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Synthesis, Structural Features and Applications of Pyrazole derived Azo Dyes : A Review

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Abstract: Pyrazole azo dyes have received significant attention due to their structural diversity, vibrant coloration, and multifunctional properties. The unique electronic and steric properties of pyrazoles enable its dual functionality as both diazo components and coupling agents, leading to a wide variety of azo derivatives with tailored chromophoric and physicochemical characteristics. Numerous studies reviewed herein highlighted their promising roles in textiles, optoelectronics, sensors, and biomedical fields. The review emphasizes the role of computational chemistry, especially DFT-based approaches, in predicting and validating structural and optical parameters, thereby guiding the rational design of next-generation dyes. Overall, this comprehensive overview underscores the potential of pyrazole azo dyes as versatile compounds for advanced scientific and industrial applications, while encouraging further exploration toward sustainable synthesis and targeted functionality.

Keywords: Diazo Compounds, Computational Chemistry, Cytotoxicity, Solvatochromism, Substituent effect

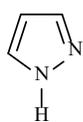
Introduction

Aromatic and heterocyclic azo dyes differ primarily in their molecular structure, which make them highly stable and commonly used in textile dyes¹, 2 printing inks and plastics³. These dyes typically produce bright red, orange, and yellow shades due to their strong conjugation systems⁴. However, their solubility in water is often low, requiring modifications for effective applications. Literature reports revealed

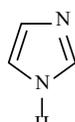
that the water solubility of azo dyes may be increased by the addition of heteroatom, such as nitrogen, oxygen, or sulfur, in the aromatic ring of an azo dyes due to enhanced polarity and hydrogen bonding. The dipole interactions make the dye more hydrophilic compared to purely benzene-based azo dyes and make them more suitable for aqueous applications⁵. Apart from the introduction of heteroatom in the ring, further modifications such as sulfonation or carboxylation may also be done for

optimal performance in industrial and commercial applications⁶⁻⁹.

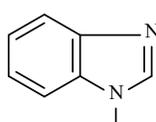
Nitrogen-containing heterocyclic azo dyes are superior to oxygen and sulfur-based azo dyes due to their higher stability, better solubility, stronger color intensity, and broader applications. Their low toxicity also makes them ideal for biomedical and food-related applications¹⁰ while their photostability ensures long-lasting colors in textiles, coatings, and UV-resistant materials¹¹. Their applications have also percolated into various fields such as LCD (liquid crystalline displays), advanced materials, optics, storage devices, polymers and food colors and in organic synthesis¹²⁻¹⁵. It is also reported that diazole (1-3) derived azo dyes exhibit a pronounced redshift compared to simple aromatic systems, making them highly valuable for various industrial, biomedical, and technological applications¹⁶⁻¹⁷.



