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Synthesis, Characterization and Study of the Effect of Nanoparticles on the Biological Activity of New Silicon Polymers and Their Nanocomposites

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Abstract

A new class of silicone polymers was synthesized based on dichlorodi(methyl)silane (DCDMS) with some organic compounds [M1-M6] containing terminal hydroxyl groups previously synthesized by different chemical reactions, and their nanocomposites were synthesized using silver nanoparticles (Ag-NPs). All polymers were synthesized using condensation polymerization and characterized by FTIR and ^{1}HNMR spectra. The biological activity of silicone polymer P5 was evaluated using different weights of silver nanoparticles (1%, 3%, 5%, and 7%) against *Escherichia coli* (Gram-negative) and *Staphylococcus aureus* (Gram-positive). The results showed that the higher the percentage of silver nanoparticles, up to 7%, the higher the biological activity, and accordingly, this percentage of silver nanoparticles was used to synthesize and measure the biological activity of nanocomposites P1-P5 and P6 against the same two types of bacteria. The nanocomposites showed antibacterial activities against *Escherichia coli* (Gram-negative) and against *Staphylococcus aureus* (Gram-positive) better than silicon polymers without silver nanoparticles (Ag-NPs). The results also showed that P6 was more antibacterial when pure than the other polymers. The polymer P'4 with silver nanoparticles (7%) was 20 times more antibacterial against *Escherichia coli* and 25 times more antibacterial against *Staphylococcus aureus*. This means that P'4 has more antibacterial activity against the same two types of bacteria than other nanocomposites.

Keywords: Antibacterial activity, nanocomposite, silicone polymer, silver nanoparticles.

1. Introduction

Silicone polymers are one of the most important polymers because they have good thermal stability and oxidation resistance, as well as valuable resistance to high and low temperatures. Because silicones are chemically inert materials, the Food and Drug Administration has approved their use in medical devices such as permanent or temporary implants, catheters, tubes, stomach bags, and prostheses. Silicone frequently finds its way into medical consumables. Silicones have attracted the attention of researchers in recent years because they are resistant to oxygen, ozone, and sunlight, so this type of polymer has superior resistance to weathering and aging [1-3]. They find many uses in oils and grease materials. Silicone oils are desirable because of their viscosity,

which meets all of the characteristics of both high and low temperatures. Other silicones are used in hydraulic fluids as well as electrical insulators. Hospitals often use invasive devices like catheters and ventilators, which can often lead to fatal bacterial infections. To tackle these issues, researchers have conducted intensive efforts and numerous studies to design antibacterial devices that incorporate antibacterial agents like antibiotics, quaternary ammonium salts, and metal nanostructures to inhibit the growth of microbes [4]. The use of Si films infused with silver for medical devices could potentially reduce the frequency of such infections. Also, using silver particles (AgNPs) and the stronger bond between Ag and bacterial cell walls could change the way bacterial membranes look, by breaking them, which could let cell contents leak out and kill the bacteria [5]. Additionally, they can destroy multiple drug-resistant pathogens and disrupt their growth formation [6,7]. There are several methods for introducing antimicrobial activity into polymeric materials, such as incorporating antimicrobial agents directly into the polymers, coating antimicrobials onto polymer surfaces [8,9], immobilizing antimicrobials by chemical grafting [10,11], or using polymers that exhibit intrinsic antimicrobial properties [12,13].

Manufacturers can manufacture various silicone polymers, such as liquids (oils), greases, synthetic rubbers, and resins, using different organic groups like Schiff bases or aryl substituents linked to dimethyl silicon dichloride. Currently, common antibacterial polymers are based on silver compounds. This is because polymers coated with silver-based compounds release silver ions into solution, known to have antibacterial properties against a wide range of microorganisms [11–15].

2. Materials and Methods

2.1 Materials

All the raw materials were supplied by Merck and SIGMA-ALDRICH CO.

