RESEARCH ARTICLE

Synthesis and Identification of Azo Disperse Dye Derived from 4-Aminoantipyrine and Their Applications to Determine Some Drugs

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ABSTRACT

Background: Azo dyes exemplify the most production volume of the chemistry dye this day, and their proportional significance might even rise in the future. regularly azo dyes utilized to applications in the pharmaceutical, food, paper.cosmetics, textile and leather industries. 4-Aminoantipyrine (AAP) is usually utilized of the synthesis of azo dye and in the pharmaceutical industry, in experiments biochemical and in environmental monitoring. 4AAP as an aromatic contaminant in the environment poses a great threat to human health.

Methods: Synthesis of azo dye by mixture from 10 mL of HCl, (10 mL) DW and 4-Aminoantipyrine (2.0 g, 0.00984 mol) was collected in to 250 elementary flask and mixturing was cooling in to ice bath to 0^{o}C. Then (0.676 g, 0.00984 mol) sodium nitrate in (5 mL) DW and adding dropwise in to mixture by kept the temp. at 0°C. A spectrophotometric, simple, Sensitive, accurate, and low-cost way have been proposed for the estimation of 4-amenoantypyren (4AAP) The way is based on the Deozonztion reaction of drug 4-amenoantypyren (4AAP) by diphenylamine in acid medium, and then reacts by diphenylamine to give a complex colored at (0^{o}C) which produce a product having maximum absorption at 555 nm.

Results: The data find that the order of addition, 4AAp –Acid - diphenylamine, gave the best sensitivity and absorbance; the best acid was (1N) hydrochloric acid requiring for emerging the product color and increase its stability. That compound, at 25°C, gave a best absorbance and was carefully chosen for further use in this study.

Conclusion: The method used to synthesize azo dye is an easy, fast, and inexpensive method and gives high sensitivity and absorbency. Also, it was found that the color of the dye is stable and stable at room temperature.

Keywords: Azo dye, Colorimetric, Diphenylamine, Drug, Synthesis, Spectrophotometric, 4-amenoantypyren (4AAP).

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INTRODUCTION

Synthetic compounds of the Azo dyes having azo bond –N=N-, acquired fundamentally from the aromatic substrate of amines like nitroso and nitro. The synthesis methods depend on a convenient reaction reducing/ oxidizing or reaction diazotization/coupling.[1] This is considered one of the utmost significant reactions in the expansion of organic chemistry. It requires the synthesis of salt diazonium and component coupling.[2] Figure 1 appear the common synthesis of azo dye.[3] Pharmaceuticals are among the most important and most dangerous emerging pollutants and are of concern due to their low biodegradability, high resistance, and ease of bioaccumulation. Among these are emerging pollutants, such as antibiotics, blood lipid regulators, anti-inflammatory agents, and hormones. Pharmaceutical factories, hospitals, and homes are the main source of drug leakage into sewage water, where pharmaceutical preparations are constantly leaked into the aquatic environment in very low concentrations. It leads to their presence in drinking water,[4] thus affecting plants, human and animal health. Therefore, there are several ways to get rid of these pollutants from water Sewage Several biological ways and physio-chemical has been used to eliminate contaminants from waste-water. Physio-chemical ways contain bio-degradation,[5] treatments biological,[6] bio-filtration,[7] zonation,[8] ion exchange,[9] chemical precipitation,[10] photocatalytic degradation,[11,12] advanced oxidation methods (AOPs),[13] adsorption,[14-16] electrocoagulation,[17] electro-chemical,[18] ultra-filtration membrane, membrane filtration.[19]

4-Aminoantipyrine powder a yellow crystalline, the properties as an inflammatory analgesic and analgesic,

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antipyretic The $C_{11}H_{13}N_3O$ is a formula, 169 L.mol^{-1} molar volume and molecular weight is 203.24 g/mol.[20,21] It is important to mention that 4AAP is a very significant intermediate in the production of anlagen. The reason for this is the effectiveness of the minute, Its ease of use and low cost, which is why it plays a fundamental role in clinical medicine, and the purity of (4AAP) greatly affects the purity of (anlagen).[22,23] In this study, a new azo dye was synthesized from the reaction of (4AAP) at a fixed temperature in the presence of sodium nitrate ($NaNO_3$) in an acidic medium as a mediating factor for the reaction to occur after the formation of the azo dye. The application of this ozo dye to determine some drug-like reacted with Diphenylamine reagent in an alkaline medium to form the complex. The colorant has a wavelength (555 nm), and the complex is stable at room temperature. The drug is estimated in an easy, simple, and sensitive way, and the optimal conditions are studied, each of the reagent concentration, base size, order of addition, temperature, and color stability time. Also, commercial pharmaceutical preparations such as capsules and syrup were used and compared with pure drugs.

