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SYNTHESIS AND EVALUATION OF BIOLOGICAL ACTIVITY OF SOME NEWSALICYLIC ACID DERIVATIVES

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ABSTRACT : The aim of this work includes the synthesis and characterization of new azo dye compounds. By coupling various derivatives aniline included(P-Nitro aniline, P-Bromo aniline, P-Amino benzoic acid and 4-chloro –o-phenylenediamine) with salicylic acid. The new prepared azo compound have a biological activity against (gram positive and gram negative bacteria) that lead to be used in the pharmaceutical industry. By conclusion The structure of the synthesized compounds were deducedby using some spectroscopic methods, FT-IR and ^{1}H NMR.

Key words : Salicylic acid, azo dye, biological activity.

INTRODUCTION

Sweet-smelling amines are a class of natural mixes in which an amino (–NH_2) bunch is straightforwardly connected to fragrant carbon. These are utilized for the blend of numerous mixes like azo colors, Schiff 's bases, plastics and polyamides (Hammad *et al*, 2015). Azo colors contain somewhere around one nitrogen-nitrogen two fold bond (N=N), the azo gatherings are commonly associated with benzene and naphthalene rings, however can likewise be connected to fragrant hetero cycles, for example, chloroquine, while depicting a color atom, nucleophiles are alluded to as auxochromes, while the sweet-smelling bunches are called chromophores. Together, the color atom is frequently depicted as a chromogen. Amalgamation of most azo colors includes diazotization of an essential sweet-smelling amine, trailed by coupling with at least one nucleophiles (Ghulam *et al*, 2017). Amino-and hydroxy-bunches are generally utilized coupling segments. In view of the assorted variety of color parts accessible for amalgamation, an extensive number of basically unique azo colors exist and are utilized in industry (Layla, 2016). The colors have been most broadly utilized in fields, for example, biting the dust material strands, biomedical investigations, propelled applications in natural amalgamation and high innovation zones like lasers and ink-stream printer (Chandravadivelu *et al*, 2017). Azo mixes likewise utilized as organic exercises, for example, antifungal, antibacterial activity (Dheefaf, 2017) nshading specialists for sustenances and beauty care products businesses. Different applications incorporate developing advances like fluid gems, natural photoconductors (Thawra *et al*, 2015).

MATERIALS AND METHODS

Melting points were measured using digital melting point, Stuart SMP 10. Infrared spectrophotometer measurements were performed using perkinelmer spectrum 65, 1H-NMR spectra were measured with a bruker spectrophotometer model ultra-shield at 300.13 MHz in DMSO-d6 and CDCl3 as solvents with TMS as internal standard, Al-Albyt University, Jordan.

Not: in 1H-NMR spectra, the signals at 2.5 ppm for DMSO-d6.

Synthesisof Azo dyes (Dheefaf, 2017)

1- (0.01 Mole) derivatives amine dissolved in (10 ml) from 50% hydrochloric acid (HCl) using ice bath.

2- (0.8 gm) sodium nitrite ($NaNO_2$), dissolvedin (4 ml) of distill water.

3- The solution of step 2 gradually added to solution of step1 with steering in ice bath, to form diazonium salt (beaker content 1).

4- (0.01 Mole) salicylic acid dissolved in (9 ml) from 10% NaOH using ice bath (beaker content 2).

5- Beaker content 1 (diazonium salt) was added gradually to beaker content 2 (salicylic acid) with steering in ice bath.

