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Synthesis new derivatives of Azo compounds contain a Schiff bases and evaluate their biological activity

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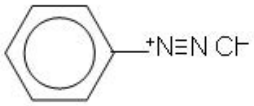
Abstract: The novel compounds have been synthesis via reaction benzidiene compund with p-amino acetophenone in the presence of ethanol absolute and reflux with stirrer to give Schiff base compound 1. The Schiff base copound treated with phosphoric acid and nitric acid, sodium nitrate and o-hydroxynaphthaldehyde, to product Azo compound 2. This compound reaction with different aromatic amines such as 2-chloro aniline, 4-hydroxyaniline, 2-carboxyaniline, 3-hydroxyaniline respectively to give compounds 3a-d. All the new compounds characterized by FT-IR spectrophotometer and HNMR spectroscopy, and evaluation The synthesized compounds shown moderate to good effectiveness against both gram positive (Streptococcus pneumoniae, Mycobacterium tuberculosis) and gram negative (Esherichia coli, Proteus mirabilis) bacteria when compared to the original medication.

Keywords: *Azo family, Antibacterial activity, Benzidiene, Schiff base.*

1. Introduction

The diazonium salts are extremely unstable; the only ones that are more stable are the aromatic salts, and even those are not very stable [1].

- This is due to the benzene ring's ability to stabilize the N-N group due to its high electron density.
- One type of diazonium salt is benzenediazonium chloride:



When phenols and diazonium ions are mixed, azo compounds with the azo group, -N=N-, are created. As an example, A yellow azo-dye is produced by an alkaline solution of benzene diazonium chloride, an electrophile, and phenol, the coupling agent. The addition of naphthalen-2-ol produces a visually arresting red precipitate [2-4]. These particular colors have little practical significance because of how little solubility they have in water. On the other hand, azo-dyes containing one or more sulphonic acid groups are far more useful and soluble. They are significant from a business standpoint in the dyestuffs industry [5-8]. Nowadays, synthetic azo compounds are widely used in many different application sectors, such as shipbuilding, the automotive industry, cable manufacture, paints, plastics, food, cosmetics, and pharmaceuticals [9–17]. However, the textile industry remains the traditional target market for synthetic azo dyes, and it is highly desirable to treat fibrous materials to impart antibacterial properties in addition to color. This is due to the biological deterioration that textile materials experience; it seems that microbes are responsible for about 40% of the damage. Fungi and bacteria are responsible for a material's decreased mechanical strength, color change, stains, and stale odor. Utilizing materials with antibacterial properties both extends their useful lifetimes and guards against damage caused by biological decay.

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Chemical reactions (adding antimicrobial chemicals via chemical bonding to functional groups of the fiber-forming polymers) or impregnation with antimicrobial compounds are two ways to manufacture biologically active materials.

2. Experimental

The effective fume cupboard was used to carry out the chemical operations for the synthesis and purification of the chemicals. The fluka Company provided the benzidiene. The other material was delivered by the chemical firms Aldrich, Across Organic, and Merck. Solvents and reagents were of analytical quality and were acquired from markets. Shimadzu's FT-IR 8300 spectrophotometer was used to obtain FT-IR spectra of KBr pellets in the 4000-400 (cm-1) area. Using silica plates and TLC, all reactions were monitored. A potassium permanganate staining chamber was employed, and ultraviolet light at 254 nm was used for visualization.

3. Synthesis of Compound [1]

In a round flask, dissolve (2.70 g-14.7 m.mol) of benzidine in (5 ml) of ethyl alcohol with the addition of (3-5) drops of glacial acetic acid. Then, add (1.99 g-14.7 m.mol) of (C 8. H 8N O) P-aminoacetophenone was heated to 67 C° while being stirred for 7 hours. The resultant solution should be poured into a bottle and left for an hour to allow the alcohol to evaporate [18], collect the precipitate, and dry it before being washed with water to remove any leftover alcohol. It is then re-crystallized. By using mobile phase TLC (hexane: ethyl acetate) with a ratio of (6:4) (V: V), it was possible to assess the purity of the produced product and the completion of the reaction using ethanol.

