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كلية الآداب والعلوم قصر الأخيار

ELMERGIB UNIVERSITY

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# مجلة العلوم الإنسانية والتطبيقية

Journal of Humanitarian and Applied Sciences

مجلة دورية نصف سنوية محكمة

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### الإخراج والإشراف الفني

أ.أحمد عياد المنتصري



لا يسمح بإعادة إصدار محتويات المجلة أو نقلها أو نسخها بأي شكل من الأشكال دون

موافقة رئيس التحرير

إن كافة البحوث تعبر عن وجهة نظر أصحابها، ولا تعبر بالضرورة عن رأي المجلة أو الكلية

جميع الحقوق محفوظة



## قواعد النشر

حرصاً من هيئة التحرير على استخدام الأسلوب العلمي في كتابة البحوث والدراسات المراد نشرها، ينبغي اتباع القواعد التالية :

الغلاف ينبغي أن يحتوي على العنوان واسم الباحث ( الباحثين ) ، والدرجة العلمية وجهة العمل ، والدولة ، والبريد الإلكتروني ، وسنة النشر .

المتن يشتمل على ملخص للبحث ( عربي - إنجليزي ) يعكس لغة البحث لا يتجاوز ورقة واحدة.

تخضع البحوث المقدمة للنشر للتحكيم العلمي ، وهيئة التحرير أن تطلب من المؤلف بناء على اقتراح المحكمين بإجراء التعديلات المطلوبة على البحث قبل الموافقة على نشره .

ضوابط ومواصفات البحوث المقدمة للنشر:

1. أن يكون البحث أو الدراسة ضمن الموضوعات التي تختص بها المجلة .
2. ألا يكون البحث قد سبق نشره في إحدى المجلات أو مستلماً من أطروحة علمية أو يكون الباحث قد تناوله بعنوان آخر في وسيلة نشر أخرى ويوتق ذلك بتعهد خطي بهذا الخصوص .
3. فيما يخص البحوث العربية تكتب هوامش البحث وقائمة المراجع وفق دليل جمعية علم النفس الأمريكية **American Psychological Association (APA)** الطبعة الخامسة بالنسبة للبحوث العربية وتكون الطباعة على وجه واحد على ورق (A4) بخط (Traditional Arabic) بحجم (14) للنص مع ترك مسافة 1 بين السطور وتكون الهوامش 2.5 سم و مع ترك هامش 3 سم من جهة التجليد ،
4. فيما يخص البحوث باللغة الإنجليزية تكتب وفق نظام **Modern Language Association (MLA)** ، بحجم خط (12) بخط (Times New Roman) مع ترك مسافة 1 بين السطور مع وجود ملخص باللغة العربية في بداية البحث بحيث لا تزيد صفحات البحث 17 صفحة ي يكون التوثيق داخل المتن ( اللقب ، السنة ، الصفحة ) .
5. عنوان البحث يجب أن يكون مختصراً قدر الإمكان وأن يعبر عن هدف البحث بوضوح ويتبع المنهجية العلمية من حيث التناول والإحاطة بأسلوب بحثي علمي ، وأن لا تزيد ورقات البحث عن 25 صفحة بما في ذلك صفحات الجداول والصور والرسومات وغيرها .
6. يجب على الباحث التقييد بأصول البحث العلمي وقواعده من حيث أسلوب العرض والمصطلحات وتوثيق المصادر والمراجع في آخر البحث ، وهو المسئول بالكامل عن صحة النقل من المصادر والمراجع المستخدمة ، وهيئة التحرير غير مسئولة عن أي نقل خاطئ "سرقاات أدبية وعلمية " قد تحدث في تلك البحوث .
7. البحوث المقدمة للمجلة تخضع للتقييم من قبل متخصصين بشكل يضمن التقييم العلمي، ويتطلب من الباحث مراعاة سلامة بحثه من الأخطاء اللغوية والإملائية .
8. تلنزم المجلة بإشعار الباحث بقبول بحثه إن كان مقبولاً للنشر أو قابلاً للتعديل بعد التقييم على أن يرسل الباحث إذا قبل بحثه سيرة ذاتية ( CV ) مختصر قدر الإمكان يتضمن الاسم الثلاثي - والدرجة العلمية - والجامعة والكلية والقسم - وأهم المؤلفات إن وجدت - البريد الإلكتروني - والهاتف .