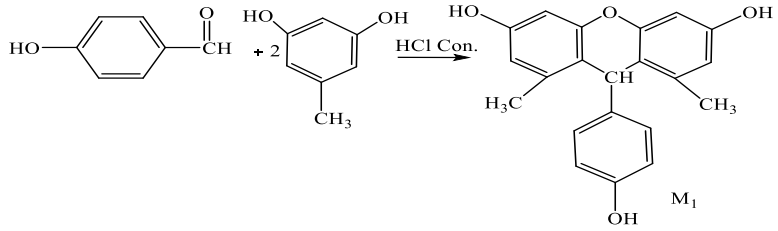
2.2 Instrumentation

The FTIR spectrum of samples was recorded on a Shimadzu (Ir Prestige-21), and 1H-NMR spectra have been performed by the following companies: Ultra Shield 500MHz, Bruker, Al-Basrah University, and an antibacterial activity test carried out against *S. aureus* and *E. coli* supplied by the Microbiology Laboratory (central environmental laboratory) in the College of Sciences/ University of Baghdad.

2.3 Synthesis method

2.3.1 Synthesis of monomer [M_1]

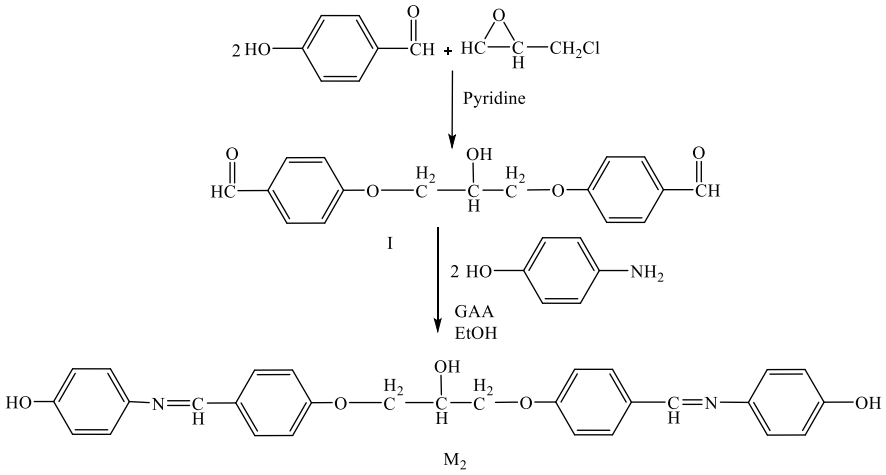
A mixture of 5-methylbenzene-1,3-diol (2.248 g, 0.02 mol) with 4-hydroxybenzaldehyde (1.22g, 0.01 mol) and HCl as a catalyst (2.5 mL) was heated in the oil bath at 60 ºC for 6 hours, then cooled, and the reaction mixture was transferred to cold water (10 mL) [16]. **Table 1** lists the physical data, the structure, and the structure of the synthesized monomer [M1], while **Scheme 1** outlines the reaction sequence leading to the formation of monomer M1.



Scheme 1. Reaction pathway for the synthesis compound M_1.

