

## Preparation , characterization and biological activity of some new tetra - azo compounds

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### Abstract

In this paper , preparation and characterization of some tetra azo compounds .Azo coupling of 3',3,4',4- tetra amino biphenyl with phenol and some phenol derivatives in alkaline solution gave azo dyes. I.R and U.V. visible spectra were recorded to identify the three azo compounds prepared . Some physical and chemical properties such as molar absorption coefficient in different pH value and maximum absorption  $\lambda_{max}$  were investigated. The biological effect of these dye against two gram positive Bacteria (*S.aureuse*) and other two gram negative Bacteria (*P.aeruginosa*) is presented .

### **Introduction:**

The azo compound class accounts for 60 – 70 % of all dyes <sup>(1)</sup> . Most azo dyes contain only one azo group, but some contain two ( diazo ), three (triazole) or more . Azo dyes can supply a complete rainbow of colors, they tend to supply yellow, orange , blue and red than other colors <sup>(2)</sup> .

In recent decades, organic color chemistry undergoes very exciting development as a result of the opportunities presented by dye applications in high technology fields: electronic devices <sup>(3)</sup>, linear and nonlinear optics<sup>(4,5)</sup>, reprography, sensors<sup>(5)</sup>, textile industry <sup>(3,4,6)</sup> and biomedical <sup>(7)</sup> . Azo dyes are important intermediates for the synthesis of some bioactive compounds such as nitron compounds <sup>(8)</sup> and there are so many search about azo compounds synthesis and it's chemical properties <sup>(9,10)</sup> . Furthermore, they are reported to show a variety of interesting biological actions<sup>(7)</sup>, including antibacterial , antifungal<sup>(11)</sup> , anti mouse hepatitis virus<sup>(12)</sup> , inhibition of herpes simplex virus and adenovirus , anticancer <sup>(13)</sup>, anti mosquito larvae and herbicidal activities<sup>(14)</sup> .

### **Experimental:**

#### **Apparatus:**

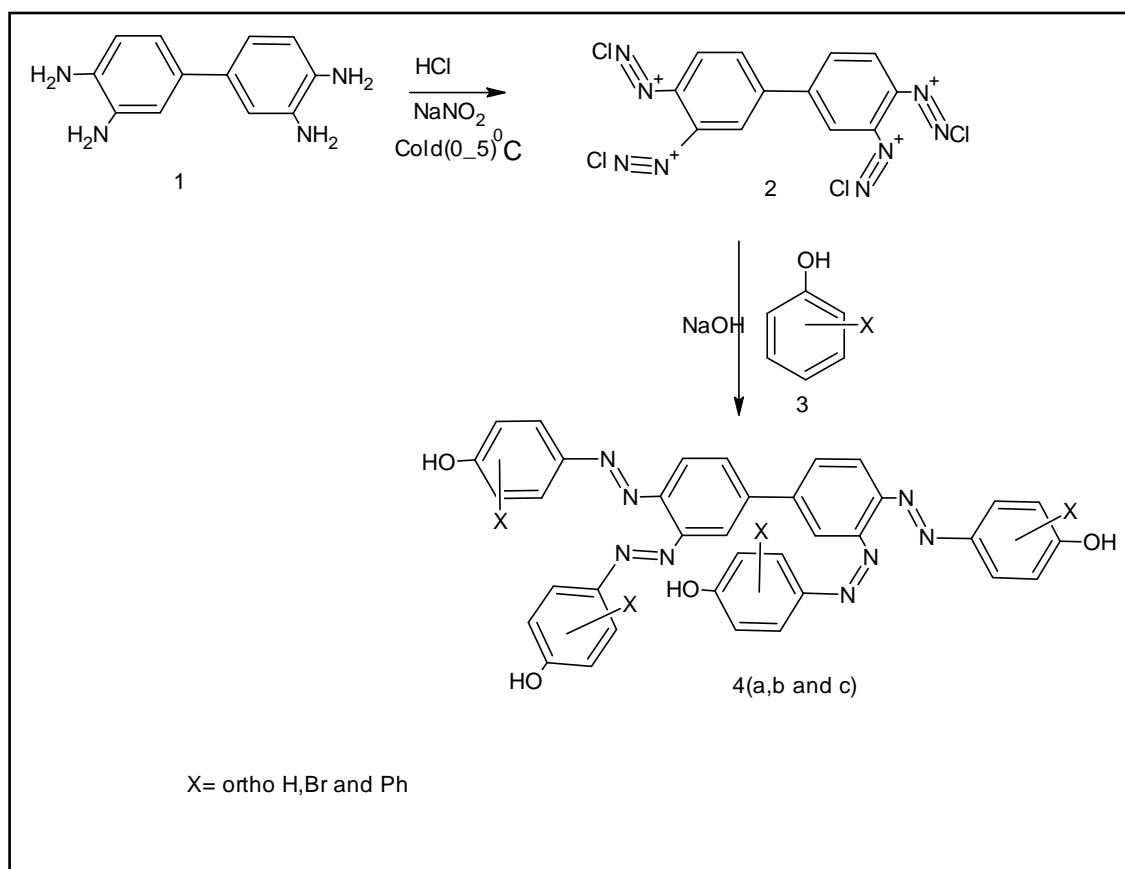
Melting points were determined by using as “ Electrothermal “ melting point and remain uncorrected .The U.V- Visible spectra were recorded on a sunny spectrophotometer , I.R. spectra were recorded on a Buck infrared spectrophotometer in Nojol solution ( $\nu$  in  $\text{cm}^{-1}$ ) . Melting points, crystallization solvents and yields percentage are listed in Table (1).