9. البحوث المقدمة للمجلة لا تعاد لأصحابها سواء نشرت أو لم تنشر ، وهي تعبر عن رأي أصحابها فهم المسئولون عنها أدبيا وقانونيا ولا يمثل بالضرورة رأي المجلة .
10. المجلة تنشر كل ما يتعلق بالجمال العلمي والبحثي وما يتعلق بالمؤتمرات والندوات والأنشطة الأكاديمية وملخصات الرسائل العلمية ونقد الكتب على أن لا تزيد عن خمس صفحات مطبوعة
11. إشعار الباحث بقبول بحثه وإرجاعه للتصحيح أو الإضافة أو التعديل على أن يقوم بتزويد المجلة بنسخة من البحث في صورته النهائية على قرص مدمج ( CD ) .
12. تعتبر البحوث قابلة للنشر من حيث صدور خطاب صلاحية النشر وتحال إلى الدور بانتظار الطبع حسب أولوية الدور وزخم الأبحاث الحالية للنشر .
13. يزود الباحث بنسخة من إعداد المجلة التي نشر بها بحثه .

هيئة تحرير المجلة

## افتتاحية العدد

### بِسْمِ اللَّهِ الرَّحْمَنِ الرَّحِيمِ

يسر هيئة محرر مجله العلوم الإنسانية و لاجتماعية و العلمية أن تقدم الى القراء الكرام العدد الثامن بعد ان يم تعديل اسمها الى مجله العلوم الإنسانية و التطبيقية بدلا من العنوان السابق بناء على ملاحظات القراء الكرام.

باي هذا العدد حافلا بمجموعة من البحوث و الدراسات المتنوعة في مجالس العلوم الإنسانية و التطبيقية أملس أن يجد القارئ الكريم في هذا العدد مبتغاه.

وفي إطار تطور المجله بعد ان بالت المجله الاعياد الدولي و الاعياد العرقي فإننا نعيد تذكير السادة الباحث و المهتمس بالبحث العلمي بسياسة المجله التي تعمل على تقديم أفضل البحوث و الدراسات وفق مهجية علمية و تقديم مادة مفيدة من أجل يجويد و يحسس الإنتاج العلمي بحيث تكون الدراسات و البحوث تتناول موضوعات شتى في مجتلف ميادس المعرفة سواء في مجال العلوم الإنسانية أو التطبيقية.

كما نذكر السادة الباحث فإن المجله تفتح أبوابها لاستقبال المزيد من الإنتاج العلمي الرصص سواء على المستوي المحلي أو العرقي أو الدولي، وفي الوقت نفسه نعتذر للسادة الباحث النس قدموا بحويهم ولم ييم استكمال تقييمها نظرا للظروف التي يمر بها البلاد فإننا سننير الصالح منها في الاعداد القادمة بعون الله تعالى.

والله ولي التوفيق

## Synthesis and Study on Biological Effect of The (1*E*,4*E*)-1,5-bis (4-nitrophenyl) penta-1,4-dien-3-one

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### Abstract

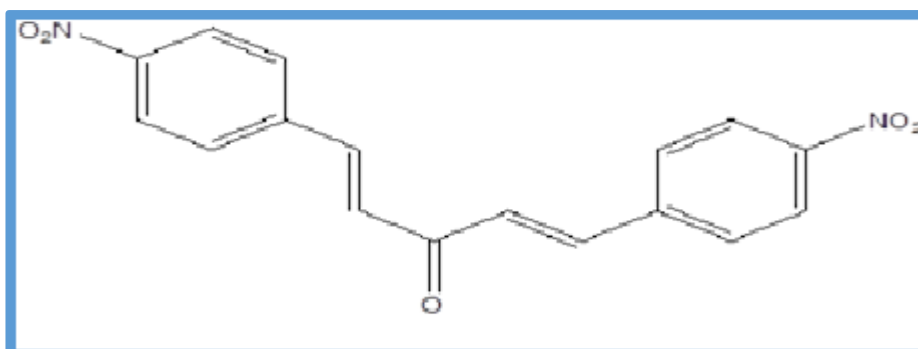
The (1*E*,4*E*)-1,5-bis (4-nitrophenyl) penta-1,4-dien-3-one (3) was synthesized by treatment of acetone with 4-nitrobenzaldehyde using 1:2 mol. respectively in the presence of basic medium of sodium hydroxide in a good yield. The structure of the prepared compound was characterized by elemental analysis, IR, <sup>1</sup>H-NMR, and mass spectra. The newly prepared compound was screened for the antibacterial activity against *Escherichia coli* (G-) and *Staphylococcus aureus* (G+) bacterial strains by the disc diffusion method, and for their antifungal activity against *Aspergillus flavus* and *Candida albicans* in N,N-dimethylformamide by the agar diffusion method. The results of the test compound exhibited low cadre of antibacterial and antifungal activities compared with the standard drug Amikacin, therefore, minimum bactericidal concentration (MBC) and minimum fungicidal concentration (MFC) were not determined for the test compound.

