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Synthesis and Characterization of Polymers Bearing Penicillinic Acid Moiety and Their Application

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Abstract

This work includes the preparation of a new monomer have an aminopenicillinic acid moiety and introducing in a homogeneous and co-polymerization reaction and studying the possibility of using them as corrosion inhibitors as well as studying some biological activity. Firstly amid acid was prepared from the reaction of 4-aminoacetophenone with maleic anhydride, then dehydration of water for ring closure of amic acid was performed to produce the corresponding imide, which in turn entered the Schiff base formation reaction with the aminopenicillinic acid compound to produce the required monomer , followed by the synthesis and preparation included conducting the polymerization process for the prepared monomer and preparing four new polymers (a homo polymer and three copolymers with vinyl monomers: acrylonitrile, methyl acrylonitrile, and methyl acrylate).Study of The biological activity of these polymers, as well as the study of the effectiveness of these polymers in protecting carbon steel from corrosion in an acidic medium, and the prepared polymers gave excellent results in both applications. Compounds and polymers prepared were identified by using techniques (FT-IR and 1H-NMR), spectroscopy, and study physical properties of compounds and polymers, including (melting points, softening points), and solubility.

Keywords: Penicillinic acid, free radical polymerization, co-polymerization, anticorrosion, antibacterial.

تخليق وتشخيص البوليمرات التي تحتوي على مجموعة حمض البنسلينيك وتطبيقاتها

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الخلاصة

يتضمن هذا البحث تحضير مونومير جديد يحتوي على مجموعة حمض الأمينو بنسلينيك وإدخاله في تفاعل بلمرة متجانسة ومشتركة ودراسة إمكانية استخدامها كمثبطات للتآكل بالإضافة إلى دراسة بعض النشاط البيولوجي. أولاً تم تحضير حمض الأميك من تفاعل 4-أمينو أسيتوفينون مع أنهيدريد الماليك، ثم تم إجراء عملية نزع الماء لإغلاق حلقة حمض الأميك لإنتاج الإيميد المقابل، والذي دخل بدوره في تفاعل تكوين قاعدة شيف مع مركب حمض الأمينو بنسلينيك لإنتاج المونومير المطلوب، تلا ذلك عملية التخليق والتحضير التي تضمنت إجراء عملية البلمرة للمونومير المحضر وتحضير أربعة بوليمرات جديدة (بوليمر متجانس وثلاثة بوليمرات مشتركة مع مونوميرات الفينيل: أكريلونيتريل، ميثيل أكريلونيتريل، ميثيل أكريلات). تم دراسة النشاط البيولوجي لهذه البوليمرات، بالإضافة إلى دراسة فعالية هذه البوليمرات في حماية الفولاذ الكربوني من التآكل في وسط حمضي، وأعطت البوليمرات المحضرة نتائج ممتازة في كلا التطبيقين. تم تشخيص المركبات والبوليمرات المحضرة باستخدام تقنيات (FT-IR و H-NMR1) والتحليل الطيفي ودراسة الخصائص الفيزيائية للمركبات والبوليمرات بما في ذلك (نقاط الانصهار ونقاط التليين) والذوبانية.

1. Introduction

Penicillin derivatives are a class of the most important antibacterial agents in the current clinical system, which are highly effective in eliminating a broad spectrum of bacteria. Therefore, there is a great emphasis on the preparation of large groups of penicillin derivatives by introducing different groups to make its composition and modifications to increase its effectiveness in eliminating bacteria because [1]- [4] . Antimicrobials are among the most important types of drugs that are taken on a daily basis, as the use of this type of medication has led to the discovery of numerous chemical compounds with biological activity. Increasing antimicrobial resistance has led to an increase in deaths from infectious diseases, which are a major contributor to global mortality [4]-[9] . Antimicrobial resistance has made many clinically effective antimicrobials that are still in use less effective, despite the World Health Organization calling them "wonder drugs" and "breakthrough weapons for the treatment of infectious diseases". Multidrug-resistant bacteria are becoming more common, which has led chemists to develop new antimicrobial agents that are effective against these resistant strains in current therapy [10]-[12] . Polyimides are one of the most important polymers that have a wide range of applications in medicine, microelectronics, fuel cells and gas separation due to their properties known for their thermochemical stability [13,14].

