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Synthesis and Characterization of New Acrylamide Derivatives and Studying Their Antibacterial Activity

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RESEARCH ARTICLE

Synthesis and Characterization of New Acrylamide Derivatives and Studying Their Antibacterial Activity

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ABSTRACT

This study aims to test the new derivatives of polymers on the biological activities of two types of bacteria work describes the synthesis of 1,3-oxazepine and self-polymerization via a cycloaddition reaction between Schiff's bases 2-methyl-N-(5-(2-methylbutanoylimino) pentylidene)but-2-enamide and succinic, maleic anhydride. The synthesized compounds were characterized by melting points, softening points, and physical properties such as swelling capacity, TGA, and FT-IR. The biological activity showed intermediate results against two bacterial isolates resistant to antibiotics.

Keywords: Acrylamide, Antibacterial activity, Heterocyclic compounds, Oxazepine, Schiff s bases

Introduction

Acrylamide has been distinguished as a poisonous material that induces carcinogenic, reproductive, and genotoxic influences on mammalian cells. Recently, significant work has been consecrated to developing specific programs to distinguish acrylamide in the environment and drinking water. As claimed by the EU report,[1] it is determined that the total acrylamide production capacity in the EU is between 80,000–100,000 mg/year, and the cumulative amount of acrylamide from all known sources discharged toward watercourses is 0.28 mg/day. Acrylamide is the principal component of polyacrylamides, non-poisonous polymers that have an extensive field of employment in raw oil production processes, wastewater and drinking water treatment, paper and textile production, and treatment for preserving soil degradation, cement, and mineral processing, production of colors and cosmetic supplements and other diverse uses - photographic emulsion, coatings, and adhesives, and poly(acrylamide). A water-soluble synthetic polymer, has been significant in many applications, such as medical productions, textile coating, and building industries.[1,2] Polyacrylamides are combined with insecticides as condensation agents.[3] Hugo Schiff declared Schiff's bases in the middle of the eighteenth century.[4] They are an essential discussion of organic composites attributed to a broad spectrum of biological activities, like ant tubercular,[5] antiviral,[6] proliferative,[7] antimalarial,[8] antifungal,[9] and antibacterial.[10] The general structural characteristic of the mentioned composites is the azomethine group with the general formula where R and R^1 are aryl, alkyl, cycloalkyl, or heterocyclic-group.[11] Heterocyclic composites are shown as building blocks of some biological molecules;[12] they usually hold five- and seven-membered rings.[13–15] The Schiff bases were developed by Schiff for the initial time[16] from condensation-response to either ketones or aldehyde with primary amines through refluxing the compound in absolute ethanol, benzene, or by any different appropriate solution for one hour or more; at times, the reaction may be stimulated with acid.[17,18] Oxazepine is an unsaturated non-symmetric seven-membered

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heterocyclic that includes O and N atoms in the first and third positions, respectively. The pericyclic cycloaddition of Schiff bases with phthalic imide, succinic imide, and 3-nitophthalic anhydride[19] develops this ring. The significance of 1,3-oxazepine is attributed to its purpose as an anti-spasm. For instance, numerous heterocyclic composites, such as oxazepine derivatives,[20] including oxygen and nitrogen atoms, were prepared to utilize the Schiff base process.[20,21] Biological activities like anticonvulsant and antibacterial activity showed oxazepine derivatives.[22,23]

Materials and methods

Polymerization of acrylamide[24]

In a screw-capped polymerization bottle (3 g., 0.041 moles), acrylamide was dissolved in 15 ml of ethanol, and 0.05% of ammonium persulphate was added as an initiator. The bottle was flashed with nitrogen for a few minutes inside a glove box and firmly stopped. The solution was maintained at 70°C using a water bath. After 1 hour, the solvent was evaporated under vacuum; the product was washed three times with diethyl ether and dried in a vacuum oven at 50°C, yielding 90%, Scheme 1 shows synthesis of Schiff base and self-polymerization.