EXPERIMENTAL DETAILS

Synthesis of Azo Dye

A mixture from 10 mL of (HCl), 10 mL distilled water, and 4-Aminoantipyrine (2.0 g, 0.00984 mol) was collected into a 250 mL Elementary flask. The mixture was cooling into an ice bath to 0°C. Then (0.676 g, 0.00984 mol) sodium nitrate in DW (5 mL) dropwise adding in mixture with kept the temperature at 0°C.

A solution from diphenylamine (1.663 g, 0.00984 mol) and HCl (10 mL, 10%) was added dropwise to salt diazonium under vigorous stirring. After the addition, the mixture was at 25°C. Leave the mixture overnight, and the mixture was neutralized by NaOH (1 M). Filtered and washing via DW was done (Figure 2).

Spectrophotometric Determination of 4-AAP

Stander solution (100 mg/L) of 4-Aminoantipyrine: The stock solution of 4-Aminoantipyrine drug was prepared by dissolving in 100 mL (0.1 g) of 4AAP in distilled water. after that using dilutions of the sequent using D.W were made to cover the range working.

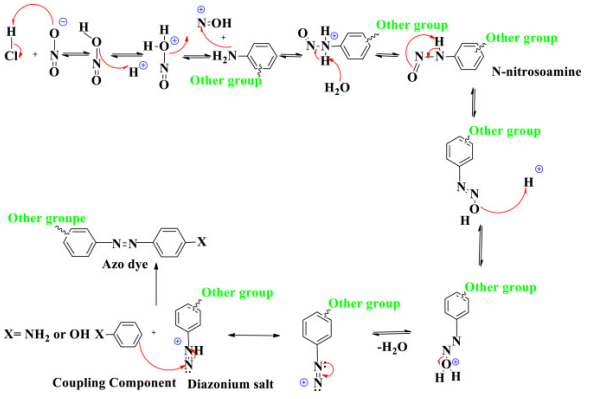

Figure 1: Synthesis of azo dye[3]

An accurately measured suitable volume of 4-Aminoantipyrine was transported from solution to Conical flasks 10 mL, which can be diluted to gain 2 mL 4AAP,each containing 3.0 mL of diphenylamine reagent were added in basic medium. After 2 minutes of mixing, to give color purple and complete the solution to 10 mL by D.W, the absorbance values were measured at 550 nm against the blank reagent, as appear in Figure 3.

RESULTS AND DISCUSSION

FTIR

The prepared azo dye was identification by infrared technique. Where it was observed from the (FT-IR) spectrum as in Figure 4, the appearance of a strong absorption band at site 1650.95 cm^{-1} indicating to the carbonyl group in the pyrazoline ring, and a strong absorption band at 1589.23 cm^{-1} due to C=C stretcher Aromatic ring, and a weak absorption band at 1496.66 cm^{-1} indicating to the -N=N- group and a weak absorption band appeared at the 3286.48 cm^{-1} due to the -NH group.

Effect of Volume Diphenylamine Reagent

When several volume diphenylamine was added to 4-amenoantypyren, that found that increased absorbance through increasing volume of reagent and reached its good value on using 2.5 mL concentration of reagent at good sensitivity give absorption 555 nm of the reagent. Here, it was observed that by increasing the reagent volume, the absorbance value decreases, and this is evident that increasing the reagent volume reduces the stability of the colored compound[24,25] as appears in (Figure 5).

