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SYNTHESIS OF SOME NEW AZO COMPOUNDS DERIVED FROM 2-HYDROXY ACETOPHENONE AND AROMATIC AMINES

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ABSTRACT
Azo compounds have very wide applications in dye industry which contain (-N=N-) group. In the present study some azo compounds have been synthesized from 2-hydroxy acetophenone and aromatic amines like p-nitro aniline, Suplhanilic acid, metanilic acid and 1-naphthyl amine. Each azo compound ethanolic solution was interacted with metal solutions to observed color change. The entire azo compounds were confirmed by melting point and FT-IR spectroscopy.

KEYWORDS: Aromatic amines, 2-hydroxy acetophenone, FT-IR spectra.

INTRODUCTION

The azo dyes constitute an important class for dyeing fibres and a high rate of growth.[1] In addition, azo compounds have shown promise in drug delivery and electronic industry.[2] The azo compounds are usually coloured compounds which can be yellow, red, orange, depending on the structural properties of the molecule. As a result this, azo compounds are very importance as pigments for a long time.[3] The use of heterocyclic intermediates in the synthesis of some azo dyes are well established, and the resultant dyes exhibit good strength and brightness.[4]

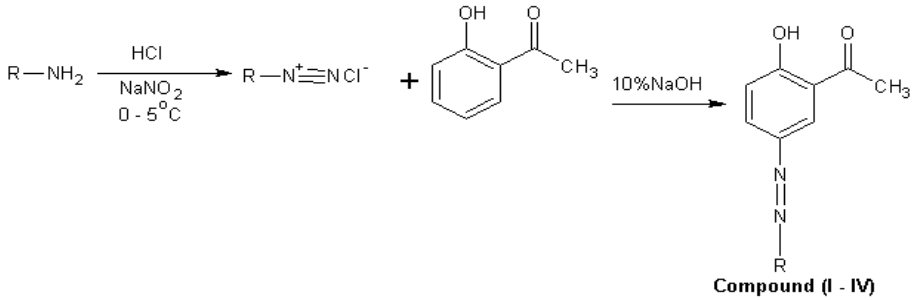
The intermediate for synthesis of azo compounds are diazonium salts, which are very unstable but aromatic diazonium salts are relatively stable because of high electron density of benzene ring.[5] Azo compounds have variety of biological applications like antidiabetics, antiseptics, anti-inflammatory and chemotherapeutic agents.[6-10]

In this study some azo compounds were synthesized by coupling of 2-hydroxy acetophenone and some aryl diazonium salts prepared from p-nitro aniline, Suplhanilic acid, metanilic acid and 1-naphthyl amine and colour change was observed by adding aqueous ethanolic solution of synthesized azo compound in the solution of some metal salts.

METHODS AND MATERIALS

Synthesis of 2-hydroxy-5-phenyazo acetophenone(I-IV).

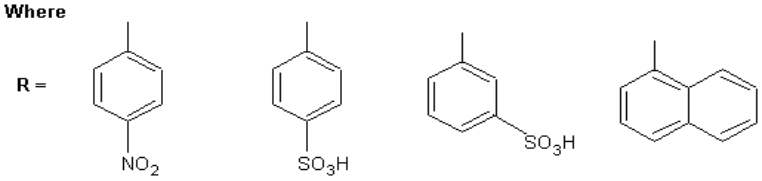
The aryl diazonium chloride was prepared by dissolving pure p-nitro aniline, Suplhanilic acid, metanilic acid and 1-naphthyl amine separately (5mmol) with concentrated HCl (5ml) and water (5ml) and allow to cool at 5^{o}C. in ice bath. $NaNO_2$ (5mmol) was dissolved in water(10ml) at 5^{o}C.The above two solutions were mixed with constant stirring. This mixture was added to the solution of 2-hydroxyacetophenone (5mmol) which was dissolved in 10% NaOH solution at 5^{o}C.The mixture were kept in ice bath with constant stirring for about 10min.The product precipitate was filtered and re-crystallized from glacial acetic acid.



Compound (I - IV)

Where

R =



(I) (II) (III) (IV)

Colour interactions of azo compound with metal salts solution.