In the current work, we sought to produce a new imide derivative with penicillin and Schiff's base, and then we polymerized the prepared monomer to produce new homo and copolymers. These compounds are considered very important because they contain three important groups, which are the imide, Schiff base, and the penicillin groups, which are important groups and have wide applications. The biological activity of these polymers was studied as well as the possibility of using them to protect metal from corrosion in an acidic medium. He studied the synthesis of these compounds by using different spectral methods (FT-IR and 1H-NMR) as well as studying their thermal and physical properties for synthesized monomer and polymers.

2. Experimental and materials

Materials: The chemicals were supplied by CDH, Merck and Aldrich Chemicals Company and used without more purification.

Techniques: FT-IR spectra were logged using KBr discs on (8400s Shimadzu) FT-IR spectrophotometer. 1H-NMR spectra were carried out by Bruker 400 MHz, DMSO was used as a solvent with TMS as an internal standard. Melting points were determined by using Gallen Kamp melting point apparatus.

2.1 Methods

2.1.1 Synthesis of 4-((4-acetylphenyl)amino)-4-oxobut-2-enoic acid (1)

Amic acid was prepared via the reaction of equal moles from 4-aminoacetophenone and maleic anhydride in acetone solvent. The reaction mixture was string at room temperature for four hours, then filtered and washed with cold acetone then dried [14]. color: yellow, in yield: 94%, m.p. = 201-203 °C. IR (v =cm-1): 3459 (OH), 3417, 3269 (NH2), 2920 (C-H, Ar.), 1710 (C=O, acid), 1633 (C=O, amide), 1537 (C=C).

2.1.2 Synthesis of 1-(4-acetylphenyl)-1H-pyrrole-2,5-dione (2)

Imide was synthesized via using acetic anhydride as dehydrating agent were 0.5 gm of enoic acid dissolved in acetic anhydride 25 mL with 10% gm of anhydrous sodium acetate and mixture was reflux for six hours on water bath, after time expired the mixture cooled then poured into crushed ice. Formed solid precipitate was filtered then washed with cold ethanol then dried [14]. color: dark red, in yield: 86%, m.p. = 279-281 °C. IR (v =cm-1): 2916 (C-H, Ar.), 1778, 1714 (C=O, imide), 1598 (C=C), 1379 (C-N, imide).

2.1.3 Synthesis of 6-((1-(4-(2,5-dioxo-2,5-dihydro-1H-pyrrol-1-yl)phenyl)ethylidene)amino)-3,3-dimethyl-7-oxo-4-thia-1-azabicyclo[3.2.0]heptane-2-carboxylic acid (3)

Synthesis of Schiff base was confirmed by reaction of 4-aminopenicillinic acid 0.01 mol dissolved in absolute ethanol with imide (2) 0.01 mole also dissolved in ethanol with three drops of G.A.A. then the mixture was reflux for six hours on water bath. The formed solid was filtered and washed with ethanol and dried[15]. color: brown, in yield: 83%, m.p. = 99-101 °C. IR (v =cm-1): 3270 (OH), 2918 (C-H, Ar.), 1786, 1712 (C=O, imide), 1665 (C=O, acid), 1645 (C=O, amide), 1592 (C=C), 1529 (C=N, imine), 1369 (C-N, imide). 1H-NMR (DMSO-d6): δ 1.33-1.52 (9H, CH3), 4.53-4.76 (3H, lactam ring), 5.39 (2H, vinylic), 7.87-7.92 (4H, Ar.), 11.19 (1H, OH).

2.1.4 polymerization of 6-((1-(4-(2,5-dioxo-2,5-dihydro-1H-pyrrol-1-yl)phenyl)ethylidene)amino)-3,3-dimethyl-7-oxo-4-thia-1-azabicyclo[3.2.0]heptane-2-carboxylic acid (4-7)

Homo polymers and copolymers were synthesized by the free radical polymerization by using of (AIBN as initiator) in tetrahydrofuran as solvent, where the polymerization mixture (monomer only in homo polymerization and monomer with vinylic monomers (acrylonitrile, methyl acrylonitrile and methyl acrylate) in copolymerization) was putted in dark polymerization bottle then flashed with nitrogen gas and stoppered. The bottle heated on water bath for 8 hours and then the mixture cold and poured on methanol to precipitate the corresponding polymer. Then washed with methanol and dried. Scheme 1 showed the synthesis route of compounds and polymers [16].