4. Synthesis of Azo Compound [2]

1.78 mmol) of compound(I) should dissolve after being heated and stirred in 8 mL of 85% phosphoric acid. (H3PO4), followed by the addition of 4 mL of concentrated nitric acid and 0.13 g, 1.87 mmol, of sodium nitrite (NaNO2) in 2 mL of distilled water, while vigorously stirring the mixture at a temperature below (5 o C) for about 10 minutes. Add the droplets of aldehyde solution that you made by thoroughly swirling 0.17 g, 1.78 mmol of meta hydroxy naphthaldehyde into 0.5 mL of distilled water [19]. The reaction was then monitored after the reaction mixture had been added to 100mL of cold water, the sediment had been repeatedly leached and rinsed with cold water, and it had been re-crystallized using ethanol.

87% yield, as off-white precipitate. IR /cm^{-1} 3464, 3316, 3198, 1983,1784, 1634, 1492, 1107, 836 cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d6) δ = 9,38 OH 8.95 (2H, s, NH), 7.85 (17H, d, *J* 9 Hz, ArH).

5. Synthesis of Compound [3]$_{a-d}$

3.60 g, 10 mmol, of compound [2] in 5 mL of ethanol, together with (3-5) drops of glacial acetic acid, should dissolve in a round flask. After that, stir for 7 hours while adding (10 m.mol) of various aromatic amines. The end result should be poured into a bottle and allowed for an hour to allow the alcohol to evaporate [18], collect the precipitate, and dry it before being rinsed with water to eliminate any remaining alcohol. The crystal is then reformed. The purity of the generated product and the success of the ethanol reaction were evaluated using mobile phase

3a; IR ν_{max}(film)/cm^{-1} 3459, 3234, 3178, 1894,1753, 1633, 1489, 1118, 943 cm^{-1}; ^{1}H NMR δ = 10,56 OH 9.04 (2H, s, NH), 6.99 (21H, , ArH).

3b; IR ν_{max}(film)/cm^{-1} 3554, 3256, 3134, 1976,1723, 1698, 1445, 1100, 845 cm^{-1}; ^{1}H NMR δ = 8,95 OH 7.98 (2H, s, NH), 6.96 (21H, , ArH).

. **3c**; IR ν_{max}(film)/cm^{-1} 3543, 3432, 3076, 1967,1743, 1645, 1494, 1176, 834 cm^{-1}; ^{1}H NMR δ = 8,84 OH 7.88 (2H, s, NH), 6.86 (21H, , ArH).

. **3d**; IR ν_{max}(film)/cm^{-1} 3554, 3345, 3145, 1965,1723, 1687, 1478, 1111, 876 cm^{-1}; ^{1}H NMR δ = 8,93 OH 7.97 (2H, s, NH), 6.87 (21H, , ArH).

