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Article

Preparation and Characterization of Some Imidazoline-4-Ones and Study of Their Biological and Laser Activity

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Abstract: Four novel imidazolidin-4-one derivatives (H7-H10) were synthesized via reaction of Schiff base compounds with glycine in the presence of triethylamine as an organic base and THF as a solvent. The synthesized derivatives were characterized using FT-IR, ^{1}H-NMR, and ^{13}C-NMR spectroscopy. Spectroscopic data confirmed the formation of the required derivatives by observing the absorption bands and signals of functional groups and heterocyclic ring in their spectra. In vitro biological evaluation revealed that all synthesized derivatives H7-H10 showed antibacterial activity against Escherichia coli and Staphylococcus aureus, and inhibition zones were increased with increase in concentration of synthesized derivatives. Compound H8 exhibited the highest antibacterial activity against E. coli with inhibition zone 18 mm at concentration 0.01 mg/mL and H9 compound exhibited the highest activity against S. aureus with inhibition zone 19 mm at same concentration. It was shown that irradiation with laser light of a wavelength of 808 nm caused a number of transformations in the parameters such as color, melting points, and Rf TLC values of synthesized compounds. The greatest changes were observed in the first 30 seconds of irradiation, and the investigated parameters became relatively constant in the time period from 40 to 60 seconds. Therefore, it can be concluded that synthesized imidazolidin-4-one derivatives are promising in terms of antibacterial activity and have a certain effect from laser radiation.

Keywords: laser activity, hydrazones, imidazoline, glycine, Schiff base.

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1. Introduction

Since they have several anti-disease qualities and are regarded as antifungals, antibacterials, and anti-inflammatory substances, these heterocyclic compounds have a wide range of biological uses [1]. Since imidazolidine derivatives are very effective and have broad biological significance, and imidazole is a crucial pharmaceutical component, most of their compounds are replaced in the (5,3) locations [2]. The presence of imidazole rings is common in natural products and pharmaceuticals; therefore, imidazole heterocycles are one of the most important nitrogen-containing heterocycles. Moreover, imidazole heterocycles are an indispensable component of medicinal chemistry and play a crucial role in curing various diseases. Nowadays, new medicinal derivatives are rapidly developed globally [3]. Because of the special structure features of imidazole heterocycle and the presence of a rich electron donor group, there is an advantage in binding of the imidazole group with different receptors and enzymes in biological organisms through weak interactions, showing various biological effects. Nowadays, numerous biologically active imidazole heterocycles are actively used in clinics as drugs for treatment of various diseases including antibacterial [4], antifungal [5], and anti-inflammatory diseases [7]. Due to the great significance of the pharmacological/biological effects of imidazole-based heterocycles and high medicinal value, medicinal chemists and synthetic chemists have found their antimicrobial [7], antiparasitic [8], and anticancer effects [9]

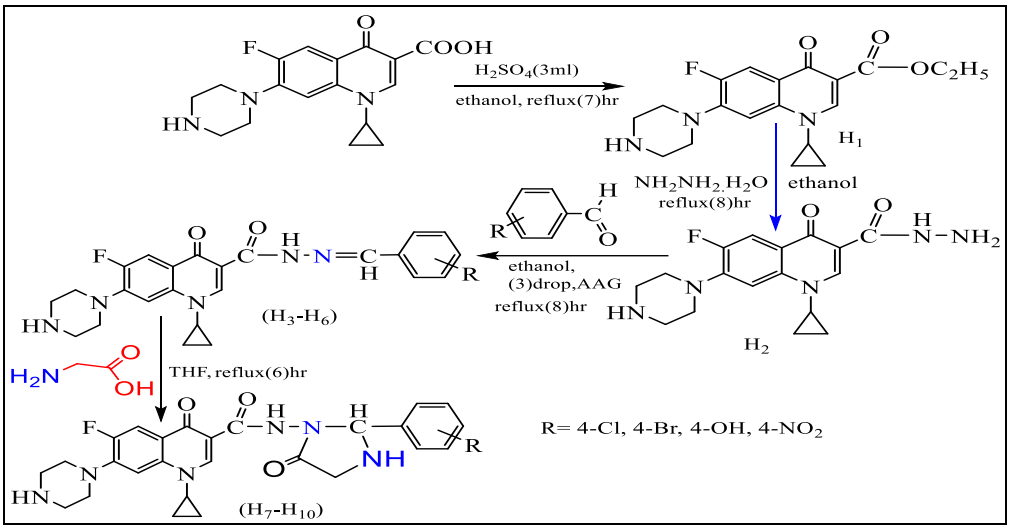
2. Materials and Methods

Chemicals used: Aldrich, BDH Thomas, Fluka, and Merck chemicals were utilised.