Preparation of Schiff base 2-methyl-N-(5-(2-methylbutanoylimino) pentylidene)but-2-enamide[25]

A solution of acrylamide (1.0 g, 14 mmol) in absolute ethanol (20 ml) was added to glutaraldehyde (70 mg, 7 mmol). After adding glacial acetic acid (4–5 drops), the mixture was refluxed for 8 hours. The resulting mixture was then cooled slowly, and the precipitate was filtered, collected, and recrystallized from absolute ethanol. Table 1 shows some physical characteristics, Scheme 1 shows the synthesis of Schiff base, and self-polymerization, preparation of Schiff baseN,N'-(ethane-1,2-diylidene)bis(2-methylbutanamide).

Preparation of Schiff base N-(4-methoxybenzylidene)-2-methylbutanamide

A solution of acrylamide (1.0 g, 14 mmol) in absolute ethanol (20 ml) was added to 4-methoxy benzaldehyde (1.9 g, 14 mmol). After adding glacial acetic acid (4–5 drops), the mixture was refluxed for 8 hours. The resulting mixture was then cooled slowly, and the precipitate was filtered, collected, and recrystallized from absolute ethanol. Table 1 shows some of the physical characteristics, Scheme 1 shows the synthesis of Schiff base and self-polymerization.

Table 1. Physical properties of the starting material and the synthetic compounds.

Compound number	Color	m.p. °C or s.p. °C	Yield %	Swelling capacity
S	White	84.5	–	–
1	Off white	132–134	92	88.94
2	Off white	128-130	93.3	60.71
3	Pale yellow	122–125	52.1	92.38
4	Yellow	110–112	72.94	93.33
5	Pale yellow	100–102	70.52	91.85
6	Pale orange	118–120	54.69	88.09
7	Pale orange	138–140	80.2	90

mp: melting point; sp: softing point.

Preparation of Schiff bases N-(2-hydroxybenzylidene)-2-methylbutanamide and N-(4-(dimethylamino)benzylidene)-2-methylbutanamide 4 to access these two compounds, a similar procedure to that used for Schiff base N-(4-methoxybenzylidene)-2-methylbutanamide synthesis has been employed.

Preparation of oxazepine compounds 6 and 7[26,27]

Synthesis of compound 5,5′,6,6′-tetramethyl-3,3′-bis(2-methylbutanoyl)-2,2′-bi(1,3-oxazepane)-4,4′,7,7′-tetraone(6)

