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Synthesis and Characterization of New Azo Dyes Based on Thiazole and Assess the Biological and Laser Efficacy for Them and Study their Dyeing Application

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Abstract

The aim of this work describes the synthesis of disperse dyes in the derivative of 2-amino -4-hydroxy thiazole, which can be used as dyes for polyester fabrics with orange and red color They were obtained by preparing, (2-amino-4-hydroxy- thiazole), then the latter compound was diazotization and couplings to produced (compound III and IV), Which were introduced by another coupling with diazonium salt of compound I diazotization to synthesize disperse (VII,VIII). The synthesized heterocyclic and synthesized dyes were studied by UV Spectroscopy, FT-IR, ^{1}H-NMR and ^{13}C-NMR. The substituted dyes Penetrate with good depth on polyester fabrics with a shade of orange and red colors, respectively. That increase heteroatoms and the conjugation in the dyes structure lead to high redshifts and the brightness of shades, color stability is high and fastness properties. The antibacterial activities were studied against different kinds of bacteria, namely *Eschershia coli* and *Klebsiella Pneumonia* Gram (-) ve, *Staphylococcus aureus* and *Staphylococcus epidermidis* Gram (+) ve. In addition, evaluation of laser efficacy was showed for the compounds (I,III,IV,VII,VIII) were radiated by laser for (10, 20, 30) seconds, It was observed that the prepared compounds were not affected and did not polymerize or degradation when measuring melting point and color.

Keywords: Thiazole, azo dyes, Disperse dyes, Heterocyclic compounds

Introduction

Dyes can be defined as unsaturated and colored organic compounds that are capable of coloring and dyeing a substrate (a textile). A disperse dyes are one of a types of water-insoluble dyes that dye acetate fibers polyester and disperse dyes have substantively for one or more hydrophobic fibers e.g. nylon, cellulose acetate, acrylic, polyester, and most of the manufactured fibers which are free from ionizing groups [1-3]. A disperse dye molecule is based on an azobenzene or anthraquinonoid molecule with nitro, amine, hydroxyl, etc. groups attached to it; these types of dyes have been continuous/ thermoset. Dyeing of polyester under high pressure and temperature. Disperse dyes have a larger molecular size, lower volatility, and hence, better sublimation properties. For the synthesis of disperse dyes, we need different chromophore compounds in the structure of disperse dyes [4-7]. Heterocyclic have been used widely in the production of disperse dyes with excellent discharge ability on cellulose acetate. These dyes are characterized also by having generally high extinction coefficients and excellent brightness, relative to azo dyes derived from substituted anilines. These distinctive properties have encouraged work on dyes prepared from heterocyclic diazo components, which are suitable for dyeing synthetic fabrics [8]. Many diazo components have been used in the production of disperse dyes in recent time [9, 10]. Derivatives of 2-aminothiazole have been used as heterocyclic components since long for different disperse dyes [11, 12]. 2-Aminothiazoles have a great impact from a therapeutic effect because it used as an intermediate in the synthesis of antibiotics such as the well-known sulfa drugs. As shown in the literature as anti-inflammatory activity, antimicrobial, antifungal, anesthetic, antiviral drugs and anti-hypertensive. 2-Aminothiazole derivatives have been also used as intermediates in the synthesis of various kinds of dyes. In continuation of our interest in the synthesis

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of arylazothiazole derivatives, this paper reports the synthesis of some 2-amino-5-arylazothiazoles and their application as disperse dyes on polyester fabrics. Furthermore, the antibacterial activities of the synthesized dyes against various pathogenic bacteria were also investigated [13-15].

Materials and Methods of work (Experimental Section)

Description of materials and products.

Materials

In FALC instrument (s.r. I) 50/60 HZ (Italy) melting points were determined. FT-IR spectra of compounds were recorded on ((Shimadzu Fourier transform infrared Spectrophoto meter FT-IR 8400 s (KBr) scale (4000 – 400) cm^{-1} infrared spectrophotometer using KBr pellets. ^{1}H NMR,^{13}CNMR spectra were recorded on ^{1}H-NMR, ^{13}C-NMR (Bruker Advance III 600,300 MHz NMR spectrometer with the cryoprobe). Thin layer chromatography TLC, This test was carried out on all the compounds prepared. It was conducted during various preparatory times. They checked using U.V Lights, sheets silk jel and mobile phase (ethanol absolute). The prepared compounds were irradiated with helium-neon laser beam (visible laser) of one milliwatt and wavelength 600-700 nanometers, 2010 model and organic elemental analysis 2400 series II CHNS/O Elemental Analyzer.

Chemical synthesis

Synthesis of 2-aminothiazol-4-ol (I) [16]

2- Amino 5-hydroxy thiazole (I) which was prepared by refluxing a mixture of (12.2 g, 0.1 mol) ethyl chloroacetate and (7.6 g, 0.1 mol) thiourea in (150 ml) ethanol absolute for three hours. After that (150 ml) of ice-water was added to mixture, the product (I) rapidly precipitated. It was purified by pouring them in water and adjusting the pH to 5 with sodium acetate. The product was obtained as a yellow solid in good yield. (72%), 145-147 C^0. Anal. Calculated for $C_3H_4N_2OS$ (116,045): C, 31.034; H, 3.448, N; 24.137; found %: C, 31.241; H, 3.391; N, 24.149.

Synthesis of (E)-2-bromo-2-((4-hydroxythiazol-2-yl) diazenyl)-1-phenylethan-1-one (III) and (E)-1-bromo-1-((4-hydroxythiazol-2-yl)diazenyl)propan-2-one (IV)

A) Synthesis of diazonium hydrochloride (II) [17]

Compound salt solution amine hydrochloride was prepared from (2mmol) of (I) in 5 ml Conc. (HCl) after that, the solution was placed in an ice bath for 10 min at (0-5) °C. Sodium nitrite solution prepared from (0.145 gm, 2.1mmol, 5ml water) was applied drop wise with stirring to the amine hydrochloride salt solution over a period of (20-25) minutes at 0 ^{0}C.where a yellow precipitate of diazonium hydrochloride salt was formed. The reaction mixture was stirred for another 15 minutes while the temperature was held at (0 °C).

