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Research Article

Synthesis and Characterization of 1-Phenyl Azo 2-Naphthol: A Comprehensive study

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ABSTRACT

1-Phenylazo-2-naphthol compound was synthesised and shows yield 13.46gm. Synthesis occur by diazotisation reaction. In Characteristic study various parameters are measure like UV spectra, TLC, IR . The synthesized compound has been tested invitro against various bacteria's in order to assess their antibacterial activity using pour plate method.

INTRODUCTION

The compound was synthesized via a diazotisation reaction, which is commonly used to create azo compounds. This typically involves the reaction of aromatic amine with nitrous acid. Azo compounds having molecular formula Ar-N=N-Ar are prepared with interaction of diazonium salt with a phenol in presence of NaOH. Phenyl azo 2-naphthol prepared by using azo dyes by diazotization reaction.[1] Azo compounds are formed by the functional group –N=N- using two symmetrical and asymmetrical identical alkyl or aryl radicals.[2] The azo group consists of a nitrogen-nitrogen double bond (N=N). This group is typically found in the general formula of azo dyes: R-N=N-R', where R and R' are organic groups. The azo group has high electron density due to the π-electron system of the N=N bond. This electron-rich system can interact with light, which is crucial for the absorption of visible wavelengths.[3] Many azo dyes exhibit antimicrobial properties against various strains of bacteria. This is an interesting aspect of azo dyes that extends beyond their role in coloration. azo dyes with specific substituents, such as nitro, amino, or hydroxyl groups, often exhibit antimicrobial activity.

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a. **Methyl Red**: Known for its antibacterial activity against certain Gram-positive bacteria.
b. **Congo Red**: Shows antimicrobial activity against some Gram-negative bacteria.
c. **Phenylazo Compounds**: These often exhibit varying degrees of antimicrobial activity depending on their substituents and structure. [4,5]

Recrystallization is indeed a vital technique for purification of solid organic compounds. The process you described involves several key steps to achieve purification:

1. **Dissolution at High Temperature**: The first step is to dissolve the impure solid in a suitable solvent at an elevated temperature. The solvent should dissolve a significant amount of the compound at high temperatures but allow the compound to crystallize upon cooling. This ensures that the compound can be separated from any impurities that are either insoluble or remain in solution when the temperature is reduced.
2. **Hot Filtration**: After the compound is dissolved, the solution is typically filtered while still hot to remove insoluble impurities, such as dust or solid particles that don't dissolve in the solvent. This step ensures that only the dissolved compound and impurities which are soluble, remain in the solution.
3. **Crystallization**: Upon cooling, the solution becomes supersaturated, and the solute (the compound of interest) begins to crystallize. The crystallized compound is often purer than the original solid because the impurities which remain dissolved in the solvent, which is then removed.
4. **Isolation of Crystals**: After crystallization, the pure compound is typically isolated by filtration (vacuum filtration is commonly used) to separate the crystals from the solvent and any remaining impurities.
5. **Drying**: Finally, the purified crystals are dried.

This process is highly effective for compounds that have a significant difference in solubility between the desired compound and the impurities, allowing for a high level of purity in the final product.[7]

UV Spectra: The UV-Vis absorption spectrum of 1-phenylazo-2-naphthol typically shows strong absorption due to its azo and naphthol chromophores, with absorption maxima generally observed in the range of 300-450 nm.

The specific UV-Vis characteristics can vary depending on the solvent and concentration; however, the presence of the azo group often causes significant shifts in the absorption peaks compared to similar compounds lacking this group. Ultraviolet absorption spectra arise from the transition of electrons within a molecule or anion from a lower to a higher electronic energy level. Ultraviolet emission spectra result from the reverse transition, where electrons return from a higher energy level to a lower one, emitting radiation in the process. For radiation to cause electronic excitation, it must be within the UV region of the electromagnetic spectrum. A UV spectrophotometer is an instrument designed to measure these spectra.[6]

The Rf (Retention factor) value in Thin Layer Chromatography (TLC) is defined as the ratio of the distance traveled by the center of a spot (the analyte) to the distance traveled by the solvent front. It is expressed as:

$$Rf = \frac{\text{Distance traveled by the solvent front}}{\text{Distance traveled by the compound}}$$

This ratio gives a dimensionless number between 0 and 1. The closer the Rf value is to 1, the further the compound has traveled, which typically indicates that it is more soluble in the mobile phase (less interaction with the stationary phase). Conversely, a lower Rf value indicates that the compound interacts more strongly with the stationary phase and is less soluble in the mobile phase.[8]

MATERIAL AND METHOD:

Reagents and Materials:

1. **Aniline** (for phenyl group)
2. **2-Naphthol**
3. **Nitrous acid** (generated in situ from sodium nitrite and hydrochloric acid)
4. **Sodium hydroxide** (for adjusting pH)
5. **Solvents** (e.g., ethanol, water)
6. **For UV Spectrophotometer:** Ethanol
7. **For Thin layer chromatography:** stationary phase: Silica Gel, solvent system: Hexane: Ethyl acetate (6:4)
8. **For antibacterial activity:** Methyl Red staining reagent, Agar medium and gram positive and gram negative bacteria.
9. **IR Spectra:** Potassium Bromide.