**Keywords:** Chalcones; Claisen-Schmidt reaction; antibacterial activity; disc diffusion method; agar diffusion method.

### Introduction

Chalcones (1,3-diaryl-2-propen-1-ones) are popular intermediates for synthesizing various heterocyclic compounds.<sup>1</sup> The compounds with the backbone of chalcones have been reported to possess various biological activities such as antimicrobial, anti-inflammatory, analgesic, antiplatelet, antiulcerative, antimalarial, anticancer, antiviral, antileishmanial, antioxidant, antitubercular, antihyperglycemic, immunomodulatory, inhibition of chemical mediators release, inhibition of leukotriene B<sub>4</sub>, inhibition of tyrokinases and inhibition of aldosereductase activities.<sup>1</sup> The presence of a reactive α,β-unsaturated keto function in chalcones is found to be responsible for their biological activities.<sup>1</sup> There are varieties of methods available for preparation of chalcones. The classical Claisen-Schmidt reaction was chosen to produce chalcones derivatives with elongated conjugated linker between the aromatic rings using 4-nitrobenzaldehyde and acetone carried out in presence of aqueous sodium hydroxide (Figure 1). Due to the reversible nature of Claisen-Schmidt condensation

reaction, the reactants were taken in excess of stoichiometric proportion in order to increase the  $\alpha,\beta$ -unsaturated product. Antimicrobial tests were examined to the synthesized compound to observe the antibacterial and antifungal actions of the synthesized compound.



**Figure 1 :Chemical structure of (1E,4E)-1,5-bis(4-nitrophenyl) penta-1,4-dien-3-one.**

$\alpha,\beta$ -Unsaturated ketones, including dimethylidene acetone derivatives, are not only important building blocks in organic synthesis, but also key chemicals in many fields including perfumery, biochemistry, agriculture, food chemistry, polymer and material science, and others<sup>2-5</sup>. Therefore, the synthesis of these compounds is of great importance in both academic and industrial circles. Among reported works, Claisen-Schmidt condensation appears to be the most practical method to prepare  $\alpha,\beta$ -unsaturated ketones owing to its directness, clean procedures and accessible starting materials. Despite being discovered over 100 years ago, the enthusiasm for Claisen-Schmidt condensations never reduces and in recent years, a series of novel catalysts have been developed for this reaction, such as solid bases<sup>6,7</sup>, nano catalysts<sup>8,9</sup>, ionic liquid catalysts<sup>10</sup>, fluoros based catalysts<sup>11,12</sup>, metal-organic frame works (MOFs)<sup>13</sup> and organocatalysts<sup>14,15</sup>. Nevertheless, cheap and abundant NaOH would be expected to be the most common catalyst for the reaction due to its availability in laboratory, and indeed this method is still widely employed up to the present<sup>16,17,18</sup>. But reactions performed in strong alkaline conditions are corrosive to equipment and generate unmanageable and corrosive solid waste. These drawbacks have limited the large-scale application of NaOH. Moreover, methods for the synthesis of dimethylidene acetone derivatives, especially for those dissymmetrically substituted compounds, have not been well documented yet. Thus, developing novel alternative synthetic methodologies with broad scope using mild and common base catalysts is not only desirable but timely for the field.

Calcium hydroxide is also a readily accessible base and compared with NaOH, it is much cheaper and less alkaline. Moreover,  $\text{Ca}(\text{OH})_2$  is easily neutralized and precipitated by  $\text{CO}_2$ , which is beneficial from the point of industrial use. However,

despite several well-known applications in industrial production, examples of the employment of  $\text{Ca}(\text{OH})_2$  as a base catalyst in organic synthesis are rare<sup>19</sup>. In some previous worked out research projects with industries in development of green synthetic methodologies<sup>20-29</sup>, an organoselenium-catalyzed green oxidation of  $\alpha,\beta$ -unsaturated ketones was used to prepare vinyl esters which serve as versatile copolymers in material science<sup>25</sup>. To facilitate industrial application, a green and practical synthesis of  $\alpha,\beta$ -unsaturated ketones (the starting material for vinyl ester synthesis) was desired. To that end,  $\text{Ca}(\text{OH})_2$ -catalyzed Claisen-Schmidt condensation was applied to prepare  $\alpha,\beta$ -unsaturated ketones. During this work, dilute aqueous ethanol was unexpectedly found to be the optimal solvent and calcium could be precipitated by  $\text{CO}_2$  and removed by filtration to afford high purity products after solvent evaporation. The method allows comprehensive access to versatile  $\alpha,\beta$ -unsaturated ketones, including the challenging dissymmetrically substituted dimethylidene acetone derivatives. Herein, we wish to report our findings.