B) Coupling diazonium salt [18]

To a cold and stirred solution of amine hydrochloride salt (II) and sodium acetate anhydrous (5 gm) in pyridine, (2mmol) phenacyl bromide was added to produced (III) and (2mmol) brome acetone was added to produced (IV) was added drop wise at (0-5 ^{0}C). Stirring lasted for 2 hrs. A colorless needle crystal. Colorless powder precipitate formed respectably and left overnight in the refrigerator. (250 ml) of Water was added to the reaction mixture. The products obtained was filtered, washed with several times and recrystallized from chloroform

Compound (III), (75% yield). m.p. = 334-336 ^{0}C. Anal. Calculated for $C_{11}H_7N_3O_2S$ (245.44): C, 53.877; H, 2.857; N; 17.714; found %: C, 53.926; H, 2.901; N, 17.592.

Compound (IV) (62% yield), m.p. 197-201 0C. Anal. calcd for $C_6H_5N_3O_2S$ (183.): C, 39.304; H, 2.809; N, 22.855; found %: C,39.412:H,3.762;N,23.014.

Synthesis of (E)-(5-hydroxy-6-((4-hydroxythiazol-2-yl)diazenyl)thiazolo[2,3-c] [1,2,4] triazol -3-yl)(phenyl)methanone (VII) and (E)-1-(5-hydroxy-6-((4-hydroxythiazol-2-yl)diazenyl)thiazole [2,3-c] [1,2,4]triazol-3-yl)ethan-1-one (VIII) [19]

(0.01) mole of either compound (V or VI) was dissolved in 40 mL of ethanol absolute containing (2.5g) of sodium acetate then, this solution was added to the diazonium salt (II) drop wise with stirring, keeping the temperature below 5 ^{0}C. Stirring was continued for 45 min. The produced solid was filtered and washed with cool water. The formed solid was recrystallized from benzene. Yield 80%. The process was monitored by TLC on silica gel plates using as an eluent system ethanol: acetone (2:1, v⁄v).

Dye (VII) red needle crystals (60% yield), m.p= 266 -268 C0.). Anal. calcd. for $C_{14}H_8N_6O_3S_2$ (372): C, 45.161; H, 2.15; N, 22.58, found%: C, 45.093; H, 2.062; N, 22.639.

Dye (VIII) green crystals (52% yield), m.p= 258-260 C^0. Anal. calcd. for $C_9H_6N_6O_3S_2$ (396): C, 34.838; H, 1.935; N, 27.096, found%: C, 34.813; H, 1.962; N, 27.179.

Dyeing and fastness properties of fabrics.

By using the recorded procedure19, all the dyes (VII) and (VIII) were applied on wood in 3% shade. The fastness to light, sublimation, and perspiration of the dye pattern was assessed according to British standard 1006-1978 and the washing fastness

according to Indian standard (ISO method) at 60 ^{0}C/30 min. The fastness was tested using crock meter (10 strokes to & fro) (Atlas) AATCC-1961. The percentage of exhaustion and fixation of dyes on the polyester fabric was determined by following the reported method [20, 21].

Reduction Clearing

The dyed material was processed for 30 minutes at 60° C in a bath containing 1.5 g / L dispersing agent, 2 g / L caustic soda, and 2 g / L sodium dithionite. This was intended to eliminate unfixed dye and carrier residues that could linger on the fabric after them dying [21]

Antibacterial Activity

The antibacterial activity of compounds (I, III, III, VII, VIII) were measured against two types of bacteria namely ***Eschershia coli*** **and** ***Klebsiella Pneumonia*** **Gram (-) ve,** ***Staphylococcus aureus*** **and** ***Staphylococcus epidermidis*** **Gram (+) ve**, using the disk diffusion method. The disks were soaked with a DMSO. Afterwards, dried in an incubator before being put in bacteria cultures. The negative control was DMSO. For two days, the plates were incubated at 37° C. The maximum inhibition zone against each type of test micro-organism was observed and calculated for study. ***Ampicillin***, ***amoxicillin***, and ***Ciprofloxacin*** were used as control samples at three concentrations [22-24].

Results and Discussion

2-amino-4-hydroxy thiazole (I) was prepared by the reaction of ethyl chloroacetate and thiourea in ethanol for three hours as summarized in Scheme 1. The structure of the product produced is assigned on the basis of analytical and spectral data. FT-IR spectrum showed a stretching band at (3452 and 3328) cm^{-1} for O-H and stretching bands NH_2, a stretching band at 1635 cm^{-1} for C=C and stretching bands at (1141-900) cm^{-1} for C-O stretching. ^{1}H-NMR revealed 3 signals corresponding to 5 hydrogen atoms and the ^{13}C-NMR spectra this spectrum showed three signals of three carbon atoms for the same compound.

Heterocyclic diazonium salts are the class of reactive substrates, and have gained recent attention to their synthetic potential. In addition, there are many heterocyclic diazo compounds, which have biological activities.

Thus, when diazonium salt of thiazole (I) reacted with phenacyl bromide and bromoacetone respectively in pyridine gave the corresponding compounds (III, IV) indicating condensation with elimination of HBr (scheme 1). In elemental analysis, the structure of the compounds formed coincided with their correct values. The most important band appeared to distinguish the dyes by IR spectral bands, appearance of stretching vibrations bands of carbonyl group(C=O) at (1681.65 -1687012) cm^{-1}, azo group (N=N) at (1500.52-1521.73) cm^{-1} respectively frequency and the disappearance of the amine group frequency is evidence of the success of the coupling process between the diazonium salt and the two compounds mentioned to give the Corresponding compounds preparation.