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Review of literature

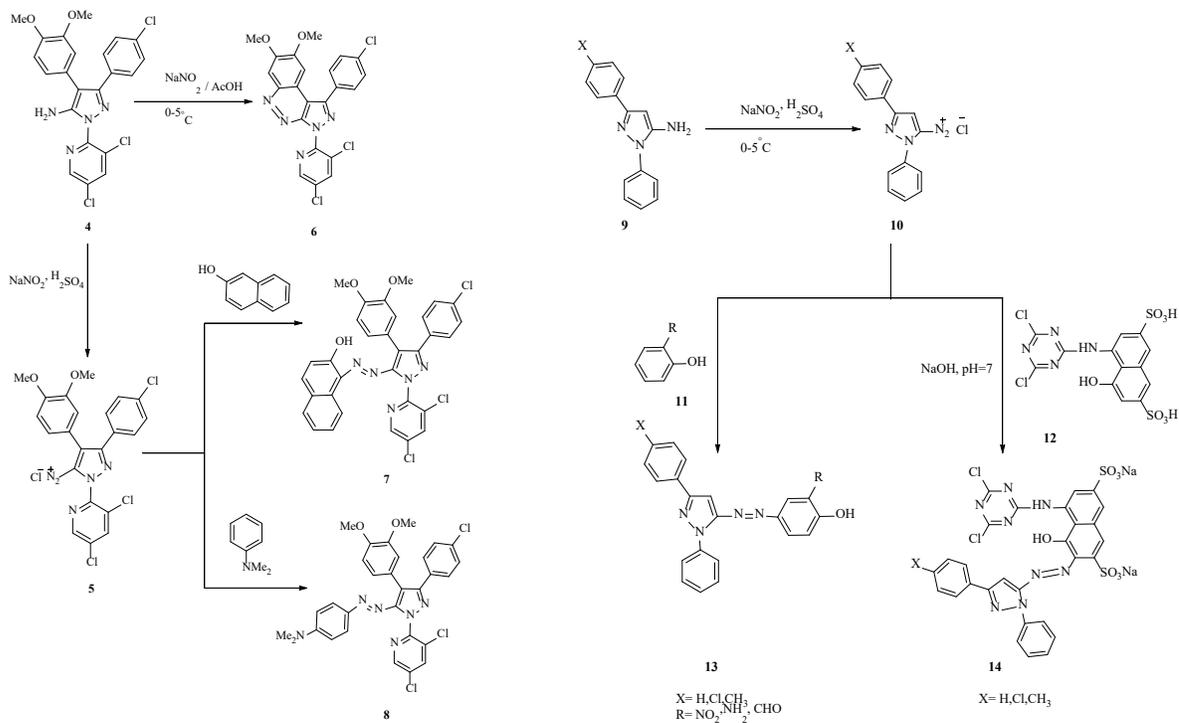
Pyrazole play a crucial role in azo dye synthesis by serving as either diazo components or coupling agents. When used as diazo components (contain free amine (-NH₂) groups), undergo diazotization to form diazonium salts, which can then couple with nucleophilic substrates to produce stable azo dyes. These dyes often exhibit enhanced thermal stability, pH sensitivity, and bioactivity due to the electronic effects of the ring. Alternatively, when pyrazole

act as coupling agents, their active positions or heteroatoms participate in azo coupling reactions, influencing dye properties such as solubility, photostability, and bathochromic shifts. This dual functionality make these azo dyes valuable in various applications, including textiles, biological staining, pH sensors, and pharmaceuticals¹²⁻¹⁷. The present review focuses on literature reports from 2000 onward, highlighting the dual role of pyrazole as both diazo components and coupling agents in azo dye synthesis along with the methodology used. Accordingly, this section is divided into two sub sections:

Use of pyrazole as a coupling component

Pavlov I. V. *et al.* studied diazotization reactions of 5-aminopyrazoles (**4**) by sodium nitrite in acetic acid, leading to pyrazolyl-5-diazonium salts (**5**) as an intermediate. Due to the presence of 3,4-dimethoxyphenyl group at position 4 of amino pyrazole, the reaction does not stopped at diazotization step and immediately underwent intramolecular cyclisation to form 1,3-disubstituted-7,8-dimethoxypyrazolo[3,4-c]cinnolines

(**6**). Use of H₂SO₄ in the reaction instead of AcOH, formed the stable diazonium salt (**5**) which was further coupled with electron rich aromatic compounds i.e. N,N-dimethyl aniline and β-naphthol to form pyrazole substituted azo dyes (**7**, **8**). These dyes exhibited high stability and found effective for coloring natural and synthetic fibres under standard conditions in Scheme 1.¹⁸⁻²¹



Scheme 1. Synthesis of pyrazole substituted azo dyes

H.F. Rizk *et al.* successfully synthesized of a series of 3-substituted-5-arylazopyrazoles (**13**, **14**) using NaNO_2 and H_2SO_4 as a diazotizing agent at low temperature. The diazonium salt (**10**) coupled with two electron donating groups i.e. phenols (**11**) and 4-amino-5-hydroxynaphthalene-2,7-disulphonic acid (**12**).

