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RESEARCH ARTICLE

Synthesis of Salicylaldehyde Derivatives and Evaluating Its Antimicrobial, Antifungal, and Anticorrosion Activities

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ABSTRACT

Chalcones are natural, unsaturated aromatic ketones used in organic synthesis and medicine. Nevertheless, obtaining synthetic analogs and studying their reactions remains relevant. Unsaturated aromatic ketones are important substrates for synthesizing various heterocyclic compounds. The new chalcone, dihydropyrazole derivatives were synthesized based on 2-hydroxy-5-phenyldiazenyl benzaldehyde, and their structures were studied by NMR. Many metal structures fail each year due to harsh conditions, causing significant damage to the global economy. To overcome this problem, chemists are constantly researching and developing new corrosion inhibitors. The compounds under test contain nitrogen, oxygen, and sulfur atoms, which can be coordinated with metals. These types of compounds can have strong corrosion-inhibiting properties. Additionally, the inhibitory properties of the synthesized chalcones and dihydropyrazole derivatives against microorganisms were investigated, and the high antibacterial and antifungal effects of dihydropyrazole derivatives were detected. The results found that, compounds 2-hydroxy-5-[(*E*)-phenyldiazenyl]benzaldehyde (**1**), (2*E*)-3-{2-hydroxy-5-[(*E*)-phenyldiazenyl]phenyl}-1-phenylprop-2-en-1-one (**2**), (2*E*)-1-(4-bromophenyl)-3-{2-hydroxy-5-[(*E*)-phenyldiazenyl]phenyl}prop-2-en-1-one (**3**) showed lower inhibition properties against the tested microorganisms than commercial antibiotics. Compound 5-{2-hydroxy-5-[(*E*)-phenyldiazenyl]phenyl}-3-phenyl-4,5-dihydro-1*H*-pyrazole-1-carbothioamide (**4**) showed the same results as *Gentamicin* and *Amoxicillin* against *Staphylococcus aureus*, but compound 3-(4-bromophenyl)-5-{2-hydroxy-5-[(*E*)-phenyldiazenyl]phenyl}-4,5-dihydro-1*H*-pyrazole-1-carbothioamide (**5**) showed higher inhibition activity than *Posaconazole* and *Caspofungin* against *Candida albicans*. Compounds **2** and **4** demonstrated the best anti-corrosion effects among the tested compounds. The inhibitory effects of these compounds were 90 and 94%, respectively. The investigated compounds (**1–5**) may be successfully used as antimicrobial, antifungal, and anticorrosion agents in the future.

Keywords: Antibacterial, Antifungal, Chalcone, Pyrazole, Salicylic aldehyde

Introduction

As we know, polyfunctional phenol compounds are important in organic synthesis and widely used as antioxidants in food products, corrosion inhibitors in the chemical industry, pharmaceuticals in medicine, etc.^{1–3} Recently, high-molecular-weight compounds based on phenols had applications as photoresist compositions in various fields of modern microelectronics

and computer technology.^{4–6} Phenol derivatives with tert-butyl ester groups are used in electron beam lithography for high-resolution patterning.⁷ Salicylic aldehyde is also a phenol derivative and an important compound in many fields of science and technology.^{8–10} It is used in perfumery, cosmetic products, dyeing, and the preparation of medicinal preparations.^{11–13}

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The presented work was carried out taking into account the practical importance of derivatives of chalcone derivatives in various fields of modern organic synthesis, medicine, and industry.^{14–16} Thus, in the framework of ongoing studies in the chemistry of chalcones, we report the synthesis and assessment of the antimicrobial and anticorrosion properties of some chalcone and pyrazole derivatives from salicylic aldehyde.^{17–19} The obtained results demonstrated that the tested compounds (**1–5**) may be successfully used as antimicrobial, antifungal, and anticorrosion agents in the future.

Materials

All reactants were purchased from Merck and Fluka as chemically pure and used without further purification.