6- After (15 mint) the precipitate was filtrate and

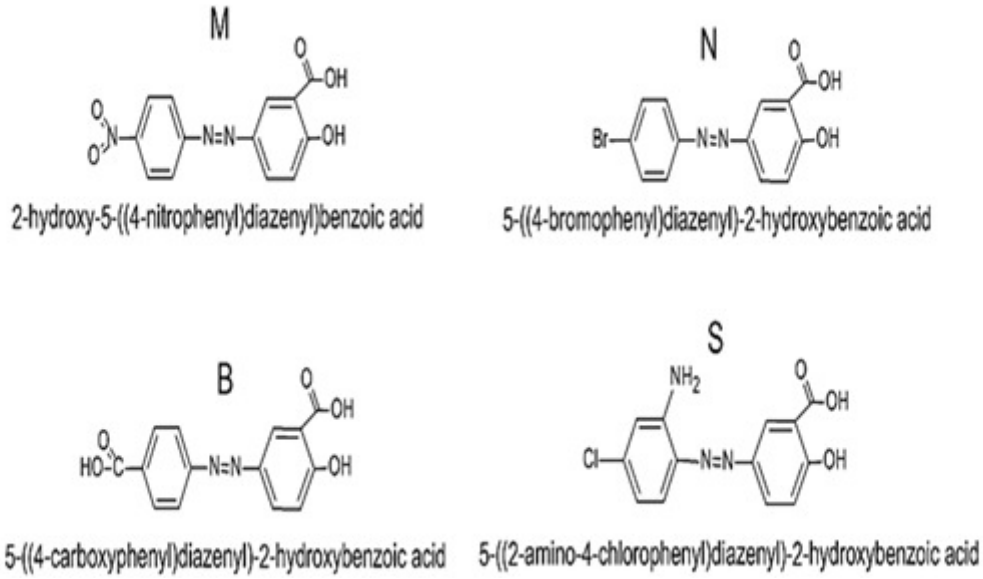


Fig. 1 : Name and chemical structure of prepared azo dye compounds.

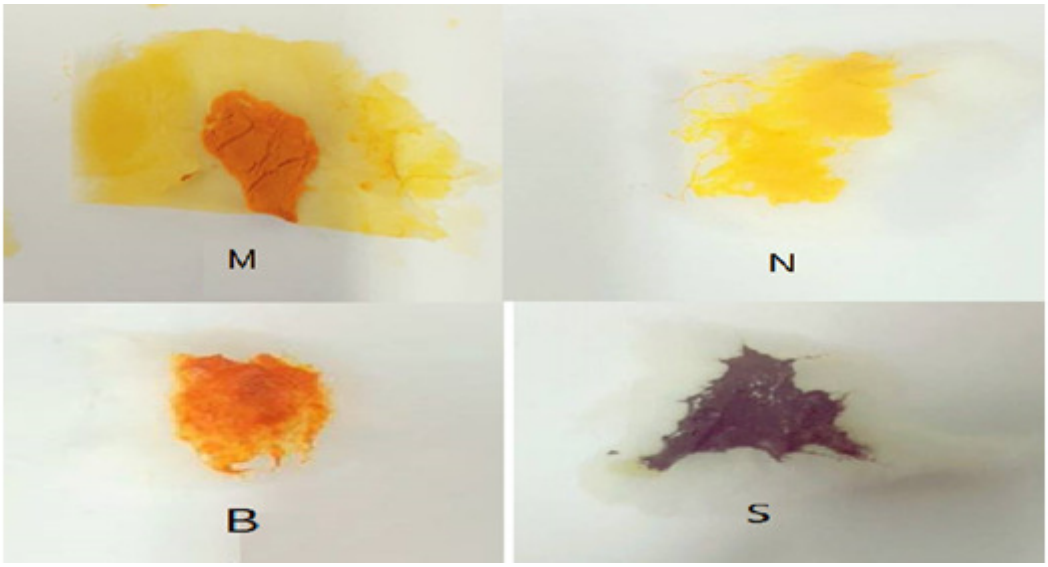


Fig. 2 : The color of prepared azo dye compounds.

Table 1 : Physical properties of prepared azo dye compound.

Comp. name	Color	Melting point (^{0}C)	Solubility in water	Solubility in DMSO	Conversion%
M	Dark yellow	173	Insoluble	soluble	98
N	Light yellow	221	Insoluble	soluble	97
B	Orange	235	Insoluble	soluble	98
S	Silver	279	Insoluble	soluble	96

dried in oven at 45^{0}C. All physical properties were listed in Table 1.