The synthesized azo compound (I-IV) (0.1gm each) was dissolved separately in de-ionized water and ethanol in the ratio of 3:2 in boiling tests tube. Then for observation six tests tubes were prepared by dissolving 0.1gm metal salt in 5ml de-ionized water and labeled them as $CuSO_4$,$CrPO_4$, $CoCl_2$, $FeCl_3$, $CdCl_2$ and $NiCl_2$.Then about four drops of synthesized azo compound solution was added to the test tubes containing metals salts solution. The colour change was noted and shown in table no-1.

RESULT AND DISCUSSION

The melting points of azo compounds I- IV were sharp. The entire azo compounds were by FT-IR spectra.

2-hydroxy-5-(4-nitrophenyl) azo acetophenone (I)

IR (KBr) cm^{-1}: The dye shows the absorption peak due to azo group, -N=N- stretching vibration at 1519.91cm^{-1}. Aromatic C-H stretching vibration bands appeared at 3068.75 and 2929.87 cm^{-1}**.** Aromatic C-H bending vibration bands appeared at 837,780 and 670 cm^{-1}. -OH stretching vibration bands appeared in the region of 3350cm^{-1}**.** -C=O stretching vibration bands appeared in the region of 1645.28cm^{-1}.**m.p.-164°C, Percent yield-88.57%.**

2-hydroxy-5-(4-sulphonylphenyl) azo acetophenone (II)

IR (KBr) cm^{-1}: The dye shown the absorption peak due to azo group, -N=N- stretching vibration at 1575cm^{-1}. The S=O stretching vibration bands appeared in the region of 1122.67 cm^{-1}**.** Aromatic C-H bending vibration bands appeared in the region of 825-855 cm^{-1}. The -OH stretching vibration bands appeared in the region of 3454cm^{-1}**.** The -C=O stretching vibration bands appeared in the region of 1643.35 cm^{-1}. **m.p.-210°C, Percent yield-47.35%.**

2-hydroxy-5-(3-sulphonylphenyl) azo acetophenone (III)

IR (KBr) cm^{-1}: The dye shown the absorption peak due to azo group, -N=N- stretching vibration at 1575.84cm^{-1}. The S=O stretching vibration bands appeared in the region of 1055 cm^{-1}**.** Aromatic C-H bending vibration bands appeared in the region of 710-900 cm^{-1}. -OH stretching vibration bands appeared in the region of 3450cm^{-1}**.** -C=O stretching vibration bands appeared in the region of 1643 cm^{-1}. The –C-N and –C-O stretching vibration bands appeared at 1481 and 1209 cm^{-1} respectively. **m.p.-108°C, Percent yield-74.77%.**

2-hydroxy-5-(1-naphthyl) azo acetophenone (IV)

IR (KBr) cm^{-1}: The dye shows the absorption peak due to azo group, -N=N- stretching vibration at 1490.97cm^{-1}. Aromatic C-H stretching vibration bands appeared at 3053.32cm^{-1}**.** Aromatic C-H bending vibration bands appeared at 636-966 cm^{-1}. -OH stretching vibration bands appeared in the region of 3381.21cm^{-1}**.** -C=O stretching vibration bands appeared in the region of 1635.64cm^{-1}. The absorption signal at 1373.32 cm^{-1} may be due to presence of –C-N stretching vibration.

m.p.-214°C, Percent yield-60%.

The all synthesized azo compounds display the absorption signal in the region of 1635.64 to 1645.28 cm-1 due to the –C=O stretching vibration. This decrease in the frequency from 1715 cm-1 to 1635.64 may be attributed due to presence of conjugation in these compounds.

The colour interactions of all synthesized azo compounds(I-IV) was observed that all the compounds shown colour changes except 2-hydroxy-5-(1-naphthyl) azo acetophenone (IV).

CONCLUSION

The synthesis of azo dyes from 2-hydroxy acetophenone and aryl diazonium salt were undertaken. The selected

method for the synthesis of azo compounds is simple and makes it possible to prepareazo compounds in high yield. On the basis of performance of the synthesized compounds in IR, Solubility, melting points etc can replace these dyes by other dyes. The colour interactions also conclude that these dyes can be acts as pigments and may be useful for dying cotton, wool and fabrics.

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