Synthesis

2-Hydroxy-5-[(E)-phenyldiazenyl]benzaldehyde (1). For the synthesis of the aldehyde, two chemical glasses were used. Stirring 0.83 g of aniline, 3 ml of HCl, 10 ml of distilled water, and 0.63 g of sodium nitrite were added to the first glass on ice. Then, 10 ml of distilled water was added to the second glass by mixing 10 ml of salicyl aldehyde, 10 ml of ethanol, 4.1 g of NaOAc, and 10 ml of distilled water to the second glass. The solution in the first glass was added to the second with intensive stirring for 2 hours. Small brown particles have formed as a result of the reaction. This reaction mixture was stored for one day. Then, it was filtered, washed with water, and left to dry. Three ethanol purifications of the resultant brown powder (yield 73%, $T_{mp} = 105^{\circ}\text{C}$) were performed.

^1H NMR spectrum (acetone- d_6 , δ , ppm., J_{HH} , Hz). 7.2 (d, 1H, arom., $^3J_{H-H} = 8.5\text{Hz}$), 7.6 (m, 3H, arom.), 7.9 (d, 2H, arom.), 8.2 (d, 1H, arom., $J = 8.5\text{Hz}$), 8.4 (s, 1H, arom.), 10.2 (s, 1H, ald.) and 11.4 (s, 1H, OH); ^{13}C NMR spektrum (acetone- d_6 , δ , m.h.). 118.3, 120.9, 122.6, 129.2, 129.3, 129.7, 131.1, 152.4, 162.3, 163.5 and 196.9.

(2E)-3-{2-hydroxy-5-[(E)-phenyldiazenyl]phenyl}-1-phenylprop-2-en-1-one (2). To synthesize chalcone (**2**), a mixture was prepared by dissolving 2.26 g of aldehyde (**1**) and 0.12 g of acetophenone in 10 ml of ethanol and 7 ml of DMSO. 5 ml of a 20% KOH solution was employed for the reaction. The reaction mixture was stirred at ambient temperature for 5 hours. After the completion of the process, the reaction mixture was carefully transferred into a container filled with ice and the formation of a

yellow precipitate was observed. Then, the solution was neutralized with hydrochloric acid, filtered, and purified by recrystallization from methanol (yield 68%, $T_{mp} = 168\text{--}170^{\circ}\text{C}$). ^1H NMR spectrum (acetone- d_6 , δ , ppm., J_{HH} , Hz). 7.2 (d, 1H, arom., $^3J_{H-H} = 8.5\text{Hz}$), 7.6–8.2 (m, 13H, arom., and $\text{CH}=\text{CH}$), 8.5 (s, 1H, arom.), 10.2 (s, 1H, OH); ^{13}C NMR spectrum (acetone- d_6 , δ , m.h.). 116.9, 122.4, 122.5, 122.6, 122.7, 124.7, 125.3, 128.4, 128.6, 129.2, 130.7, 132.7, 138.4, 138.6, 146.2, 152.6, 159.9, 189.3.

(2E)-1-(4-bromophenyl)-3-{2-hydroxy-5-[(E)-phenyldiazenyl]phenyl}prop-2-en-1-one (3) was obtained according to the compound **2** synthesis procedure (brown powder, yield 69%, $T_{mp} = 156\text{--}157^{\circ}\text{C}$). ^1H NMR spectrum (DMSO- d_6 , δ , ppm.). 7.1–8.1 (m, 13H, arom. and $\text{CH}=\text{CH}$), 8.5 (s, 1H, arom.), 10.3 (s, 1H, OH); ^{13}C NMR spektrum (DMSO- d_6 , δ , m.h.). 116.9, 122.4, 122.5, 122.6, 122.7, 124.7, 125.3, 128.4, 128.6, 129.2, 130.7, 132.7, 138.4, 138.6, 146.2, 152.6, 159.9, 189.3.