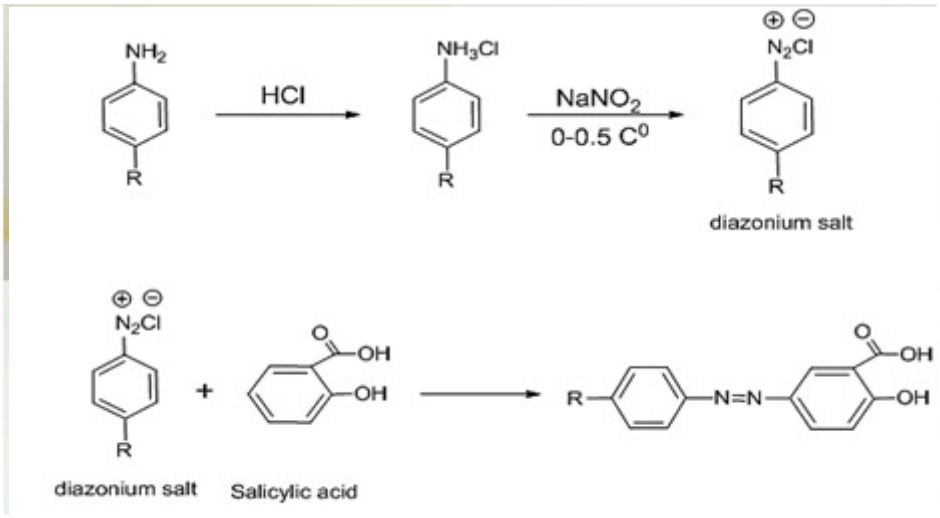
RESULTS AND DISCUSSION

The new azo dye compounds were prepared by coupling dizonium salt with salicylic acid, its show in scheme (1), the reaction applied in ice path between (0-5^{0}C). All the synthesized compounds were characterized by IR, ^{1}HNMR and UV-Visible and the biological activity were determined with gentamicin as standard.

Identification of prepared azo dye compounds [M-S] by FT-IR spectra (Firyal *et al*, 2017; Luma*et al*, 2018).

FTIR spectrum of prepared dye (M), 2-hydroxy-5-((4-nitrophenyl) diazenyl) benzoic acid, showed absorption peaks at (1485 cm^{-1}) of (N=N) azo group and (C=C) aromatic absorption peak at (1533-1612 cm^{-1}), peak at (3068 cm^{-1}) due to (C-H aromatic) stretching.

FTIR spectrum of (N) 5-((4-bromophenyl) diazenyl)-



Scheme 1 : Reaction coupling of dizonium salt with salicylic acid.

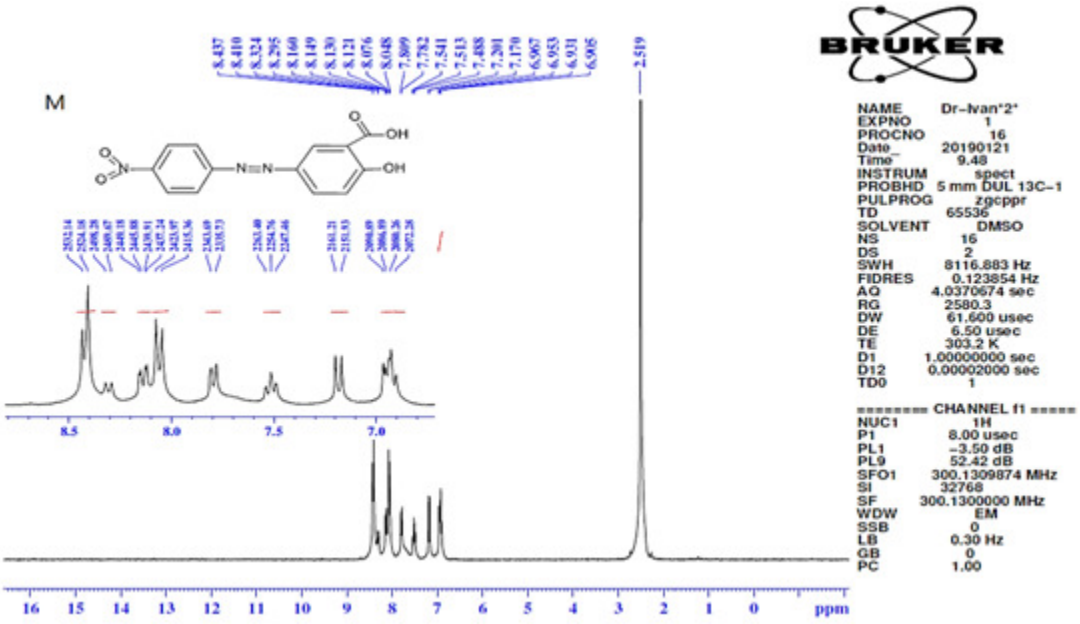


Fig. 3 : ^{1}H-NMR Spectrum of (M) [2-hydroxy-5-((4-nitrophenyl) diazenyl) benzoic acid].