Effect of the Concentration Base

Determine the beater base (Figure 6 and Table 1). generally used of the base KIOH, NaOH, NH_4OH, Na_2CO_3, and $NaHCO_3$ because the development color of the complex is reliant on, kind of quantity of base and nature of base. Utmost of these bases bear the values minimal absorbance when used more and can be arranged in high NaOH<KOH< Na_2CO_3 <$NaHCO_3$<NH_4OH. NaOH is used as the alkaline agent. Because it gives better absorbance and the best sensitivity. The concentration of the best base was also studied, and it was found that 0.1 N give clear higher stable colored complex, best sensitivity, major intensity, and the maximum absorbance.[26,27]

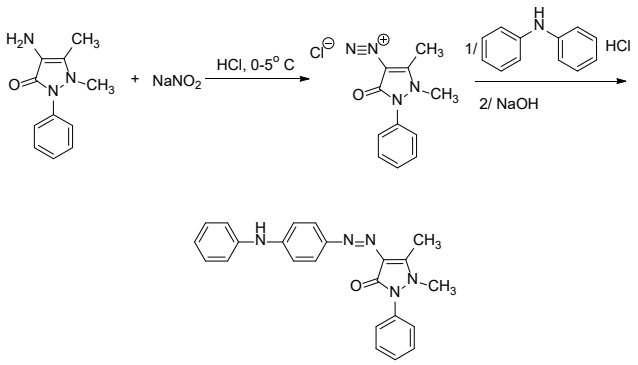

Figure 2: Synthesis of azo dye and coupling reaction with a reagent to give color complex

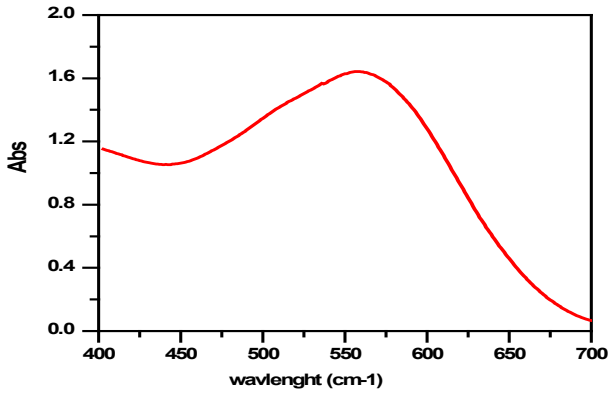

Figure 3: Absorption spectra of (25 mg.L^{-1}) solution of 4AAP

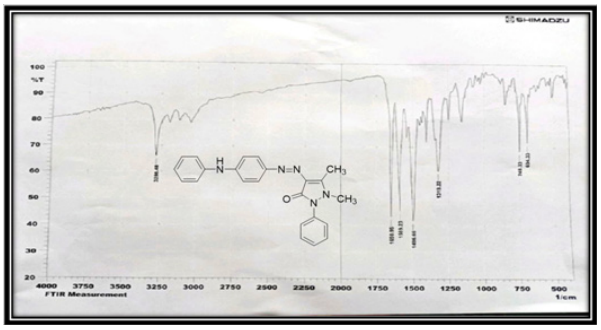

Figure 4: FT-IR for compound azo dye

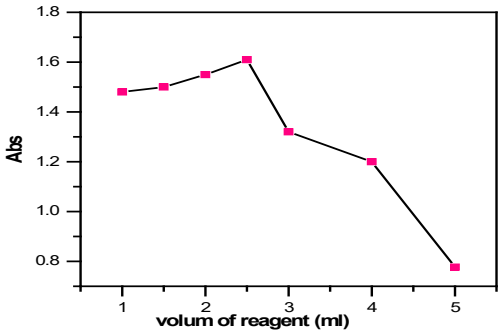

Figure 5: Absorption of azo dye found in several volumes of diphenylamine

Effect of the Order Adding

The impact of adding reagents on the reaction product was studied and looked at in (Table 2) the best order adding taking the following order (4AAP+ + NaOH + reagent).[21,28] Through the results in Table 2, the order sequence of addition is observed, which is very important in stabilizing the color of the complex for a longer period and giving the best absorbency.

The Influence of Time on the Color Stability of Azo Dye

Time plays a significant and basic role in the complex's color stability under the optimum experimental conditions, both the size of the reagent and the type and size of the base used.[21,29] Its stability appears in Figure 7.