#### **Preparation:**

The three dyes were prepared by adding 0.025 mole of amine to 25 ml of (1:1) distilled water and hydrochloric acid mixture. 0.025 mole of sodium nitrite solution was added drop wise with stirring at 0 - 5°C.

The diazonium salt was prepared according to a reported method <sup>(11)</sup> then was added drop wise to phenol and it's derivatives which prepared by dissolving of 0.025 mole of phenols into 25 ml of 10% sodium hydroxide . The yielding compounds were left at cold system then it were separated by filtering process and washed with cold water for several times . The dyes were purified and recrystallization by aqueous methanol to give the pure material 78-89% yield (scheme1) <sup>(15)</sup> .

The biological test was determined by prepare (0.1, 0.5, 1) gm/L solution of dyes against gram positive (*Staphylococcus aureus*) and gram negative (*Pseudomonas aeruginosa*) bacteria.



**Scheme ( 1 ) : the structure of prepared dyes**

### Results and discussion:

Azo dyes are well known characterize with special properties as a simple and easy method of preparation in a desire colors , high solubility into common solvents and high stable adsorption on cotton and wool textiles surfaces <sup>(16)</sup> .

Tetra amine was used so that the dyes were prepared have four azo groups lead to large structure and it have a dark color.

UV visible spectra (200- 800) nm were recorded. Table (1) shows that the  $\lambda$  max belonging to the  $\pi - \pi^*$  transition which conformed by measured in acid medium (pH=5) show that the  $\lambda$  max shift to higher wave length <sup>(16 - 19)</sup> (red shift). Table (1) show the  $\lambda$  max in both pH =5, 7 and other properties molar absorption coefficient in both media figures (1,2and 3).