Homopolymer (4); color: brown, in yield: 87%, softening point: 55-63 °C. IR (v =cm-1): 3343 (OH), 2922 (C-H, Ar.), 1712 (C=O, imide), 1665 (C=O, acid), 1643 (C=O, amide), 1599 (C=C), 1531 (C=N, imine), 1380 (C-N, imide). 1H-NMR (DMSO-d6): δ 1.25-1.76 (9H, CH3), 3.42 (2H, CH-CO), 4.33-4.56 (3H, lactam ring), 7.51-7.71 (4H, Ar.), 11.47 (1H, OH).

Copolymer (5); color: pale yellow, in yield: 81%, softening point: 74-88 °C. IR (v =cm-1): 3329 (OH), 2922 (C-H, Ar.), 2243 (C≡N), 1718 (C=O, imide), 1681 (C=O, acid), 1651 (C=O, amide), 1600 (C=C), 1531 (C=N, imine), 1371 (C-N, imide). 1H-NMR (DMSO-d6): δ 1.17-1.55 (9H, CH3), 2.51 (2H, CH-CO), 3.17 (CH2), 4.14-4.97 (3H, lactam ring), 7.43 (4H, Ar.), 11.27 (1H, OH).

Copolymer (6); color: brown, in yield: 85%, softening point: 110-118 °C. IR (v =cm-1): 3321 (OH), 2983 (C-H, Ar.), 2235 (C≡N), 1716 (C=O, imide), 1683 (C=O, acid), 1650 (C=O, amide), 1600 (C=C), 1533 (C=N, imine), 1375 (C-N, imide). 1718 (C=O, imide), 1681 (C=O,

acid), 1651 (C=O, amide), 1600 (C=C), 1531 (C=N, imine), 1371 (C-N, imide). 1H-NMR (DMSO-d6): δ 1.25-1.58 (12H, CH3), 2.53 (2H, CH-CO), 2.55 (CH2), 4.18-4.58 (3H, lactam ring), 7.58-8.03 (4H, Ar.), 11.12 (1H, OH).

Copolymer (7); color: brown, in yield: 79%, softening point: gummy. IR (v =cm-1): 3324 (OH), 2885 (C-H, Ar.), 1712 (C=O, imide), 1660 (C=O, acid), 1644 (C=O, amide), 1599 (C=C), 1530 (C=N, imine), 1380 (C-N, imide). 1H-NMR (DMSO-d6): δ 1.12-1.43 (12H, CH3), 2.47 (2H, CH-CO), 2.53 (CH2), 4.19-4.93 (3H, lactam ring), 7.37-7.42 (4H, Ar.), 10.08 (1H, OH).

2.1.5 Antibacterial activity study

Antibacterial activity of synthesized compounds and polymers was studied via the (cup plate technique) against two types of micro-organisms (E.coli and S.aureus). By using DMSO solvent to dissolve the compounds, medium of nutrient agar was engaged. Sample volume and sample solution for all the studied polymers were retitled (0.1 mL). The petri dishes were incubated in sterilized cup on incubator. The investigation was completed in the cups, (0.1 mL) of compounds solution was added in small holes in agar, then petri dishes of tests were incubated at (37 C˚) for (24 hr.). Inhibition zones were measured in millimeters for polymers solution and the solvent (DMSO) diameter was determined by the same method [17].

2.1.6 Corrosion measurement

The potentiostat set up includes a (Host computer, thermostat, magnetic stirrer), (EmStat 4s, Palm Sens, Holland) potentiostat, and galvanostat. The cell is one liter volume consisting of two bowls (internal and external). Three electrodes were made the electrochemical corrosion cell. An auxiliary electrode is 10 cm length of platinum, carbon steel as a working electrode used to detect the potential of it rendering to the reference electrode and reference electrode saturated calomel (Hg/Hg2Cl2 sat. KCl). The working electrode was dipped in the test solution for (15 minute) to find steady state open circuit potential (Eocp). The electrochemical measurements were achieved in a potential range of (± 200) mV. All tests were carried out at 298 K by using a water bath for cooling-heating [18, 19].