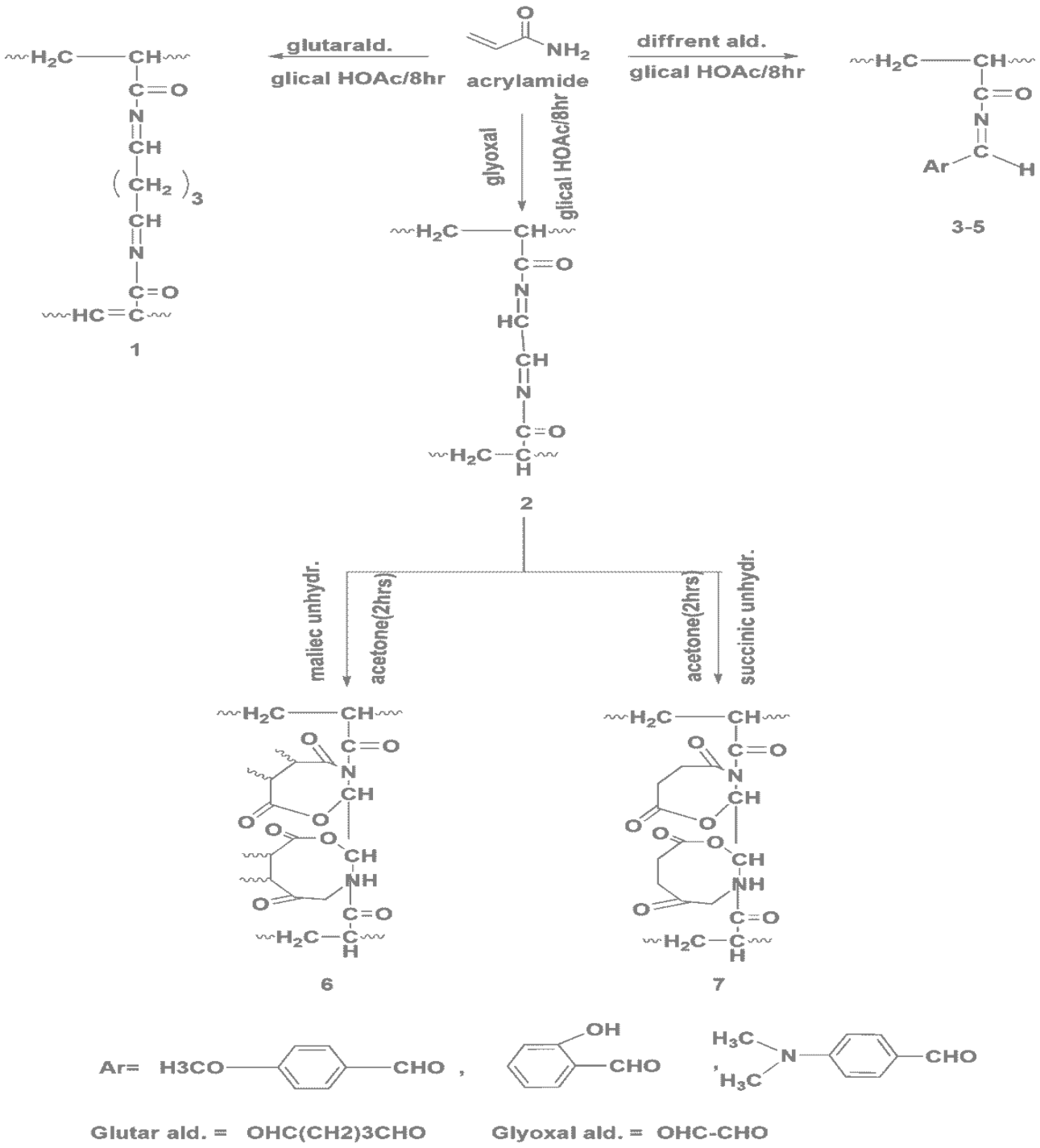
A solution of sodium acetate (0.2 g, 0.002 mol) and maleic anhydride (0.5 g .0.006 mol) were dissolved in acetone (25 ml). The resulting solution was added to (0.5, 0.003 mol) Schiff base 2. The reaction mixture was refluxed for 2 hours before cooling to room temperature. The solid-crude material was filtrated. Table 1 shows some of the physical characteristics, and Scheme 1 shows synthesis of Schiff base and self-polymerization.

Preparation of compound 3,3′-bis(2-methylbutanoyl)-2,2′-bi(1,3-oxazepane)-4,4′,7,7′-tetraone(7)

To access these two compounds, a similar procedure to that used for 5,5′,6,6′ -tetramethyl-3,3′-bis(2-methylbutanoyl)-2,2′-bi(1,3-oxazepane)-4,4′,7,7′-tetra one synthesis was employed.

The antibacterial activity[28–30]

The agar diffusion approach determined the antibacterial activity of these compounds 1,2 and 3. Utilizing *Staphylococcus aureus* (G−) and *Pseudomonas* (G+), 5 mM of these compounds were seated on an agar planted with the test organism. The plate was incubated at a suitable temperature 37 °C for 24 hours.



Scheme 1. Synthesis of Schiff base and Self Polymerization.

Results and discussion

This work included the preparation of new polyacrylamide derivatives of Schiff bases **1–5** and then internal polymerization to give a new heterocyclic polymer. Regarding compounds **6** and **7**, there were two steps for synthesizing: the first step from two moles of the acrylamide with one mole of the glyoxal in the existence of ethanol as a solvent to afford Schiff base **2**. The FT-IR spectral data of compounds **1** and **2** revealed absorption bands at 2813 and 2812 cm^{-1} for C-H aliphatic, and absorptions at 1676, 1674, and 1612 cm^{-1} are attributed to C=O amide, C=N groups, Fig. 1.

