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SYNTHESIS AND CHARACTERIZATION OF DYES DERIVED FROM 1-AMINOANTHRAQUINONE

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Abstract

This study investigated the synthesis and characterization of three anthraquinone-based dyes derived from 1-aminoanthraquinone through diazotization and coupling reactions using H-acid, J-acid, and γ-acid as coupling agents. Diazotization was carried out by reacting 1-aminoanthraquinone (0.89 g, 0.004 mol) with sodium nitrite (0.28 g, 0.004 mol) in hydrochloric acid at 0–5°C to form a diazonium salt, which was subsequently coupled with H-acid, J-acid, and γ-acid to produce Dye A, Dye B, and Dye C, respectively. The synthesized dyes were characterized using UV–Visible spectroscopy, Fourier Transform Infrared (FTIR) spectroscopy, melting point determination, and solubility tests. Dye A and Dye C exhibited orange coloration, while Dye B was maroon. Melting points ranged from 279.4°C to 321.2°C, indicating high thermal stability. Percentage yields were 99.98%, 87.02%, and 97.29% for Dyes A, B, and C, respectively. UV–Visible analysis revealed absorption maxima at 474, 477, and 466 nm for Dyes A, B, and C, respectively, corresponding to π→π* electronic transitions. The FTIR spectra confirmed the presence of O–H and N–H stretching vibrations in Dyes A and C, strong C=N and O–H (carboxylic) bands in Dye B and aromatic C=C and C=O stretches across all samples, verifying successful coupling and anthraquinone framework integrity. Solubility tests in methanol, distilled water, propanol and hydrochloric acid revealed high solubility in polar solvents and limited solubility in less polar or acidic media, consistent with the presence of hydroxyl and sulfonic groups. As such, these results demonstrate that 1-aminoanthraquinone is a suitable precursor for synthesizing thermally stable, vividly coloured, and thermally stable and vividly coloured dyes with potential applications in textile, polymer and optoelectronic industries.

Keywords: 1-Aminoanthraquinone, Synthesis, Dyes, Characterization, Colourants

1. INTRODUCTION

Anthraquinone derivatives represent one of the most important classes of organic colourants widely used in the textile, leather, and printing industries (Routoula and Patwardhan, 2020). Their structural framework, built around the anthracene-

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9,10-dione nucleus, provides a stable conjugated system that supports intense colouration and excellent resistance to light, heat and chemical degradation (Gupta and Sekar, 2024). Over the years, anthraquinone-based dyes have gained attention for their brilliant shades, ranging from violet to deep blue, and for their ability to maintain chromatic strength under diverse dyeing conditions. These properties have made them valuable alternatives in applications where long-term colour fastness and oxidative stability are important, such as in high-quality fabrics and polymeric materials (Choudhary *et al.*, 2025).

When compared to azo and triphenylmethane dyes, anthraquinone derivatives exhibit superior photostability, better resistance to washing and bleaching and minimal tendency to fade under UV exposure (Riaz *et al.,* 2023). While azo dyes are more common due to their low production cost and wide colour range, they often suffer from poor resistance to sunlight and may degrade to release toxic aromatic amines (Zafar *et al.,* 2022). Triphenylmethane dyes, on the other hand, are known for their bright hues but lack thermal and chemical stability (Liu *et al.*, 2021). As such, anthraquinone dyes offer intense and durable colours while maintaining excellent stability, which makes them highly suitable for advanced dyeing processes and valuable as intermediates in the manufacture of pigments. (Penthala *et al.,* 2019; Gupta and Sekar, 2024).