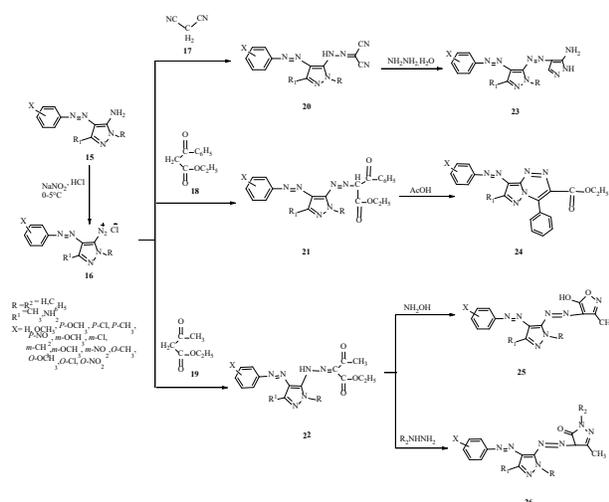
The synthesized dyes were characterized by elemental analysis and spectral data and further investigated for their dyeing characteristics on cotton, wool, polyester, and wool/polyester blend fabrics. The dyed fabrics displayed very good to excellent washing, perspiration, light and sublimation fastness and moderate to excellent dry and wet rubbing fastness in Scheme 2.²²⁻²⁷

Scheme 2. Synthesis of 3-substituted 5-arylazopyrazoles

Karci *et al.* diazotized 5-amino-4-arylaazo-3-methyl-1*H*/1phenyl-pyrazoles (**15**) using NaNO_2 and HCl at low temperature. which was further coupled with active methylene groups i.e. malononitrile (**17**), ethyl benzoylacetate (**18**) and ethyl acetoacetate (**19**) as a coupling agent to synthesize a series of forty-eight bisazo dyes (**23-26**) respectively.

The synthesized dyes were characterized by elemental analysis and spectral methods. Solvent, substituent and acid–base influence on the wavelength of maximum absorption have been studied.²⁸⁻³² The absorption maxima of these dyes were studied in various solvents, and the effects of acidic and alkaline solutions on their absorption spectra was also investigated. The absorption spectra of these bisazo dyes in

methanol were found to be quite sensitive to the addition of base. The dyes exhibited solvatochromic effects, with most dyes showing single tautomeric forms. The synthesized dyes having chloro or methoxy groups as a substituent showed significant antibacterial, antifungal activity in Scheme 3.³⁴⁻³⁷

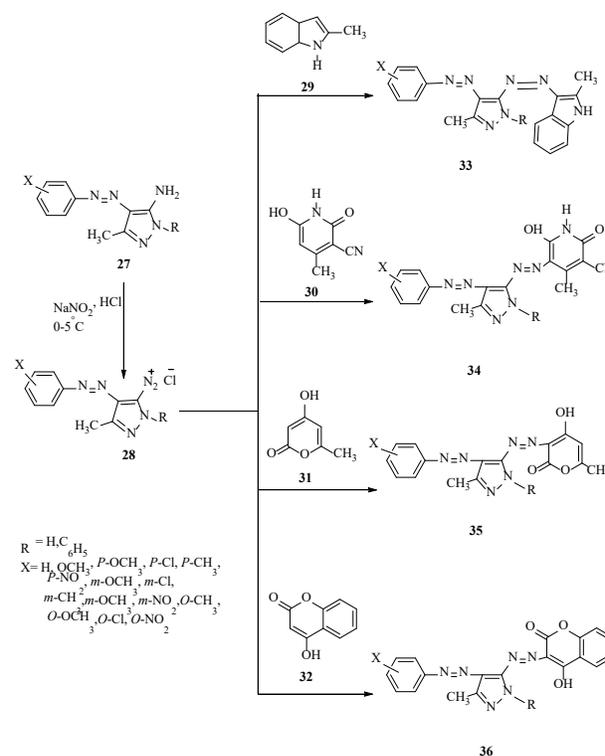


Scheme 3. Synthesis of disperse bisazo dyes

Using the same methodology, Karci also synthesized a series of forty-four disperse bisazo dyes (**33-36**) by the reaction of (**27**) with azo heterocycles (**29, 30**) and benzopyrones (**31, 32**) as a coupling agent. The dyes were characterized using UV-Vis, FT-IR, ¹H NMR, and elemental analysis. The effects of acid and base on the visible absorption spectra in different solvents were also explored. Theoretical vibrational frequencies, along with ¹H NMR chemical shifts calculated using the Gauge-Independent Atomic Orbital (GIAO) method, were compared to experimental data.