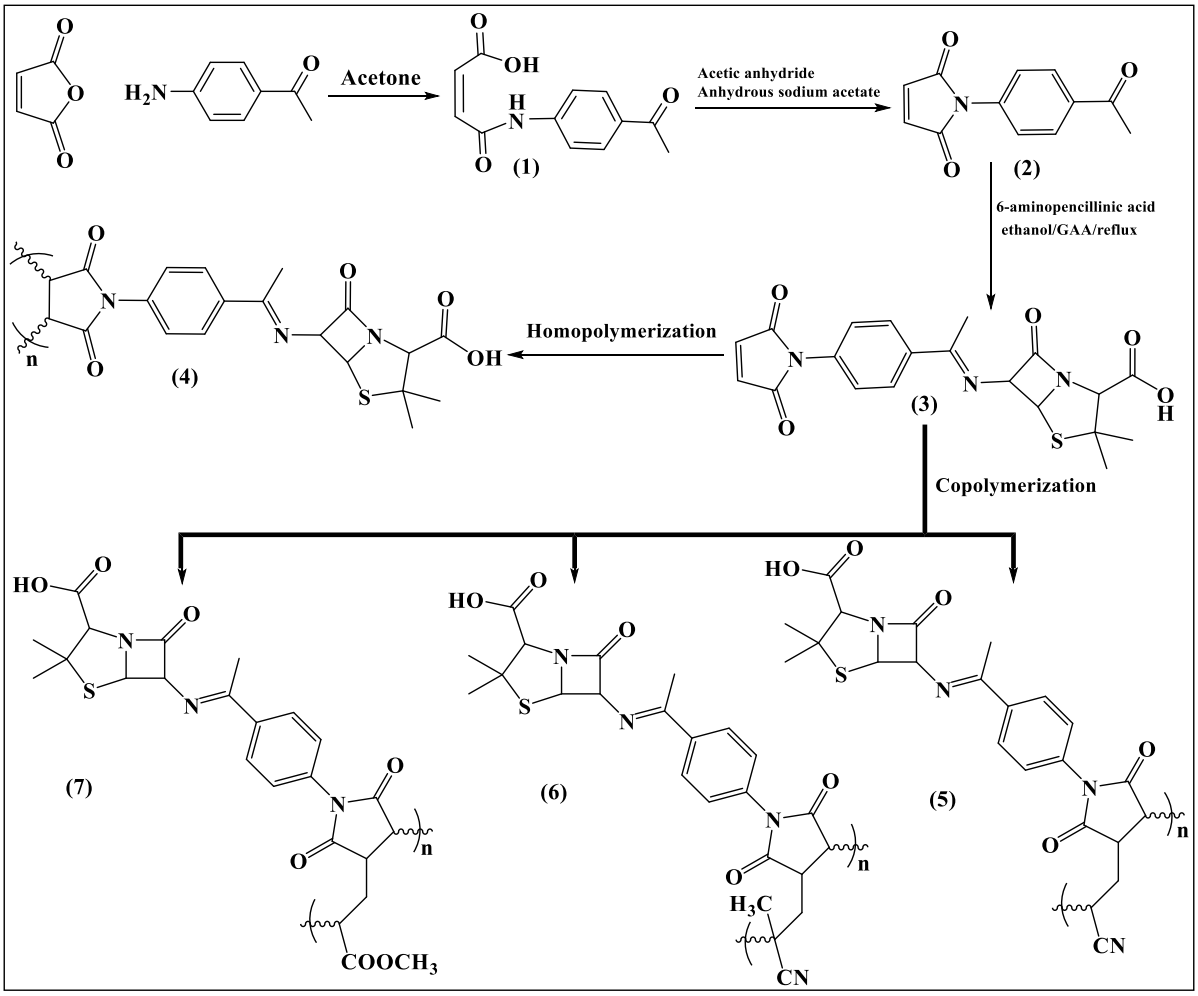
The Corrosion Cell electrodes

The three electrodes can be explained as follows:

A. A reference electrode is used to control the working electrode potential rendering to the potential of a reference electrode. The potential of reference electrode is well identified and precise. It is the joining of two tubes; the inner tube contains Hg/Hg2Cl2 saturated KCl, the reference electrode stand at a distance (2 mm) from the working electrode.

B. the Auxiliary Electrode contains platinum metal (high purity); and has a length of (10 cm).

C. the Working Electrode used in this study, where its Electrode potential should be recorded. This electrode was made from metallic wire (20 cm) and connected to the mounted specimen.