The other and strongest evidence for the formation of compounds is the study of spectra ^{1}H-NMR and ^{13}C-NMR spectra. ^{1}H-NMR spectra of II revealed, multiple signals attributed to aromatic protons and two signals At 6.65 and 10.08 ppm and the integration of one proton to the other, due to tautomerism (OH)and (=CH). H-NMR spectra of compound (III) as revealed3signals corresponding to5 hydrogen atoms, due to (OH), (=CH) and (CH_3),while ^{13}C-NMR spectrum showed 9 signals for the compound (III) and 6 signals for the compound (IV) in different chemical shifts Which is in line with our proposal for chemical formulas for the duality product.

Characterization of I

IR, (ν/cm^{-1}): 3452-3328 (OH, NH_2), 3104 (HC=C), 1635 (C=C).

^{1}H-NMR, δ: 5.16 (s, 2H, NH_2), 6.81 (s, 1H, thiazole H-3), 9.76 (s, 1H, OH).

^{13}C-NMR: 131.98 (thiazole C-3), 146.91 (C-NH_2), 162.26 (C-OH).

Characterization of Compound III

IR, (ν/cm^{-1}): 3480–3327 (OH), 3.114, 3047 (CH, aromatic), 1673 (C=O), 1639 (C=C), 1583 (C=N).

^{1}H-NMR, δ: 6.65 (s, 1H, thiazole), 7.09–7.56 (m, 5H, C6H5), 10.08 (s, 1H, OH).

^{13}C-NMR, δ: 122.92, 123.74, 124.99, 126.35, 129.20, 129.37, (Ar), 131.79 (thiazole C-5), 147.09 (thiazole C-2) 152.78 (-C-C=N), 166.96 (C-OH), 188.91 (C=O).

Characterization of Compound IV

IR, (ν/cm^{-1}): 3340–3315 (OH), 3102 (HC=C), 2978-2811 (CH3), 1684 (C=O), 1636 (C=C), 1587 (C=N).

^{1}H-NMR, δ: 2.54 (s, 3H, CH3), 6.68 (s, 1H, thiazole-H-3), 9.98 (s, 1H, OH).

^{13}C-NMR, δ: 17.8 (CH_3), 133.4 (thiazole C-5), 141.6 (Thiazole C-2), 148.19 (-C-C=N), 159.67 (C-OH), 178.53 (C=O).

Characterization of Dye (VII)

IR (ν/cm^{-1}): 3300-3260 (OH), 1668 (C=O) 1626 cm^{-1} (C=C), 1517 cm^{-1} (C=N), 1440 cm^{-1}(N=N).

^{1}H-NMR (DMSO-d6): δ/ppm= 9.85 (s, H, OH thiazole), 10.90 (s, H, OH thiazole), 7.20 (s, 1H, thiazole), 7.23-8.31 (m, 8H Ar-H).

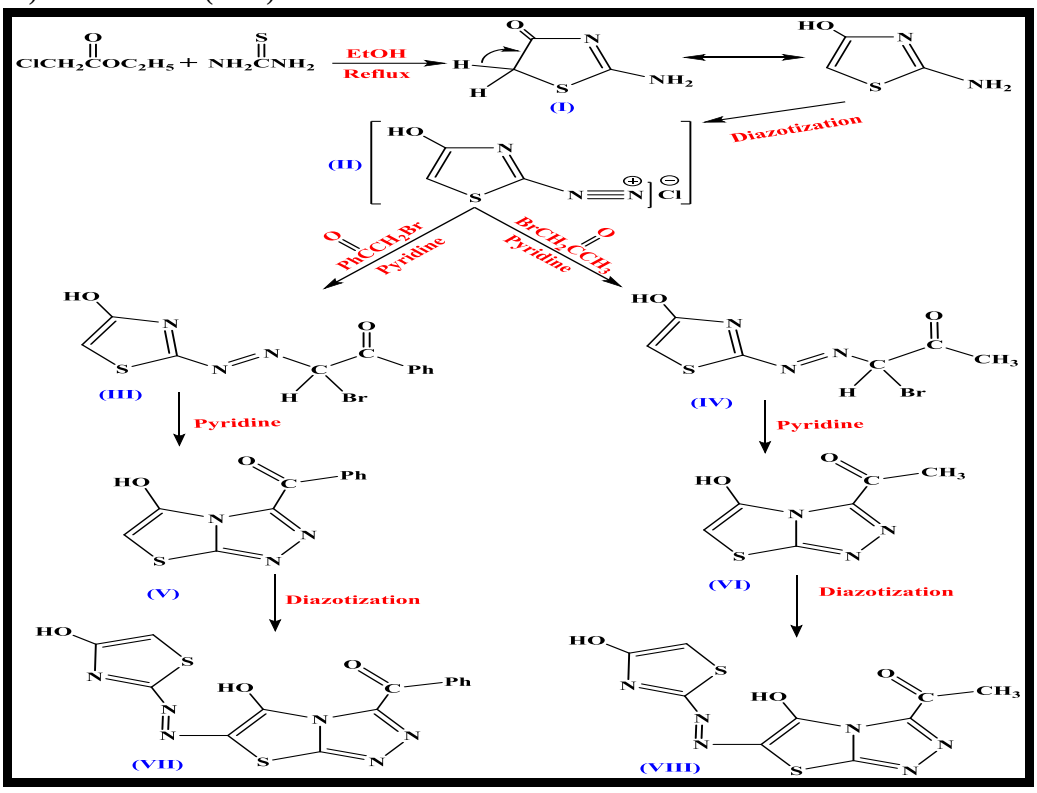
^{13}C-NMR, δ: 122.4, 123.7, 124.6, 128.7, 129.2, 131.5 (Ar), 133.85-134.26 (thiazole C-5), 147.45 (-C-C=N) 156.39- 160.14 (thiazole C-2), 169.06-171.98 (C-OH), 187.21 (C=O).