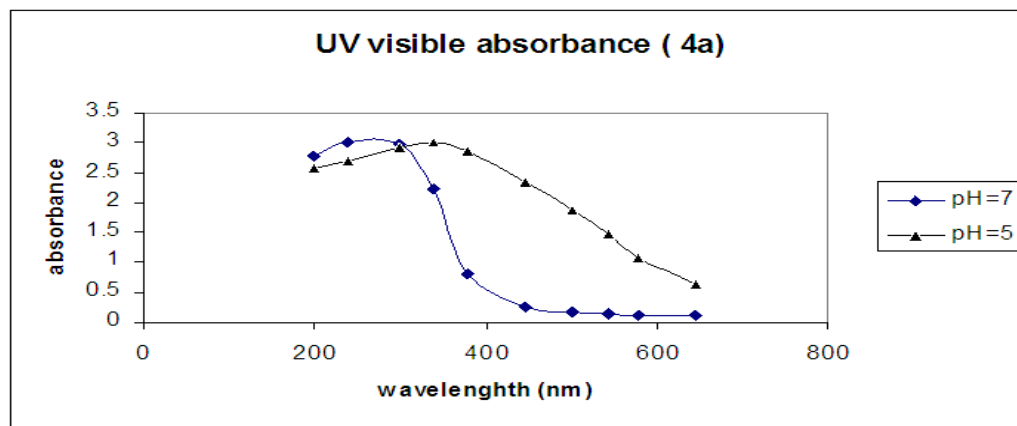


Fig. (1) : The absorption spectra of (4a) compound .

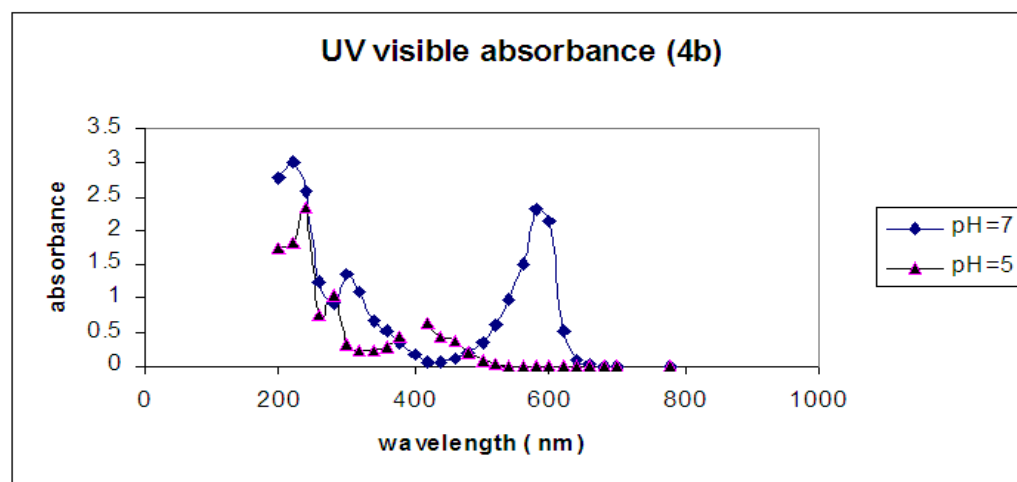


Fig. (2) : The absorption spectra of (4b) compound .