Scheme 1- Synthesis route of compounds and polymers.

3. Result and discussion

Polyimides is considered one of the important polymers, where in this work synthesized with new moiety (Penicillin moiety) liked with polymer by Schiff base linkage. And synthesized two types of polymers (homo and copolymers) all one of these polymers have a special property depending on the composition of it. The prepared compounds and polymers were proven to be synthesized by observing the changes that occurred in the physical properties of the resulting compounds in terms of color, melting and softening point, and following up the reaction using (TLC) thin layer chromatography. As for the spectral identified of the compounds it was done by using spectral methods (FT-IR infrared spectrum and 1H-NMR spectrum).

Amic acid (1) gave basic peak for the infrared spectrum due to the stretching vibration of the carboxyl group at 3453 cm-1, as well as the amine group at 3277 cm-1, while the groups (C=O) gave values of 1710, 1678 and 1630 cm-1 belonging to the acid, ketone, and the amide respectively, the stretching of the benzene ring is 1598 cm-1.

As for the imide compound (2), it showed the disappearance of the peaks of the carboxylic acid, amide, amine and hydroxyl groups, and the appearance stretching of new groups, which are the (C=O) imide group at 1780 and 1716 cm-1, as well as the presence of stretching (C=O) ketone bonds at 1677 cm-1.

Schiff base compound (3) in addition to the presence of stretching peak of the imide groups (C=O) around 1786 and 1716 cm-1, showed the presence of a new stretching peak at 1529 cm-1 belonging to the new bond of the Schiff base group[20].

Polymers (4-7) the basic peaks that were clear are the values of the methylene group, which appear at 2872-2983 cm-1, which is good evidence of the formation of polymeric bonds, in addition to the presence of other basic groups that were included within the vinyl monomers in the copolymerization, which are groups the nitriles appear at 2235 and 2243 cm-1 and the ester groups in the polymer at 1722 cm-1.

1H-NMR of monomer (3) give the main protons signal presence in compound were give signal at δ= (1.33-1.52) ppm fit to methyl protons and methylene group protons linked to lactam ring give signals at δ= (4.53-4.76) ppm. Signal at δ= (5.39) ppm fit to vinylic protons of imide ring. Aromatic protons signal at δ=(7.87-7.92) ppm and carboxylic hydroxyl proton give signal at δ=(11.19) ppm. 1H-NMR of polymers gives the main protons signals at δ= (1.25-1.76) ppm fit to methyl group protons and signals at δ= (4.14-4.97) ppm belong to lactam ring protons. In addition to disappear of vinylic protons signal, that is good proof for formation of polymers. Other signals of aromatic protons and carboxylic hydroxyl proton gives signals at δ= (7.37-8.03) and δ=(10.08-11.47) ppm respectively[21].

3.1 Corrosion polarization curves

Table 1 and figures 1-3 showed the corrosion parameters, the corrosion current density (icorr) and corrosion potential (Ecorr) were gotten by the extrapolation of the cathodic and anodic Tafel in nonappearance and attendance the inhibitors molecules in HCl (0.1M) solution. The anodic (ba) and cathodic (bc) Tafel slopes were also calculated from figures. Table shows the resultant data of the corrosion current density icorr (A/cm2), corrosion potential Ecorr (mV), cathodic and anodic Tafel slopes (mV/Dec), and protection efficiency PE% :

$$\%\mathrm{IE} = \frac{(\mathrm{icorr})\mathrm{o} - (\mathrm{icorr})}{(\mathrm{icorr})\mathrm{o}} * 100 \qquad (1)$$

Where (icorr)ois the corrosion current density in without of inhibitors, (icorr) is the corrosion current density with found of inhibitors. The synthesized polymers presented good protection efficiency, and this is because they contain groups of heterogeneous elements and contain sulfur, nitrogen, and oxygen, which absorb on the surface of the alloy and protect it from corrosive media.

Table 1- Corrosion parameters for blank and compounds (3–7) in acidic solutions.