5-{2-Hydroxy-5-[(E)-phenyldiazenyl]phenyl}-3-phenyl-4,5-dihydro-1H-pyrazole-1-carbothioamide (4). 0.1 g of chalcone (**2**) and 0.1 g of thiosemicarbazide were dissolved in 25 ml of ethanol and 10 ml of DMSO. 5 ml of a 5% KOH solution was used as a catalyst and refluxed for 6 hours. After completion, the reaction mixture was poured into the ice, and a red precipitate formed. The medium was neutralized with hydrochloric acid and filtered. The pure reaction product was recrystallized from methanol (yield 78%, $T_{mp} = 195^{\circ}\text{C}$). ^1H NMR spectrum (DMSO- d_6 , δ , ppm., J_{HH} , Hz). 3.6 v 4.3 (d, 2H, CH_2 , $^3J_{H-H} = 4.9\text{Hz}$), 6.7 (d, 1H, CH, $^3J_{H-H} = 4.9\text{Hz}$), 7.5–8.9 (m, 13H, arom.), 11.2 (d, 2H, NH_2), 11.9 (s, 1H, OH); ^{13}C NMR spectrum: (DMSO- d_6 , δ , ppm.). 41.8, 59.8, 116.7, 117.6, 122.7, 122.9, 124.0, 124.7, 127.7, 129.3, 129.7, 131.2, 140.0, 146.0, 156.3, 158.4, 177.6.

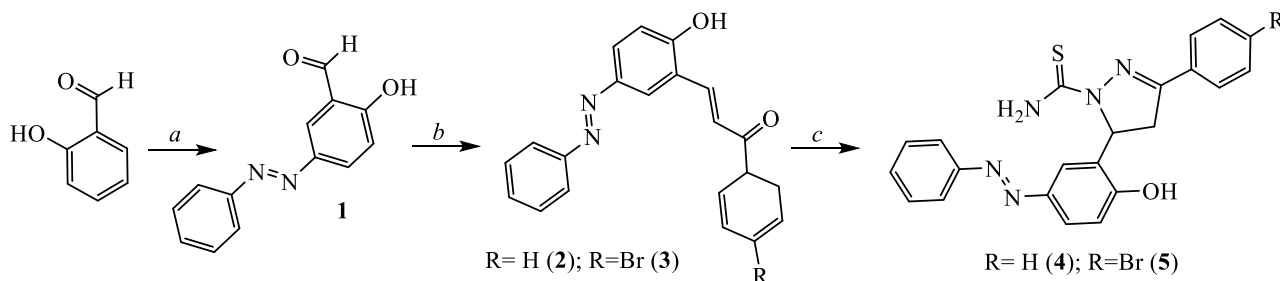
3-(4-Bromophenyl)-5-{2-hydroxy-5-[(E)-phenyldiazenyl]phenyl}-4,5-dihydro-1H-pyrazole-1-carbothioamide (5) was obtained according to the compound **4** synthesis procedure (yellow powder, yield 75%, $T_{mp} = 170^{\circ}\text{C}$). ^1H NMR spectrum (DMSO- d_6 , δ , ppm., J_{HH} , Hz). 3.6 v 4.3 (d, 2H, CH_2 , $^3J_{H-H} = 5.1\text{Hz}$), 6.6 (d, 1H, CH, $^3J_{H-H} = 5.1\text{Hz}$), 7.6–8.5 (m, 12H, arom.), 11.2 (d, 2H, NH_2), 11.9 (s, 1H, OH).

The synthesis route of the tested compounds is given below in Fig. 1.

Methods

NMR spectra

^1H NMR and ^{13}C NMR were obtained in acetone- d_6 and DMSO- d_6 using a Bruker Avance 300 NMR device



Reaction conditions; a) $\text{C}_6\text{H}_5\text{NH}_2$, NaNO_2 , HCl , r.t., 24 h.; b) $\text{C}_6\text{H}_5\text{COCH}_3$ or $4\text{-Br-C}_6\text{H}_4\text{COCH}_3$, $\text{C}_2\text{H}_5\text{OH}$, DMSO , KOH , r.t., 5 h.; c) $\text{NH}_2\text{NH}_2\text{CSNH}_2$, $\text{C}_2\text{H}_5\text{OH}$, DMSO , KOH , reflux., 6 h.;

Fig. 1. The synthesis route of the tested compounds.