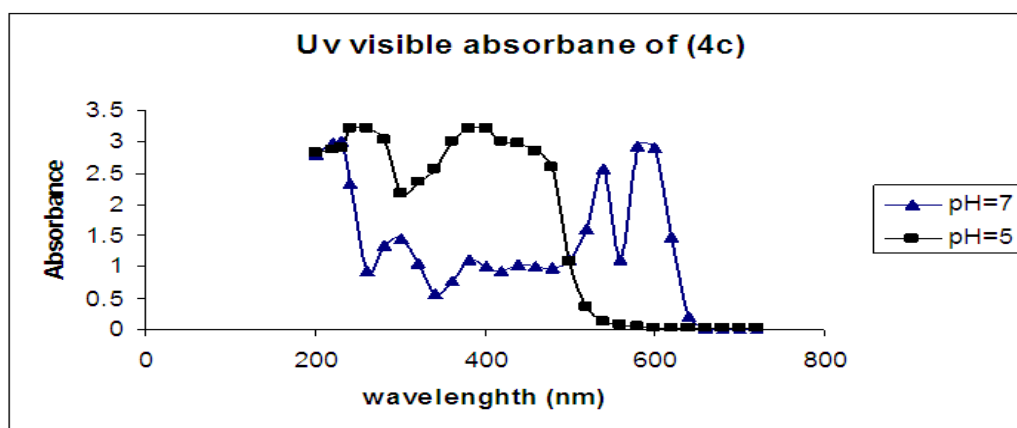


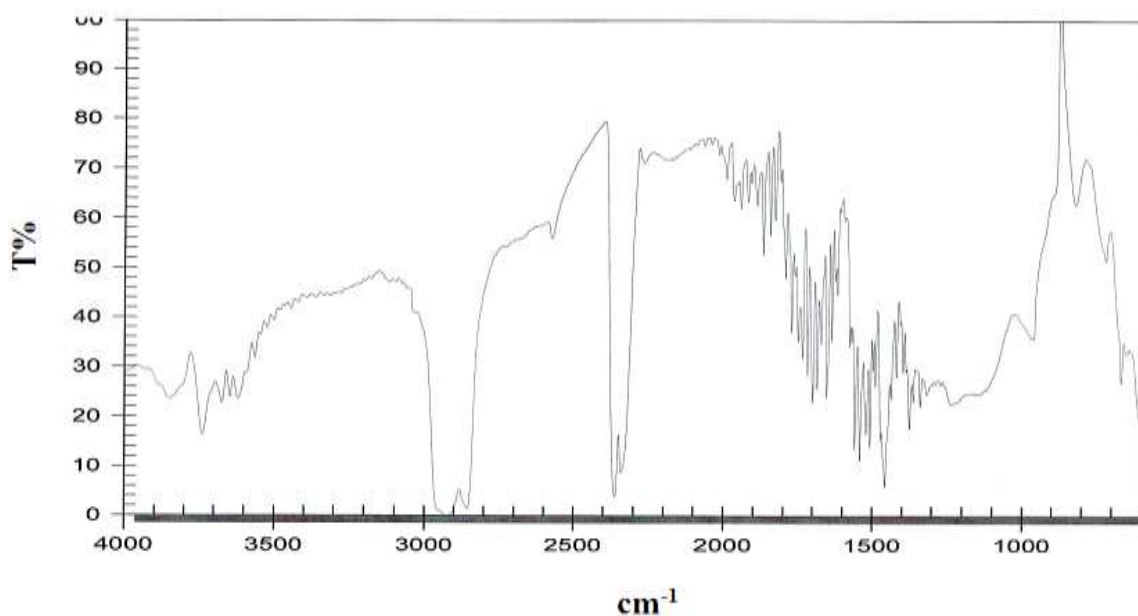
Fig. (3) : The absorption spectra of (4c) compound .

**Table (1) : Show the physical and chemical properties for the three azo dyes that has been prepared in this study**

Comp.	m.p °C	Color	$\lambda_{\max}$ nm pH=7	$\lambda_{\max}$ nm pH=5	$\epsilon$	$\epsilon$	Yield
4a	112	Dark brown	245	265	301	2985	78%
4b	174	Blue	224	245	301	2358	85%
4c	204	brown	239	241	301	3214	89%

IR spectrum of three azo compounds were prepared in this study , show the presence band of above  $3800\text{ cm}^{-1}$  belong to (OH) group<sup>(20)</sup> , absence of band between  $3100\text{-}3300\text{ cm}^{-1}$  this give essential indicator that no (NH<sub>2</sub>) group in the compound that has been formed and band  $1455\text{-}1470\text{ cm}^{-1}$  belong to the (N=N) group as shown below .

Table (2): Show the I.R bands for which group it belong for three azo dyes were prepared in this study<sup>(21-23)</sup> . Fig. (4) Show the I.R. spectrum for the (4a, 4b, 4c) respectively.