2-hydroxy benzoic acid, gave the characteristic absorption of (N=N) azo group peak was appeared at (1478 cm^{-1}), and (C=C) aromatic absorption peak at (1500-1574cm^{-1}).

FTIR spectrum of (B), 5-((4-carboxyphenyl) diazenyl)-2-hydroxy benzoic acid, showed absorption peaks at (1484 cm^{-1}) of (N=N) azo group and (C=C) aromatic absorption peak at (1579-1609 cm^{-1}), peak at (3068 cm^{-1}) due to (C-H aromatic) stretching.

FTIR spectrum of (S), 5-((2-amino-4-chlorophenyl) diazenyl)-2-hydroxy benzoic acid, showed absorption peaks at (1489 cm^{-1}) of (N=N) azo group and (C=C) aromatic absorption peak at (1528-1613 cm^{-1}) peak at (753 cm^{-1}) due to (Cl) stretching vibration, other bands of prepared compounds are listed in Table 2.

Proton nuclear magnetic resonance (1H-NMR) (Suaad *et al*, 2019; Firyal *et al*, 2017)

1H-NMR spectra of prepared azo dye compounds were obtained using DMSOas a solvent with TMS as internal standard. The 1H-NMR spectrum of prepared azo dye compound (M) [2-hydroxy-5-((4-nitrophenyl) diazenyl) benzoic acid] indicated the signal assignments in the corresponding formula, which shown the following signals: 6.5-8.5 ppm (Multiple, 7H, Ar-H), 2.5 ppm (singlet, 6H, $2CH_3$) fordimethysulfoxide (DMSO) solvent as shown in Fig. 3.

The 1H-NMR spectrum of prepared azo dye

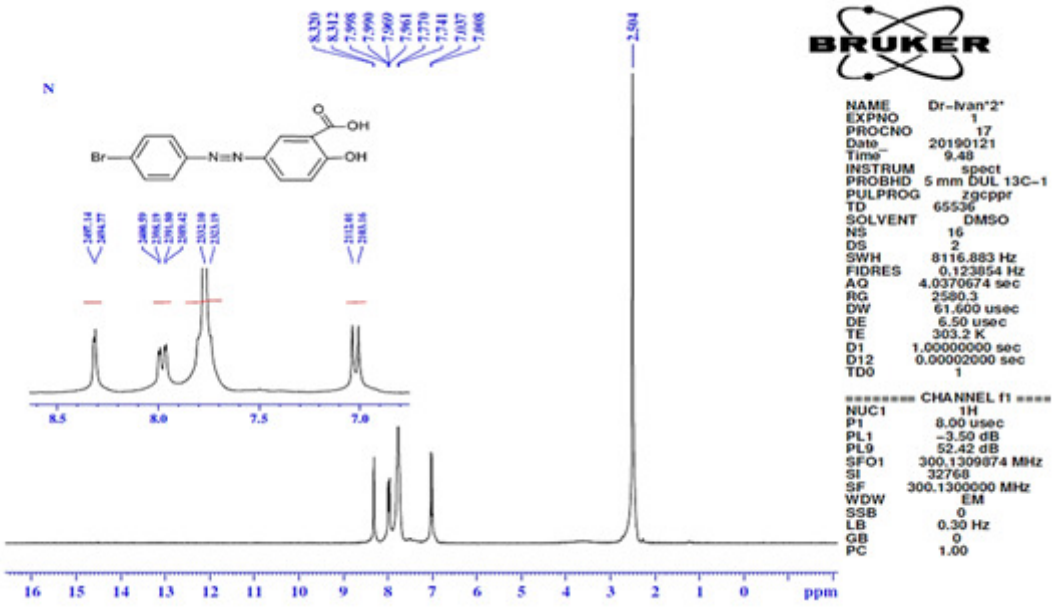


Fig. 4 : ^{1}H-NMR Spectrum of (N) 5-((4-bromophenyl) diazenyl)-2-hydroxy benzoic acid.

Table 2 : FT-IR absorptions of prepared azo dye compounds from [M-S].