(Germany, 300.13 and 75.47 MHz, respectively). The chemical shifts (δ) are given in ppm and referenced to the used solvents: 2.06 ppm (acetone-d_6) and 2.50 ppm (DMSO-d_6) for ^1H , 29.9 ppm (acetone-d_6) and 39.5 ppm (DMSO-d_6), respectively (coupling constants: J in Hz). The ^1H NMR and ^{13}C NMR spectra of tested substances are given in SI (Figs. S1–S9).

TLC (Silufol UV-254, eluent 1:3 hexane/ethyl acetate) tested the purity of the synthesized substances.

Testing against bacteria and fungi. Substances 1–5 were tested for their in vitro inhibition activities against microorganisms by the disc-diffusion method,^{20–22} which used Tryptone Soy Agar growth mediums. The reproducibility of the disc diffusion measurements was assessed by performing the tests in triplicate for each experimental condition. The evaluated solutions were diluted with DMSO (1 mg/ml compound concentrations). The chemical glasses with bacteria-fungi suspensions and disks of tested substances were incubated at 37°C for 24 hours. After incubation, the growth of the inhibition zone's size (or diameter) was measured. The DMSO alone was used as a control, and it was revealed that solvent does not influence antibacterial-antifungal properties (zone of inhibition was 1–1.5 mm).

In our investigations, we used differential microorganisms and culture media from “Liofil-chem” (Italy): *Staphylococcus aureus* (ATCC 99213), *Escherichia coli* (ATCC 25922), *Klebsiella pneumoniae* (ATCC 13883), *Salmonella enterica* (ATCC 13076), *Candida albicans* (ATCC 90028), and *Aspergillus niger* (ATCC 16404).

Gravimetric measurements. The aggressive corrosion medium was seawater with the following inhibitor concentrations: 0 (blank), 10, 30, and 60 mg/l. Gravimetric investigations are carried out in the presence (or absence) of tested compounds in 0.5L chemical glass with a mixing. The steel-3 specimens ($241\text{ mm} \times 191\text{ mm} \times 39\text{ mm}$ in size) were abraded and dried, then weighed on an electronic balance

with an accuracy of 0.00001 g. The weight losses of St-3 specimens in seawater media are calculated for 5 hours of testing time at 25°C .

Corrosion rate (C_R), inhibition efficiency (IE , $\eta\%$), coverage of surface (θ), and the value of free energy of adsorption (ΔG_{ads}^0) are estimated by the following Eqs. (1) to (4):

$$C_R = \frac{W}{St}; \quad \eta\% = \frac{C_R - C_{R(i)}}{C_R} \times 100 \quad (1)$$

$$\theta = \frac{C_R - C_{R(i)}}{C_R} \quad (2)$$

$$\frac{C_{\text{inh}}}{\theta} = \frac{1}{K_{\text{ads}}} + C_{\text{inh}} \quad (3)$$

$$\Delta G_{\text{ads}}^0 = -RT \ln (55.5K_{\text{ads}}) \quad (4)$$

C_R and $C_{R(i)}$ are the corrosion rate values with (or without) tested compounds, K_{ads} is the equilibrium constant of the adsorption-desorption process, and C_{inh} is the molar concentration of investigated substances.

Results and discussion

The literature demonstrated high antibacterial and antioxidant activities of different chalcone derivatives and Schiff bases.^{21–23} Considering it, the in vitro antibacterial activities of compounds 1–5 (1 mg/ml DMSO solutions) were evaluated against gram-positive and gram-negative bacteria and fungi using the cultures of different standard microorganisms: the inhibition properties of synthesized compounds 1–5 against *Staphylococcus aureus* (*S.aureus*), *Escherichia coli* (*E.coli*), *Klebsiella pneumoniae* (*K.pneumoniae*), *Salmonella enterica* (*S.enterica*) bacteria, *Candida albicans* (*C. albicans*), *Aspergillus niger* (*A.niger*) fungi

Table 1. The Antimicrobial activity of the compounds **1–5** against bacteria and fungi in DMSO solutions (1 mg/ml).






