>
<td>465.911</td>
</tr>
</tbody>
</table>

Table 3: Inhibition zones (mm) of compound [c-1] and [c-2]. The activity of 2.5mg/ml of sample Amoxicilline was used standard

<table>
<thead>
<tr>
<th>Compound standard</th>
<th>Staphylococcus aureus</th>
<th>Escherichia coli</th>
<th>Candida albicans</th>
</tr>
</thead>
<tbody>
<tr>
<td>c-1</td>
<td>14</td>
<td>26</td>
<td>13</td>
</tr>
<tr>
<td>c-2</td>
<td>18</td>
<td>20</td>
<td>17</td>
</tr>
<tr>
<td>Amoxicilline</td>
<td>23</td>
<td>21</td>
<td>15</td>
</tr>
</tbody>
</table>

References

7. Sprung MM. A Summary of the reaction of aldehydes with amines chem Rev.1940; 26:297-338.