Characterization of Dye (VIII)

IR (ν/cm^{-1}): 3300 (OH), 1669 (C=O), 1626 (C=N), 1517 (C=C) aromatic 1440 (N=N).

^{1}H-NMR (DMSO-d6): δ/ppm= 2.85 (s, 3H, CH_3), 6.92 (s, 1H, thiazole), 9.2 (s, H, OH thiazole), 10.85 (s, H, OH thiazole).

^{13}C-NMR, δ: 21.48 (CH3), 131.23-131.79, (thiazole C-5), 147.06 (-C-C=N), 152.76-159.76 (thiazole C-2), 164.43-166.96 (C-OH), 188.91 (C=O).



Scheme 1. Route of prepared compounds (I - VIII)

Discussion Dyeing and fastness properties of fabrics

Since the purpose of this research is to prepare dyes with desirable qualities and because we have reached the compounds that did not give us strong and stable dyes to achieve our research objective, the chemical properties of the compounds prepared from the start were examined. Where we show the following chemical properties, the diazonium salts (II) reacts as an electrophile with an electron-rich coupling component, through an electrophilic aromatic substitution mechanism. The hydroxyl group directs the aryldiazonium ion to the parasite unless that position is occupied, in which case the ion attaches ortho. On this base, that is why the coupling between a compound (III), and (IV) separately with a diazonium salt. In order to validate the new pathway in the research to synthesize the distinguishing dyes, the spectra of these dyes were studied to verify their chemical composition and to determine the binding location of the diazonium salt coupling. IR spectrophotometers as FT-IR which was used in the present study to characteristics the presence of functional groups such as carbonyl (C=O), azo (N=N), nitrile (C–N), hydroxyl (OH). The evidence that proves the authenticity of the synthesized dyes is through spectra ^{1}HNMR and ^{13}CNMR that show different signals and undoubtedly support the correct chemical structures of the synthesized dyes. ^{1}H-NMR spectra of (VII) dye revealed, multiple signals attributed to aromatic protons, which appeared in the same integration that appeared in the compound (III), this shows that the diazonium salt (II) did not couple on the phenyl ring, while these signals did not appear in the (VIII) dye, it showed a signal at (2.87ppm) the integration of three protons attributed to the methyl group. Also in ^{1}HNMR spectra for VII and VIII dyes, two converged single signals appeared, almost identical in integration and at high frequency. The first signal to proton (OH) group in the thiazole ring and the second reference to the other (OH) group, which appeared at high frequency because of the

replacement of hydrogen with the azo group on the neighboring atom. Finally, it showed a single signal and integration of one proton returns to (=CH) in thiazole ring. This is evidence that the coupling occurred at atom 4C. The results of the ^{13}CNMR spectroscopy of the dyes synthesis were consistent with our prediction of the chemical formula of the two dyes and the suggested path of the reaction.

While spectrum ^{13}C-NMRa another proof of the correctness of the structure of the two dyes prepared.

The Uv-visible confirm of the dyes showed that dyes(VII and VIII) absorbed light at high wavelengths up to 549.0 nm (dye VII) and 376.5 nm (dye VIII).

The dye VII exhausted well, gave leveled dyeing, and given good shades upon application of the dyes to polyester. This is due to the composition of the coupling portion that contains the aromatic ring structure so increases the dye's stability.

The dyeing properties such as fastness to washing, light, and perspiration of the dyes were evaluated and the results as shown in Table 1 indicate that the dyes have very high fastness to washing, perspiration, and light.

The dispersed dyes were placed on polyester fabric at a depth of 2 %. The result of dye bath exhaustion and the fixation of the dyed fabric are given in Table 2**.**

Antibacterial activity [25, 26]

The effect of the prepared compounds (I) on the growth of bacteria, namely ***Eschershia coli, Klebsiella Pneumonia*** **Gram (-ve),** ***Staphylococcus aureus*** **and** ***Staphylococcus epidermidis*** **Gram (+ve)**. Antibacterial activity of the prepared compounds were studied and the findings showed good antibacterial activity in some of the prepared compounds. The results of inhibition zone (IZD) in millimeter are shown in table (3), see scheme (2- 5)**.**

Influence of lasers on prepared compounds (I, II, III, VII, VIII) [27]

A laser apparatus with a capacity of (5) milliwatts which gives laser rays in the visible area of the spectrum with a wavelength (600-700) nm in continuous waves. The compounds (I, III, IV, VII, VIII) were radiated by laser for (10, 20, 30) seconds. It was observed that the compounds were not affected. They did not disintegrate or polymerize when the melting point and color were measured. This denotes that the laser beams used did not affect the compounds. Since they are stable, as shown in the table (4).

Conclusion

Disperse dyes (VII, VIII) were synthesized by coupling diazotized **diazonium salt of thiazole** with III and IV. The nature of the substituent in the coupling components have more effect on the visible absorption and the shade of the dying. The presence of groups that increase the stability (OH, N=N, C=O, ph, S) and increase the resonance leads to Improve pigment properties. In addition, some of the prepared compounds showed good antibacterial activity against the antibacterial such as ***Eschershia coli, Klebislla Pneumonia*** **Gram (-ve),** ***Staphylococcus aureus*** **and** ***Staphylococcus epidermidis*** **Gram (+ve)**. The compounds (I, III, IV, VII, VIII) were radiated by laser for (10, 20, 30) seconds. It was observed that the compounds were not affected and did not disintegrate or polymerize when color and the melting point were measured. This denotes that the laser beams used did not affect the compounds. Since they are stable.

Acknowledgements

The authors are grateful for everyone who supported us in getting the chemicals. The authors also thank for everyone who helped us calculate, analyze and interpret the results.