Bacteria and fungi	Zone of inhibition - mm										
	1	2	3	4	5	antibiotics				antifungals	
											
<i>S.aureus</i>	7	12	14	26	18	26	27	37	38		
<i>E.coli</i>	6	7	10	14	15	18	13	11	16		
<i>K.pneumoniae</i>	5	6	9	12	13	18	16	17	19		
<i>S.enterica</i>	7	8	9	13	12	14	12	10	8		
<i>C. albicans</i>	8	9	16	10	26					24	21
<i>A.niger</i>	7	8	12	9	11					24	28

Table 2. Evaluation of synthesized compounds as corrosion inhibitors in seawater.

C_{inh} , mg/l	1		2		3		4		5	
	CR*	IE*	CR	IE	CR	IE	CR	IE	CR	IE
10	$1,11 \cdot 10^{-5}$	40	$5,33 \cdot 10^{-5}$	71	$6,57 \cdot 10^{-5}$	64	$4,61 \cdot 10^{-5}$	75	$3,94 \cdot 10^{-5}$	78
30	$9,5 \cdot 10^{-5}$	48	$5,02 \cdot 10^{-5}$	73	$5,56 \cdot 10^{-5}$	70	$3,94 \cdot 10^{-5}$	78	$3,79 \cdot 10^{-5}$	79
60	$8,26 \cdot 10^{-5}$	55	$1,85 \cdot 10^{-5}$	90	$4,64 \cdot 10^{-5}$	75	$1,08 \cdot 10^{-5}$	94	$3,7 \cdot 10^{-5}$	80

Table 3. Thermodynamic parameters of adsorption on the steel-3 surface.

C_{inh}	$K_{ads} \times 10^{-3}$, l/mol	$-\Delta C_{ads}$, kJ/mol	C_{inh} , mg/l	$K_{ads} \times 10^{-3}$, l/mol	$-\Delta C_{ads}$, kJ/mol
(1) 10	22	34	(2) 10	44	41
30	68	37	30	9	32
60	145	38	60	37	35
(3) 10	75	37	(4) 10	108	38
30	41	35	30	64	36
60	46	36	60	92	37
(5) 10	4	30			
30	14	33			
60	37	35			

were studied. For the comparative characterization of compounds, we investigated the sensitivity of current medicinal antibiotics such as *Gentamicin*, *Amoxicillin*, *Cefazolin*, and *Tetracycline* against *S.aureus*, *E.coli*, *K.neumoniae*, *S.enterica*, *Posaconazole*, *Caspofungin* against *C.albicans*, *A.niger*. The obtained results are given in [Table 1](#).

As shown in [Table 1](#), compounds **1–3** showed lower inhibition properties against the indicated microorganisms than antibiotics currently on sale. Compound **4** showed the same results as *Gentamicin* and *Amoxicillin* against *S. aureus*, but compound **5** showed higher inhibition activity than *Posaconazole* and *Caspofungin* against *C. albicans*.

The presence of a hydroxyl group, Schiff base, unsaturated ketone fragments, and a pyrazole ring in molecules may connect the high antifungal activity of **1–5**.

The anti-corrosion (inhibitor) properties of the synthesized compounds for saving the Steel-3 sample in seawater were tested, and the obtained results are given in [Tables 2 and 3](#).

As can be seen from [Table 3](#), chemical interaction takes place on the steel surface. In other words, the synthesized compounds were adsorbed on the surface of the steel by chemical interaction, formed a stable complex, and were able to prevent corrosion.

We are currently continuing our investigation into the synthesis of new chalcone derivatives and testing their properties.

Conclusion

In this study, dihydropyrazole derivatives were synthesized from 2-hydroxy-5-phenyldiazenebenzaldehyde, and their structures were confirmed using NMR spectroscopy. The antimicrobial activities of these compounds were evaluated, revealing that derivatives **4** and **5** exhibited significant antibacterial and antifungal effects, particularly against *E. coli* and *C. albicans*. The enhanced activity of these compounds is attributed to the presence of the pyrazole ring, which is supported by the efficacy of related

medicinal drugs such as betazole and sulfaphenazole. Additionally, the anti-corrosion properties of these compounds were investigated. Results showed that compounds **2** and **4** provided substantial corrosion protection, with 90% and 94% inhibition efficiencies, respectively. This protection is likely due to chemical interactions between the compounds and the metal surfaces. Our findings underscore the potential of chalcones and their derivatives as valuable agents in both medicinal and industrial applications.