The above findings and reports prompted us to synthesize the new chalcone derivative using Claisen-Schmidt condensation reaction between appropriate molar quantities of suitable reactants in basic medium, and its antimicrobiological screening in view of search of better antimicrobiological agent. This study was aimed to synthesize a new chalcone derivative to investigate its antimicrobiological activity. The pertinent literature revealed that chalcone derivatives have shown numerous biological activities of significant levels. Therefore, this study may be useful and provide us a new compound of chalcone series as a novel microbiological agent against bacterial and fungal stains. Further, new addition of various substituents in the nucleus of chalcone derivative might be useful in view to investigate biological active compounds showing higher order of potency.

## 2. Experimental

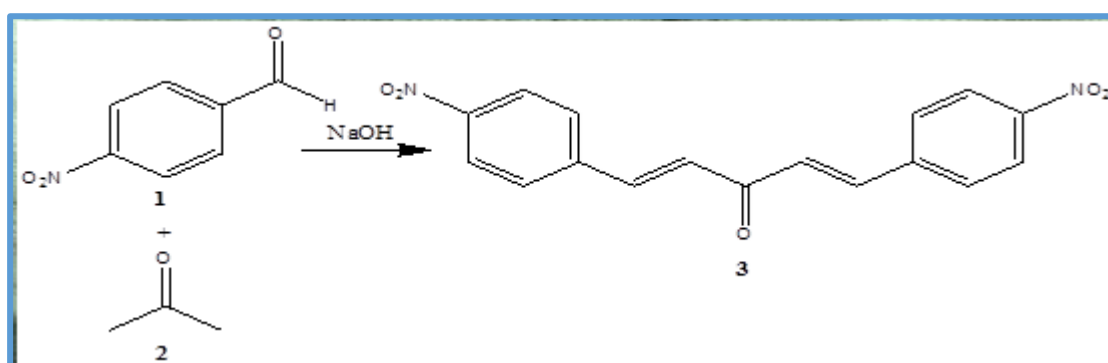
### 2.1. Chemicals and Instrumentation

All chemicals were used of high purity and analytical grade (AR) for the synthesis of compound ((1*E*,4*E*)-1,5-bis(4-nitrophenyl) penta-1,4-dien-3-one). The 4-nitrobenzaldehyde and acetone were purchased from Aldrich Co. (Germany). Melting point was recorded on an Electro thermal 9100 melting point apparatus. IR was run on a pye – Unicam SP – 1100 Spectrophotometer using KBr disc. <sup>1</sup>H NMR spectra was recorded on a Jeol GLM Ex 300 MHz FTNMR Spectrophotometer in DMSO *d*<sub>6</sub> solvent using TMS as internal standard, (chemical shift in  $\sigma$  ppm). Mass spectra was recorded in 70 eV varian MAT 300A. Elemental analysis was performed on Perkin Elmer Model 240 C Analyser. The purity of the compound was monitored on precoated TLC plates and visualizing the spots under UV light lamp.

### 2.2. Synthesis of Compound ((1*E*, 4*E*)-1,5-bis(4-nitrophenyl) penta -1,4-dien-3-one)



The compound ((1*E*, 4*E*)-1,5-bis(4-nitrophenyl) penta-1,4-dien-3-one) was prepared by treatment of acetone with 4-nitrobenzaldehyde in presence of basic medium of sodium hydroxide. 60 ml ethanol was taken in around bottom flask fixed with an air condenser followed by the addition of 80 ml sodium hydroxide (10 % w / v), 4.0 ml distilled acetone and 8.0 ml 4-nitrobenzaldehyde. The mixture was boiled gently for 5 minutes with shaking frequently. The product was cooled, filtered under suction pump, washed thoroughly with cold water, drained well and dried in air. The product was crystallized from ethanol.