Preparations of Imidazolidine: Imidazolidine was prepared from previously prepared Schiff bases (0.002) mole with glycine (0.004) mole, where Schiff base and glycine were dissolved in (10 ml) of (THF) solvent, mixed in a flask, and (3-5) drops of triethylamine (TEA) were added as a catalyst, and the reaction was escalated for (6) hours. Precipitates of different colors and proportions were obtained. They were purified and their melting points were measured [10], as in Table (1).

Table 1: Physical properties of the prepared compounds (H6 - H10)

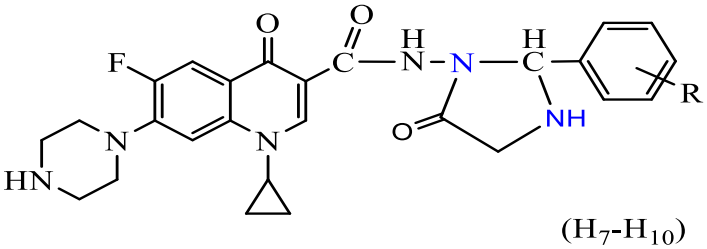
Comp. No.	R	Molecular formula	M.P C)⁰(	Yield %	Color
H_7	OH	$C_{31}H_{29}FN_6O_4$	243-245	57	grand
H_8	Cl	$C_{31}H_{28}ClFN_6O_3$	267-269	61	Yellowish
H_9	Br	$C_{31}H_{28}BrFN_6O_3$	254-256	60	Orange
H_{10}	$N(CH_3)_2$	$C_{33}H_{34}FN_7O_3$	268-270	51	White



Scheme: Prepared compounds path

3. Results

Characterization of the compounds: (H_7-H_{10}): prepared Imidazolidin-4-one from previously prepared Schiff bases with glycine, where Schiff base and glycine were dissolved of (THF) solvent, mixed in a flask, and (3-5) drops of triethylamine (TEA) were added as a catalyst, and precipitates of different colors and proportions were obtained



(H_7-H_{10})

Table 2. shows the compounds' IR spectrum [H_7, H_8, H_9, H_{10}].

Comp. No	R	ν N-H	ν Ar -H	ν C=O	ν C=C Ar	ν C-N	Others
H_7	4-OH	3388	3073	1669	1594,1530	1333	νOH 3477
H_8	4-Cl	3419	3056	1676	1604,1521	1388	ν C-Cl 808
H_9	4-Br	3433	3045	1697	1602,1621	1386	νC-Br 675

Comp. No	R	ν N-H	ν Ar -H	ν C=O	ν C=C Ar	ν C-N	Others
H_{10}	4-$N(CH_3)_2$	3397	3043	1682	1601,1533	1351	νCH_3 sy 2847 asy 2923

The proton NMR spectrum of the compound (1H-NMR) (H8) showed a signal at (δ=2.93,3.29) for the piperazine protons, a signal at (δ=3.97) for (NH) Imidazole, a signal at (δ=7.56-8.02) of the aromatic rings, a signal at (δ=8.82) of (CH) Imidazoline, a signal at (δ=10.11) of (O=C-NH) amide[11,12], as in Figure (1).

The proton NMR spectrum of the compound (1H-NMR) (H9) showed signals at (δ=2.14,3.48) of the piperazine protons, a signal at (δ=3.51) of (N-H) Imidazoline, a signal at (δ= 6.07-7.15) of the aromatic rings, a signal at (δ=7.18) of (CH) Imidazoline, a signal at (δ=11.51) of (O=C-NH) amide, as in Figure (2).

When examining the produced compound's (H8) ^{13}C-NMR spectrum, a signal at frequency (δ=36.33,50.99) goes back to carbon in aliphatic, a signal at(δ=106.59-161.05) of (Ar-C=C), a signal at (δ=166.46) of (C=O) amide, and a signal at (δ=176.82) of (C=O) ketone, as in Figure (3).

When examining the produced compound's (H9) ^{13}C-NMR spectrum, a signal at (δ=34.70,49.36) goes back to carbon in aliphatic, a signal at(δ=104.96-150.26) at (Ar-C=C), a signal at (δ=159.41) of (C=O) amide, a signal at (δ=175.18) of (C=O) ketone, as in Figure (4).