Recent researches have focused on the modification of 1-aminoanthraquinone to develop new dyes with enhanced colour intensity, solubility and ecological safety. Studies by Takeda *et al.* (2021), Masilamani *et al.* (2022), Zhou *et al.* (2023), Wang *et al.* (2018) and Li *et al.* (2017) reported various synthetic routes for producing aminoanthraquinone-based dyes and highlighted their strong bathochromic shifts in UV–Vis spectra, indicating extended conjugation in their chromophoric systems. These researches suggest that functional group substitutions on the aminoanthraquinone core can fine-tune optical and dyeing properties. Therefore, this study aims to synthesize and characterize novel dyes derived from 1-aminoanthraquinone using H-acid, J-acid and γ-acid as coupling agents. The study focuses on evaluating their physical properties, UV-Visible absorption

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characteristics, FTIR functional groups and solubility behaviour for potential textile and industrial applications.

2. MATERIALS AND METHODS

2.1 Reagents

All reagents used were of analytical grade and were used without further purification, these include; 1-aminoanthraquinone (purchased from Sigma-Aldrich), Sulfuric acid (H_2SO_4) (purchased from Merck), Sodium nitrite ($NaNO_2$) (obtained from BDH Chemicals), Hydrochloric acid (HCl) (37% purity), Sodium acetate (99% purity), Ethanol (C_2H_5OH) (99.9% purity), Acetone (C_3H_6O), Dimethyl sulfoxide (DMSO) and Distilled water.

2.2 Equipment

The equipment used included a magnetic stirrer (IKA C-MAG HS7) for uniform mixing of reaction mixtures, a UV–Visible spectrophotometer (Agilent Cary 60 UV-Vis) for measuring absorbance and determining λmax of the dyes, and a Fourier-transform infrared spectrometer (PerkinElmer Spectrum 2) for identifying functional groups in the synthesized compounds. A melting point apparatus (Stuart SMP30) was used to determine purity and thermal stability, while an analytical balance (Mettler Toledo XS205) ensured precise weighing of samples. The oven (Memmert UF110) was used for drying samples, and the autoclave (HIRAYAMA HV-85) for sterilizing glassware. A water bath (Grant SUB Aqua Pro) provided controlled heating during synthesis. Other essential glassware and materials such as Petri dishes, glass rods, beakers, conical flasks, and filter paper were used for handling, mixing and filtration throughout the experiment.

2.3 Synthesis of the Dye (Diazotization and Coupling Reaction)

The synthesis of dyes derived from 1-aminoanthraquinone was carried out through diazotization and coupling reactions. In the diazotization step, 1-aminoanthraquinone (0.89g, 0.004mol) was suspended in 60mL of distilled water, and 15mL of concentrated hydrochloric acid was added dropwise with constant stirring. The mixture was heated to 70°C until a clear solution was obtained, then cooled to 0–5°C in an ice bath (As shown in **Fig. 1**). A precooled solution of sodium nitrite (0.28g, 0.004 mol) in 10mL of water was added gradually over 10 minutes while maintaining the same temperature, and stirring was continued for 1 hour to complete diazotization. The resulting

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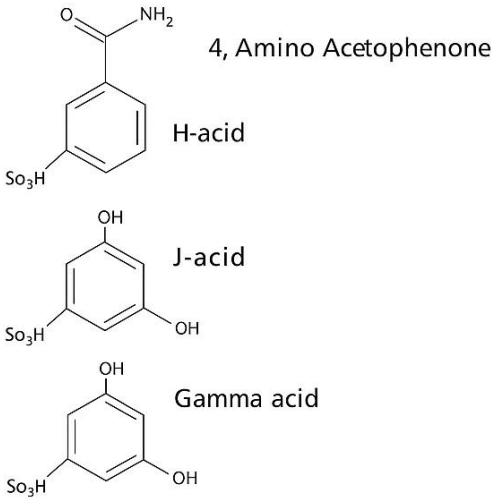
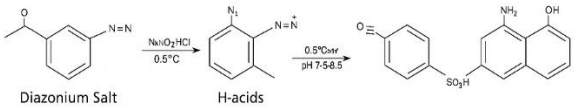
diazonium salt solution was kept at 0–5°C for subsequent coupling. For the coupling reaction, three separate aqueous solutions were prepared; H-acid (1-amino-8-naphthol-3,6-disulfonic acid, 1.28g, 0.004mol), J-acid (5-amino-2-naphthol-7-sulfonic acid, 0.96g, 0.004mol), and γ-acid (2-amino-8-naphthol-6-sulfonic acid, 0.96g, 0.004mol). Each served as a coupling component providing hydroxyl, amino and sulfonic groups essential for solubility and colour development. The freshly prepared diazonium solution (0.54 g, 0.004 mol) was added dropwise over 15 minutes to each of the three coupling mixtures while maintaining the pH between 7.5 and 8.5 using sodium hydroxide (0.16 g, 0.004 mol). The resulting dye products were labeled as Dye A (H-acid dye), Dye B (J-acid dye) and Dye C (γ-acid dye) for identification and subsequent analytical characterization.

