How to Cite:
Al-Azzawi, A. K. A., & AlAsli, N. J. A. (2022). Preparation, characterization and polymerization of some new compounds for esters derived from cephalosporins and some drugs, the study of some physical applications and evaluation of their biological activity. *International Journal of Health Sciences*, *6*(S1), 14510–14526. https://doi.org/10.53730/ijhs.v6nS1.8900

Preparation, characterization and polymerization of some new compounds for esters derived from cephalosporins and some drugs, the study of some physical applications and evaluation of their biological activity

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***Abstract*---**In this research, esters and polymers were prepared and loaded from the reaction of cephalosporins with carboxylic acids and their interaction with polyvinyl alcohol by the sublimation method. During spectroscopic measurements, including Uv, FTIR, ^{1}H-NMR, ^{13}C-NMR, mass spectrometry, elemental microanalysis, scanning electron microscopy (S.E.M.) analysis, and microanalysis for the elements (C.H.N.), the electrical conductivity and the biological activity of some prepared compounds and on four types of pathogenic bacteria were studied, two of them were Gram-positive, and two were negative. (Gram-negative), which are (Staphylococcus aureus, Enterococcus faecalis, Klebsiella pneumonia, and Pseudomonas putida), and the use of the Acker Muller-Hinton culture medium (Molar Huntin Agar) Aqueous solutions of the two compounds [DM26, DM18] with concentrations (0.01,0.001,0.0001,0.00001) mg/ml were also prepared using a dimethyl solvent. Sulfoxide (DMSO) The sensitivity test of bacterial isolates used in the study was conducted by diffusion method, and the antibiotics Cefixime, Ceftriaxone, and Ampicillin were used as a control sample.

***Keywords*---**Cephalosporins, Esters, Enterococcus Faecalis, Staphylococcus Aureus, Klebsiella Pneumonia, Pseudomonas Putida.

1. Introduction

They are organic compounds (R-CO-OR'), which is one of the derivatives of carboxylic acids that may be aromatic or aliphatic, saturated or unsaturated, and esters are polar compounds because they contain a carbonyl group, they have a boiling point slightly lower than the boiling point of aldehydes And ketones, whose molecular weights are close to them [1], and esters have a pleasant smell, and are responsible for the scent of many flowers and fruits, and there are manufactured ester materials that are added to other materials to give a fruity smell, including octyl acetate (A factory taste of orange), dipentyl acetate (a factory taste of bananas) [2], and it can also be included in the composition of medicines such as the anti-tumor drug Quedron, a compound such as taxol for the treatment of breast and ovarian cancer [3] Esters can be used in many industries because they are easy to form, whether they are cyclic or noncyclic, and they are used as protective bases for the carboxyl and hydroxyl groups, as their formation results in a group of natural products, including peptides and sugars, which are more prevalent than others [4].

2. Experimental

2.1. Chemicals used: All chemicals used in this work were purchased from BDH, Aldrich and Fluka companies and were used without further purification.

2.2. Devices used: The melting points were measured using Electrothermal Melting Apparatus 9300. The FT-IR spectra were captured using a Shimadzu FT-IR 8400S spectrophotometer with a (400-4000) cm^{-1} by KBr disc. DMSO-d^6 as solvents were used to capture ^{1}H-NMR and ^{13}C-NMR spectra on Bruker instruments running at 400 MHZ.

2.3.1. Preparation of the compound (DM17) [5]

In a round-bottom flask with a capacity of 100 ml, 0.01 mmol) of methanol was dissolved in dioxane with the addition of 7-6 drops) of hydrochloric acid, and from the separating funnel was added 0.01) mole) of cefotaxime dissolved in dioxane drop by drop, and no precipitation occurred. Then, the mixture was raised in a water bath for (3) hours, after which the mixture was cooled and then the mixture was evaporated. The colour has a smell similar to the smell of coffee.

2.3.2. Preparation of compound DM18 [6]

In a round-bottom flask with a capacity of 100 ml, 0.01 mol) of glycol was dissolved in dioxane with the addition of 7-6 drops) of hydrochloric acid, and from the separating funnel was added 0.01) mol) of trimethoprim dissolved in dioxane drop by drop; no precipitation occurred, then the mixture was raised on a water bath for (3) hours, after which the mixture was cooled, then the mixture was evaporated where a thick brown substance was formed colour.

2.3.3. Preparation of the compound DM25 [7]

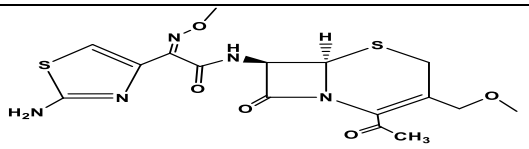
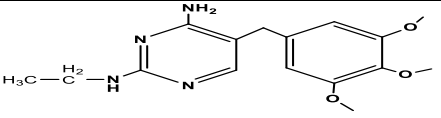
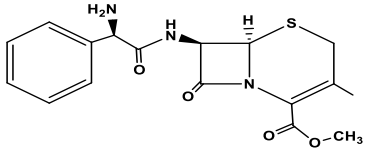
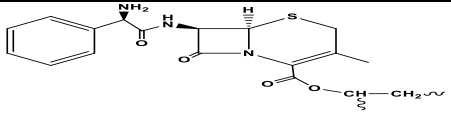
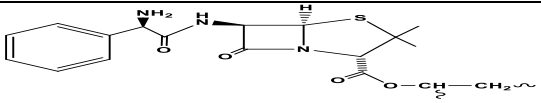
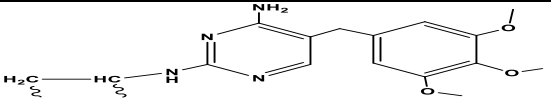
In a round flask of 100 ml capacity (0.01 mol) of polyvinyl alcohol was dissolved in distilled water, then ten drops of HCl were added to it, and from the separating funnel, 0.01) was added. mole) of ampicillin dissolved in distilled water, and there was no precipitation. Then the mixture was raised in a water bath for (3) hours,

after which the mixture was cooled. It was evaporated at a temperature of 120 (^{O}C), Where we noticed an orange resinous substance on top of the mix that combines from time to time; after drying it, we see that it forms a substance similar to glass.