A. Materials

All chemicals and solvents were obtained from Company Ltd (AR-Grade). Melting point recorded by melting point apparatus, UV spectra was recorded on double beam shimadzu uv spectrophotometer. The antibacterial activity of synthesized compound was analyzed in Pharmaceutical microbiology lab of Pravara Rural Education Society's, Institute of Pharmacy college, Loni.

B. Procedure for synthesis:

i. Diazotization:

1. Dissolve 10 ml of aniline in 16 ml HCl acid to form a solution of the aniline hydrochloride.
2. Add 16 gm of sodium nitrite to the solution under cold conditions (0-5^{0}C) to form the diazonium salt.
3. Ensure the reaction mixture remains cold to prevent decomposition of the diazonium salt.

ii. Coupling Reaction:

4. Prepare a solution of 16 gm of 2-naphthol in 180 ml of 10% NaOH solution in 250 ml beaker and cool the solution to 10^0 C to form the sodium salt of 2-naphthol.
5. Add the diazonium salt solution to the 2-naphthol solution while maintaining the temperature between 0-5^{0}C to facilitate the coupling reaction.
6. The 1-Phenylazo-2-naphthol will precipitate out.

iii. Isolation and Purification:

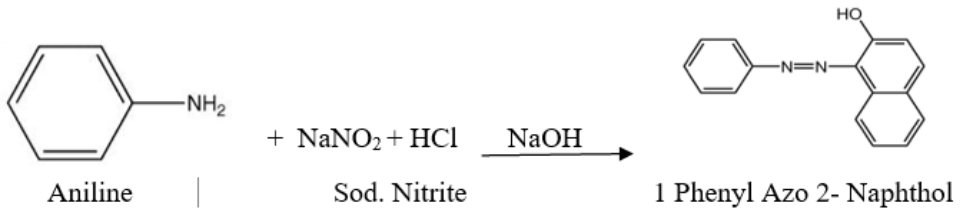
7. Filter the precipitated product.
8. Wash with cold water to remove impurities.
9. Recrystallize half product from an ethanol and half from glacial acetic acid solvent to obtain pure 1-Phenylazo-2-naphthol.

Table 1. Solvent used for Recrystalisation

Sr. No.	Solvent	Boiling Point
1.	Ethanol	78^0
2.	glacial acetic acid	117.9^0

Reaction:

Reaction of 1 Phenyl Azo 2 Naphthol



RESULT AND DISCUSSION:

1. Theoretical Yield: 27.43gm
2. Practical Yield: a) by recrystallise with Ethanol= 13.46gm

b) by recrystallise with Glacial acetic acid solution= 9.27gm

3. Solubility: Benzene.

4. Rf Value: a) Distance traveled by solvent: 7.4cm

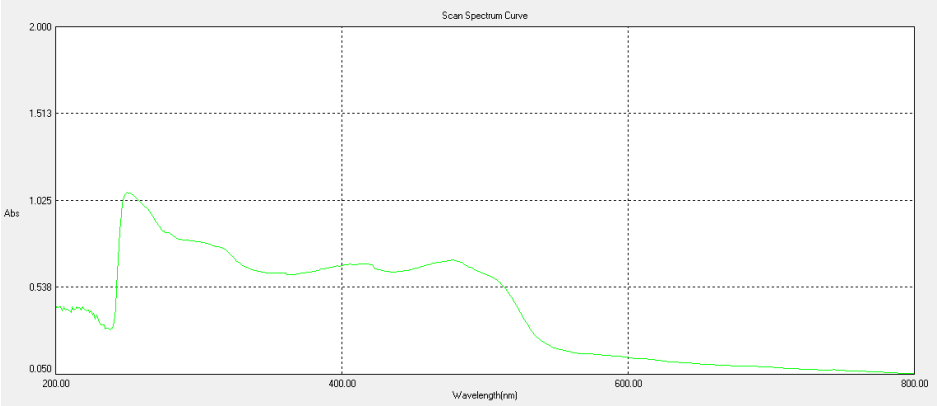
b) Distance traveled by compound: 5.6cm

c) Rf value= Distance traveled by compound/ Distance traveled by solvent = 5.6/7.4= 0.75

Table 2. Comparative study:

Sr. No.	Parameter	With Ethanol	With Glacial acetic acid
1.	Practical yield	13.46gm	9.27gm
2.	LOD	35%	46%
3.	Rf Value	0.75	0.77

4. **UV Spectra:** UV Solutions:- 0.01gm substance in 100 ml ethanol =100 ppm from which to prepare 5ppm solution & 10 ppm solution.



