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SYNTHESIS OF GLYCINE BASED AZO DYE AND ITS SPECTRAL CHARACTERIZATION

Savita Sharma

Research Scholar , Department of Applied Sciences ,
Singhania University, Rajasthan (India)

ABSTRACT

A new amino acid based azo dye- N,N dimethyl azoglycine has been synthesized. Elemental analysis FT-IR, ^{1}HNMR spectral studies has been shown in this paper.

Keywords* : *Azo dye, Auxochromes , Conjugation, FT-IR, ^{1}HNMR.

I. INTRODUCTION

Azo dyes are the largest class of synthetic dyes. In azo dyes, chromophore is an aromaric system joined to azo group and common auxochromes are NH^2, NH^2OH. Method of preparation of azo dye is direct coupling between a diazonium salt and phenol or amine. Product will absorb longer wavelength (visible region) due to extended conjugation. So, azo compounds are brightly coloured due to extended conjugation. Azo compounds have wide range of colour, brightness good dying performance. Azo dyes are used in dyeing textiles, leather, paper, food and cosmetic products. They have medicinal performance like 1,2,4 Triazoles are known as drugs for treatment of cancer. They are also used as acid base indicators like yellow orange, aniline yellow etc. Glycine based dye can also be used in formation of lanthanide complexes which have magnetic and optical properties. Some azo dyes like DEET (N,N- Dimethyl meta toluamide) used as insect repellant. The amino acid based dyes are much more photostable compared to fluroscein and exhibit constant pH independent fluroscence that is advantageous in biological application.[3] Amino acid based dyes are widely used for complex synthesis due to presence of electron rich N atom in amino acid also enhances many applications of lanthanide and transition metal complex compound.[1] Amino acid based dyes are widely used for ligand labeling due to its fluroscent nature to investigate receptor- binding properties.[2] Amino acid based dyes are also used for flouroscence study, photochemistry and photobiology.[4]

A new dye has been synthesized , named – N,N –Dimethyl azoglycine composed of glycine ($HOOC\text{-}CH_2\text{-}NH_2$) as basic moiety and 1,2–dimethyl aniline ($C_6H_5N(CH_3)_2$). Spectrochemical studies of glycine based azo dye was done.

II.EXPERIMENTAL

2.1 CHEMICALS AND MEASUREMENT

Elemental analysis was performed with Perkin Elemer2400(SAIF Chandigarh, Punjab University). FT-IR spectra were recorded with KBr pellets in 4000 to 400/cm on Nicoletet Protease 460 Spectrograph EFT (IIT Delhi).

^{1}HNMR spectra were recorded with DMSO- d6 medium on DPX-dix 300 MHz Bruker Avance Spectrometer using TMS as an internal reference from 0 to 9 δ ppm (IIT Delhi).

Glycine (99%), sodium nitrite (99%) and N,N dimethyl aniline(99%) were purchased from E. Merck, India. The solvents were of AR or spectroscopic grade and the chemicals were used as received.

2.2 PREPERATION OF AZO DYE

Synthesis of azo dye was consisting of two processes:

2.2.1 DIAZOTIZATION

2.2.2 COUPLING

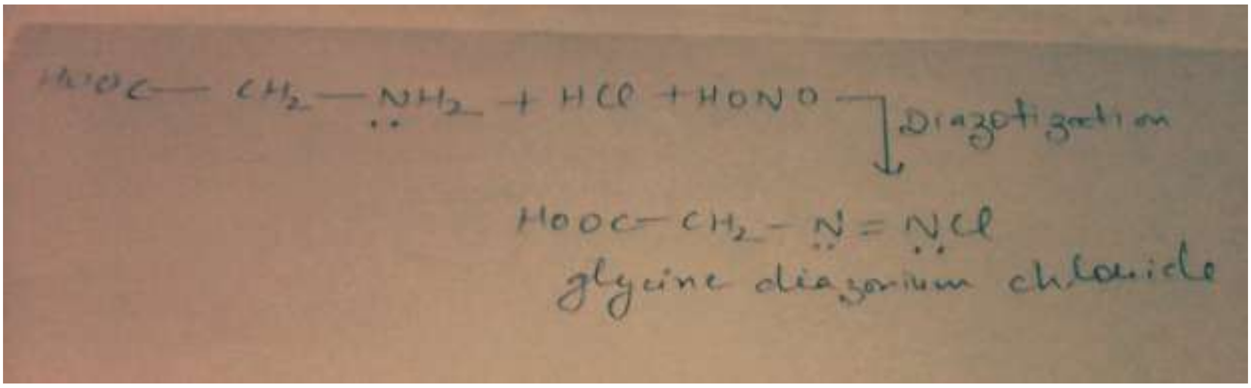

FIGURE 1. : Diazotization

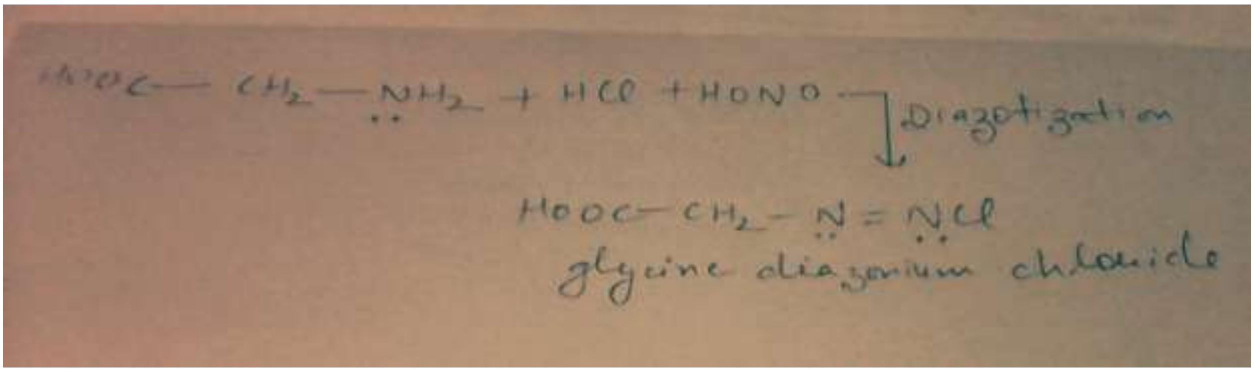

FIGURE 2. : Coupling

SCHEME : Synthesis of n,n – dimethyl azoglycine

2.2.1 DIAZOTIZATION:

6.0 g of glycine (HOOOC- CH_2-NH_2) was dissolved with 90 ml Millipore water and warmed till a complete dissolution. Now, new solution of sodium nitrite ($NaNO_2$) 6.0 g with 30 ml H_2O was added in the above reaction mixture and then allowed to cool in ice bath where temp. was maintained between 0^0 and 5^0 C. Cold diluted solution of HCl (24 ml) was added dropwise in reaction mixture (in ice bath) with continous stirring to a complete diazotization process.

2.2.2 COUPLING

Again a cold solution of N,N Dimethyl aniline ($C_6H_5(CH_3)_2$) 12 ml in dilute HCl was added to the above prepared reaction mixture and shaked thoroughly for few minutes followed by addition of 20% NaOH (90 ml). Finally, the resultant reaction mixture was heated to 60^0C with addition of24 NaCl and kept in ice bath (0^0 -5^0 C) for 3-4 hours to obtain precipitate. A dark brown colour ppt. was filtered and gently washed several times with cold water till it was free completely of all impurities. Then the ppt. was dried at room temp. and then stored in vaccum dessicator with P_2O_5.

III. RESULT AND DISCUSSION

3.1 FT-IR: FT-IR spectrum reveals type of functional group present. It involves change in vibrational quantum no. (v) with change in rotational quantum no.

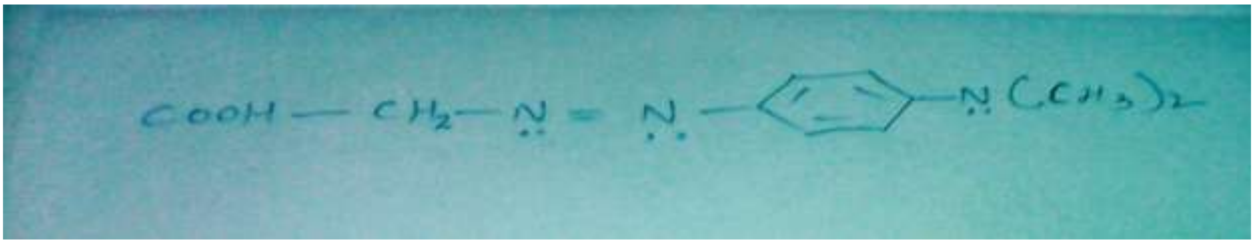

FIGURE 3. : Structure of N,N – dimethyl azoglycine

TABLE 1: Stretching frequencies (cm^{-1}) of functional group of N,N – dimethyl azoglycine

Compound	OH	C=O	N=N	C-H	Aromatic group
N,N-dimethyl azoglycine	3400	1601.95	1518.30	763.91	1518.30,1443.77,1365.29,1311.03

3.1.1 FT-IR SPECTRA OF GLYCINE BASED AZO DYE :

Absorpion band at 3400 /cm is due to OH sretching vibraton of carboxylic group of glycine. Strong and sharp absorption band at 1601.95 and 1518.30 is due to overlap of vc=0 vN=N stretching mode of carboxylic group and azo group. Multiple and dominant peaks at 1518.30 to 1311.03 /cm supported the presence of carboxylic group and azo group. Absorption peak at 763.91 indicates C-H out of plane bending vibration of disubstituted benezene ring.