2.3.2 Synthesis of monomer [M_2]

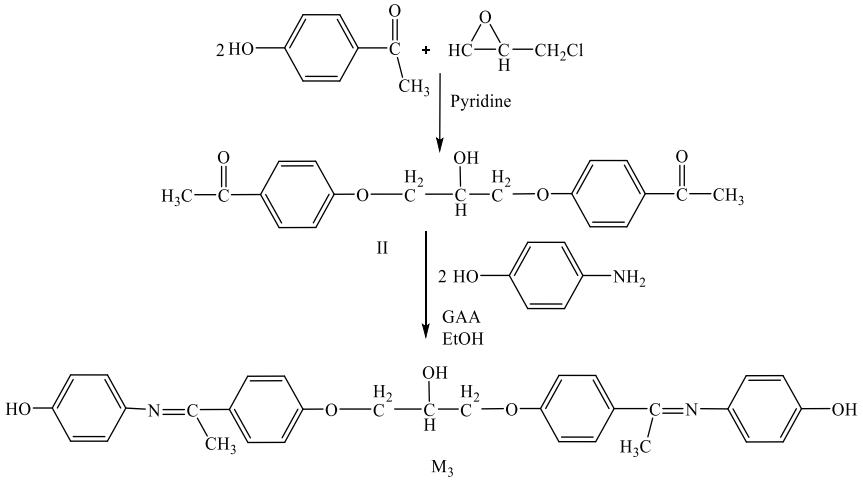
A quantity of 4-hydroxy benzaldehyde (2.44 g, 0.02 mol) was dissolved in 2 mL of pyridine in a flask placed in an oil bath at 60 ºC. The mixture was refluxed with stirring for 1 hour in an oil bath, followed by the addition of epichlorohydrin (0.925 g, 0.01 mol), then the temperature rose to over 90 ºC, and the reaction continued for another 2 hours. Until the precipitate separated, the precipitate was filtered, washed many times with distilled water, and neutralized with 5% HCl [16], then the product [I] was dried. After that, a mixture of compound [I] (3.00 g, 0.01 mol) with 4-aminophenol (2.18 g, 0.02 mol) and 3 drops of glacial acetic acid (GAA) in a minimum amount of alcohol was refluxed for 4 hours [17–20]. After cooling, it was collected by filtration and re-crystallized from ethanol to give M2. The physical data of these compounds are given in **Table 1**, and the reaction sequence leading to the formation of the monomer M2 is outlined in **Scheme 2**.



Scheme 2. Reaction pathway for the synthesis monomer M_2.

2.3.3 Synthesis of monomer [M_3]

This monomer was synthesized using the same steps given for the [M2] monomer synthesis, excluding the use of the compound 4-hydroxyacetophenone instead of 4-hydroxybenzaldehyde. **Table 1** provides the physical data of this compound, while **Scheme 3** outlines the reaction sequence that leads to the formation of compound M3.

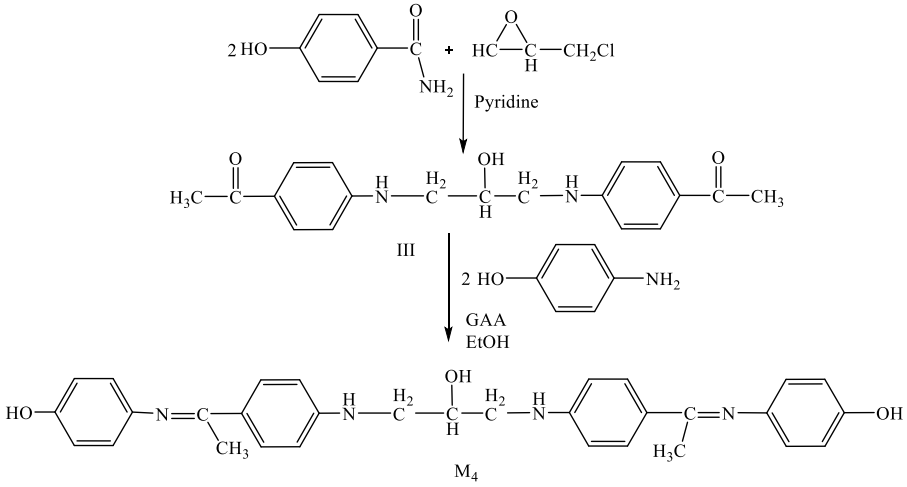


Scheme 3. Reaction pathway for the synthesis monomer M_3.