**Fig. (4) I.R spectrum for (4a compound)**

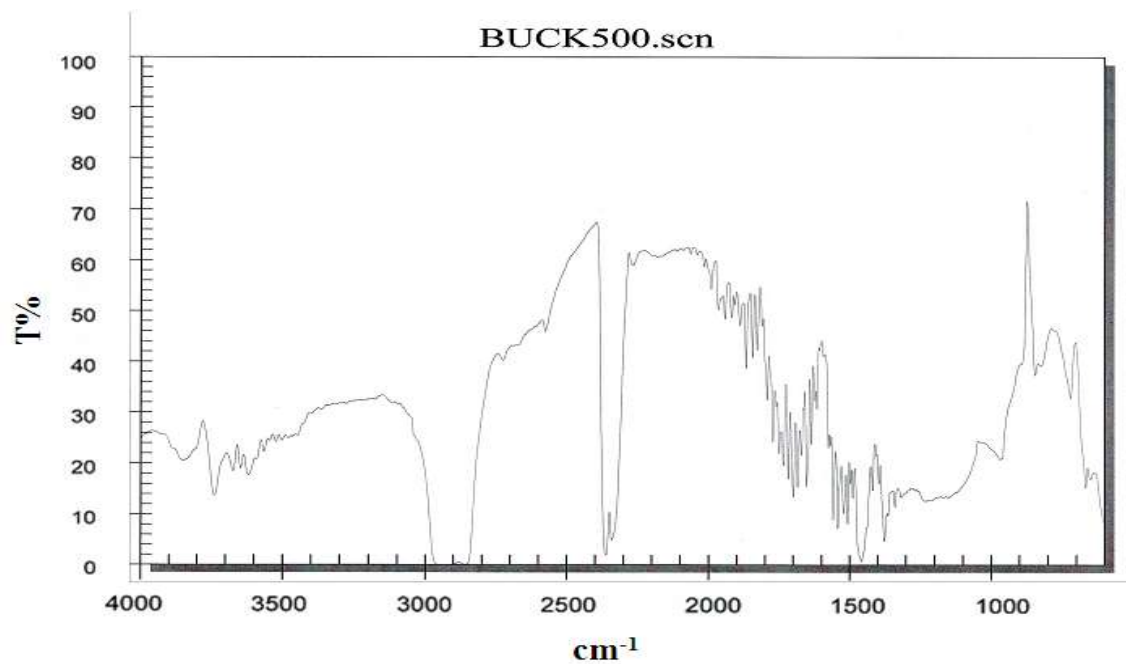


Fig. (5) I.R spectrum for (4b compound)