DFT calculations provided insights into the electronic and optical properties,

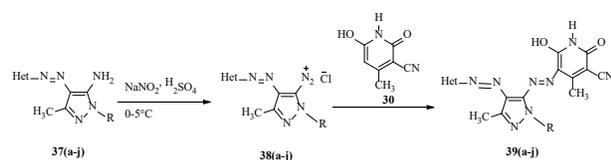
including absorption wavelengths, excitation energy, dipole moments, and charge transfer behavior. HOMO-LUMO energy gaps revealed that dye (**33**) had a smaller gap, suggesting potential charge transfer, and the dipole moments were more responsive to polar molecules. Antimicrobial studies revealed broad-spectrum activity, with these dyes (**34-36**) showed promising antimicrobial potential and displayed selective activity against *S. faecalis*. Most dyes demonstrated fungicidal effects, particularly against *C. utilis*, suggesting potential applications for other pathogenic *Candida* species. The all-synthesized dyes were also applied to polyester and polyamide fibers as disperse dyes and used in biological-medical and complexation studies in Scheme 4.³⁸⁻⁴⁰



Scheme 4. Synthesis of disperse bisazo dyes

Instead of aryl group at position 4 in compound (27), Karci F. *et al.* introduced the azole group (37) and carried out the similar reaction using 3-cyano-6-hydroxy-4-methyl-2-pyridone (30) as a coupling agent. The synthesized ten novel bisazo dyes were characterized by elemental analysis and spectral methods.

The solvatochromic behavior of the dyes in various solvents was evaluated, the effect of substituent, acid and base on the visible absorption maxima was also reported. The introduction of electron-donating groups i.e. methyl and methoxy group into the azole ring resulted bathochromic shifts in all solvents predominantly. Dyes (39a-e) were found as a mixture of tautomeric forms and an anionic form in basic solutions. Furthermore, dyes (39f-j) showed single tautomeric form in all used solvents in Scheme 5.⁴¹⁻⁴³

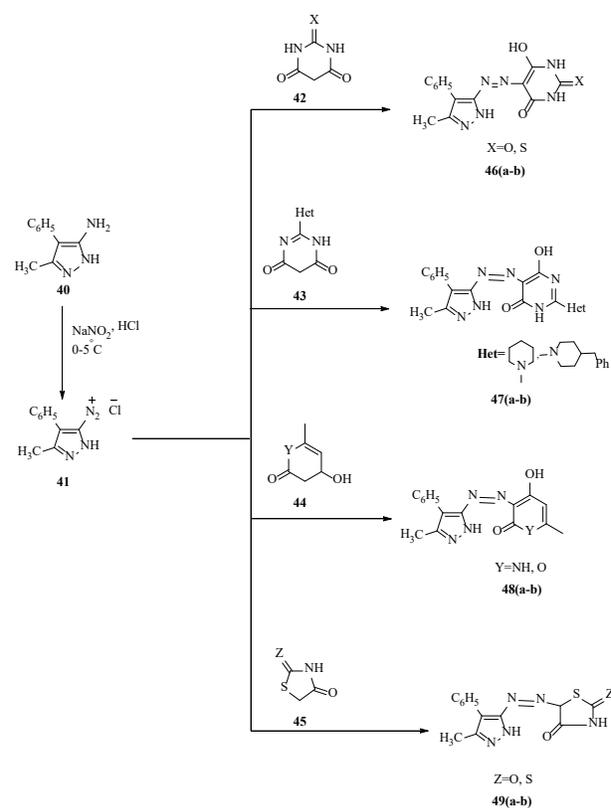


	a	b	c	d	E	F	G	h	i	j
R	H	H	H	H	H	C ₆ H ₅				
Het										

Scheme 5. Synthesis of 5-amino-3-methyl-4-hetarylazo-1h/phenylpyrazoles

The use of active methylene groups i.e. barbituric (42a) and thiobarbituric (42b) acid, 2-hetarylpyrimidine-4,6-dione (43a-b), 4-hydroxy-6-methylpyridin-