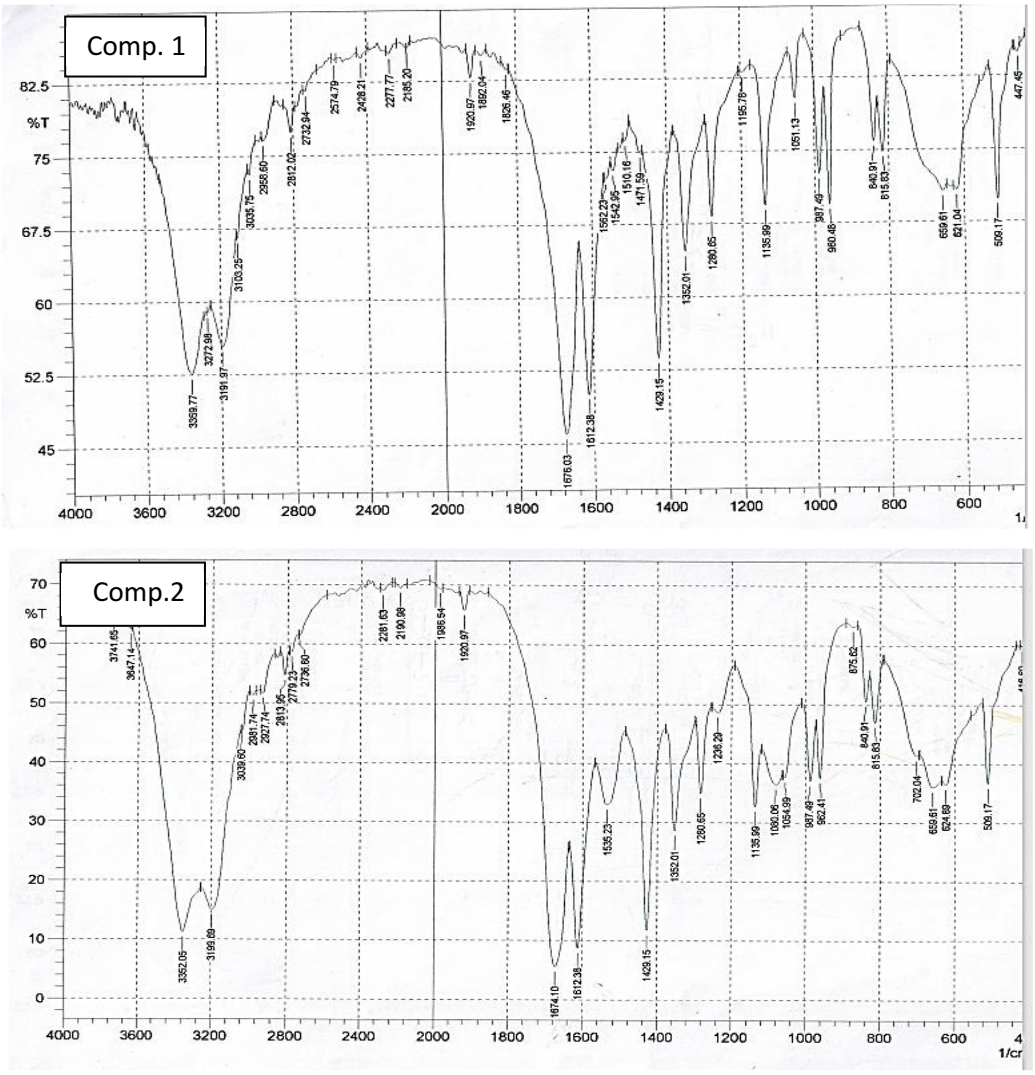


Fig. 1. Fourier transfer Infra-Red spectra of compounds C1 and C2.