2.3.4 Synthesis of monomer [M_4]

This monomer was synthesized using the same steps given for the [M2] monomer synthesis, excluding the use of the compound 4-aminoacetophenone instead of 4-hydroxybenzaldehyde. The

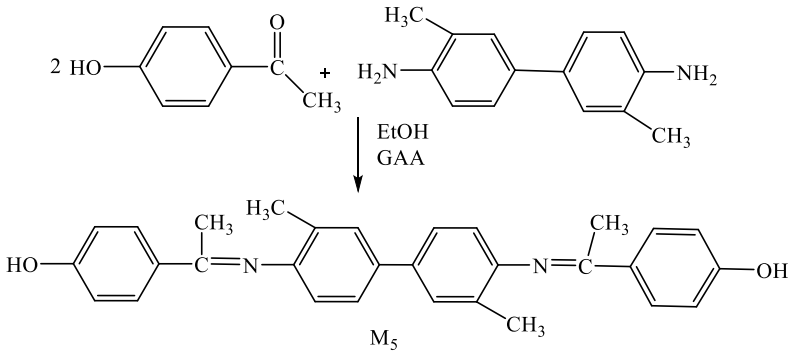
physical data of this compound are given in **Table 1**, and the reaction sequence leading to the formation of monomer M4 is outlined in **Scheme 4**.



Scheme 4. Reaction pathway for the synthesis monomer M_4.

2.3.5 Preparation of monomer [M_5]

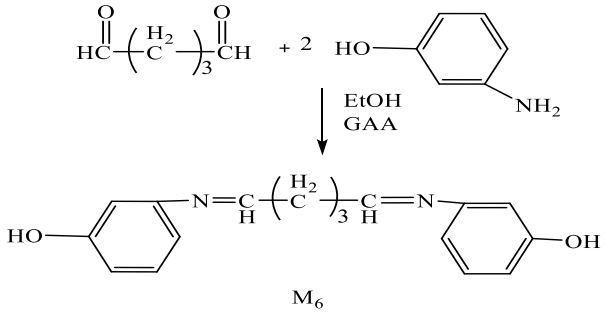
This monomer was synthesized using the same steps given for the [M2] monomer synthesis, excluding the use of a compound [3,3′-Dimethyl-[1,1′-biphenyl]-4,4′-diamine] instead of 4-aminophenol and 4-hydroxybenzaldehyde instead of compound [I]. **Table 1** shows the structural formula and physical data of M5, and the reaction sequence leading to the formation of monomer M5 is outlined in **Scheme 5**.



Scheme 5. Reaction pathway for the synthesis monomer M_5.

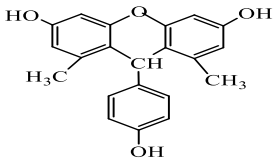
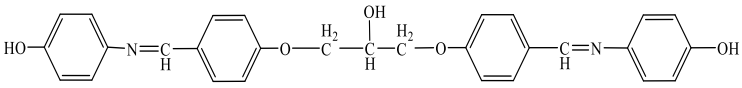
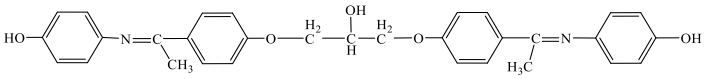
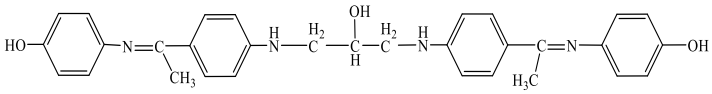
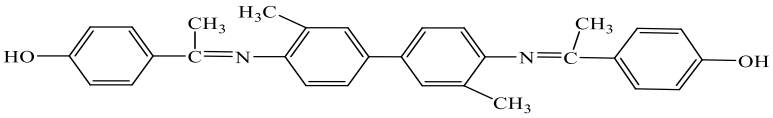
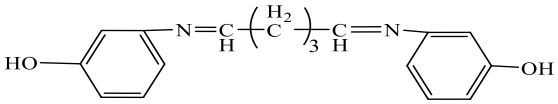
2.3.6 Preparation of monomer [M_6]

The monomer M6 was prepared according to the literature [19]. **Table 1** listed the structural formula and physical data of M6, while **Scheme 6** outlined the reaction leading to the formation of the monomer M6.