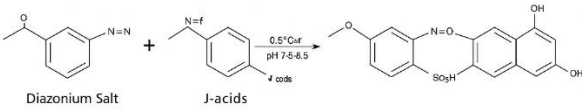
2.4 Experimental Reactions of the Synthesized Dye



Scheme 1: Diazotation Reaction of 4, Amino Acetophenone



Scheme 2: Coupling reaction with H-Acids



Scheme 3: Coupling reaction with Gamma Acids

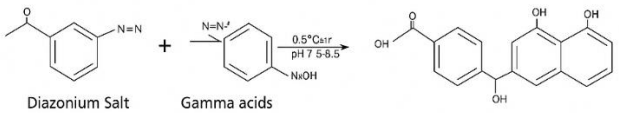


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Fig. 1: Experimental Process

2.5 Characterization of the Synthesized Dye

2.5.1 Melting Point, Decomposition Temperature and Colour Assessment

Using a Stuart SMP30 melting point apparatus, 0.5g of the synthesized dye was placed in a capillary tube and heated gradually. The melting point range was recorded when the dye transitioned from solid to liquid, and the decomposition temperature was noted when the sample began to darken or char (Pishgar *et al.*, 2022). For the colour assessment, the powdered sample of the dye was placed in a white ceramic dish under standard lighting conditions and compared to a colour reference chart; any deviation from expected anthraquinone-derived dye shade was noted.

2.5.2 UV–Visible Spectroscopic Characterization

The method of Perkampus, (2013) was employed in which the dye solution (10mg dissolved in 10 mL ethanol) was analysed using a UV–Visible spectrophotometer (Agilent Cary 60) over the wavelength range 200–800 nm. The maximum absorbance wavelength (λ_{max}) was recorded and the molar extinction coefficient (ε) calculated using Beer–Lambert's law ($A = \varepsilon \cdot \ell \cdot c$).

2.5.3 Fourier-Transform Infrared (FTIR) Spectroscopic Analysis

FTIR spectra were obtained on a PerkinElmer Spectrum 2 instrument using the method of Rotich *et al.* (2022). The synthesized dyes were mixed with KBr, pressed into a pellet, and measured in the range 4000–400 cm^{-1}. Key absorption bands such as $–NH_2$, C=O, aromatic C=C were identified and compared to reference compounds to confirm functional groups present.

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2.5.4 Solubility Test

Solubility was evaluated at room temperature by adding 0.1g of the synthesized dyes to 10mL of various solvents of differing polarity which includes methanol, distilled water, propanol and hydrochloric acid (HCl). The test judged whether the dye dissolved completely, partially or stayed insoluble. Solubility profile helps determine reaction and application compatibility, solvent choice for processing, and hints at polarity of the molecule.

3. RESULTS AND DISCUSSION

3.1 Physical Properties of the Synthesized Dyes

Table I presents the physical characteristics of the synthesized dyes (A–C) derived from 1-aminoanthraquinone coupled with different acids. The dyes exhibited distinct colours where orange was noted for Dye A and C, and maroon for Dye B. This indicates that the electronic effects of the coupling components (H-, J-, and γ-acids) where influenced by their chromophoric behaviour. Melting points ranged from 279.4°C to 321.2°C, reflecting high thermal stability, which is typical of anthraquinone-based dyes due to extensive conjugation and intermolecular hydrogen bonding (Ajayi and Olatunji, 2016). Dye A recorded the highest melting point (321.2°C), suggesting stronger crystal packing and resonance stabilization compared to Dye B and C. Yields percent were also impressive, with Dye A (99.98 %), Dye C (97.29 %) and Dye B (87.02 %), This high yield shows efficient diazotization and coupling reactions during the synthesis which aligns with the findings of Obi *et al.* (2022).