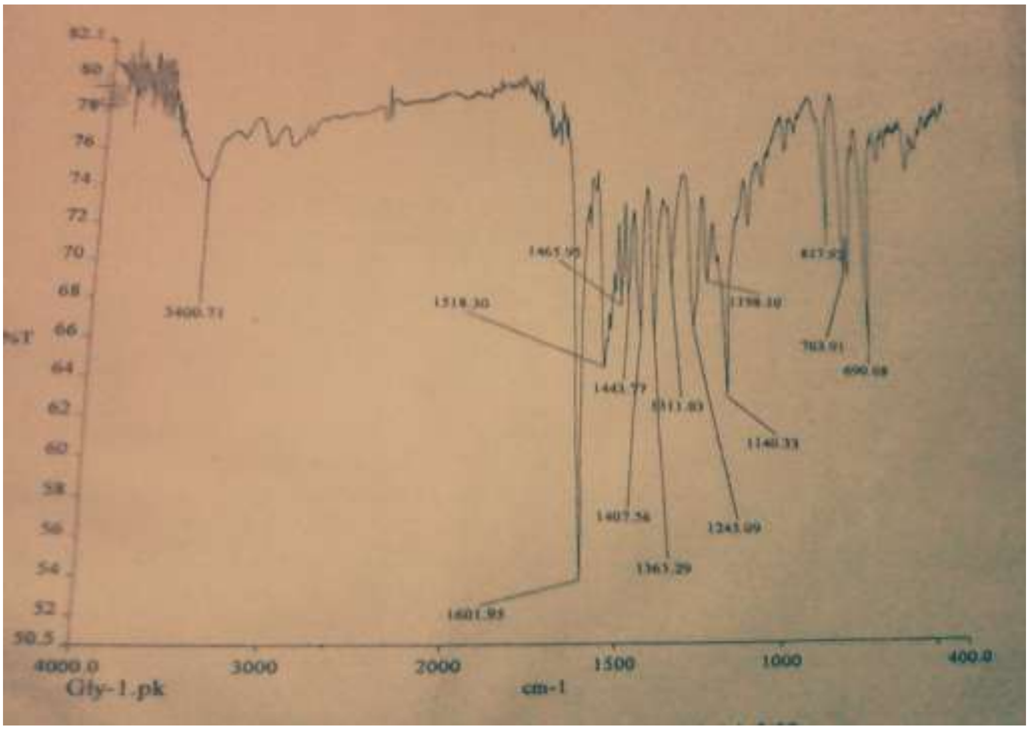

FIGURE 4. : FT- IR Spectra of azo dye

3.2 ^{1}HNMR: It involves change in direction of nuclear spin quantum no. (I) in presence of magnetic field.

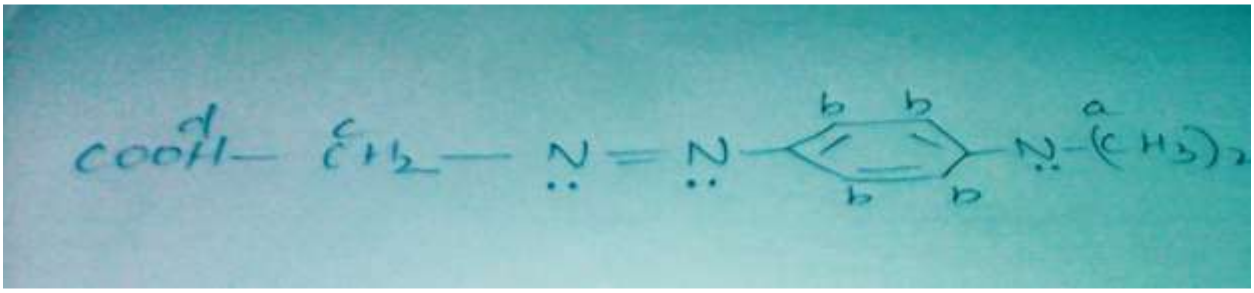

FIGURE 5. : Structure of N,N – dimethyl azoglycine

Table 2: Proton magnetic resonance data(chemical shift) of N,N-Dimethyl azoglycine