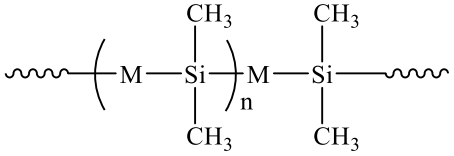
Scheme 6. Reaction pathway for the synthesis monomer M_6.

Table 1. Summary of physical properties for monomers.

NO. Monomer	Structure	Color of Monomer	M.P. of Monomer
M1		Dark Red	122-125
M2		Dark Yellow	228-230
M3		Dark Brown	152-155
M4		Brown	137-140
M5		Brown	146-150
M6		Olive	218-220

2.3.7 Synthesis of polymers [P_1-P_6]

The synthesis of these silicon polymers involved the condensation reaction (0.1 mol) of one of the monomers (M1-M6) in dry benzene with (0.1 mol) dimethyldichlorosilane, stirred under a temperature of 0–4 in an ice water bath for 48 hours. The resultant solid was poured into a 10 mL solution of dilute 5% HCl, filtered, dried, and recrystallized the precipitate in ethyl acetate [21]. **Scheme 7** provides a general formula for synthesized polymers. The characteristic FTIR absorption bands of polymers P1–P6 are listed in **Table 2**.



M=M_1-M_6

Scheme 7. Structure of silicon polymers with different M [P_1-P_6].

2.3.8 Synthesis of silver nanocomposites [P'_1-P'_6]

To prepare the nanocomposites by the solution casting method, 1 g of one of the polymers (P1-P6) was placed in 5 mL of DMF with stirring using a magnetic stirrer for 24 hours. Then, nanoparticles AgNPs in the concentration of 7% were disperse in the polymer media, ultrasonic for 2 hours at 25ºC was used to ensure preparation of a homogenous mixture of nanoparticles and the silicon polymers and then the mixture was poured into petri dishes [22,23].

3. Results

3.1 FTIR and ^{1}HNMR characterization

The characteristic FT-IR absorption bands for monomers M1-M_6 and polymers P_1-P_6 are listed in **Table 2** [24].

Table 2. Summary of FTIR spectra of the monomers and polymers.

Compound index	cm^{-1}							
	OH	C-H arom.	C-H aliph.	C=N end, exocyclic	C=C	Si-CH_3 asymmetric,symetric	Si-OPh	Others
M_1	3275	3020	2962,2732	—	1599	—	—	C-O-C 1234,1091
P_1	3433	3020	2958,2700	—	1600	1425,1263	993	
I	3367	3000	2960,2800	—	1598	—	—	C=O aldehyde1678
M_2	3357	3030	2974,2707	1616	1593	—	—	
P_2	3417	3010	2974,2707	1616	1593	1365,1261	890	
II	3367	3000	2931,2742	—	1581	—	—	C=0 ketone1681
M_3	3303	3024	2900,2819	1595		—	—	
P_3	3425	3000	2931,2800	1629	1602	1396,1271	950	
III	3334	3039	2974,2707	—	1593	—	—	C=O ketone 1647,NH 3398
M_4	3380	3037	2974,2702	1627	1595	—	—	NH 3465
P_4	3200	3064	2964,2800	1627	1598	1398,1263	956	NH 3363
M_5	3313	3020	2920,2700	1604	1577	—	—	
P_5	3387	3005	2989,2710	1604	1577	1423,1261	900	
M_6	3367	3024	2931,2843	1612	1597	—	—	
P_6	3353		2964,2891	1620		1404,1230	960	