Table (1): shows some physical properties of esters [DM26-DM21, DM17]

Comp. No.	Molecular Formula	Color	M.P ^{O}C	Yield %
DM17	$C_{16}H_{17}N_5O_7S_2$	White	175-177Dec.	75%
DM18	$C_{14}H_{18}N_4O_3$	Brown	93-95	79%
DM21	$C_{16}H_{17}N_3O_4S$	Brown	127-129	78%
DM24	$C_{16}H_{17}N_3O_4S$	Brown	127-129	78%
DM25	$C_{16}H_{19}N_3O_4S$	yellow	96-98	79%
DM26	$C_{14}H_{18}N_4O_3$	Light Brown	93-95	79%

Table (2) shows the prepared ester compounds [DM26-DM21, DM18, DM17]

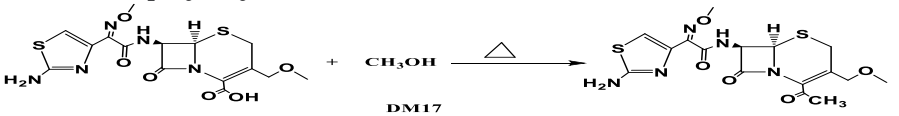
Comp. NO.	**Structure & Name**
DM17	 **(*Z*)-*N*-((6*R*,7*R*)-2-acetyl-3-(methoxymethyl)-8-oxo-5-thia-1-azabicyclo[4.2.0]oct-2-en-7-yl)-2-(2-aminothiazol-4-yl)-2-(methoxyimino)acetamide**
DM18	 **N^2-ethyl-5-(3,4,5-trimethoxybenzyl)pyrimidine-2,4-diamine**
DM21	 **methyl (6*R*,7*R*)-7-((*R*)-2-amino-2-phenylacetamido)-3-methyl-8-oxo-5-thia-1-azabicyclo[4.2.0]oct-2-ene-2-carboxylate**
DM24	 ***sec*-butyl (6*R*,7*R*)-7-((*R*)-2-amino-2-phenylacetamido)-3-methyl-8-oxo-5-thia-1-azabicyclo[4.2.0]oct-2-ene-2-carboxylate**
DM25	 ***sec*-butyl (2*S*,5*R*,6*R*)-6-((*R*)-2-amino-2-phenylacetamido)-3,3-dimethyl-7-oxo-4-thia-1-azabicyclo[3.2.0]heptane-2-carboxylate**
DM26	 **N^2-(*sec*-butyl)-5-(3,4,5-trimethoxybenzyl)pyrimidine-2,4-diamine**

2.4. Study of biological activity:

This Study was used on four types of pathogenic bacteria, two of which are gram-positive, two are gram-negative, and they are Staphylococcus aureus and Enterococcus faecalis, Klebsiella pneumonia, and Pseudomonas putida) are important in the medical field because of their resistance to antibiotics. These bacteria were taken from the laboratories of the College of Education for Pure Sciences, Department of Life Sciences, and a Mueller-Hinton-Akar culture medium was used. Molar Huntin Agar) is used to measure the biological activity of antibiotics and chemicals with medical uses and is used to measure and determine the minimum inhibitor (MIC). Aqueous solutions of the two compounds [DM26, DM17] were also prepared. At concentrations of 0.01), 0.001, 0.0001 (0.00001, mg/ml) and using a solvent dimethyl sulfoxide (DMSO), a sensitivity test was performed for the bacterial isolates that were used in the Study by diffusion method in the nutrient medium of Mueller-Hinton agar, which is a transparent nutrient medium with a dark yellow colour. D in the sensitivity test of microorganisms towards antibiotics because it contains an animal infusion extracted from casein and starch. (Autoclave, then distributed in dishes and left to harden, then small pits were made at the rate of four holes in each dish, then incubated at 37 ^{0}C for 24 hours then the results were read the next day to show the sensitivity of the derivatives used, which depends on the diameter of inhibition evident in the dishes around the holes used, as the increase in the diameter of the inhibition means an increase in the biological activity of the prepared compounds and compared that with the diameter of inhibition for antibiotics [8-11].

3. Results and Discussion

Esters were prepared by reacting one mole of cephalosporins or drugs with one mole of alcohol or polyvinyl alcohol.



3.1. Spectroscopic interpretation (UV, FT-IR.,^{1}H-NMR, ^{13}C-NMR, Mass)

The reaction of esters [DM26-DM17] was confirmed by diagnosing the prepared esters by measurements of UV, FT-IR and ^{1}H-NMR and ^{13}C-NMR spectrum. When studying the ultraviolet (U.V.) spectrum of the prepared esters, we notice the appearance of an absorption band of greater intensity and lower wavelength due to the electronic transitions π→π * and caused by (C=C) bonds, which appear Path chromia displacement" in the prepared esters within the range (280) nm also, a beam of greater wavelength and lower intensity appears, attributed to the electronic transitions *n→π, caused by the unshared electron pairs on the oxygen and nitrogen atoms. OXO and succession totals within a range of (425) nm [12-14].

When studying the infrared (I.R.) spectrum of the [DM21] esters, it was noticed that the stretching band of the hydroxyl group (O.H.) disappeared in the prepared compounds with the appearance of several bands within the range of (3589-3400) cm-1 that goes back to (N.H.) - (NH2), we also notice the appearance of a beam

within the range (3223-3120), which belongs to the stretch of the group (C.H.) olefin, as well as bundles within the field (3063), which belong to the aromatic (C.H.) group, also appeared in the area of (2914) which It refers to the stretching of the aliphatic (C.H.) group, also a band seems within the area (1726) cm-1, which refers to the stretching frequency of the ester carbonyl group, and the band is within the scope of (1688) cm. 1 due to the stretching frequency of the imide carbonyl group, a bar appears within the field (1626), which belongs to the stretching of the (C=C) olefinic group, as well as a band seems within the scope of (1593-1500) cm-1 which belongs to the stretching frequency of the (C=C) aromatic group, and the beam is within the field (1404-1357) cm-1 belonging to the frequency of the (C=C) group curvature of the group (C-N) appeared in the areas (1237-1201) cm^{-1} and beam bending gathering (C-O) appeared in areas (1192-1159) cm^{-1} [15], and as shown in table (3).