Comp Name	υ(N=N)cm^{-1} Azo	υ(C=C)cm^{-1} Aromatic	υ(C-H)cm^{-1} Aromatic	υ(O-H)cm^{-1} Carboxylic	υ(C=O)cm^{-1} Carboxylic	υ other band cm^{-1}
M	1485Strong	1533-1612	3068	2400-3400broad	1675	Nitro (NO_2) 1347Strong
N	1478Strong	1500-1574	3050overlap	2400-3400broad	1661	Bromide661strong
B	1484Strong	1579-1609	3068	2400-3400broad	1675	(C-N)1294)strong
S	1489Strong	1528-1613	3062	2400-3400broad	1665	Chloride753Strong

Table 3 : Biological activity for azo dye compound with gentamicin as standard.

Azo dye compounds	Inhibition zone diameter (mm) forAzo dye compounds				
	Gram positive		Gram negative		Fungi
	Staphylococcus aureus	*Staphylococcus epidermidis*	*Escherichia coli*	*Klebsiella* sp.	*Candida albicans*
M	15	12	10	12	16
N	21	21	10	14	22
B	-	-	10	13	-
S	12	10	10	10	11
Gentamicin	36	29	27	27	15

compound (N) 5-((4-bromophenyl) diazenyl)-2-hydroxy benzoic acid.indicated the signal assignments in the corresponding formula, which shown the following signals:

7-8.3 ppm (Multiple, 7H, Ar-H), 2.5 ppm (singlet, 6H, $2CH_3$) fordimethysulfoxide (DMSO) solvent as shown in Fig. 4.

The 1H-NMR spectrum of prepared azo dye compound (B) 5-((4-carboxyphenyl) diazenyl)-2-hydroxy benzoic acid.indicated the signal assignments in the corresponding formula, which shown the following signals:

6.8-8.4 ppm (Multiple, 7H, Ar-H), 2.5 ppm (singlet, 6H, 2CH3) fordimethysulfoxide (DMSO) solvent as shown in Fig. 5.

Biological activity (Amal *et al*, 2018; Siham *et al*, 2018)

Biological activity of prepared azo dye compounds were determined with gentamicin as standard, by agar diffusion method, all azo dye compounds were tested and the plates were incubated at 37^{0}C for 24 hours, the inhibition zone measured in (mm). Azo dye compound (M, N, B, S) were evaluated for antibacterial activity against different bacterial strain, (*Staphylococcus aureus*, *Staphylococcus epidermidis*, *Escherichia coli*, *Klebsiella* sp.) and fungi such as (*Candida albicans*) as shown in Fig. 6, inhibition zones caused by these compounds are determined and listed in Table 3.