The ^{1}HNMR spectrum for some polymers was in DMSO as a solvent. The ^{1}HNMR spectrum for [P1] showed the following signals: signal type singlet in δ (9.720) ppm for proton of OH phenol ring, multiple signal between δ (8.617–6.018) ppm that attributed for protons of benzene rings and proton of pyran ring, besides a singlet signal at δ2.662 ppm for three protons of CH_3-Ph groups. While the protons of $(CH_3)_2$-Si groups appeared in region δ (0.00–0.016) ppm, the ^{1}HNMR spectrum for [P3] showed the following signals: a signal in region δ (9.109) ppm for proton of OH group, signals in region δ (8.998–6.19) ppm that attributed for protons of benzene rings, and the one proton of CH-OH appeared at δ 4.016 ppm, in addition, a singlet signal at δ 2.443 ppm and δ 2.404 ppm for protons of CH_2-O groups and CH_3-C=N group, respectively. Another signal at δ (0.090) ppm is due to protons of $(CH_3)_2$-Si groups. The ^{1}HNMR spectrum for [P5] showed the following signals: signal type singlet in δ(9.023) ppm for proton of OH phenol ring, signals in region δ (8.870-6.545) ppm that attributed for protons of benzene rings and signals at δ 2.800 ppm and 2.600 ppm could be attributed for protons of CH_3-C=N and CH_3-ph groups respectively, signals in region δ (0.013-0.994) ppm for protons of $(CH_3)_2$-Si groups.

3.2 Antibacterial activity test

The rate of inhibition of the polymer P_5 with different loading ratios of silver nanoparticles (1, 3, 5, and 7)% was investigated to observe the effect of different amounts of nanoparticles on antimicrobial polymeric films. Investigations against two types of bacteria; *Escherichia coli (G-) and Staphylococcus aureus* (G+), were performed according to the agar diffusion method, using DMSO to prepare polymer solutions, and the Petri dishes were sterilized for 25 min at 37ºC. All the plates were incubated at 37°C for 24 hours before removing them. The load of 1% did not produce sufficient inhibition, while the percentages of 5% and 7% showed distinct efficacy against *E.coli* better than others [25–28], while the samples had low activity against *Staphylococcus* bacteria. The present results are shown in **Table 3** and **Figure 1**. However, 7% is the best percentage for both types of bacteria. The experiment was repeated for all the polymers P_1-P_6 and silver nanocomposites P_1'-P_6' with loading ratios of 7% wt. of silver nanoparticles to observe the effect

of amounts of 7% wt. from nanoparticles to develop antibacterial polymers and used DMSO for the preparation of polymer solutions except for the polymer P6, which was prepared by the DMF.

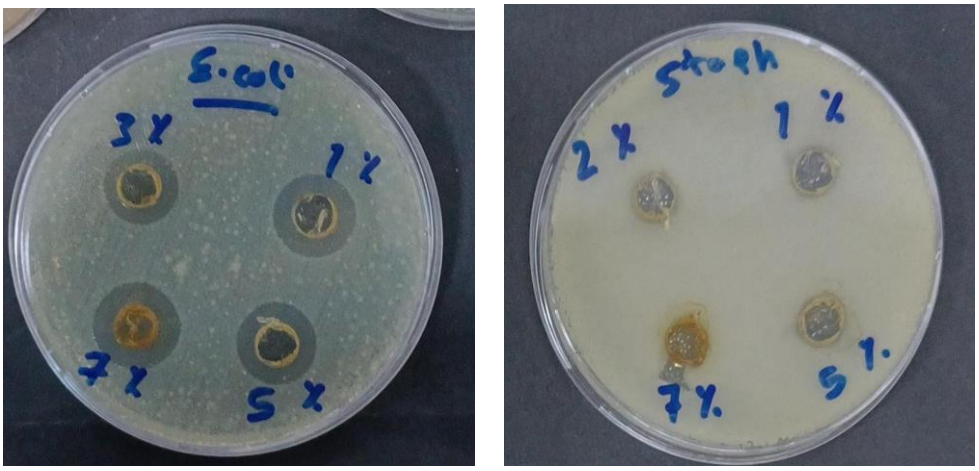


Figure 1. Antibacterial test against *Esherichia coli* and *Staphylococcus aureus*, for nanocomposite P_5 with different weight of Ag 1%, 3%, 5% and 7%.