In the second step, the oxazepine compounds **6** and **7** were prepared by the maleic and succinic anhydride reaction with Schiff base **2** in the presence of anhydrous sodium acetate. The structure of compounds **6** and **7** was proved by FT-IR spectroscopy. The FT-IR spectra of compounds **6** and **7** revealed absorption bands at 2999 and 2933 cm^{-1} for C-H aliphatic. The absorption band at 1612 cm^{-1} for C=N imine of compound **2** disappeared, indicating the cyclization reaction. In addition, the absorption band at 1708 cm^{-1} is for C=O ester, indicating cyclization, Fig. 2.

The other compounds (hydrozones) **3**, **4,** and **5** were prepared *by reacting* several aromatic aldehydes with acrylamide. The FT-IR spectra of compounds **3**, **4,** and **5** showed absorptions at 1610, 1612, and 1600 cm^{-1} are attributed to the C=N group, Fig. 4. Also, the absorptions at 1512, 1521, and 1550 cm^{-1} are attributed to C=C for aromatic rings. In addition, the absorption of C=O amide appeared in 1676, 1672, and 1668 cm^{-1} for the oxazepine cyclic cycle.[28]

Thermal analysis

Thermogravimetric analysis of compound **1**, Fig. 4, displayed four weight-loss steps. The first step, at 198 °C, is attributed to the water evaporation in the compound: the weight was reduced by 97.84%. At the temperature of 248 °C, the second step is attributed to the decomposition of acrylamide bonds with glutaraldehyde and the decomposition of part of glutaraldehyde with a weight loss of 58.85%. The weight percentage was relatively small, representing the proportion of the reacted glutaraldehyde.