Table I: Physical Properties of the Synthesized Dye

Dyes	Colour	Melting Point (°C)	Yield (%)	Molecular Weight ($g \cdot mol^{-1}$)
Dye A	Orange	321.2	99.98	553
Dye B	Maroon	279.4	87.02	505
Dye C	Orange	298.2	97.29	505

Keys; Dye A = H-acid, Dye Dye B = J-acid Dye, Dye C = γ-acid dye

3.2 UV–Visible Spectral Characteristics

The UV–Visible spectral results presented in **Table II** and illustrated in **Fig. 2, 3** and **4** showed that absorption maxima (λ_{max})

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between 466 nm and 477 nm, which fall within the visible region and correspond to $\pi \rightarrow \pi^*$ transitions in the anthraquinone chromophore. Dye B exhibited the longest λ_{max} (477 nm), indicating the most extensive conjugation or electron-donating substituent effect from J-acid. Dye C, though slightly blue-shifted (466 nm), recorded the highest absorbance (0.239), implying strong chromophoric intensity and efficient electronic delocalization and this was found to be in relation to the study of Obi *et al.* (2022).

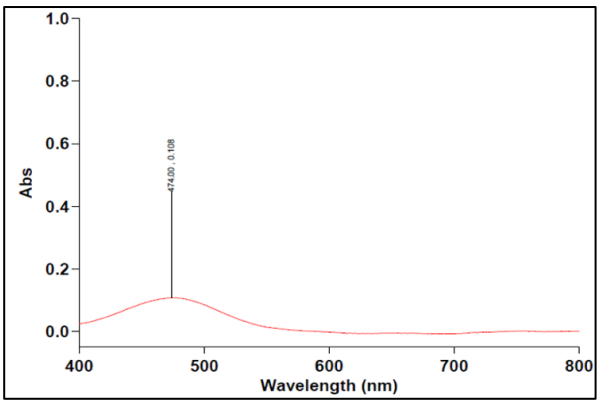

Fig. 2: UV-VIS spectra for Dye A

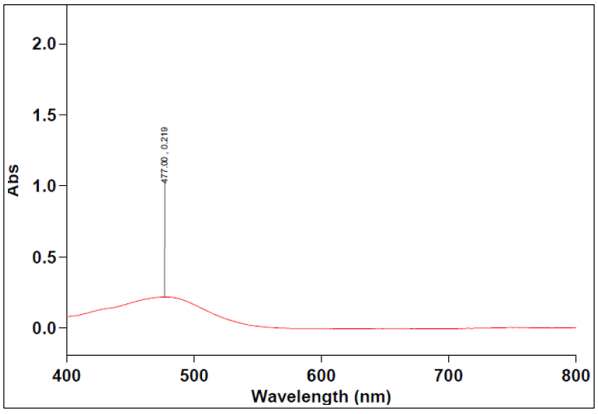

Fig. 3: UV-VIS spectra for Dye B

Table II: UV-VIS Spectroscopy of the Synthesized Dye

Dye	Wavelength (λmax, nm)	Absorbance (A)	Molar Extinction Coefficient (ε, L·mol⁻¹·cm⁻¹)
Dye A	474	0.209	2.45×10^1
Dye B	477	0.219	9.06×10^1
Dye C	466	0.239	7.47×10^1