In this study, a comparison between the inhibition rate of silicone polymers and nanocomposites against two types of bacterial species [(*Esherichia Coli* (Gram-negative) *and Staphylococcus aureus* (Gram-positive)] showed fluctuating activity between low and moderate activity, as shown in **Table 4** and **Figure 2**. In general, the silicon polymers showed less antibacterial activity than the nanocomposites, which means that the silver nanoparticles improved the inhibition against bacteria.

Table 3. Result of bacterial activity test for polymer P_5/Ag with different weights of nanoparticles AgNPs.

Compound	Ag%	*Escherichia Coli*	*Staphylococcus aureus*
P_5/Ag	1%	16	8
	3%	14	8
	5%	16	8
	7%	18	10

Table 4. Result of bacterial activity test for polymers P_1-P_6 and silver nanocomposites P'_1-P'_6 /Ag with 7% weight of nanoparticles AgNPs.

Compound	*E. Coli*	*Staphylococcus aureus*
P1	8	8
P2	8	8
P3	6	12
P4	9	10
P5	8	8
P6	11	15
P'_1	15	6
P'_2	-	10
P'_3	-	8
P'_4	20	25
P'_5	18	10
P'_6	7	12

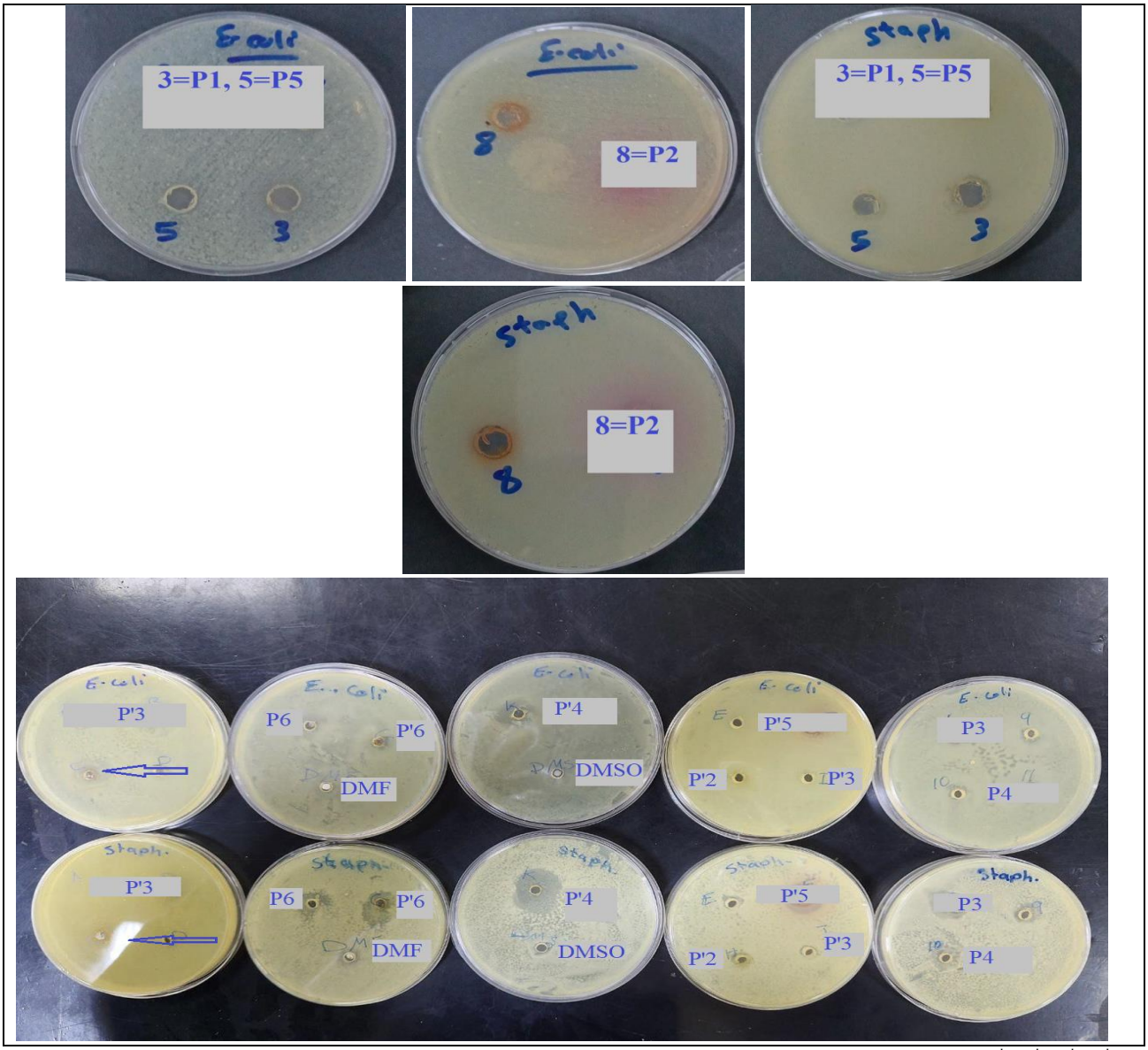


Figure 2: Antibacterial test against *Escherichia coli and Staphylococcus aureus*, P_1, P_2, P_3, P_4, P_5,P_6, P'_1, P'_2, P'_3, P'_4, P'_5 and P'_6.

4. Discussion

The experiment's results to detect the inhibition rate of the polymer P5 with different loading ratios of silver nanoparticles showed that the inhibition rate of polymer 1% did not produce sufficient inhibition. In comparison, the percentages of 5% and 7% showed distinct efficacy against *E. coli* better than others [25-28], while the samples had low activity against Staphylococcus bacteria. However, 7% is the best percentage for both types of bacteria, by repeating the same experiment for polymers P1-P6 and silver nanocomposites P1-P6 with loading ratios of 7% wt. of silver nanoparticles, the