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Authors' declaration

- Conflicts of Interest: None.
- We hereby confirm that all the figures and tables in the manuscript are ours. Furthermore, figures and images that are not ours have been included with the necessary permission for re-publication, which is attached to the manuscript.
- No animal studies are present in the manuscript.
- No human studies are present in the manuscript.
- Ethical Clearance: The project was approved by the local ethical committee at university of Baku State University.

Authors' contribution statement

I.M. contributed to the design, the research implementation, the results analysis, and the manuscript's writing. A.K. contributed to the design, manuscript drafting, revision, and proofreading. All authors have read and agreed to the final draft of the manuscript.

Supplementary material

Supplementary material is available online at <https://doi.org/10.21123/2411-7986.4990>.

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التوليف وتقييم المواد المضادة للميكروبات ومكافحة التآكل لمشتقات الساليسيلك الديهايد

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الخلاصة

كما هو معروف، فإن الجالكونات أو الكيتونات العطرية غير المشبعة هي منتجات تكثيف كلايسن شميدت (Klaissen-Schmidt condensation) والتي لها أهمية عملية كبيرة في الطب والتركيب العضوي الحديث. تتواجد Chalcones على نطاق واسع في الطبيعة. ومع ذلك، لا يزال الحصول على النظائر الاصطناعية لهذه المركبات ودراسة تحولاتها الكيميائية في التفاعلات المختلفة أمراً صعباً. تجدر الإشارة إلى أن الكيتونات العطرية غير المشبعة هي ركائز قيمة في تركيب المركبات الحلقية غير المتجانسة. وكما نعلم أيضاً، تفشل كمية كبيرة من الإنشاءات المعدنية كل عام نتيجة للتأثيرات العدوانية والتي تسبب أضراراً جسيمة للاقتصاد العالمي. للتغلب على هذه المشكلة، يقوم الكيميائيون باستمرار بالبحث وتطوير مثبطات جديدة للتآكل. تم تصنيع مشتقات الجالكون الجديدة وثنائي هيدروبيرازول على أساس 2-هيدروكسي-5-فينيلديازينيل بنزالديهايد وتمت دراسة تركيباتها بواسطة جهاز الرنين المغناطيسي النووي. تم دراسة الخواص التثبيطية للجالكونات المصنعة ومشتقات الديهيدروبيرازول ضد الكائنات الحية الدقيقة وتم الكشف عن التأثيرات المضادة للبكتيريا والفطريات لمشتقات الديهيدروبيرازول. تحتوي المركبات التي تم اختبارها على ذرات النيتروجين والأكسجين والكبريت، والتي يمكن تنسيقها مع المعادن. يمكن أن يكون لهذه الأنواع من المركبات خصائص قوية تمنع التآكل.

بينت دراستنا امتلاك المركبات 1-3 خصائص تثبيطية ضد الكائنات الحية الدقيقة التي تم اختبارها بالمقارنة مع المضادات الحيوية المعروفة بالأسواق المحلية. أظهر المركب الرابع نفس النتائج التي أظهرها الجنتاميسين والأموكسيسيلين ضد بكتريا *S. aureus*، لكن المركب 5 أظهر نشاط تثبيطي أعلى من البوساكونازول والكاسوفونجين ضد خميرة *Candida albicans*. من بين المركبات التي تم اختبارها، تم إثبات أفضل التأثيرات المضادة للتآكل في 2 و 4. وكانت التأثيرات المثبطة لهذه المركبات 90 و 94٪ على التوالي.

الكلمات المفتاحية: الساليسيلك الديهايد، بايرازون، الجالكونات، المواد المضادة للبكتيرية، المواد المضادة الفطرية.