Table (3): results of the absorption spectra of ultraviolet (nm) and infrared rays (cm^{-1}) for (DM17-DM21, DM24-DM26)

Comp. No.	UV, λmax (nm), DMSO	ν (NH_2) ν (N-H) ν (OH)	ν(CH) olefinic ν(CH) aromatic ν(CH) aliphatic	ν (C=O) amide ν (C=O) carboxylic	ν(C=C) olefinic ν(C=C) aromatic δ (C-H) alkyl	δ (NH_2) δ (CH)aromatic out of plane ν(C-N), ν(C-O)	-------
DM17	287	3433 3344	3099,3045 2937,2899, 2820	1752, 1728 1650	1610 1537 1437,1386	1650 866,810 1354 1284,1242	-------
DM18	286	3479,3443 3396,3336 ——	3167 3070 2933,2881,	— —	1643,1606, 1595 1531,1500 1419,1342	1676,1643 1315 1263,1240	-------
DM21	286	3446 3446 —	3246 3064 2962,2914	1752 ———	—— 1525 1450,1425,	869 1338 1292,1253, 1234	-------
Comp. No.	λmax	ν (NH_2) and residues (OH), ν(NH)	ν (CH) olefinic ν(CH) aromatic ν(C-H) aliphatic	ν (C=O) ester ν (C=O) amide	ν(C=C) olefinic ν(C=C) aromatic δC-H alkyl	ν (C- N) ν (C-O)	δNH2 δC-C
DM24	285	3423-3257 3433	3257 3072 2941	1776 1691,1664	1633 1556,1523 1502	1333 1247	1650 1523,150 2 1444,141 1
DM25	288	3435-3298 3435	3219,3151 3059,3032 2968,2941	1750 1687,1680	1614 1514 1454,1375	1324 1247	1656 1514,145 4
DM26	287	3470-3242 3471	3144,3113 3090 2993,2929, 2895,2841	— —	1620 1595,1504, 1471,1438 1365,1309	1267 1188	1653 1562,151 2,1444

When performing the NMR analysis of the compound [DM21] shown in Figure (3), we notice a weak signal at (8.6-8.4 ppm) belonging to the (NH_2) group protons). As we note, the protons of the benzene ring appeared as two overlapping signals in the range of (7.5-7.4) ppm, and a signal appeared at (5.4-5.0 ppm). (Belongs to the protons of the olefins that are close to the acid carbonyl group, as well as the

appearance of protons of alkyl groups at the 3.6-3.4) position and a signal appeared at (2.2-1.5) It belongs to the protons of the solvent (DMSO-d^6) [16].

When studying the NMR spectrum of carbon for the compound [DM21] shown in figure (4), it was observed that signals appeared at the position (149.52-110) ppm belonging to the aromatic benzene ring carbons, as well as the appearance of a signal at the site (70.42) ppm attributed to the (CH, aliphatic) group carbon, and the appearance of a signal at the site (62.68) ppm attributed to the group carbon. (CH_2), and the formation of movements at the range 39.98-38.27) ppm attributed to the solvent carbonate (DMSO-d^6) [17].

When studying the mass spectrum of the compound [DM21], a weak peak appears at m/z = 347.1 [$C_{16}H_{17}N_3O_4S$], a second peak appears at m/z=319.12 [$C_{16}H_{21}N_3O_2S$] and the emergence of a third peak m/z=303.1 [$C_{15}H_{17}N_3O_2S$], and a fourth peak [$C_{13}H_{15}N_3O_2S_2$] m/z = 277.1, a fifth peak m/z = 257.2 [C12H21N2O2S], the sixth peak appeared at m/z = 241.1 [$C_{11}H_{17}N_2O_2S$], and a seventh peak appeared at m/z=215.1 [$C_{10}H_{19}N_2OS$], an eighth peak appeared m/z = 190.1 [C10H9NOS], and a ninth peak appeared at m/z = 161.1 [C10H11NO], A tenth peak m/z = 132.0 [C8H6NO], and an eleventh peak appeared at [C6H4NO] m/z = 106.0, which is the peak of the baseline. Also, twelve peaks appeared at m/z = 77.0. [C6H5], and the last peak at m/z = 57.9 [C4H7], and the peak of the baseline proves the validity of the compound, while the rest of the peaks prove the structural shape of the compound, and figure (5) explains it [18].

3.2. Microanalysis of the Elements (C.H.N)

A precise analysis of the elements was carried out for some of the prepared compounds, and the measurements were identical or close to the calculated ratio, as shown in the following table (4):

Table (4): Microanalysis of the Elements (C.H.N)

Comp.	Molecular	Calculated			Found		
No.	Formula	C%	H%	N%	C%	H%	N%
DM17	$C_{16}H_{19}N_5O_5S_2$	45.50	4.50	16.46	45.17	4.46	16.46
DM21	$C_{17}H_{19}N_3O_4S$	56.67	5.30	11.67	56.50	5.30	11.63

3.3. SEM analysis of the scanning electron microscope:

The effect of laser bombardment by scanning electron microscopy for the two compounds [DM25-DM17]. The compound [DM21] at 10μm) appears as rocky cliffs scattered with rocky pieces of different sizes and densities, where we notice that the trenches have expanded and become more scattered. The compound [DM26] appears at the same distance. The surface seems like scattered wooden fragments containing channels and voids between them. Compound [DM21] appears at (1μm) as large ice chunks scattered over them with different densities and deep trenches and gaps between them. At the same distance, a compound [DM26] appeared Arrayed with wooden poles. The compound [DM21] at 500nm appears as if it were accumulated rocks interspersed with deep valleys with voids between them. The compound [DM21] appears at (200 nm) as large rocks scattered on it with small rock pieces interspersed with voids, and the

phenomenon of nanoparticles became clear. At the same distance, the compound [DM26] appeared as if it was large rocks scattered on it with small rock pieces interspersed with voids, and the phenomenon of nanoparticles became clear.

3.4. Electrical conductivity

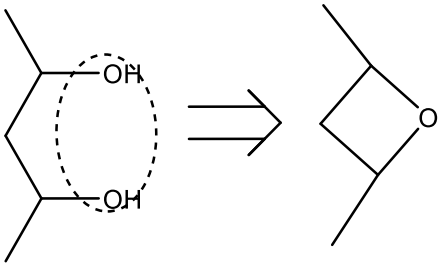
Electrical conductivity is widely used in coordination chemistry to find possible ionic formulas. It occurs in a compound when it is a solution or a solid, and the degree of conductivity is greater when the number of ions that it releases in the solution is more. At the same time, the degree of conductivity in the complex is low.