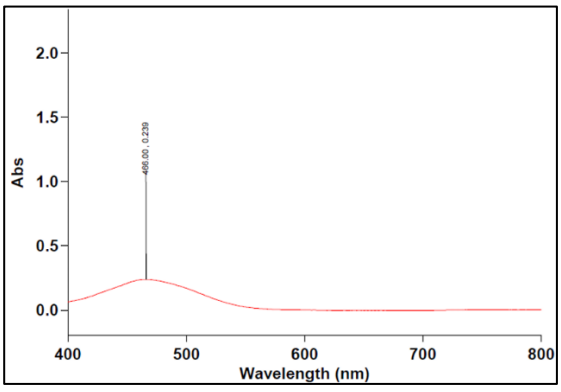

Fig. 4: UV-VIS spectra for Dye C

3.3 FTIR Spectral Analysis

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On **Table III**, the Fourier Transform Infrared (FTIR) spectra of the synthesized dyes confirmed the incorporation of key functional groups expected in anthraquinone-based azo dyes. Broad absorption bands observed between 3410–3496 cm^{-1} indicated O–H stretching vibrations of hydrogen-bonded hydroxyl groups, most prominent in Dyes A and C, reflecting the influence of H-acid and γ-acid coupling components. N–H stretching vibrations between 2877–3302 cm^{-1} verified amine functionalities, with Dye A showing a strong band at 2877 cm^{-1} (amine salt) and Dye C a medium band at 3302 cm^{-1} (primary amine). Distinct N–O and C=N bands at 1500–1659 cm^{-1} confirmed the presence of nitro and imine linkages, signifying successful diazotization and coupling, while additional C=C and C≡C absorptions (1614–2106 cm^{-1}) revealed aromatic and alkyne conjugations responsible for color intensity. Dye B exhibited strong C=N and O–H (carboxylic) peaks at 1659 cm^{-1} and 2643 cm^{-1}, indicating imine formation and acidic substitution from J-acid. Thus, the functional groups confirm the anthraquinone framework of the synthesized dyes and show that each coupling acid uniquely influences their structure, functionality, colour properties and general stability (Shahab *et al.*, 2017; Celik *et al.*, 2022).

Table III: FTIR Absorption Bands (cm^{-1}) of Synthesized Dyes

Bands Causing Absorption	Vibrational Assignment	Dye A	Dye B	Dye C
O–H stretching (Alcohol / Phenol)	Hydrogen-bonded O–H vibration	3410, 3496 (strong, broad)	–	3414, 3060 (strong / weak)
N–H stretching (Amine)	Primary / secondary amine vibration	2877 (strong, broad)	–	3302 (medium)
C–H stretching (Alkane / Aromatic)	Symmetric and asymmetric C–H stretch	3041 (medium)	3060, 3298 (medium – strong)	2929 (medium)

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C=O / CO–O–CO stretch	Carbonyl / Anhydride vibration	1044 (strong, broad)	–	–
C=N stretching (Imine / Oxime)	Azomethine linkage formation	–	1659 (strong)	–
C=C / C≡C stretching	Aromatic and alkyne conjugation	2106 (weak)	1908 (medium)	1614, 1919 (medium)
C–N stretching (Aromatic / Aliphatic Amine)	C–N bond formation	1103 (medium)	1279, 1454 (strong / medium)	–
N–O stretching (Nitro Compound)	Symmetric / asymmetric N–O stretch	1502 (strong)	1528 (strong)	1517 (strong)
O–H stretching (Carboxylic Acid)	Free O–H vibration	–	2643 (strong)	2620 (strong)