Comp.	E corr.	I corr.	I corr./ r	Resis.	Anodic β	Cathodic β	Corr. rate,	IE%
Blank	-0.535	101.77	1.875E-4	169.3	0.103	0.057	0.920	-
3	-0.626	11.89	2.378E-5	3021	0.130	0.229	0.117	88
4	-0.617	16.04	3.208E-5	1615	0.094	0.164	0.157	84
5	-0.611	18.55	3.709E-5	1840	0.131	0.196	0.182	82
6	-0.641	11.69	2.338E-5	2258	0.105	0.145	0.115	89
7	-0.628	13.17	2.634E-5	1766	0.090	0.133	0.129	87

Where , E corrosion, (V) ,I corrosion,(μA) , I corrosion per surface area, (A/cm²) , Polarization Resistance,(Ω) , Anodic β Tafel constant, (V/decade) , Cathodic β Tafel constant, (V/decade) , Corrosion rate (mm/year) ,and IE% inhibition efficiency.

3.2 Antibacterial activity

Antibiotics for β-lactam compounds are widely used to eliminate a wide range of bacteria and microorganisms, as is the case for compounds containing lipid groups, as they are included in the composition of many compounds that are used as treatments against a wide range of diseases and their causes. The synthesized polymers give good activities against two types of micro-organisms (S.aureus and E.coli) and the result listed in table (2) and figure (4) and (5).

Table 2- biological activity of compounds and polymers.

Comp.	*Staphylococcus aureus*	*E.coli*
3	8	8
4	6	8
5	6	12
6	6	16
7	6	12

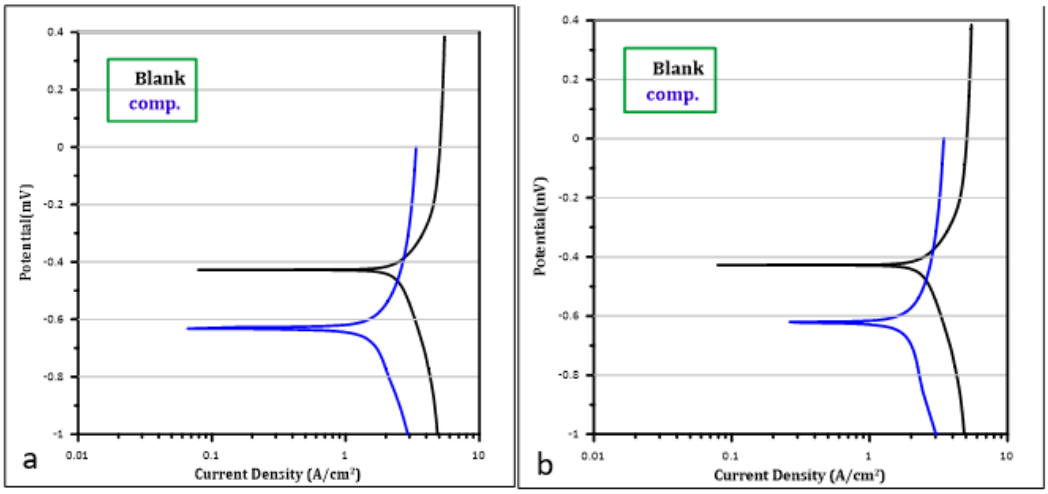


Figure 1- (a) polarization curve of monomer (3) , and (b) polarization curve of polymer (4)

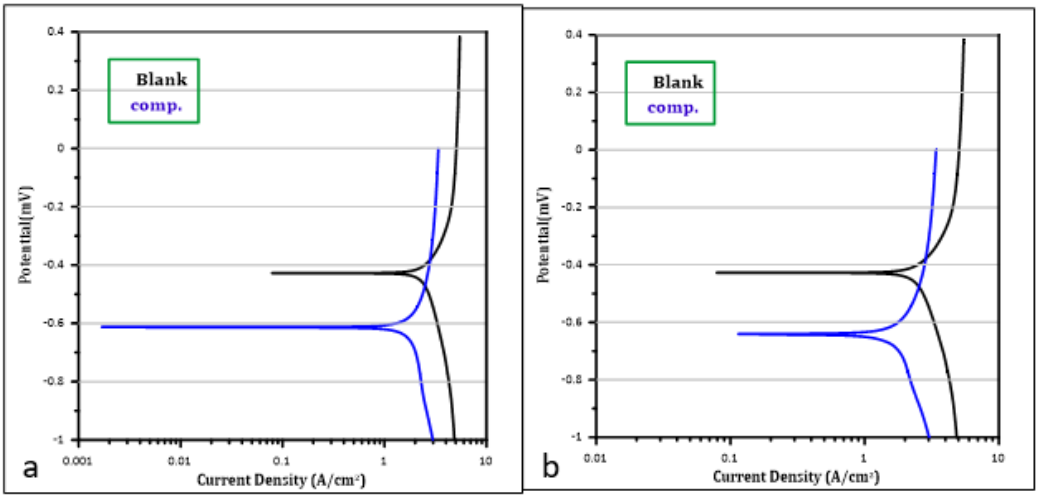


Figure 2- (a) polarization curve of polymer (5) , and (b) polarization curve of polymer (6).