Table (5) shows the electrical conductivity values of some compounds prepared using the DMSO solvent

Test	DM17	DM18	DM 21	DM25	DM26
Conductivity .us/cm	4.5	4.2	1.6	5	2.8
C°	18.4	18.3	17.8	18.0	15.6

3.5. Differential scanning calorimetry

The study of the thermal transformations that occur on the crystallized polymer or the glass transition are examples of the studied thermal changes, where the thermal analyzes showed the stability of the compound [DM26] for temperatures exceeding 600 ° C at (225-150) A water molecule is lost from the polymeric chains of unlinked polyvinyl alcohol, and this may lead to the formation of quaternary rings of the corresponding epoxide. The evidence for this is that the crystal occurs with the appearance of epoxides, and after melting, it may be polyether before the dissolution process occurs in it and eat all the chains.



3.6. Biological activity of some prepared compounds

The study of the biological activity of the compounds prepared with certain concentrations showed that most of these compounds have antagonistic activity against the types of bacteria studied, compared to the antibiotic Ceftriaxone, Ampicillin (Cefixime, which is a broad-spectrum antibiotic with an antibacterial activity). It has both positive and negative bacteria (6). It also has a large inhibitory diameter as it gives a high selectivity when studying the sensitivity of bacteria to the prepared compounds, and since this antibiotic is used to treat many infections and diseases such as urinary tract infections (9 -8-7), especially those that occur as a result of infection with colon bacteria and Staphylococcus aureus bacteria. It also treats simple cystitis in females caused by colon bacteria and treats chronic bacterial prostatitis caused by colon bacteria, in addition to infections of the lower respiratory tract and sinusitis, arthritis and bone (11-10). It is also used to treat diarrhea caused by colon bacteria and effectively treat

typhoid. Therefore, two compounds of the compounds prepared in this research [DM26, DM17] were studied on different types of chromium-positive and negative bacteria, which recorded a global antagonistic activity against the bacteria studied, and compared with the mentioned antibiotics, it is possible to use these compounds As a treatment for the same infections and pathological conditions above, after investigating the biological path of these compounds, their side effects, and the amount of their accumulation in animal tissues. Different compounds (0.01,0.001,0.0001,0.00001 mg/ml), where the diameter of the inhibition ranges between (0 mm minimum diameter of inhibition to 40 mm maximum diameter of inhibition measured) and the table below shows the inhibitory activity of some of the prepared compounds. The figures show that the value of the inhibition varies according to the compound, and this is due to the low baseline and the presence of resonance.

Table (6): The inhibitory activity of the two compounds [DM26, DM17] in the growth of several positive and negative bacteria (the diameter of inhibition measured in mm)

Test	*Staph aureus*	*Enterococcus faecalis*	*Pseudomonas putida*	*Klebsiella pneumoniae*	*Antibiotic*
DM17 0.0 1	20	34	30	36	S.a 0
A 0.001	12	26	20	20	E.F 18
B0.0001	10	24	22	18	
C 0.00001	6	9	20	4	
DM26 0.0 1	36	46	40	40	E.F 32
A 0.001	34	42	36	30	P.P 10
B 0.0001	22	40	30	20	
C 0.00001	-	24	18	18	