Table 1. Results of dispersing dyeing and various fastness properties of the dye on polyester fabric

Dye	Color shades on	Light	Washing	Perspiration Fastness		Sublimation	Rubbing fastness	
No.	polyester fabric	fastness	fastness	Acid	Alkaline	fastness	Dry	Wet
VII	Red	5	5	4.5	5	5	3	3
VIII	Light orange	5	4	5	5	5	3.4	3.4

Table 2. Absorption maxima (λ max), intensities (log ε), fixation (F) and exhaustion (E) of disperse dyes on polyester fabric

Dye	Absorption maxima		Disperse dyeing on polyester	
No.	λ_{max}/nm in DMF	log ε	% F	% E
VII	500	5.33	82	77
VIII	367	3.15	67	56

Table 3. Antibacterial activity of the prepared compounds (I, III, IV, VII, VIII) with control antibiotic

Comp. No.	*E. Coil* Conc. mg/ml			*K. Pneumonia* Conc. mg/ml			*S. Aureus* Conc. mg/ml			*S. Epidermidis* Conc. mg/ml		
	25	**50**	**100**	**25**	**50**	**100**	**25**	**50**	**100**	**25**	**50**	**100**
I	1	3	4	1	2	3	1	3	3	2	3	4
III	0	2	4	0	2	3	1	2	4	1	2	4
IV	1	2	3	0	3	4	1	3	3	0	3	4
VII	1	1	2	2	3	3	1	2	3	1	3	4
VIII	1	2	4	0	2	4	1	2	4	1	2	4
Amoxicillin	2	3	3	1	3	3	2	2	4	1	2	4
Ampicillin	1	3	4	2	3	4	1	2	4	2	2	3
Ciprofloxacin	1	2	4	2	2	4	1	2	3	1	3	4
Blank disk	0	0	0	0	0	0	0	0	0	0	0	0

Table 4. The results of the irradiation of the compounds by laser beams

Comp. No.	10 S		20 S		30 S	
	M.P. ^{0}C	**Color**	**M.P. ^{0}C**	**Color**	**M.P. ^{0}C**	**Color**
I	145-147	Yellow	145-147	Yellow	145-147	Yellow
III	334-336	Colorless	334-336	Colorless	334-336	Colorless
IV	197-201	Colorless	197-201	Colorless	197-201	Colorless
VII	266 -268	Red	266 -268	Red	266-268	Red
VIII	258-260	Green	258-260	Green	258-260	Green

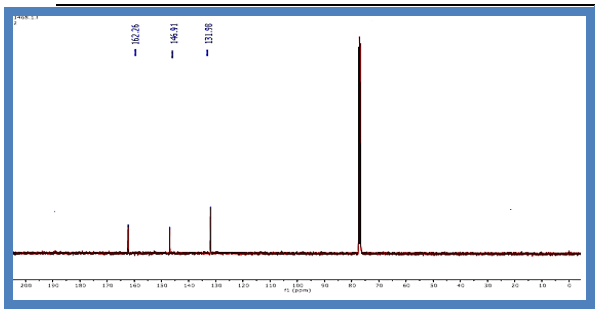


Fig. 1. ^{13}C-NMR spectrum of (I)

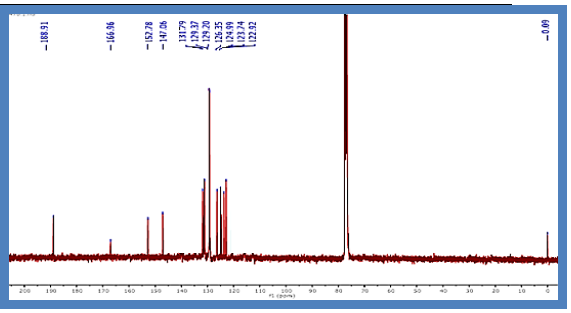


Fig. 2. ^{13}C-NMR spectrum of (III)

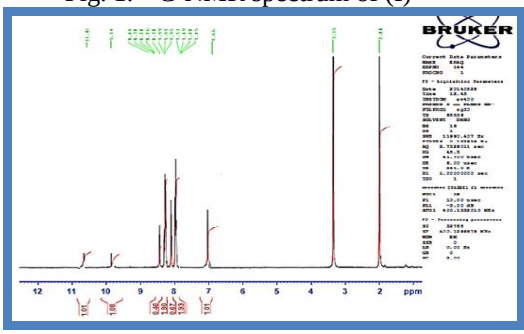


Fig. 3. ^{1}H-NMR spectrum of Azo dyes (VII)