Analytical Applications

The equivalent of (12 tablets and capsules) of the commercial drug (50 mg, 100 mg) were weighed and ground to a fine

Table 1: Effect of the type base added of 4AAP

Type of base	*Absorbance*
Na_2CO_3	0.55
$NaHCO_3$	0.333
KOH	1.51
NaOH	1.61
NH_4OH	0.122

Table 2: Effect of order addition of 4AAP and diphenylamine

No.	*Order Addition*	*Absorbance*
1	4AAP + NaOH + reagent	1.61
2	4AAP + reagent+ NaOH	1.55
3	Reagent + 4AAP + NaOH	0.998
4	Reagent +NaOH + 4AAP	0.899

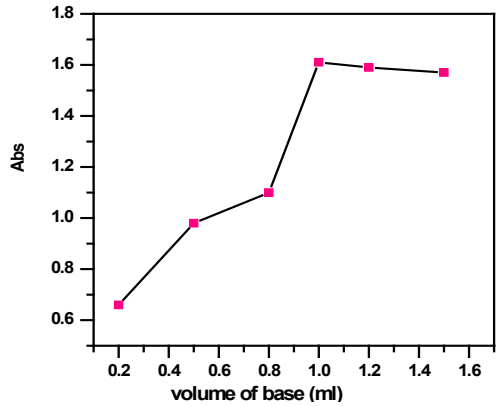

Figure 6: Absorption of azo dye in the found of several volume base added of 4AAP

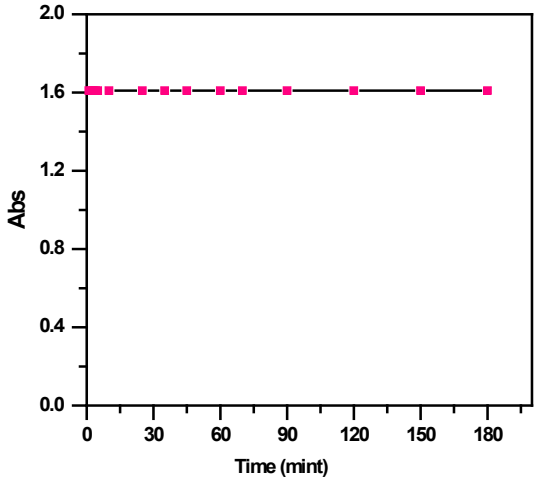

Figure 7: Effect of time on the color stability of azo dye

powder and dissolved in (50 mL) distilled water to ensure complete dissolution. It was placed in an ultrasound device for a period of (15 minutes) then filtered the solution through a filter paper to obtain a clear solution and complete the volume with water Distilled to (100 mL) where the proposed method was useful for analyzing pharmaceutical preparations containing

Table 3: Determination of 4AAP in some formulations using the official and proposed way

	Conc. Of 4AAP (mg L^{-1})		*E %*	*Rec%*
Pharmaceutical preparation	*Present*	*Found*		
4-Aminoantipyrine tablets 50 mg (DSI), Iraq	10	9.911	-0.89	99.11
	20	19.783	-1.085	98.915
	50	49.383	-1.234	98.33
4-Aminoantipyrine tablets 50 mg (DSI), Iraq	10	9.933	-0.67	99.33
	20	20.183	0.915	99.085
	50	51.016	2.032	97.968
4-Aminoantipyrine tablets 100 mg Iran	10	9.666	-3.34	96.66
	20	19.883	-0.585	98.415
	50	49.123	-1.754	99.183
4-Aminoantipyrine tablets 50 mg	10	9.850	-1.5	98.5
	20	19.650	-1.75	98.25
	50	49.223	-1.554	98.446
4-Aminoantipyrine capsule 50 mg (Iran)	10	9.665	-3.33	96.65
	20	19.877	-1.501	98.511
	50	51.116	2.022	97.977
4-Aminoantipyrine capsule 100 mg (DSI), Iraq	10	9.841	-1.51	98.51
	20	20.11	0.915	99.022
	50	51.12	2.031	97.965

(4AAP) and through the table 3 through the spectroscopic method using a reagent characterized by high accuracy and selectivity.[30,31]

CONCLUSION

In this study, the synthesis of azo dye is done by a mixture of 10 mL of acid in 10 mL. DW and drug (0.00984 mol) and mixing were cooling into an ice bath to 0^{o}C. It is an easy, simple, and inexpensive method. The color of the dye is stable at room temperature for three days. Improving the color of the azo dye relies on the kind of Nature base, the quantity of NaOH, and found (0.1 N) NaOH give the best absorbance and higher sensitivity**.**

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