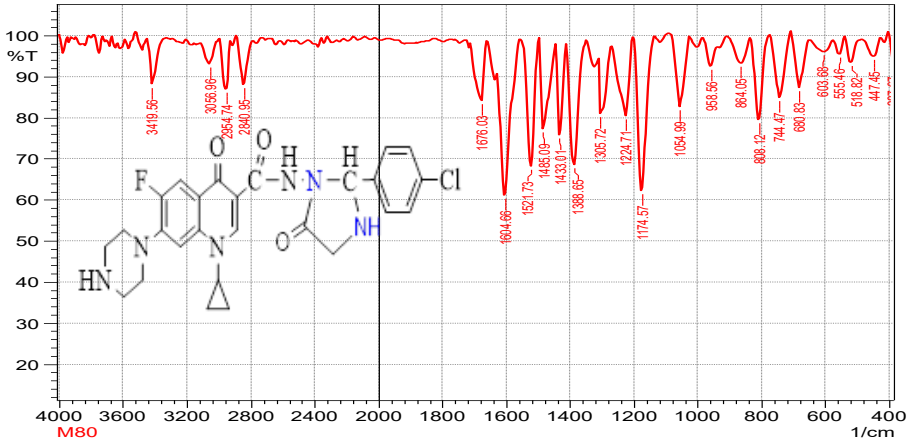


Figure 1. FT-IR for H8

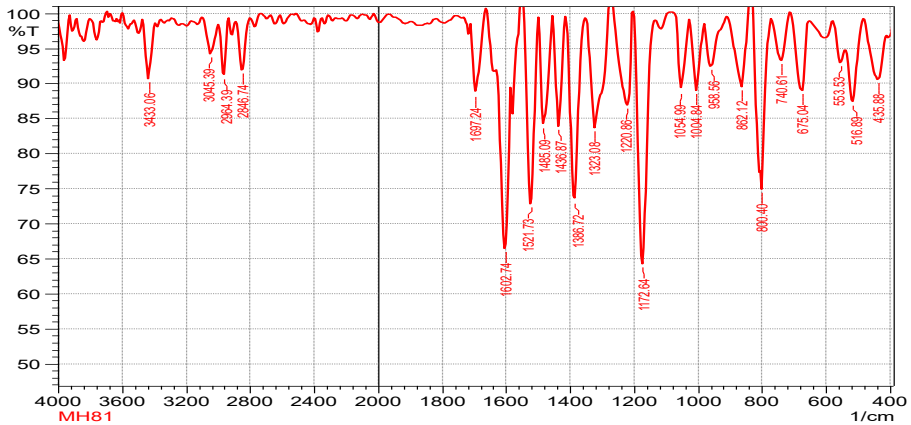


Figure 2. FT-IR for H9

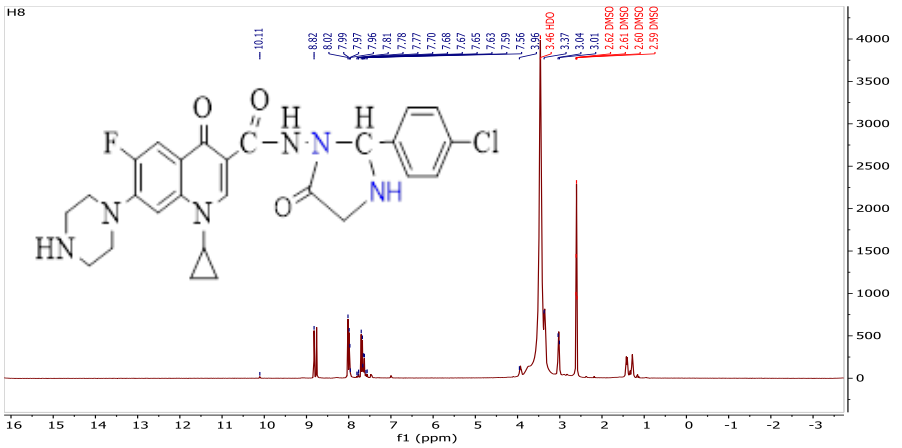


Figure 3. (1H.NMR) for (H_8).