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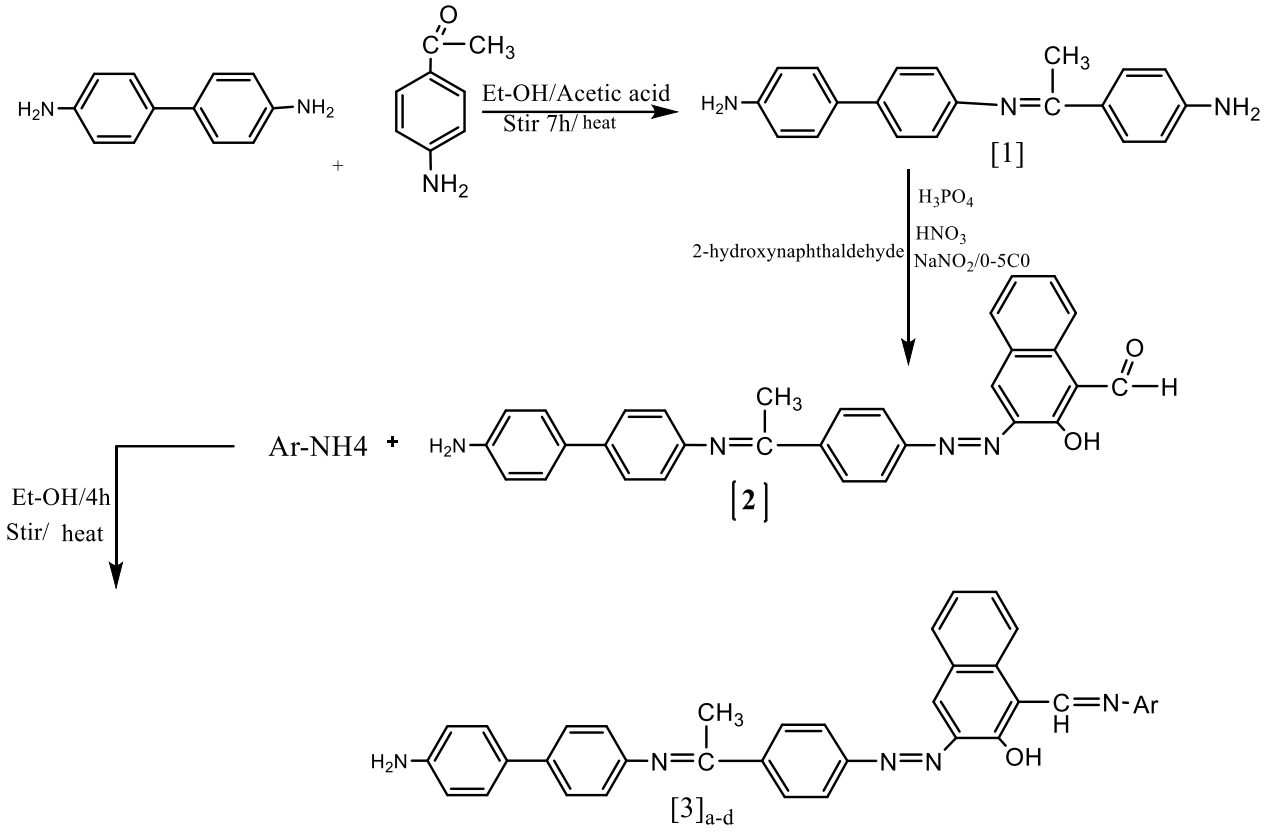
6. Antibacterial Activity

Treatment of infectious diseases is a very difficult issue because of a number of significant factors, one of which is bacterial therapeutic resistance. The synthetic compound's biological effects have been screened out of gram-ve and gram-positive bacteria. Table 1 illustrates that certain compounds had moderate to good antibacterial activity in comparison to amoxicillin. The antibacterial qualities of the generated compounds were assessed using the disc diffusion method against gram positive and gram-



Ar-=2-Cl-C_6H_4-NH_2 ,4-OH-C_6H_4-NH_2 ,2-COOH-C_6H_4-NH_2 ,3-OH-C_6H_4-NH_2,

negative bacteria, such as Proteus mirabilis and Esherichia coli, as well as Mycobacterium TB and Streptococcus pneumoniae. Here, the paper disc diffusion method was used to conduct the test, and Benzidine 1 was assessed as a reference chemical to compare the activity. Table 1 unequivocally demonstrates how the selected strains' antibacterial activity was boosted by dapsone derivatives. Data show that the chemical under examination has better effects than the reference against S. pneumoniae, M. tuberculosis, P. mirabilis, and E. coli.

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Table 1.

Comp no.	Inhibitory zone measured in (mm)			
	M tb	S. pneumoniae	E. Coli	P. mirabilis
1	17	19	18	14
2	22	24	21	23
3a	25	22	21	18
3b	20	25	16	20
3c	18	33	16	16
3d	18	20	21	24
Amox	16	12	15	13
DMSO solvent	0	0	0	0

7. Conclusion

In this study, Synthesis of new derivatives containing Azo compound, characterization of these compounds and the biological activity of the prepared compounds was studied,

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