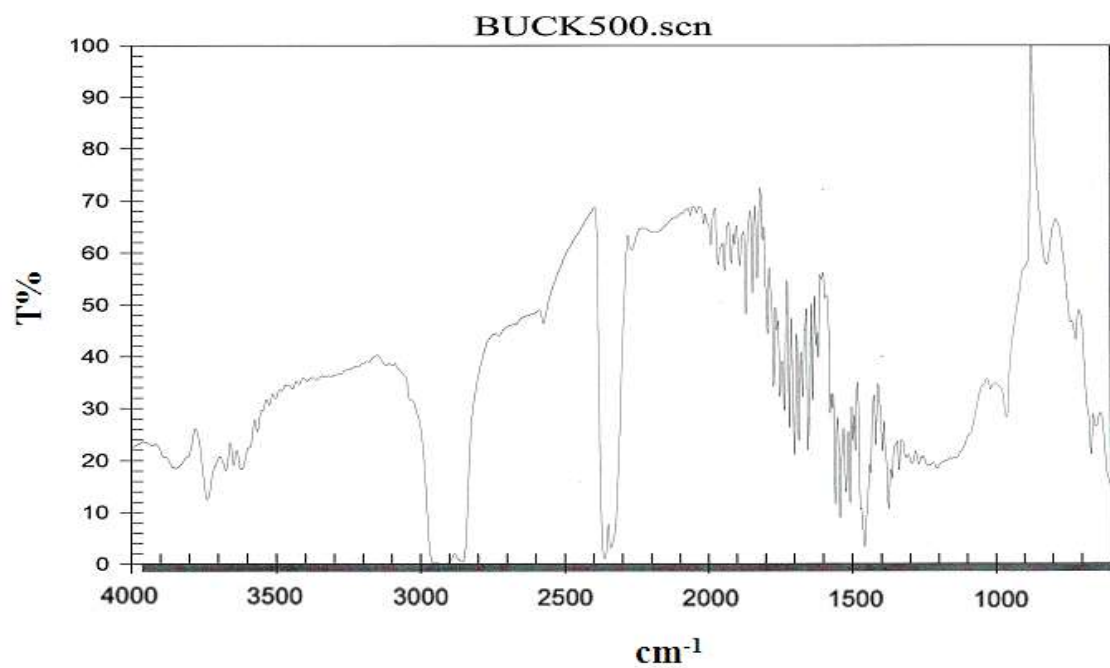


Fig. (6) I.R spectrum for (4c compound)

**Table (2): show the major band of I.R spectrum of (4a, 4b and 4c) compounds**

Comp.	OH Stretch cm <sup>-1</sup>	C=C Stretch cm <sup>-1</sup>	N=N Stretch cm <sup>-1</sup>	C-O,C-N bending cm <sup>-1</sup>	OH bending cm <sup>-1</sup>
<b>4a</b>	<b>3616</b>	<b>1540</b>	<b>1455</b>	<b>1378</b>	<b>975</b>
<b>4b</b>	<b>3632</b>	<b>1467</b>	<b>1470</b>	<b>1367</b>	<b>968</b>
<b>4c</b>	<b>3675</b>	<b>1540</b>	<b>1459</b>	<b>1366</b>	<b>971</b>

### **Biological activity:**

Three chemicals solution was detected their activity against bacterial growth. Two types of bacterial broth were used in this study, which are *Staphylococcus aureus* gram positive and *Pseudomonas aeruginosa* gram negative bacteria <sup>(24)</sup>. There bacteria pathogenic produced several medical conditions supportive infections, food Poisoning, broucho pneumonia, meningitis, wound infections, and urinary tract infection.

According to the result of presented study, these solutions in different concentration have bacterial effect on fresh culturing media and used a method similar to Antibiotics sensitivity test <sup>(25-27)</sup>.

**Table (3) : The biological activity of (4a) azo dye**

Bacteria \ Comp.4a	Gram positive	Gram negative
	<i>S.aureuse</i>	<i>P.aeruginosa</i>
0.1gm/L	+	+
0.5 gm/L	+	+
1 gm/L	+	+

Table (4) : The biological activity of (4b) azo dye

Bacteria	Gram positive	Gram negative
	Comp.4b	<i>S.aureuse</i>
0.1gm/L	+	+
0.5 gm/L	+	+
1 gm/L	+	+

Table (5) : The biological activity of (4c) azo dye

Bacteria	Gram positive	Gram negative
	Comp.4c	<i>S.aureuse</i>
0.1gm/L	+	+
0.5 gm	+	+
1 gm/L	+	+

Conclusion:

All the three azo compounds prepared in this study show absorbance belonging to  $\pi - \pi^*$  and it has been confirmed by measuring its absorbance in acid medium. Three compounds have a biological activity against bacteria both gram positive and gram negative.



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### الملخص

تناولت الدراسة الحالية تحضير ودراسة صفات بعض المركبات الجديدة لصبغات رباعية الازو تم تحضيرها للمركب 3,4,4'-ثلاثي امينو بايفنايل مع الفينول وبعض مشتقاته في محاليل قاعدية . تم تشخيص المركبات باستخدام أطياف الأشعة تحت الحمراء وأطياف الأشعة المرئية وفوق البنفسجية كما تم قياس الخواص الكيميائية والفيزيائية مثل معامل الامتصاص المولاري والطول الموجي الأعظم في محاليل مختلفة الأس الهيدروجيني . تناولت الدراسة أيضا قياس الفعالية البايولوجية للمركبات المحضرة ضد نوعين من الجراثيم الأولى موجبة لصبغة كرام (*S.aureuse*) والثانية سالبة لصبغة كرام (*P.aeruginosa*).