**Scheme 1. Chalcone derivative preparation**

### 2.3. Biological Activity

The compound (3) was screened for *in vitro* antimicrobial activity. Antibacterial activity of the compound was measured by using disc diffusion method<sup>30</sup> against two bacterial stains, staphylococcus aureas and Escherichia coli. Antifungal activity of the compound was evaluated by using poisoned food techniqu<sup>32</sup> against two fungal stains, Aspergillus flavus and Candida albicans. The test compound was dissolved in *N,N*-dimethyl formamide (DMF) to get 1mg ml<sup>-1</sup> concentration of stock solution. The inhibition zones were measured in millimeter (mm) at the end of an incubation period of 48 hrs. at (35±2°C). DMF solvent showed no inhibition activity. Nutrient Agar (NA) and potato dextrose sugar (PDA) were used as basal media to test the bacterial and fungal growth inhibition activity, respectively. The results were compared with standard commercial antibacterial and antifungal drug under similar test conditions for comparison<sup>31, 32</sup>. Graded amounts of test compound was incorporated into measured amount of media used and the minimum inhibitory concentration (MIC) values of the test compound which completely inhibited the growth of bacteria / fungi were noted.

## 3. Results and Discussion

### 3.1. Infrared, <sup>1</sup>H NMR and mass spectra of ((1*E*, 4*E*)-1,5-bis (4-nitrophenyl) penta-1,4-dien-3-one):

The structure of the synthesized compound was characterized by physical, analytical and spectral data, which are given in Tables 1 and 2.

**Table 1. Physical data of compound (3)**

Compound No.	M.p. °C Colour	Solvent (Yield)	MF (M wt.)	Elemental Analysis(%) Calculated / Observed		
				C	H	N
3	158 -160	Ethanol	C <sub>17</sub> H <sub>12</sub> N <sub>2</sub> O <sub>5</sub>	62.96	3.73	8.64
	Brown	94	324.29	62.90	3.68	0.57

**Table 2. IR, and <sup>1</sup>H NMR Spectroscopic data for compound (3)**

Compound No.	IR (KBr) $\nu$ cm <sup>-1</sup>	<sup>1</sup> H NMR $\delta$ (ppm)
3	1723 (C = O) 1562 (C = C)	7.56 (d, CH = CH, J = 8.7 Hz) 6.98 (m, Phenyl Protons)

The infrared spectrum (IR) of compound (3) ( Table 2 ) exhibited a strong stretching frequency band for the carbonyl group at 1723 cm<sup>-1</sup> and two strong bands at 1562 and 1608 cm<sup>-1</sup> due to (C=C) of aromatic rings, and conjugated system (-C=C-CO-C=C-) of  $\alpha,\beta$ -unsaturated carbonyl respectively.

The <sup>1</sup>H NMR spectrum of compound (3) in deuterated DMSO-d<sub>6</sub> (Table 2) showed a doublet signal at 7.56 ppm due to (-CH=CH, J=8.7 Hz), as well as multiplets in range 6.98 – 7.25 ppm due to phenyl protons. The mass spectrum of compound (3) showed the following peaks of (m/z) values followed by (%) of relative abundances: [M-3] = 321 (12%), 279 (10%), 232 (10%), 176 (74%), 102 (52%), 63 (100%).

The results of the spectral data of the synthesized compound (3) are consistent with the spectral data of similar compounds of previous studies.<sup>33, 34</sup> Thus, the elemental analysis and spectral data of the synthesized compound fully support its final structure as (1E, 4E) -1,5- bis(4- nitrophenyl) penta-1,4-dien-3-one.