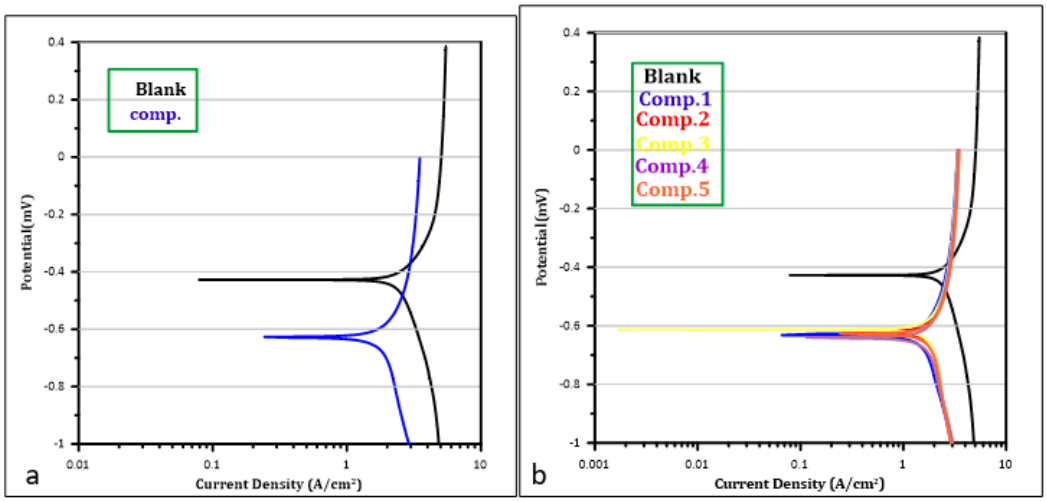


Figure 3- (a) polarization curve of polymer (7) , and (b) polarization curve of polymers and monomer