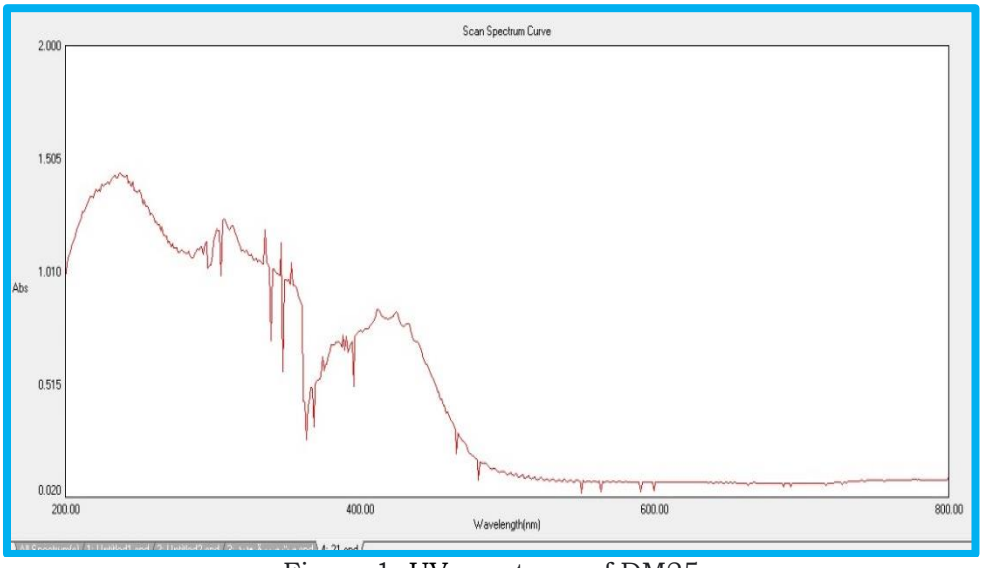


Figure 1: UV spectrum of DM25

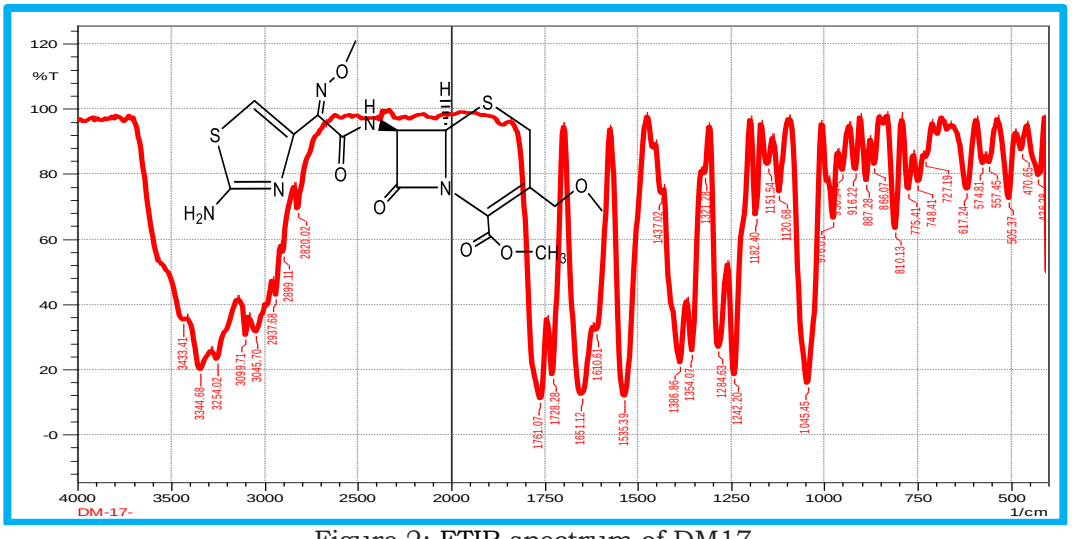


Figure 2: FTIR spectrum of DM17

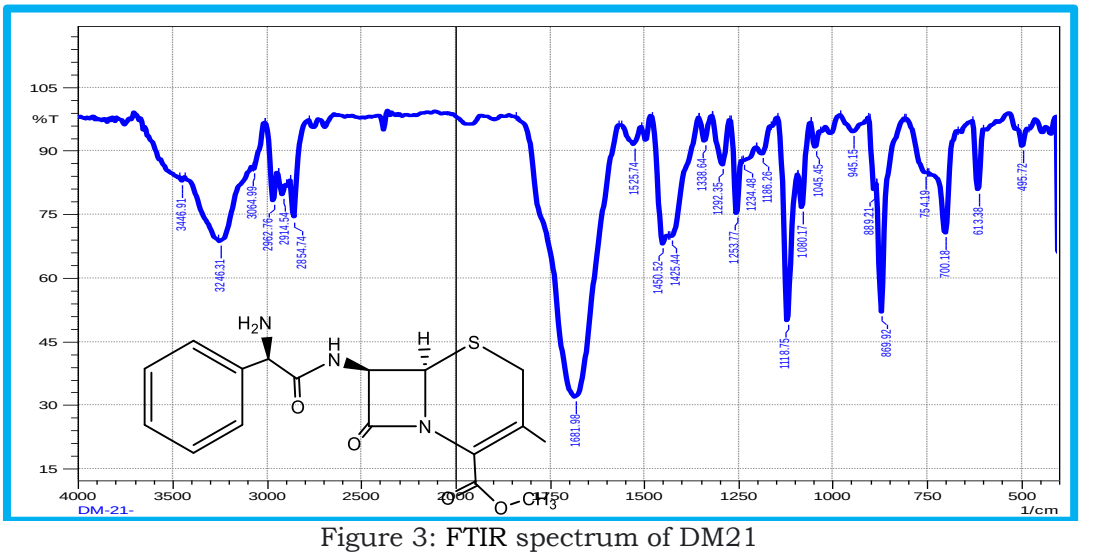


Figure 3: FTIR spectrum of DM21

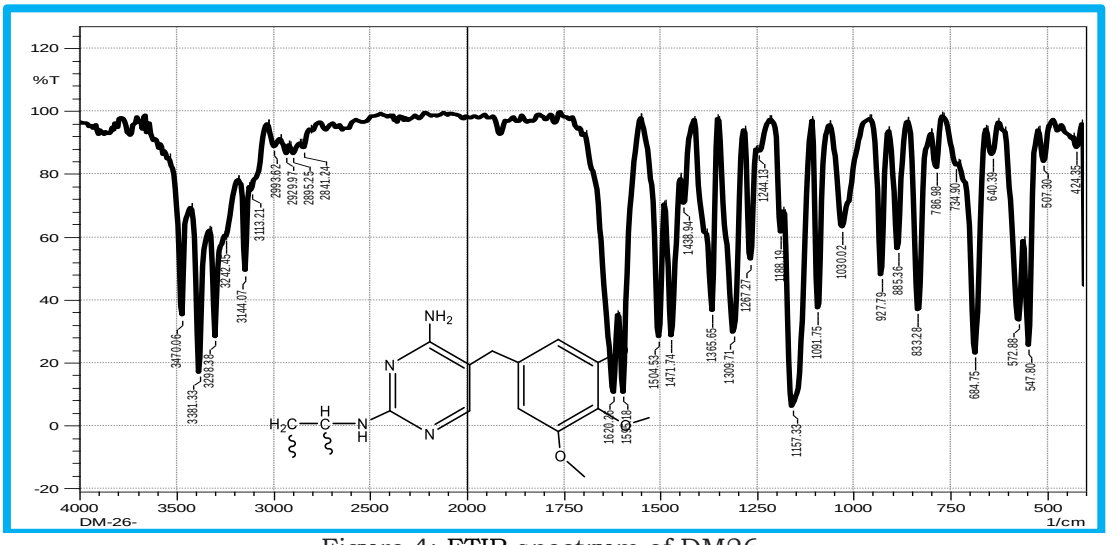


Figure 4: FTIR spectrum of DM26

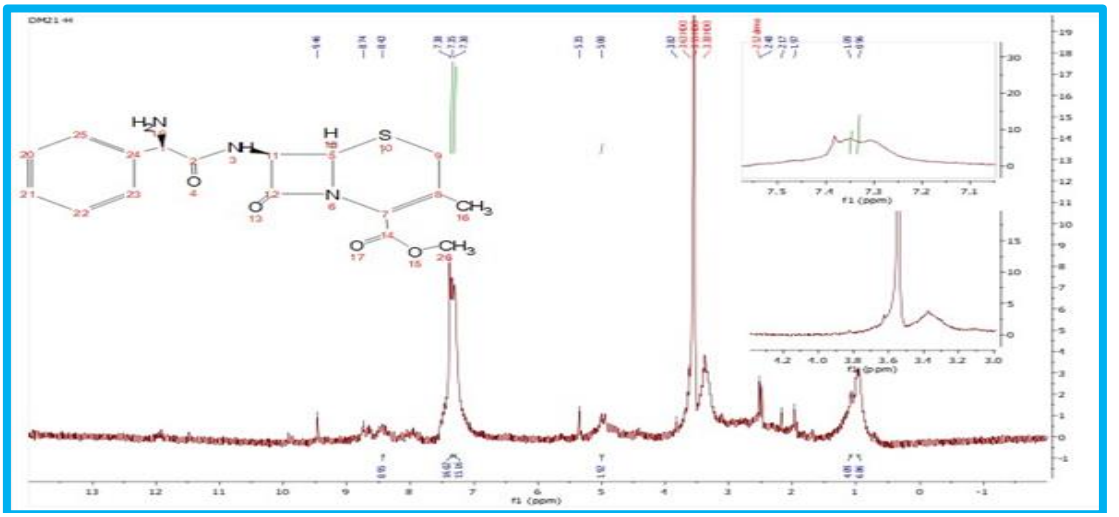


Figure 5: ^{1}H-NMR spectrum of DM21