2(1*H*)-one (44a), 4-hydroxy-6-methyl-2*H*-pyran-2-one (44b), 1,3-thiazolidine-2,4-dione (45a), 2-thioxo-1,3-thiazolidin-4 one (45b) was also reported by Ledenyova I. V. *et al.* in the diazotization reaction of 3-methyl-4-phenyl-1*H*-pyrazol-5-amine (40) using NaNO₂ and hydrochloric acid. Total eight new pyrazolyl azo derivatives were synthesized and characterized by the spectral and analytical techniques in Scheme 6.⁴⁴⁻⁴⁸

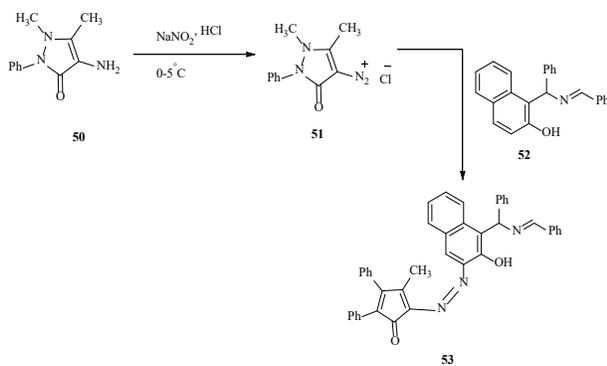


Scheme 6. Synthesis of azo pyrazolyl derivatives

Jathi K. *et al.* synthesized new disperse azo dye (54) through diazotization of 4-amino-1,5-dimethyl-2-phenyl-1*H*-3(2*H*)-one (51) by NaNO₂ and HCl at

0-5°C and further coupling with (*E*)-1-((benzylideneamino)(phenyl)methyl)naphthalen-2-ol (**53**). The synthesized dye was characterized by various physico-chemical techniques. The structure of dye was optimized using DFT with the B3LYP method at a 6-31g basis set. The HOMO-LUMO energy, IR, and UV-Visible spectra were obtained and compared with experimental results⁴⁷⁻⁵².

Cyclic voltammetry on a glassy carbon electrode revealed the dyes exhibited irreversible redox behavior, involving a two-step reduction to form hydrazo and primary amines. The overall electrode process involved a $4e^-/4H^+$ transfer, was diffusion-controlled, and the reduction peak potential decreased with increasing sulfuric acid concentration in DMF in Scheme 7.



Scheme 7. Synthesis of 2-((*Z*)-(4-(((*E*)-benzylideneamino)(phenyl)methyl)-3-hydroxynaphthalen-2-yl)diazenyl)-3-methyl-4,5-dihencyclopenta-2,4-dienone

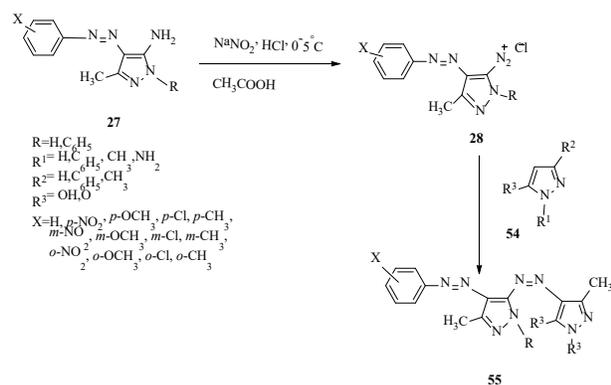
Use of pyrazole as a coupling component

Karci F. *et al.* described the synthesis

and spectroscopic properties of a series of thirty-five novel disperse bisazo dyes and four 3-methyl-1*H*/phenyl-pyrazolin-5-one (**55**) including pyrazole rings. In this synthesis, 5-amino-4-aryazo-3-methyl-1*H*/phenyl-pyrazoles (**27**) were diazotized using NaNO_2 and HCl at 0-5°C coupled with 3-amino-5-hydroxy-1*H*/phenylpyrazole (**54**) to give disperse and bisazo dyes (**55**) respectively.