comparison between the inhibition rate of silicone polymers and nanocomposites against two types of bacterial species (*Escherichia Coli* (Gram-negative) and *Staphylococcus aureus* (Gram-positive)) showed fluctuating activity between low and moderate activity. In general, the silicon polymers showed less antibacterial activity than the nanocomposites, which means that the silver nanoparticles improved the inhibition against bacteria. The positively charged silver has an antibacterial impact due to its strong binding to the electron donor groups like nitrogen, sulfur, or oxygen found in microbial cell walls. The capacity of silver nanoparticles to attach to and infiltrate the bacterial cell wall, as well as the formation of free radicals by Ag NPs,

which might harm the cell and perforate its membrane [29,30], all contribute to the action of AgNPs on bacterial cells, so the combination of silicone polymers and AgNPs important for medical applications.

5. Conclusion

Silicone polymers were synthesized by condensation polymerization using dichloro(dimethyl) silane (DCDMS) and different organic compounds and structurally characterized using FTIR, ^{1}HNMR techniques. Its efficiency was evaluated in vitro against two bacteria, Gram (+) (*staphylococcus aureus*) and Gram (-): (E. coli), using the agar diffusion technique. The presence of silver nanoparticles (Ag-NPs) significantly increased the antibacterial activities of silicone polymers.

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Conflict of Interest

There are no conflicts of interest.

Funding

None.

Ethical Clearance

This work has been approved by the Scientific Committee at the University of Baghdad at the College of Education for Pure Science (Ibn Al-Haitjam).

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