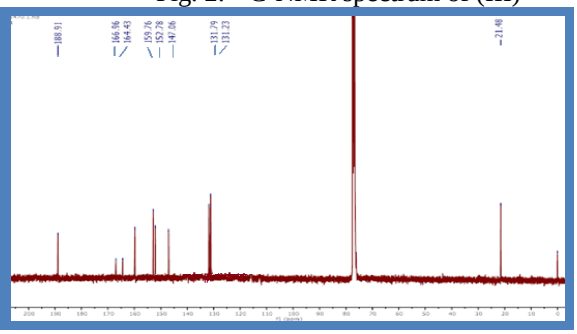
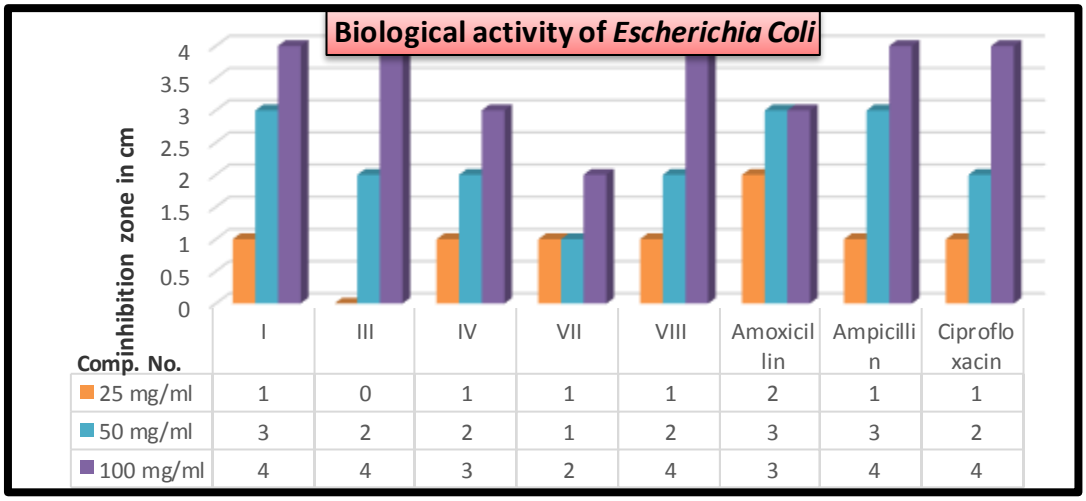
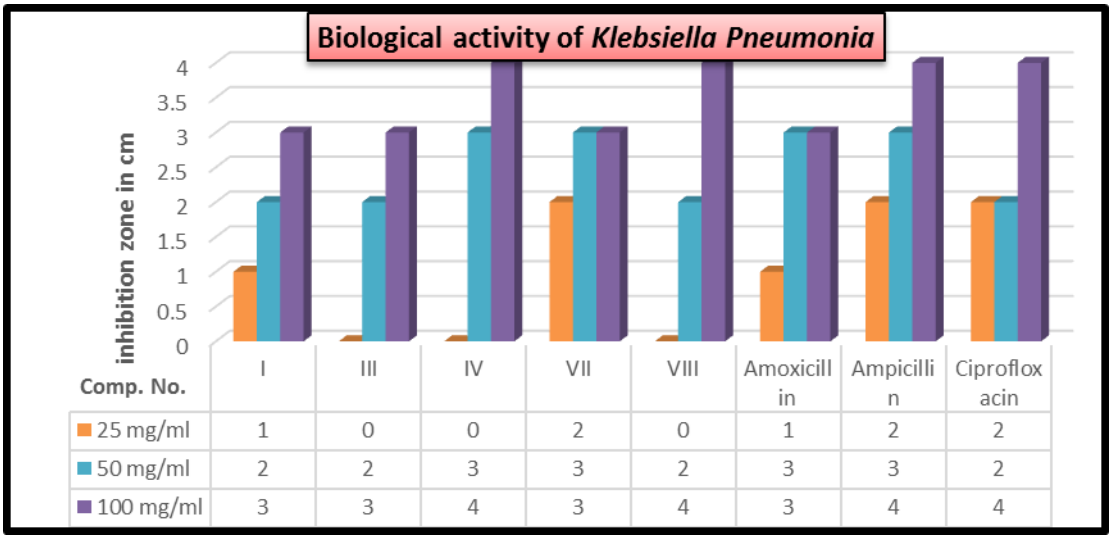


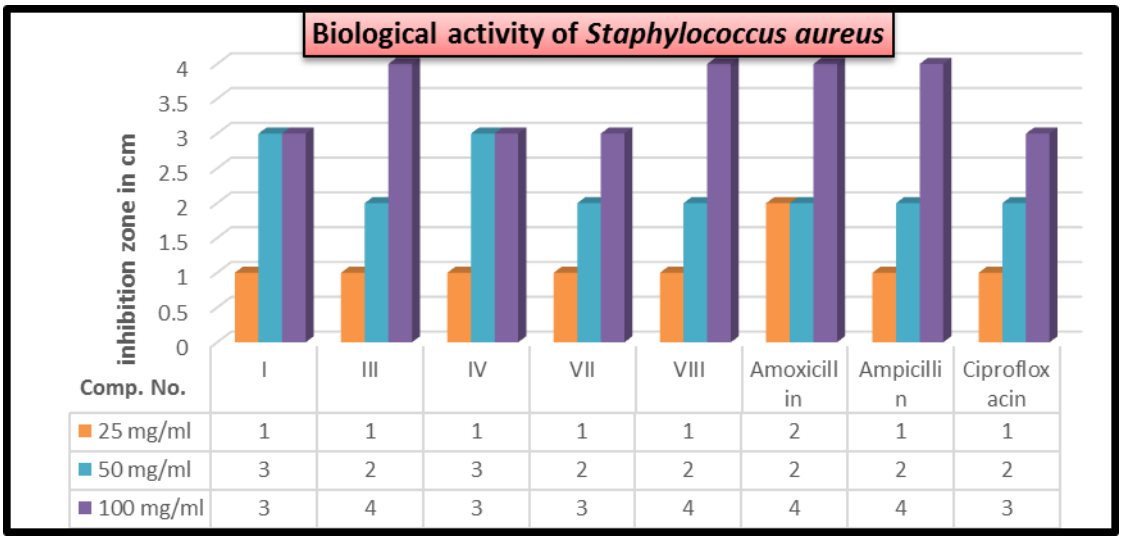
Fig. 4. ^{13}C-NMR spectrum of Azo dyes (VIII)