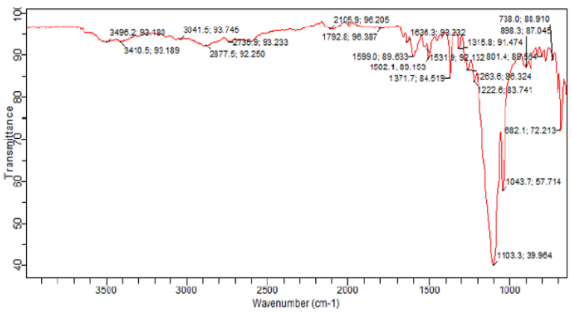

Fig. 5: FTIR Spectrum for Dye A

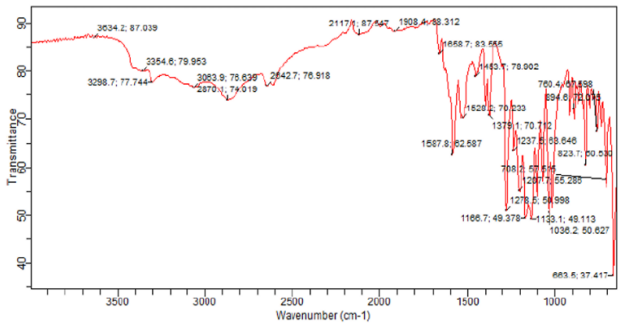

Fig. 6: FTIR Spectrum for Dye B

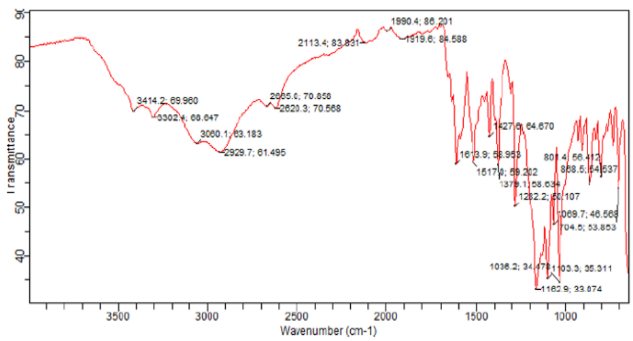

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Fig. 7: FTIR Spectrum for Dye C

3.4 Solubility Behaviour

Table IV presented the solubility test of the synthesized dyes which showed that all dyes were soluble in methanol and distilled water, indicating the presence of polar substituents such as hydroxyl and sulfonic acid groups. Dye C displayed the highest solubility across all solvents, signifying that γ-acid enhances dye polarity and molecular symmetry. The dyes were only sparingly soluble in HCl, due to protonation of amino groups which reduced their solvation capacity, and Dye A was insoluble in propanol likely due to its highly polar nature. Therefore, this findings is consistent with Myek *et al.,* (2021), who reported that azo dyes bearing multiple hydroxyl or sulfonic groups dissolve readily in protic solvents (water, methanol) but poorly in non-polar or acidic media

Table IV: Solubility Test for the Synthesized Dyes

Dyes	Methanol	Distilled Water	Propanol	HCl
Dye A	S	S	IS	SS
Dye B	SS	S	S	SS
Dye C	S	S	S	SS

Keys; S = Soluble SS = Sparingly Soluble IS = Insoluble

4. CONCLUSION

The successful synthesis and characterization of three novel anthraquinone-based dyes derived from 1-aminoanthraquinone and different coupling components (H-acid, J-acid, and γ-acid) was achieved, demonstrating excellent yield, thermal stability and distinct chromatic properties. The variations in colour and spectral features among Dyes A–C reflected the influence of substituent type and position on the electronic distribution within the anthraquinone framework. The UV–Visible spectra confirmed strong π→π* transitions in the visible region, while FTIR analysis verified the presence of functional groups characteristic of well-defined anthraquinone chromophores, including hydroxyl, amine, carbonyl, and azomethine linkages. The high melting points observed signify structural rigidity and extensive conjugation, further supporting the thermal and chemical stability of these dyes. Moreover, the solubility patterns across different solvents indicated that the incorporation of sulfonic and hydroxyl substituents enhanced dye polarity, rendering them suitable for aqueous and ethanol-based dyeing systems. Collectively, this study shows that 1-aminoanthraquinone remains an excellent precursor for developing thermally stable, intensely coloured, and environmentally safer dye molecules. The promising properties of these synthesized dyes suggest potential applications in textile coloration, polymer finishing and optoelectronic materials, marking a significant

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contribution toward sustainable and high-performance dye molecules.

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