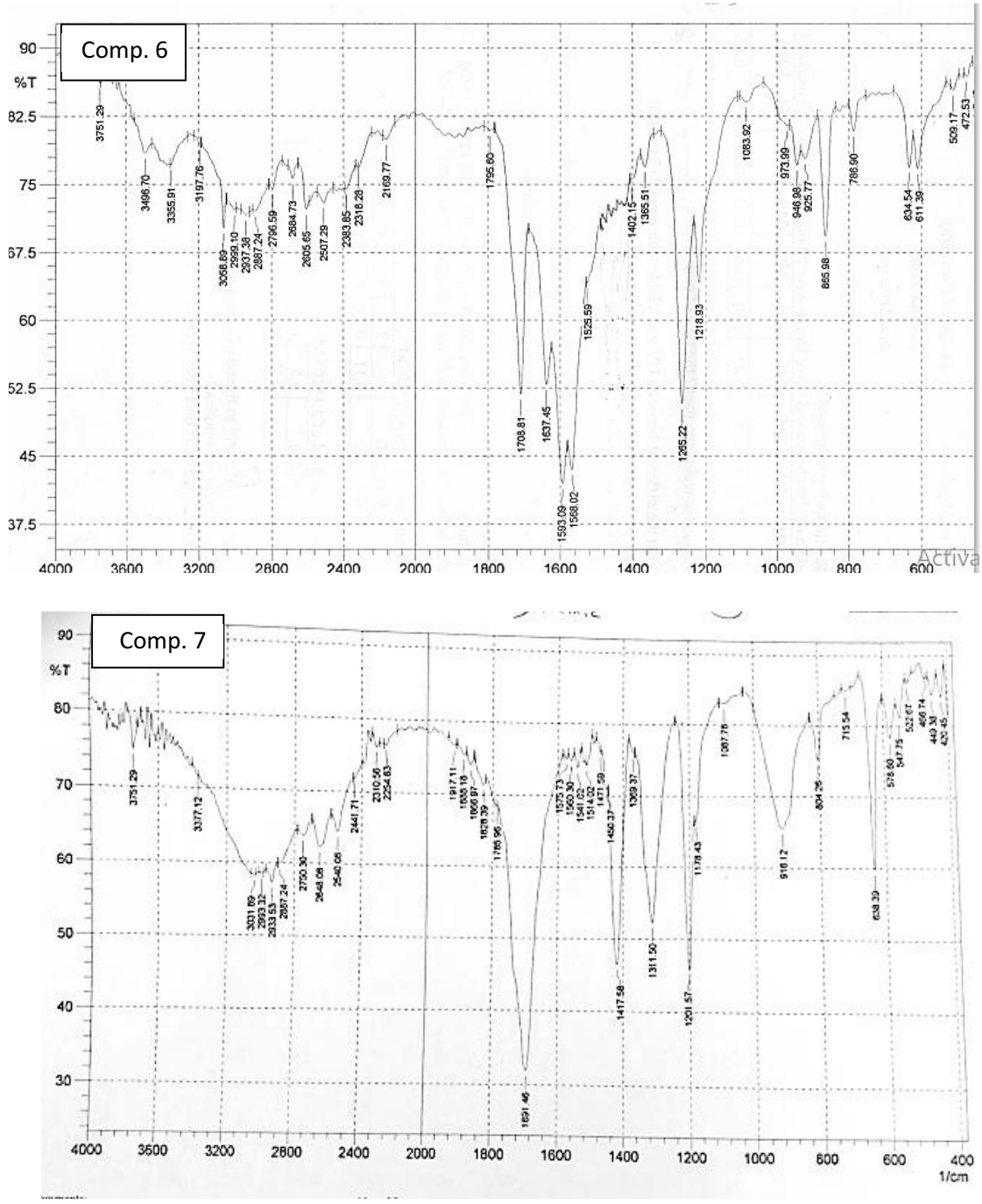


Fig. 2. Fourier transfer Infra-Red spectra of compounds C6 and C7.

The third step showed a loss of 50.01% at 300 °C, which is due to the decomposition of acrylamide bonds forming and the residual glutaraldehyde structure. Finally, the fourth step, which showed a weight loss of 15.76% at 899 °C, refers to the continued degradation of Acrylamide and the release of carbon dioxide (CO_2) and the residual weight of carbon waste.

Thermogravimetric analysis of compound **7** Fig. 5 represented three weight loss steps. The first step, at 190.1 °C, is attributed to the water evaporation in the compound: the weight was reduced by 93.06%. At the temperature of 290.7 °C, the second step belongs to the decomposition of Schiff base **2** bonds with compound **7** and the part decomposition of compound **7** with a weighting loss of 11.29%. The weight

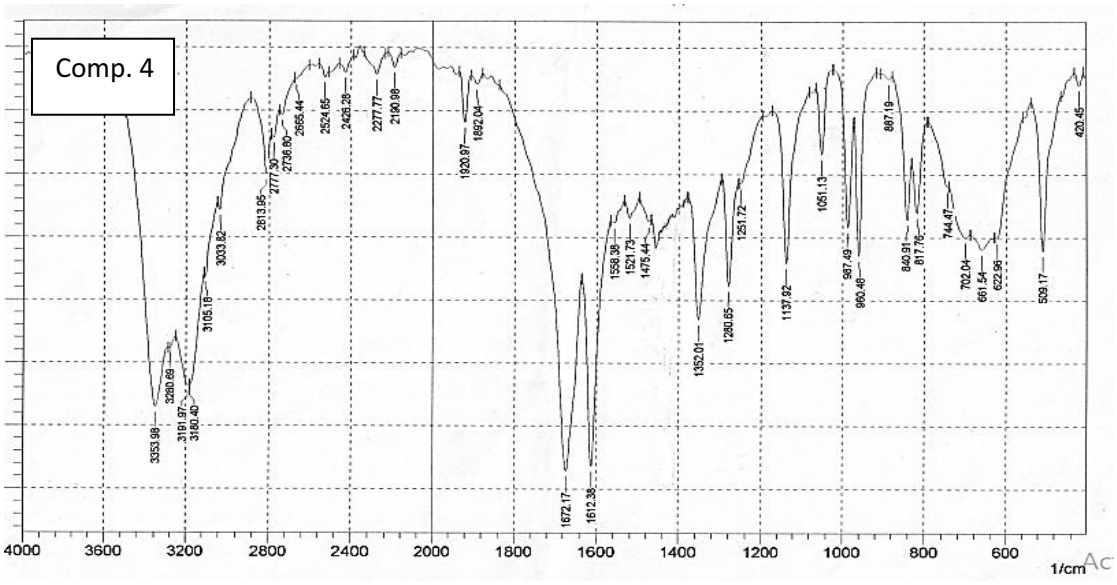


Fig. 3. Fourier transfer Infra-Red spectrum of compound 4.