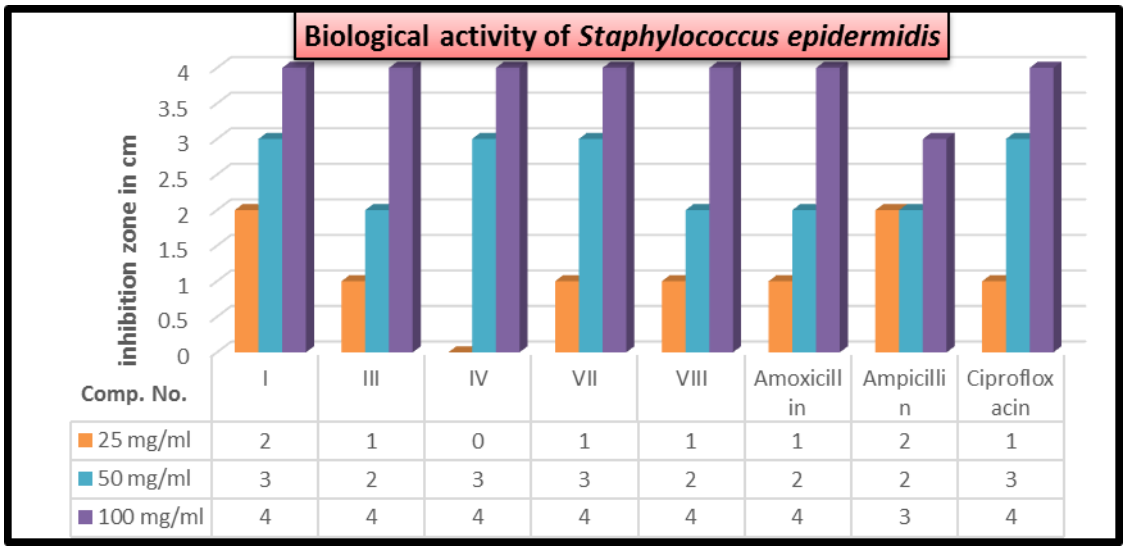
Scheme 2. Evaluation of inhibitory activity of compounds prepared with control antibiotic for *E. Coli*



Scheme 3. Evaluation of inhibitory activity of compounds prepared with control antibiotic for *K. Pneumonia*



Scheme 4. Evaluation of inhibitory activity of compounds prepared with control antibiotic for *S. Aureus*



Comp. No.	I	III	IV	VII	VIII	Amoxicillin	Ampicillin	Ciproflox acin
25 mg/ml	2	1	0	1	1	1	2	1
50 mg/ml	3	2	3	3	2	2	2	3
100 mg/ml	4	4	4	4	4	4	3	4

Scheme 5. Evaluation of inhibitory activity of compounds prepared with control antibiotic for *S. Epidermidis*

References

[1] Al-Etaibi, A. M., Alnassar, H. S., & El-Apasery, M. A. (2016). Dyeing of polyester with disperse dyes: Part 2. Synthesis and dyeing characteristics of some azo disperse dyes for polyester fabrics. Molecules, 21(7), 855.

1. Al-Etaibi, A. M., El-Apasery, M. A., Mahmoud, H. M., & Al-Awadi, N. A. (2012). One-pot synthesis of disperse dyes under microwave irradiation: Dyebath reuse in dyeing of polyester fabrics. Molecules, 17(4), 4266-4280.
2. Al-Etaibi, A. M., El-Apasery, M. A., Ibrahim, M. R., & Al-Awadi, N. A. (2012). A facile synthesis of new monoazo disperse dyes derived from 4-hydroxyphenylazopyrazole-5-amines: Evaluation of microwave assisted dyeing behavior. Molecules, 17(12), 13891-13909.
3. Qiu, J., Xiao, J., Tang, B., Ju, B., & Zhang, S. (2019). Facile synthesis of novel disperse azo dyes with aromatic hydroxyl group. Dyes and Pigments, 160, 524-529.
4. Lams, Y. Y., Nkeonye, P. O., Bello, K. A., Yakubu, M. K., & Lawal, A. O. (2014). Synthesis of disperse dyes from pyridone and resorcinol coupled to diazotized 2-amino-4-chloro-5-formylthiazole and application to polyester. Advances in Chemistry, 2014.
5. Fadda, A. A., & Elattar, K. M. (2012). Synthesis of novel azo disperse dyes derived from 4-aminoantipyrine and their applications to polyester fabrics. Am. J. Org. Chem, 2(3), 52-57.
6. Choi, Y. S., Lee, K. S., Kim, H. J., Choi, J. Y., Kang, S. B., Lee, E. J., & Keum, G. (2013). Synthesis, spectral property and dyeing assessment of azo disperse dyes containing carbonyl and dicyanovinyl groups. Bulletin of the Korean Chemical Society, 34(3), 863-867.
7. Maradiya, H. R., & Patel, V. S. (2001). Synthesis and dyeing performance of some novel heterocyclic azo disperse dyes. Journal of the Brazilian Chemical Society, 12(6), 710-714.
8. Racane, L., Stojkovic, R., Tralic-Kulenovic, V., & Karminski-Zamola, G. (2006). Synthesis and antitumor evaluation of novel derivatives of 6-amino-2-phenylbenzothiazoles. Molecules, 11(5), 325-333.
9. Modi, B. R., Vashi, D. M., & Desai, K. R. (1994). Synthesis of 8-triazinylamino coumarin derivatives and their fluorescent properties.
10. Metwally, M. A., Abdel-Latif, E., Amer, F. A., & Kaupp, G. (2004). Synthesis of new 5-thiazolyl azo-disperse dyes for dyeing polyester fabrics. Dyes and Pigments, 60(3), 249-264.
11. Maradiya, H. R. (2002). Monoazo disperse dyes based on 2-amino-1, 3, 4-thiadiazole derivatives. Journal of the Serbian Chemical Society, 67(11), 709-718.
12. Gafer, H. E., & Abdel-Latif, E. (2011). Antimicrobial activity of some new 4-arylazo-3-methylthiophene disperse dyes on polyester fabrics. Journal of Applied Polymer Science, 122(1), 83-89.
13. Gouda, M. A., Gafer, H. E., & Gouda, M. (2012). Synthesis and anti-hypertensive activity of novel sulphadimidine derivatives. Medicinal Chemistry Research, 21(11), 3902-3906.
14. Metwally, M. A., Abdel-Latif, E., Khalil, A. M., Amer, F. A., & Kaupp, G. (2004). New azodisperse dyes with thiazole ring for dyeing polyester fabrics. Dyes and Pigments, 62(2), 181-195.
15. Li, X., Ye, X., Wei, C., Shan, C., Wojtas, L., Wang, Q., & Shi, X. (2020). Diazo Activation