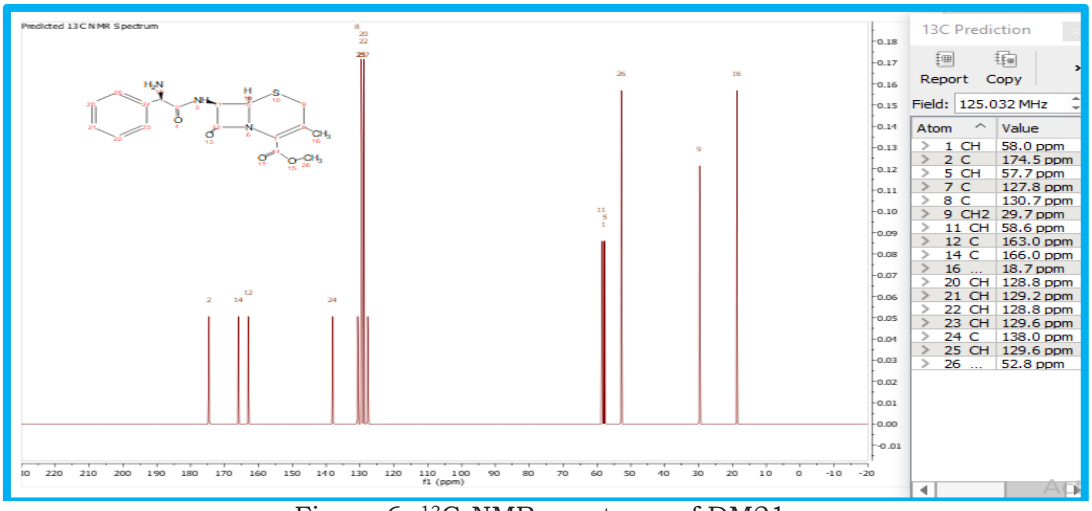


Figure 6: ^{13}C-NMR spectrum of DM21

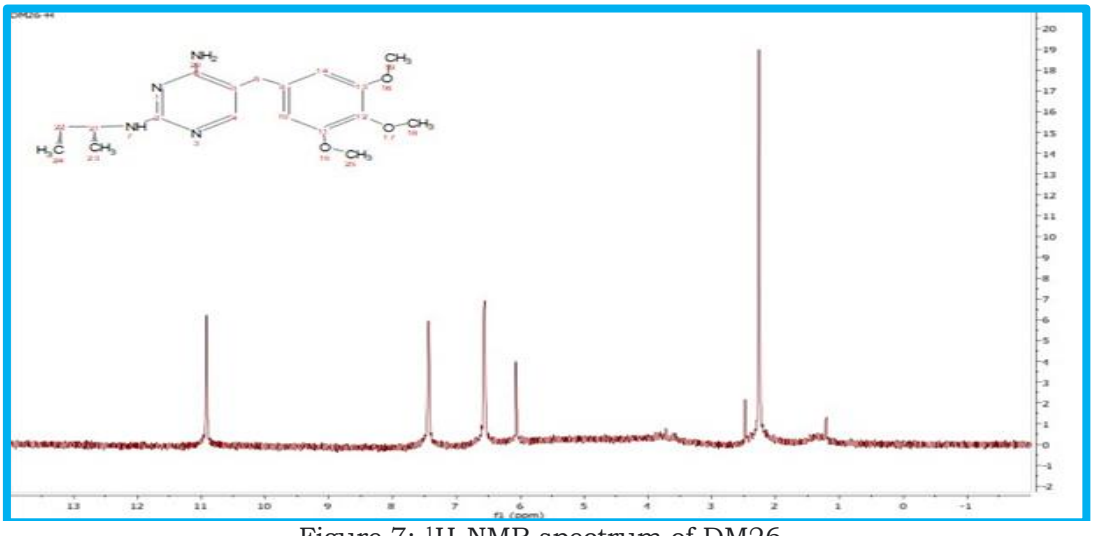


Figure 7: ^{1}H-NMR spectrum of DM26

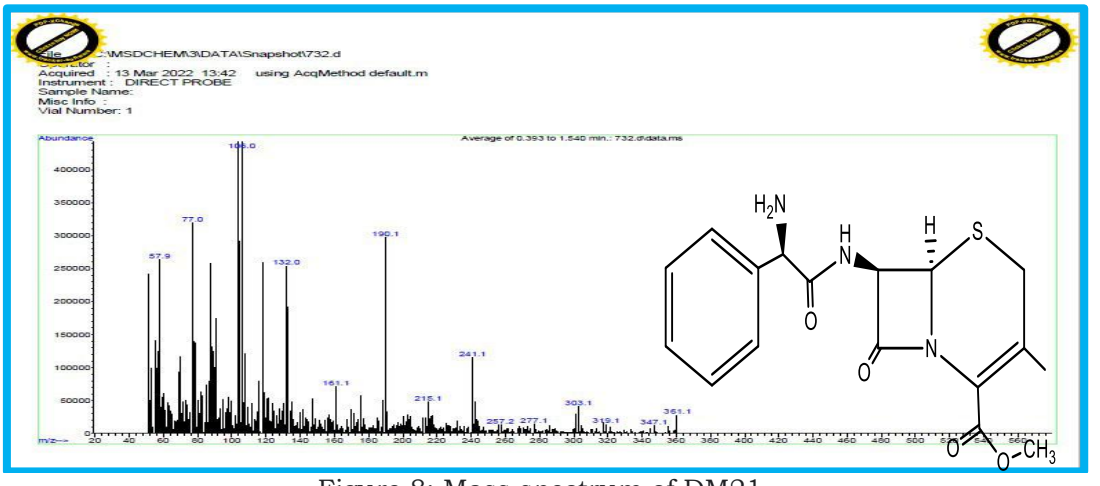


Figure 8: Mass spectrum of DM21

Eager 300 Summarize Results

Date : 2002/01/03 at 00:12:32
Method Name : Nitrogen/Carbon/Hydrogen/Sulphur
Method Filename : N C H S system.mth