The newly synthesized disperse bisazo dyes were characterized by elemental analysis and spectral methods. The solvatochromic behavior of these disperse bisazo dyes in various solvents showed bathochromic shifts in most polar solvents, such as DMSO and DMF. Substituent, acid and base effects on the visible absorption spectra of the disperse bisazo dyes substituted with electron-withdrawing and electron-donating groups at their *o*-, *m*-, *p*-position were also reported. It was observed that the absorption spectra of these dyes in methanol were quite sensitive to the addition of base. The vibration frequencies, optimized molecular structure and corresponding vibration assignments of dyes (**55**) have been calculated using DFT method at 6-31G+(d,p) basis set level. The calculated shielding constants, vibration frequencies and visible absorption values were found to be in good agreement with experiment values for these compounds. Antimicrobial studies revealed broad-spectrum activity, with these dyes (**55**) showing promising antimicrobial potential and displaying selective activity against *S. faecalis*. Karabacak C. *et al.* reported⁵³⁻⁵⁸ that the combination of two pyrazole heterocyclic rings reduced the pharmacological effect and hence they were not found to be suitable for

further modification to produce more efficacious antibacterial compounds. Most dyes demonstrated fungicidal effects, particularly against *C. utilis*, suggesting potential applications for other pathogenic *Candida* species. The dyes were also applied to polyester and polyamide fibres as disperse dyes and used in biological-medical and complexation studies⁵⁹⁻⁶³ in Scheme 8.

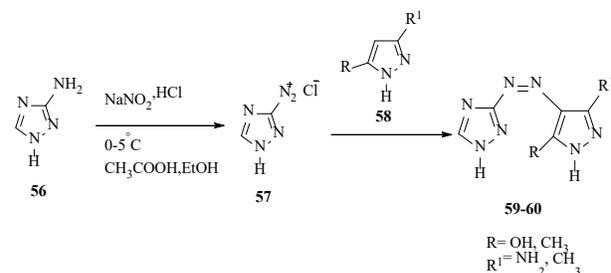


Scheme 8. Synthesis of disperse bisazo dyes

This group also synthesized⁶⁴⁻⁶⁶ two triazole-based azo dyes (**59-60**) by diazotization of 3-amino-1H-1,2,4-triazole (**56**) using NaNO_2 and HCl at $0-5^\circ\text{C}$, the diazonium salt (**57**) was coupled with substituted pyrazole (**58**). The synthesized azo compounds were characterized by ^1H NMR, FT-IR, UV-Vis, and mass spectrometry. DFT/B3LYP calculations with a 6-311G(d,p) basis set were used to optimize the molecular structures and calculate vibrational frequencies, chemical shifts, electronic transitions, and molecular electrostatic potentials (MEP).

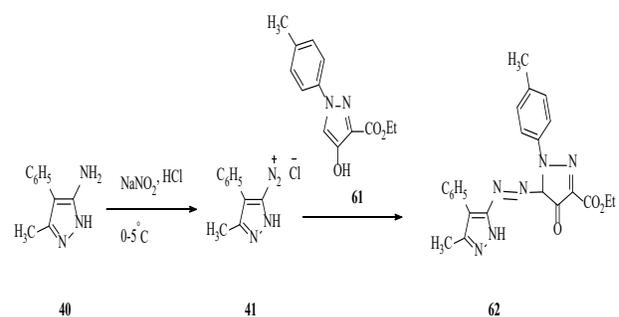
The experimental data showed good agreement with the theoretical results. The antibacterial activity of the compounds was tested against four bacterial strains,

with compound (**60**) showing good activity against *Staphylococcus aureus* in Scheme 9.