Table 2. Infrared absorption (cm^{-1}) for compounds 1–7.

Compound number	υC-H Aliphatic	υC-H Aromatic	υC=N Schiff	υC=O Amide	Others
1	2812	–	1612	1676	–
2	2813	–	1612	1674	–
3	2947	3195	1610	1676	*p*-OCH_3, 837
4	2813	3180	1612	1672	O-H, 3353
5	2904	3195	1600	1668	*p*-NCH_3, 811
6	2999	–	–	1708	–
7	2933	–	–	1691	–

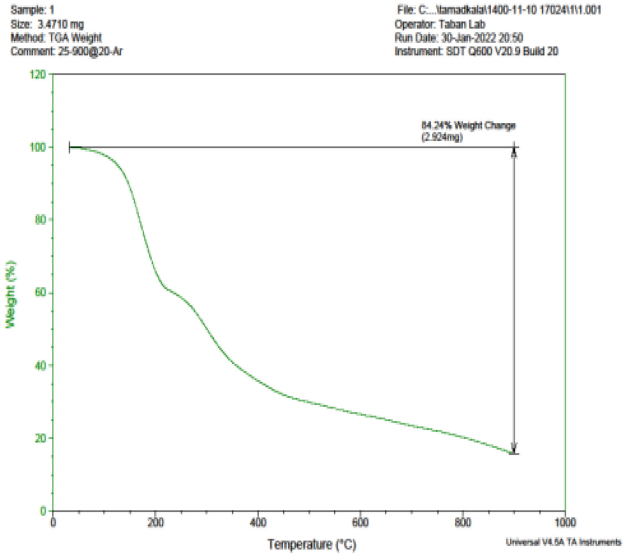


Fig. 4. Thermogravimetric analysis of compound 1.

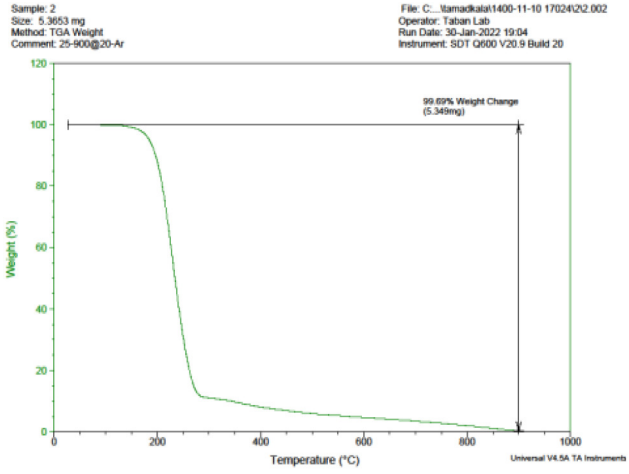


Fig. 5. Thermogravimetric analysis of compound 7.

percentage was relatively small, representing the reacted Schiff base **2** and succinic anhydride proportions. Finally, the third step, which showed a weight loss of 3.53% at 699 °C, refers to the continued degradation of Schiff base **2** and the release of carbon dioxide (CO_2) and the residual weight of carbon waste.