Filename	AS Method	Vial
DM21		

#	Group	Sample Name	Type	Weig.	Pro.F	---
58	12	DM21	UNK	2.369	6.25	---

Component name	Element %
Nitrogen%	11.67007542
Carbon%	56.67710495
Hydrogen%	5.680978775
Sulphur%	0

1 Sample(s) in Group No : 12

Component Name	Average	Std. Dev.	% Rel. S. D.	Variance
Nitrogen%	11.67007542	0	0.0000	0.0000
Carbon%	56.67710495	0	0.0000	0.0000
Hydrogen%	5.680978775	0	0.0000	0.0000
Sulphur%	0	0	0.0000	0.0000

Figure 8: C.H.N. of DM21

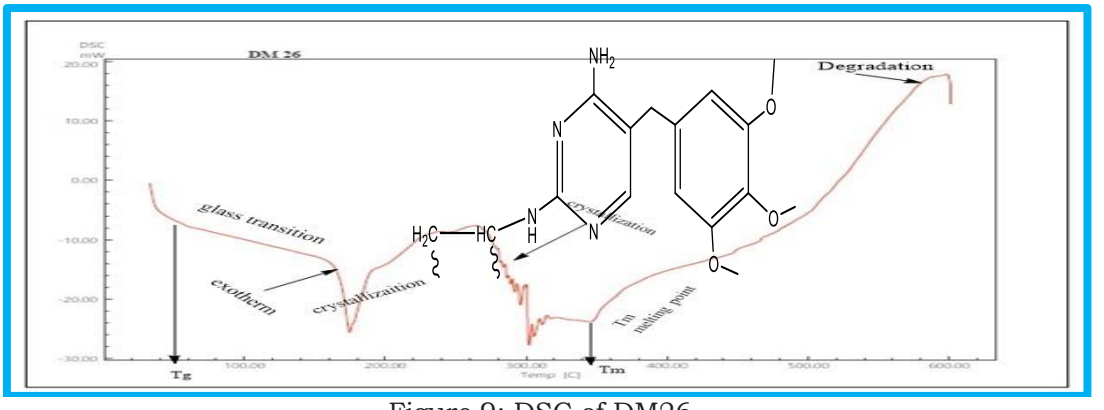


Figure 9: DSC of DM26

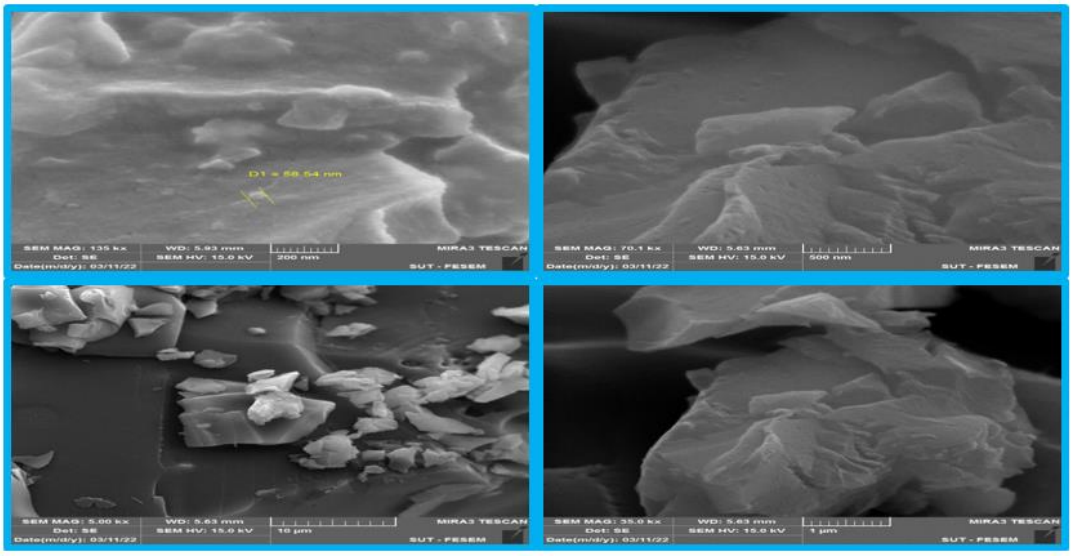