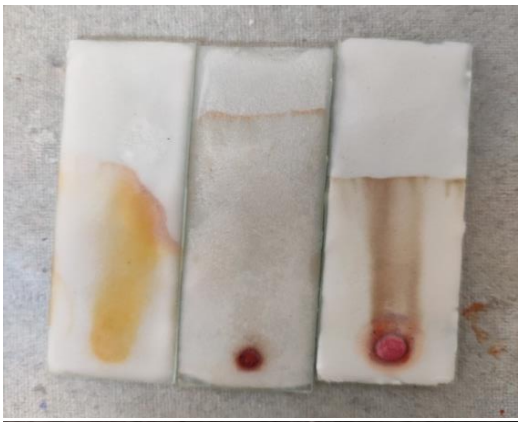
λ max found at 250nm for phenyl azo-2 naphthol

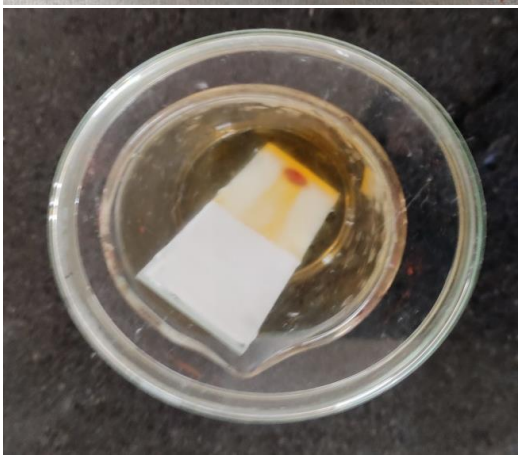
5. IR Spectra: IR absorption bands are determined by using a **KBr disk.** The sample was prepared by mixing the compound with potassium bromide (KBr), which is transparent to infrared radiation.

Azo Group (–N=N–) Stretching: appears in the range of **1200-1400 cm⁻¹.**

O-H Stretching: appears in the range of **3600-3800 cm⁻¹.**

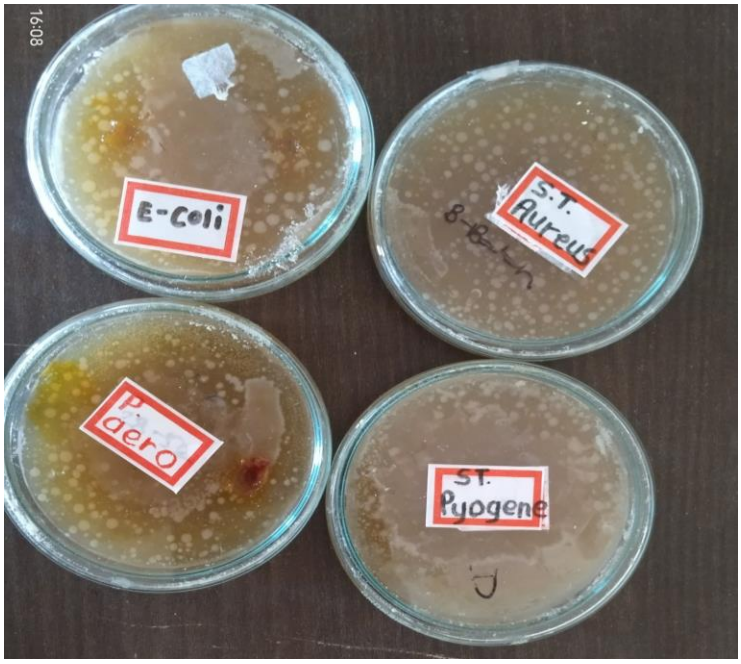
Aromatic C=C Stretching: appears in the range of **1500-1700 cm⁻¹**





6. Result of Antibacterial Activity:

Sr. No.	Bacteria	Name of drug	Concentration	Zone of Inhibition
1.	*E.coli* (G.positive)	1-Phenyl azo 2 Naphthol	50mg/ml	0.4
2.	*S.aureus* (G.positive)	1-Phenyl azo 2 Naphthol	50mg/ml	0.6
3.	*S.pyogenes* (G.positive)	1-Phenyl azo 2 Naphthol	50mg/ml	0.4
4.	*P. aeruginosa* (G.negative)	1-Phenyl azo 2 Naphthol	50mg/ml	0.7



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