Scheme 9. Synthesis of triazole based azo dyes

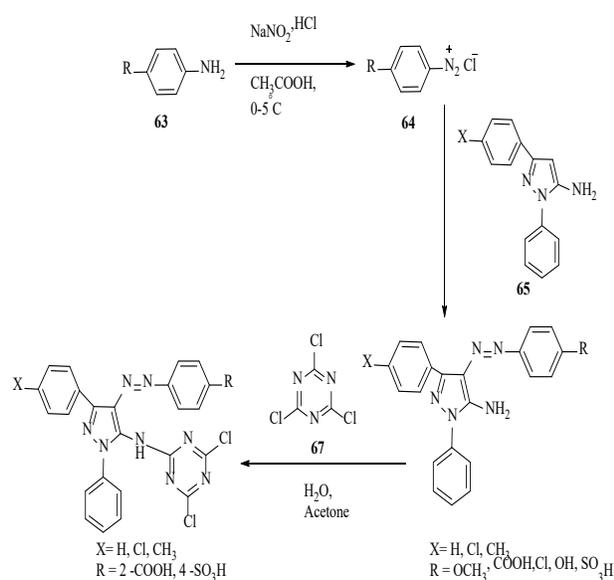
Ledenyova I. V. *et al.* reported⁴⁴ the diazotization of 3-methyl-4-phenyl-1H-pyrazol-5-amine (**40**) in hydrochloric to afford the corresponding diazonium salt (**41**). The latter underwent azo coupling with an ethyl 4-hydroxy-1-(p-tolyl)-1H-pyrazole-3-carboxylate (**61**) to yield new pyrazolyl azo derivatives (**62**). The synthesized compounds were characterized by spectral and analytical techniques in Scheme 10.



Scheme 10. Synthesis of pyrazolyl azo derivatives

H.F. Rizk *et al.* successfully synthesized²¹⁻²³ a series of sixteen new 3-substituted-5-amino-4-arylazopyrazoles (**66**) characterized by elemental analysis and spectral data. The synthesized azo dyes

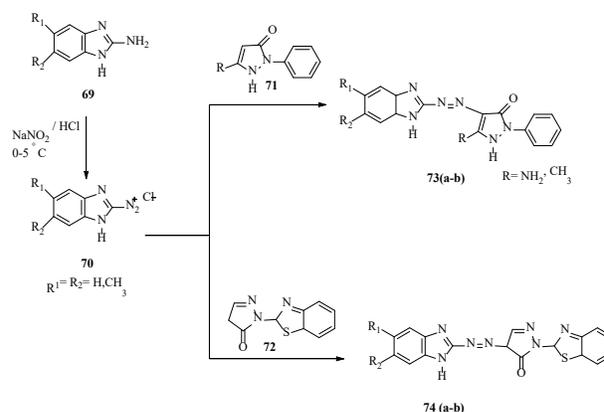
(66) were further reacted with cyanuric chloride (67) to afford six new reactive azo dyes (68). All the synthesized azo dyes were investigated for their dyeing characteristics on cotton, wool, polyester, and wool/polyester blend fabrics. The dyed fabrics displayed very good to excellent washing, perspiration, and sublimation fastness, moderate to excellent dry and wet rubbing fastness, and light fastness properties rated as very good to excellent. Investigation of antimicrobial and antifungal activity of the synthesized azo dye (68) exhibited the strong activity against all strains of tested organisms in Scheme 11.



Scheme 11. Synthesis of disperse azo dyes

As reported earlier, pyrazole derivatives (71-72) were also used as a coupling agent when 2-amino-1*H*-benzimidazole (69) was used as a diazo component. The reaction proceeded with the same methodology⁶⁷⁻⁶⁸ at low temperature. The synthesized four new azo dyes were

characterized by spectral techniques and investigated for biomedical application in Scheme 12.



Scheme 12. Synthesis of pyrazole azo derivatives and hetarylazopyrazolone Conclusion

The present review comprehensively summarizes the synthesis, structural variations, and multifaceted applications of diazole-based azo dyes, particularly those containing pyrazole, imidazole, and benzimidazole heterocycles. These dyes stand out due to their enhanced physicochemical properties such as high thermal and photostability, better aqueous solubility, strong chromophoric characteristics, and structural diversity. The ability of diazole rings to act as both diazo components and coupling agents significantly expands the range of accessible azo compounds, offering opportunities for tailored functionality.

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