Figure 10: SEM of DM21

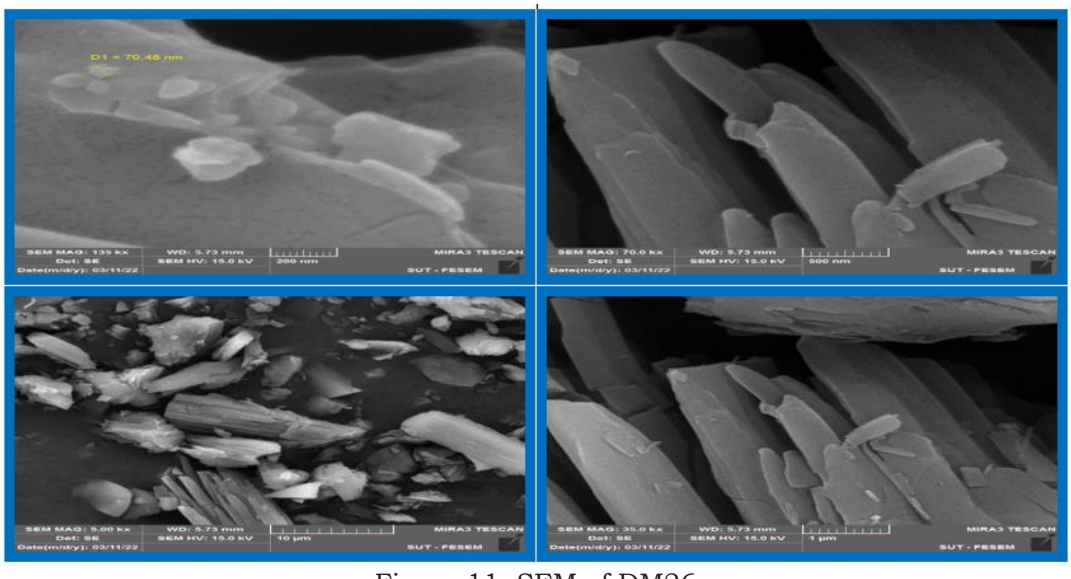


Figure 11: SEM of DM26

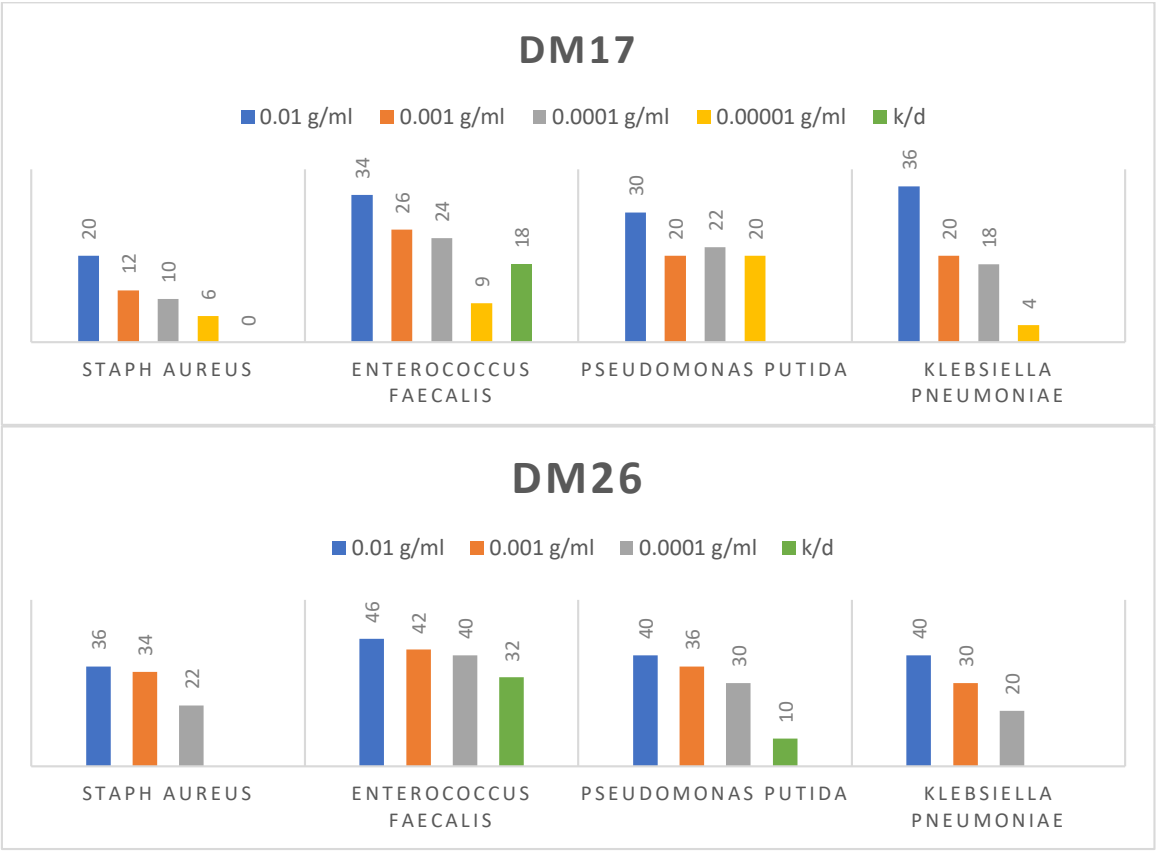


Figure (12): Inhibitory activity of (DM17 and DM26) on the four types of bacteria

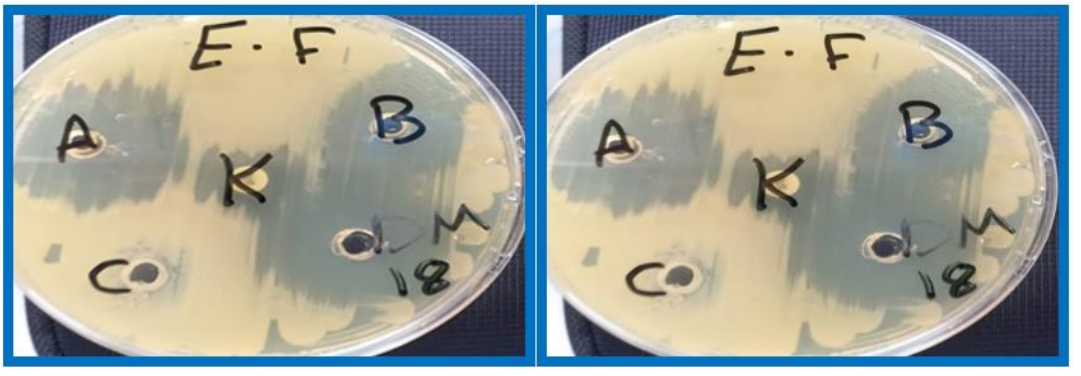


Figure 13: Compound (DM18) inhibits the growth of bacteria Pseudomonas putida and Enterococcus faecalis

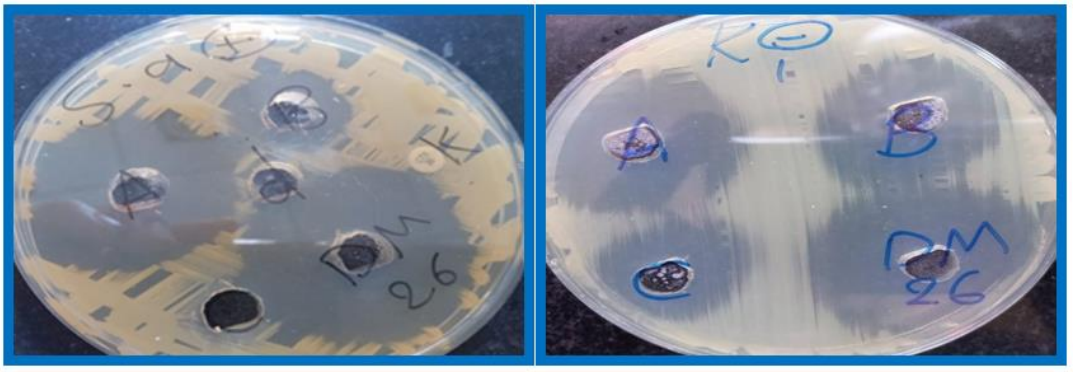


Figure 14: Compound (DM26) inhibits the growth of bacteria Klebsiella pneumoniae and Staph aureus

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