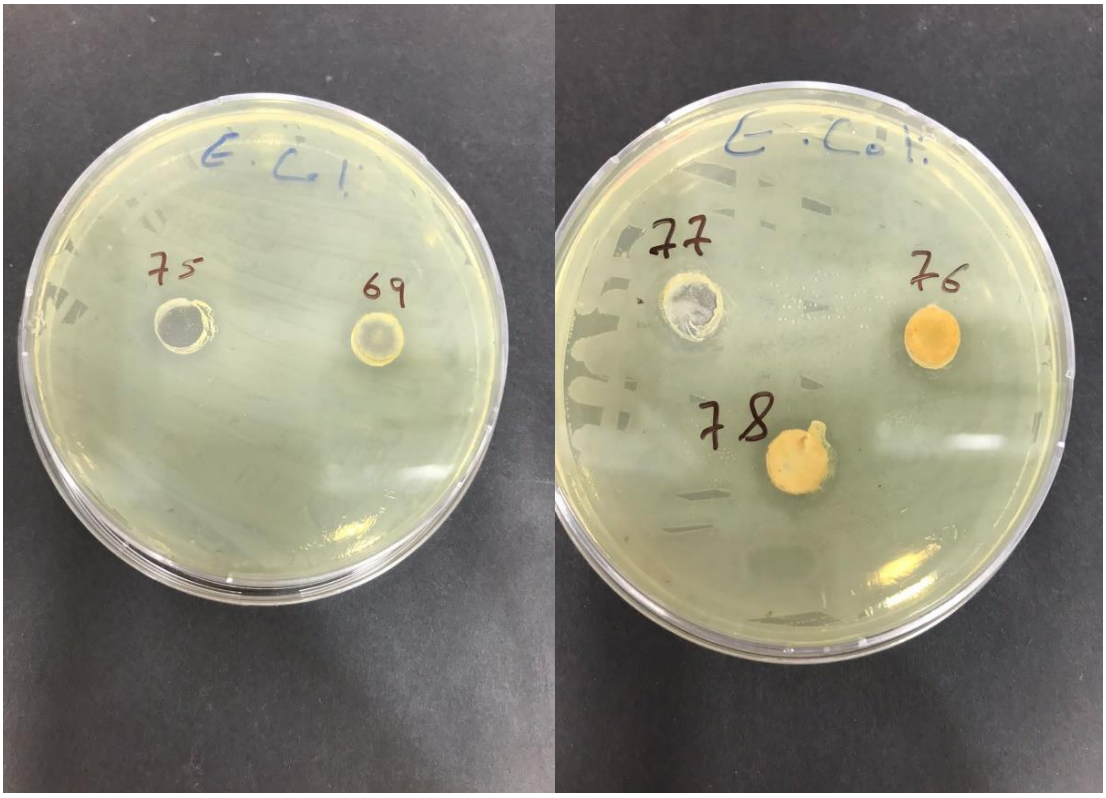


Figure 4- biological activity and inhibition zone against *E-coli*

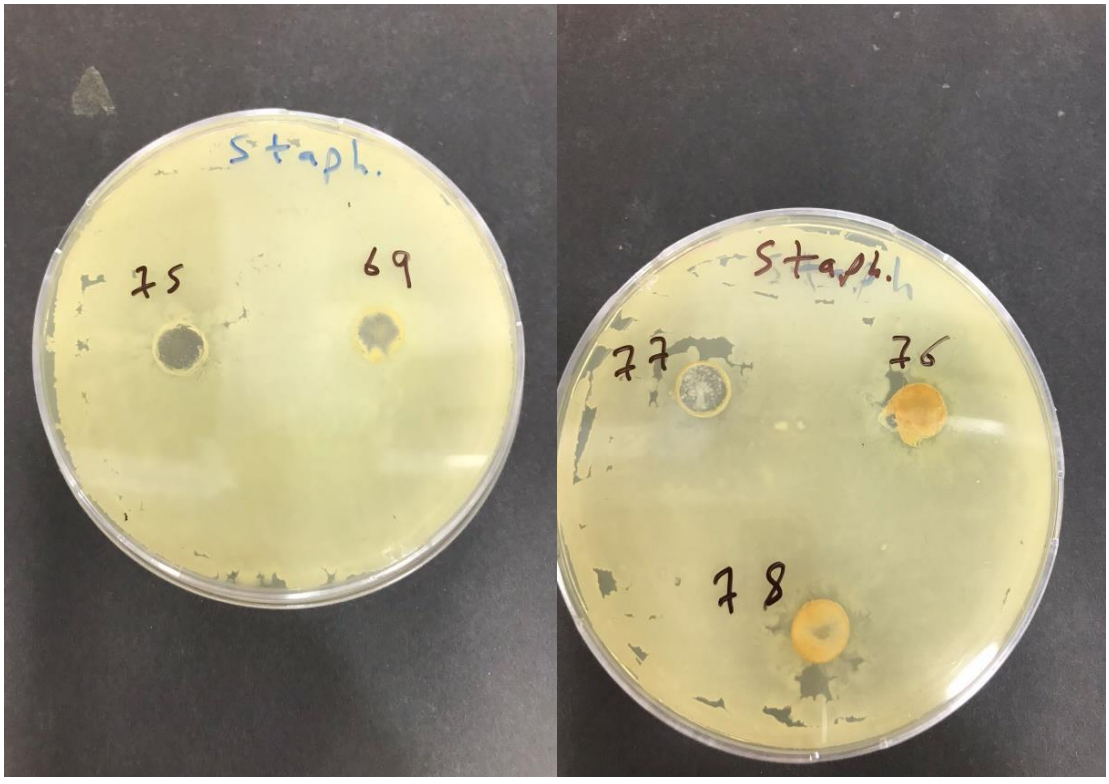


Figure 5- biological activity and inhibition zone against *Staphylococcus aureus*

4. Conclusion

In brief , I have synthesized and characterized a new set of polymers based on the maleimide and pancillinic acid subunits. They were successfully obtained through the direct polymerization reaction. These type of polymers have high solubility and thermal stability. This may be due to the presence of Schiff base and pancillinic groups. The presence of the pancillinic acid and Schiff base groups in the polymers increase the ability of using these polymers as anti-corrosion and antibacterial.

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