Thermogravimetric analysis of this compound 6, Fig. 6 represented three weight loss steps: The first step, at 120.02 °C, is attributed to the water evaporation in the compound, which reduced the weight by 94.28%. The second step, at 240.1 °C, is attributed to the decomposition of Schiff base 2 bonds with the compound 6 and the decomposition of part of the compound 6, with a weighting loss of

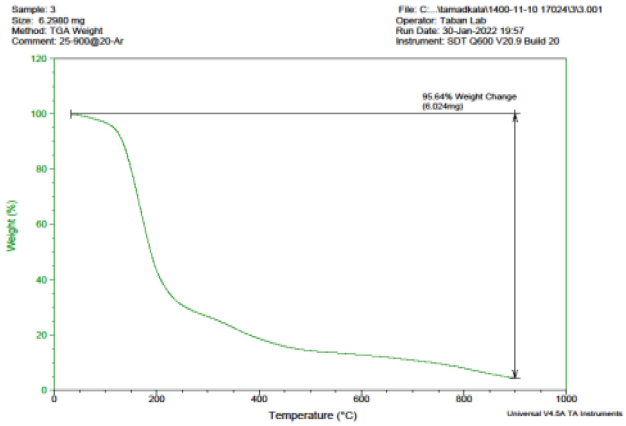


Fig. 6. Thermogravimetric analysis of compound 6.

Table 3. The antibacterial activities of compounds 1–4.

Sample number	*Staphylococcus*	*Pseudomonas*
1	–	–
2	2.9	2.2
3	2.3	2
4	5	4

Fig. 7. The antibacterial activities of compounds 1–4.

32.06%. The weight percentage was relatively small, representing the proportion of the reacted Schiff base 2 and maliec unhydried. Finally, the third step, which showed a weight loss of 10.70% at 706.2 °C, refers to the continued degradation of Schiff base 2, the release of (CO_2) carbon dioxide, and the residual weight of carbon waste.

The antibacterial activity[29,30]

The antibacterial activities of compounds 1, 2, and 3 were resistant to two types of bacteria, *Staphylococcus aureus* (gram-negative) and *Pseudomonas* (gram-positive), using DMSO as a solvent and used gentamicin as standard 4. The results express that compound **2** expresses higher activity than two kinds of bacteria, whereas compound **1** expresses no activity against two. All the results are displayed in Table 3 and Fig. 7.

Conclusion

The test of new derivatives of polymers on the antibacterial activities of two types of bacteria work describes the synthesis of 1,3-oxazepine and self-polymerization via a cycloaddition reaction between Schiff's bases 1,2,3 and succinic, maleic anhydride. The antibacterial activity showed intermediate results against two types of bacteria in compression with gentamicin as a standard reference.

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Author's declaration

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

Authors' contribution statement

The first author, R. M. D., prepared compounds and polymers, performed the IR test, and wrote the manuscript. The second author, S. H. A., performed the TGA in the third data analysis, and S. N. F. conducted the antibacterial activity.

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تحضير وتشخيص مشتقات جديدة لبوليمرات من بولي اكريل امايد ودراسة فعاليتها البكتيرية

رؤى محمد ضيدان، سناء هتور عواد، سهى نصر فاضل

قسم الكيمياء، كلية العلوم للبنات، جامعة بغداد، بغداد، العراق.

الخلاصة

تهدف هذه الدراسة إلى اختبار تأثير المشتقات الجديدة للبوليمرات على الأنشطة البيولوجية لنوعين من البكتيريا.في هذا العمل، تم وصف تحضير مركبات 1,3- اوكسازبين و حدوث عملية البلمرة الذاتية عن طريق تفاعل الغلق الحلقي بين احد قواعد شيف 2-مثيل-ن-(5-(2-مثيلبيوتانيول امينو)بنتايلدين)بيت-2انمايد ومع كل من حوامض السكسينك والماليك الغير مائي. شخصت المركبات المحضرة بواسطة درجات الانصهار ودرجة المرونة والخواص الفيزيائية كدرجة الانتفاخ وقياس الاشعة تحت الحمراء FT-IR وقياس التحليل الحراري TGA. الفعالية البيولوجية والتي اظهرت نتائج متوسطة الفعالية ضد نوعين من العزلات البكتيرية المقاومة للمضادات الحيوية.

الكلمات المفتاحية: اكريل امايد، الفعالية البكتيرية، المركبات الحلقية الغير متجانسة، اوكسازبين، قواعد شيف.