### 3.2. Antimicrobial activity

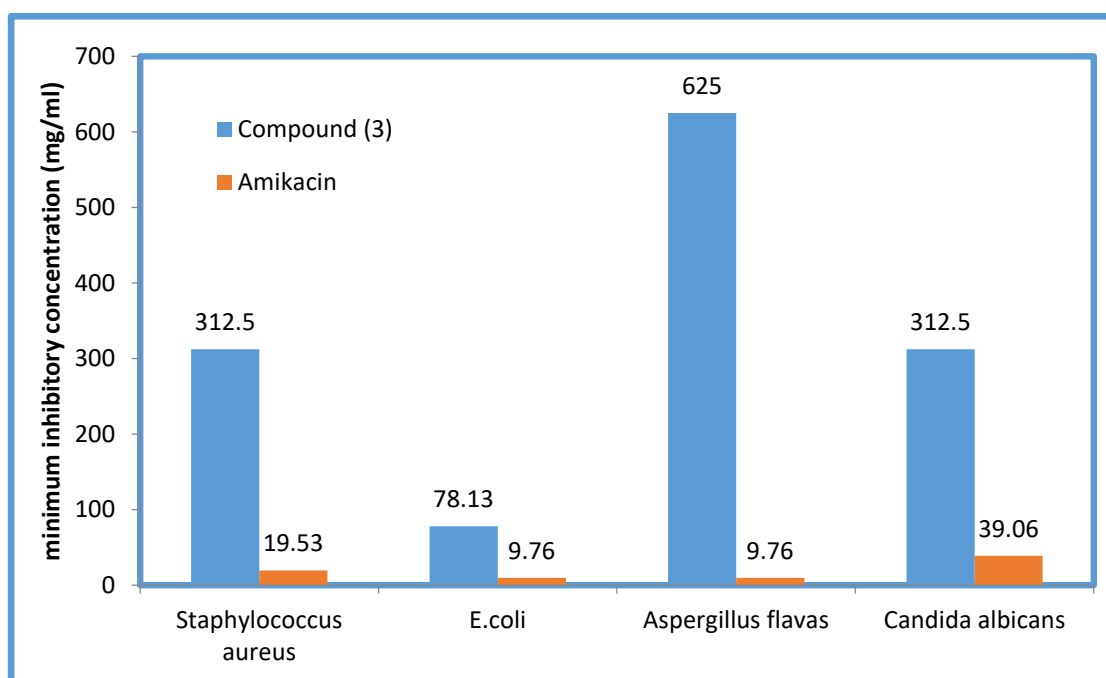
The results of antimicrobial activity of the test compound are presented in Table 3 and 4.

**Table 3. Antimicrobial activity data of compound (3) (Inhibition zones, mm), Amikacin was used as standard.**

Sample	Staphylococcus Aureas	Escherichia Coli	Aspergillus flavas	Candida albicans
Compound (3)	15	30	22	28
Amikacin	18	29	24	31

**Table 4. Antimicrobial activity data of compound (3) (MIC, mg/ml), Amikacin was used as standard.**

Samples	Minimum Inhibitory Concentration(MIC) mg/ml			
	Staphylococcus aureus	E.coli	Aspergillus flavas	Candida albicans
Compound (3)	312.5	78.13	625	312.5
Amikacin	19.53	9.76	9.76	39.06



**Figure 2. Minimum Inhibitory Concentration (MIC)**

Table 3 shows the antibacterial activity data of the test compound and standard drug Amikacin. Results data indicate that test compound has less antibacterial action (inhibition zone: 15 mm) than Standard drug (inhibition zone: 18 mm) against *Staphylococcus aureus* bacterial stain, however, slightly more antibacterial activity was found in test compound (inhibition zone: 30 mm) than standard drug (inhibition zone : 29 mm) against *Escherichia coli* bacterial stains.

Table 3 also represents the antifungal activity data of the test compound (3) and standard drug, Amikacin. It is apparent from the results that test compound (3) has lesser antifungal activity than the standard drug, Amikacin against both fungal stains (test drug inhibition zone: 22 mm and 28 mm and standard drug inhibition zone: 24 mm and 31 mm against *Aspergillus* and *Candida albicans* fungal stains, respectively).

Table 4 and Figure 2 shows the comparative antibacterial and antifungal activity data of the test compound and standard drug Amikacin given in minimum inhibitory concentration (MIC).

The comparative analysis of the antibacterial activity data shows that Minimum inhibition concentration of test compound is about 16 times and 8 times more than the standard drug against *Staphylococcus aureus* and *E. coli* bacterial stains. This indicates that test compound is more effective against inhibition of growth of *E. coli* than *Staphylococcus aureus*, lowerg, the minimum inhibitory concentration of test compound was 8 times higher to standard drug.

The comparative analysis of the results of antifungal activity of the test compound and standard drug Amikacin indicates that test compound has antifungal activity but requires 8 times and 64 times more minimum inhibitory concentration than standard drug against *Candida albicans* and *Aspergillus flavus* fungal stains. Results show that test compound is 8 times more potent against *Candida albicans* than *Aspergillus flavus* fungal stain. Thus, it is apparent from the antimicrobial screening of test compound (3) that it has good antibacterial and antifungal activities against pathogenic microbes almost similar to the standard drug but potency of the test compound (3) is many fold lower than the standard drug. Standard drug is able to produce antimicrobial activity in very low concentration than test compound (3). It is important to mention here that minimum bactericidal concentration (MBC) and minimum fungicidal concentration (MFC) of the test compound were not determined because the test compound exhibited very low cadre of potency in comparison to standard drug Amikacin.

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