with Diazonium Salts: Synthesis of Indazole and 1, 2, 4-Triazole. Organic Letters.
16. Bello, K. A. (1995). Long wavelength absorbing azo dyes derived from 2-amino-4-chloro-5-formylthiazole as diazo component. Dyes and Pigments, 27(1), 45-54.
17. Maradiya, H. R., & Patel, V. S. (2001). Synthesis and dyeing performance of some novel heterocyclic azo disperse dyes. Journal of the Brazilian Chemical Society, 12(6), 710-714.
18. Dixit, B. C., Patel, H., & Desai, D. J. (2007). Synthesis and application of new mordent and disperse azo dyes based on 2, 4-dihydroxybenzophenone. Journal of the Serbian Chemical Society, 72(2), 119-127.
19. Prakash, S., Somiya, G., Elavarasan, N., Subashini, K., Kanaga, S., Dhandapani, R. & Sujatha, V. (2020). Synthesis and characterization of novel bioactive azo compounds fused with benzothiazole and their versatile biological applications. Journal of Molecular Structure, 129016.
20. Siddiqua, U. H., Irfan, M., Ali, S., Sahar, A., Khalid, M., Mahr, M. S., & Iqbal, J. (2020). Computational and experimental study of heterofunctional azo reactive dyes synthesized for cellulosic fabric. Journal of Molecular Structure, 1221, 128753.
21. Dalaf, A. H., Jumaa, F. H., & Jabbar, S. A. S. (2018). Synthesis and Characterization of some 2, 3-dihydroquinozoline and evaluation of their biological activity. Tikrit Journal of Pure Science, 23(8), 66-67.
22. Abdulla, I. Q. (2014). Synthesis and antimicrobial activity of Ibuprofen derivatives. Natural Science, 2014.
23. Saleh, R. H., Rashid, W. M., Dalaf, A. H., Al-Badrany, K. A., & Mohammed, O. A. (2020). Synthesis of Some New Thiazolidinone Compounds Derived from Schiff Bases Compounds and Evaluation of Their Laser and Biological Efficacy. Ann Trop & Public Health, 23(7): 1012-1031. 2020.
24. Salwa, A. J., Ali, L. H., Adil, H. D., Hossam, S. A. (2020). Synthesis and Characterization of Azetidine and Oxazepine Compounds Using Ethyl-4-((4-Bromo Benzylidene) Amino) Benzoate as Precursor and Evalution of Their Biological Activity. Journal of Education and Scientific Studies, ISSN: 24134732. 16(5): 39-52.
25. Abd, I. Q., Ibrahim, H. I., Jirjes, H. M., & Dalaf, A. H. (2020). Synthesis and Identification of new compounds have Antioxidant activity Beta-carotene, from Natural Auxin Phenyl Acetic Acid. Research Journal of Pharmacy and Technology, 13(1): 40-46.
26. Salih, B. D., Dalaf, A. H., Alheety, M. A., Rashed, W. M., & Abdullah, I. Q. (2020). Biological activity and laser efficacy of new Co (II), Ni (II), Cu (II), Mn (II) and Zn (II) complexes with phthalic anhydride. Materials Today: Proceedings. 1-6

تحضير وتشخيص أصباغ أزو جديدة مبنية على الثايازول وتقييم الفعالية البايولوجية والليزرية ودراسة تطبيق الصباغة لها

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الخلاصة

يهدف هذا العمل إلى تحضير أصباغ ازو المشتتة والمحضرة من مشتق 2-أمينو-4-هيدروكسي ثايازول، والذي يمكن استعمالها كصبغات لأقمشة البوليستر ذات اللون البرتقالي والأحمر وتم الحصول عليها عن طريق تحضير (2-أمينو-4-هيدروكسي- ثايازول)، ومن المركب الأخير تم تحضير المركب (IV وIII) بعملية الديازة، والتي تم استعمالها عن طريق اقتران آخر مع ملح الديازونيوم المحضر (II) للحصول على المركبات (VII, VIII). تم تشخيص ودراسة المركبات المحضرة بواسطة جهاز طيف الأشعة فوق البنفسجية UV spectrophotometer، وجهاز طيف الاشعة تحت الحمراء FT-IR وجهاز طيف الرنين النووي المغناطيسي للبروتون والكاربون ^{1}H-NMR, ^{13}C-NMR. وقد لوحظ ان الأصباغ المحضرة تتغلغل بعمق جيد داخل أقمشة البوليستر باللون الأحمر والبرتقالي على التوالي، ان زيادة الذرات غير المتجانسة والاقتران في بنية الصبغة يؤدي إلى إزاحة حمراء عالية وسطوع الظلال وأيضاً استقرار عالي باللون وزيادة خصائص الثبات. تمت دراسة الفعالية المضادة للبكتيريا ضد أنواع مختلفة من البكتيريا، وهي *Eschershia coli* Gram (-) ve و*Klebsiella Pneumonia*، و*Staphylococcus aureus* و *Staphylococcus epidermidis* Gram (+) ve. إضافة إلى ذلك، تم تقييم الفعالية الليزرية للمركبات (I،III،IV،VII،VIII) التي تم إشعاعها بالليزر لمدة (10، 20، 30) ثانية، وقد لوحظ أن المركبات المحضرة لم تتأثر ولم تتبلمر أو تتحلل عند قياس درجة الانصهار واللون لها.