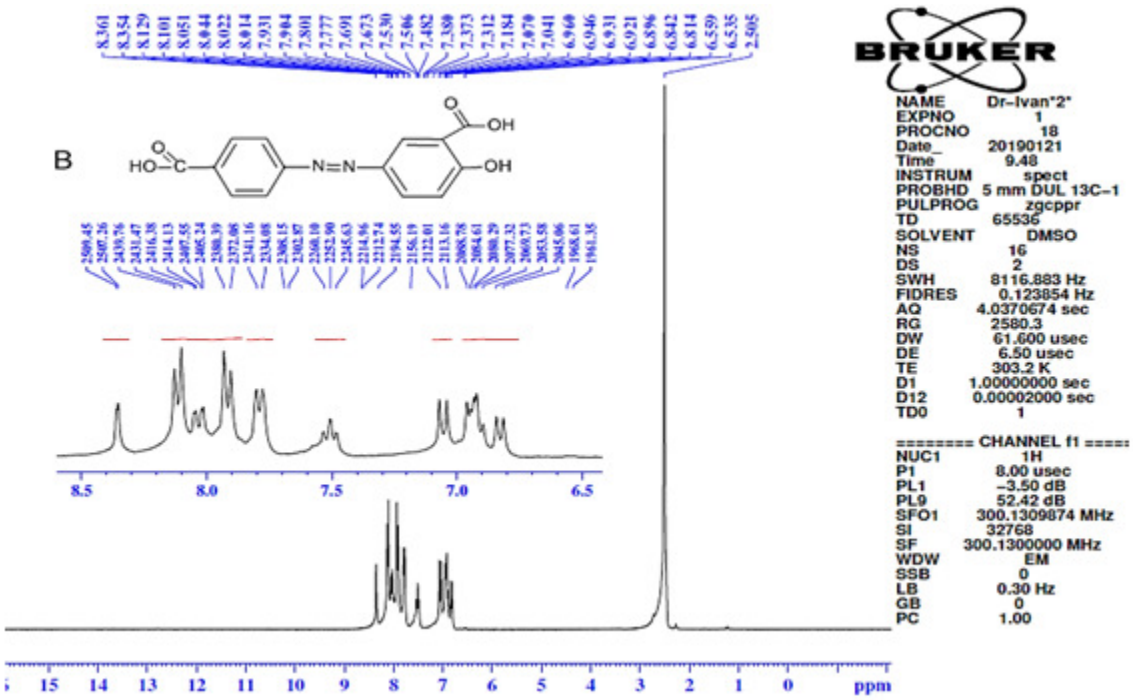


Fig. 5 : ^{1}H-NMR Spectrum of (B) 5-((4-carboxyphenyl) diazenyl)-2-hydroxy benzoic acid.

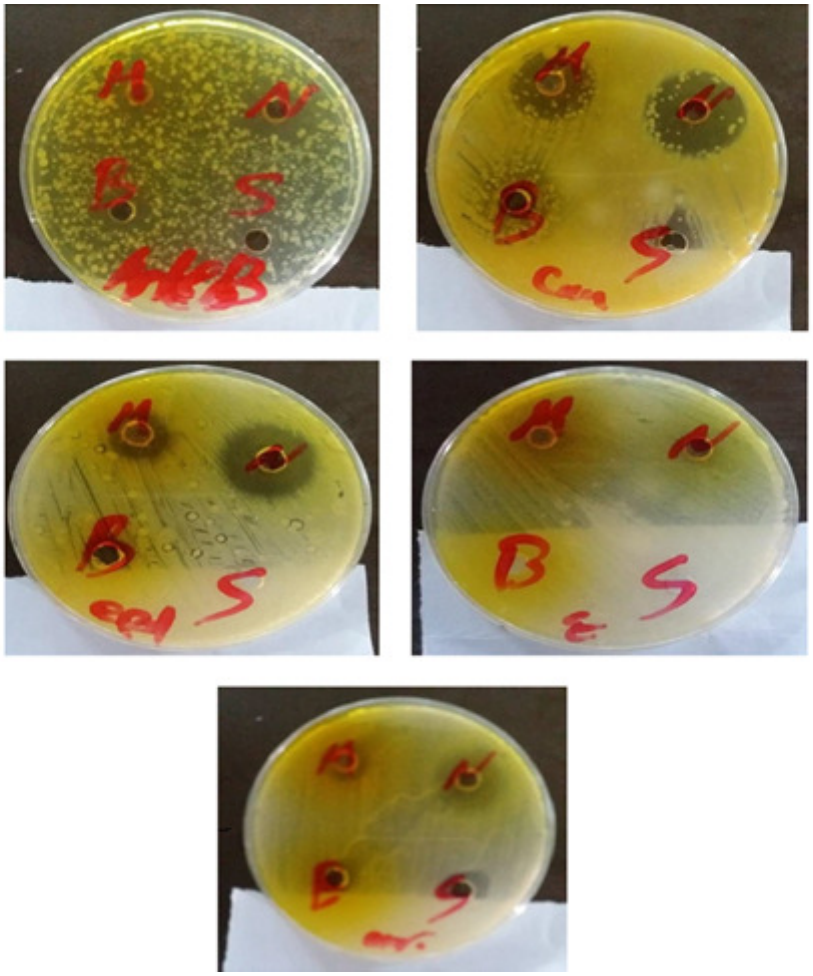


Fig. 6 : Agar diffusion method of prepared azo dye compounds.

The results in Table 3 indicate that the biological activity for azo dye compound (N) has higher than other compounds, its approach the standard (gentamicin). The compound (B) gave less biological activity from other compounds, its just show biological activity against gram negative bacteria (*Escherichia coli*, *Klebsiella* sp.). The azo dye compounds are shown more biological activity against (gram +ve) than (gram –ve) bacteria.

CONCLUSION

We conclude from this research that the compounds prepared and after diagnosis are not used only as dyes but can also be used in the field of medicines because of its have the biological activity against different type of bacteria and fungi and also found through the diagnosis that these compounds prepared have high melting point that lead to high expire date.

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