Compound	Ha	Hb	Hc	Hd
N,N-Dimethyl azoglycine	2.507	7.030 to 7.141	7.225,7.251,7.359	3.03 to 3.689

3.2.2 ^{1}HNMR SPECTRA OF GLYCINE BASED AZO DYE :

^{1}HNMR of azo dye shows resonating proton signals in four sets of signals between δ 0-10 ppm. First upfield signal appears as singlet at 2.507 ppm for Ha protons of methyl group. Second multiplet resonate in upfield between 3.03 to 3.689 ppm assigned to Hd of COOH. Third signal is a downfield doublet with signal between 7.030 to 7.141 for Hb of aromatic ring of glycine based azo dye. Fourth downfield at δ 7.225 ppm, δ 7.251 ppm and 7.359 ppm appeared due to Hc of CH_2group.

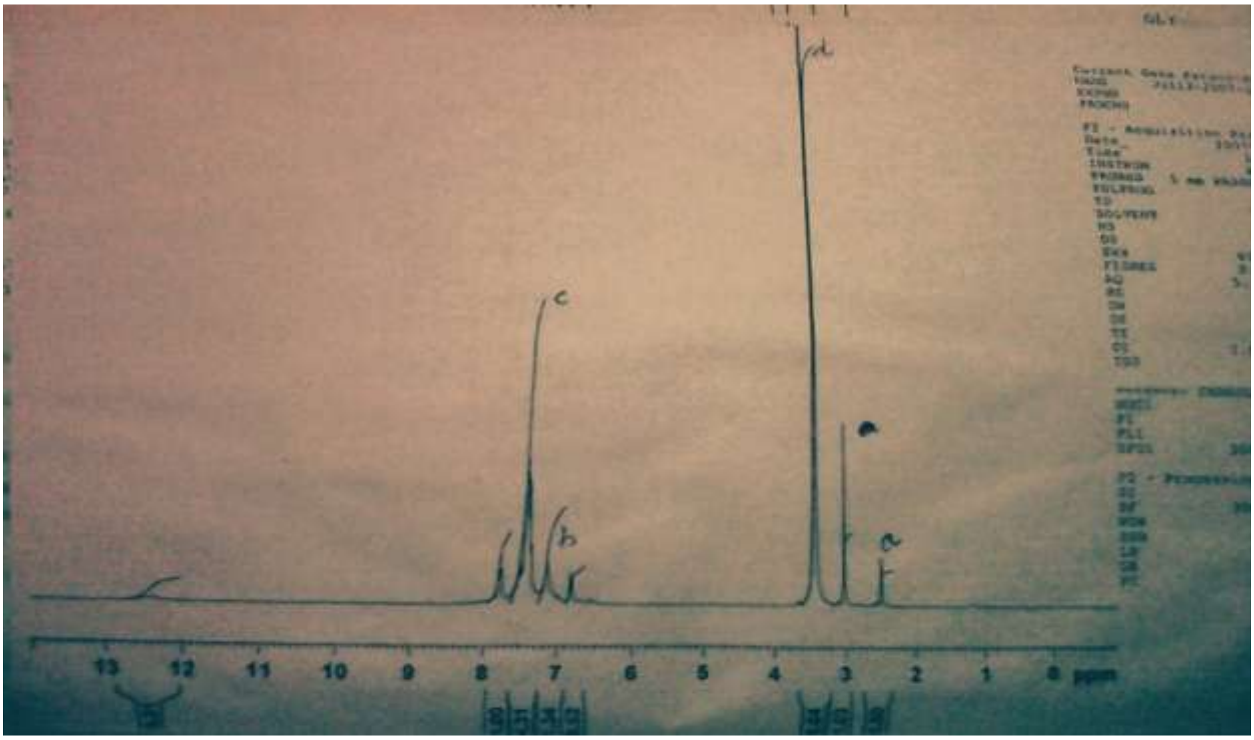

FIGURE 6. : ^{1}HNMR Spectra of azo dye

IV.CONCLUSION

A new glycine based azo dye was synthesized and characterized successfully. FT-IR spectra of glycine at 3400cm^{-1} indicated the presence of carboxylic group. Multiple and dominant peak at 1518.30 to 1311.03 cm^{-1} suppored the presence of aromatic group in dye. Prominent multiplet resonate in upfield between 3.03 to 3.689 ppm assigned to Hd of COOH group of glycine based azo dye. The molecular structure of dye has been well established with techniques as FT-IR and ^{1}HNMR described in this paper.

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