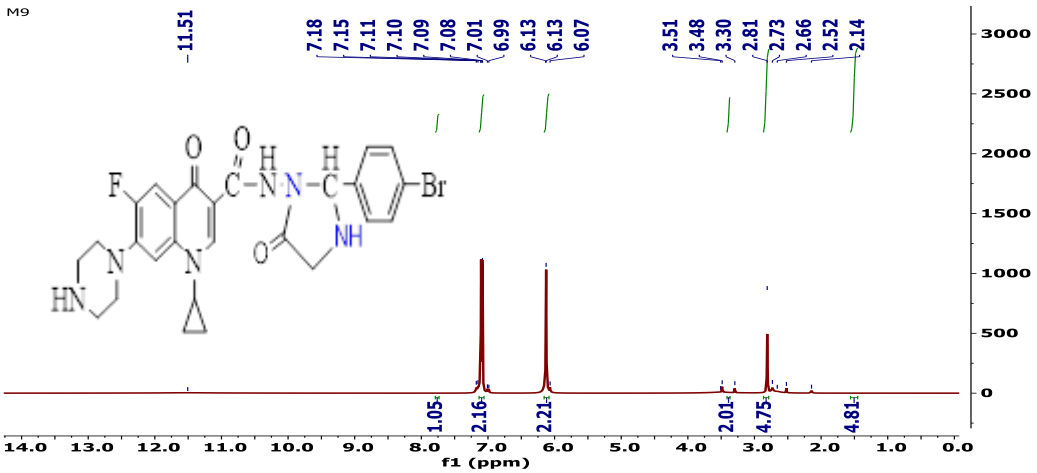


Figure 4. ^{1}H.NMR for (H_9).

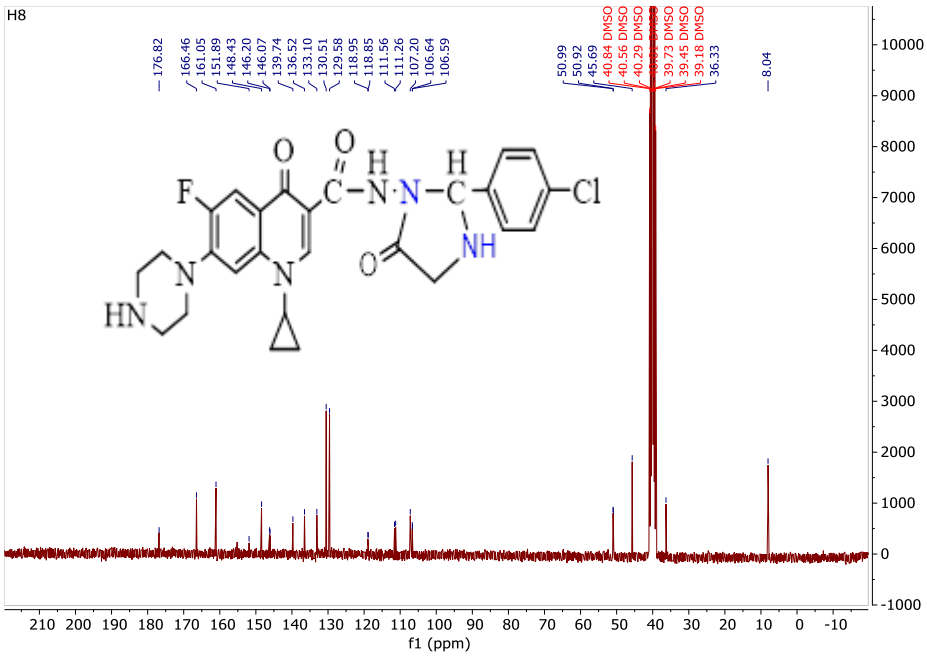


Figure 5. ^{13}C.MNR for (H_8).

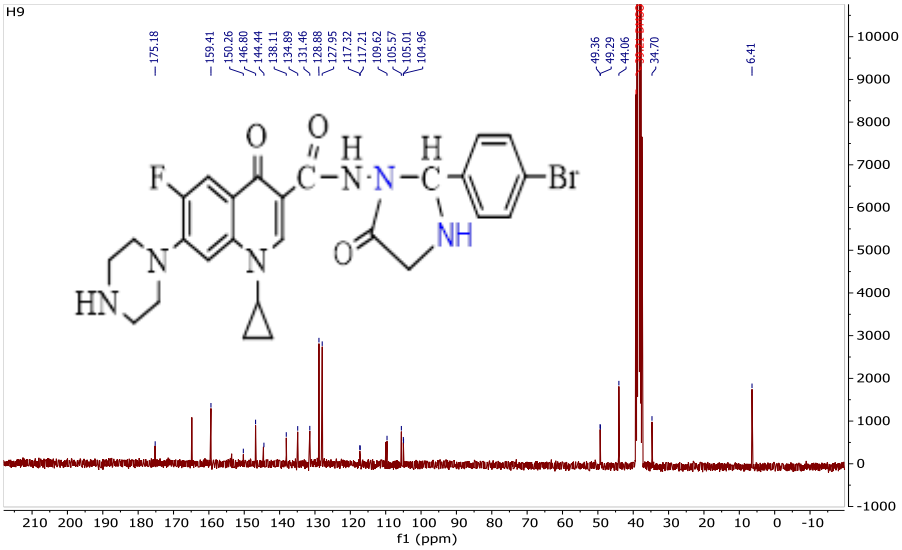


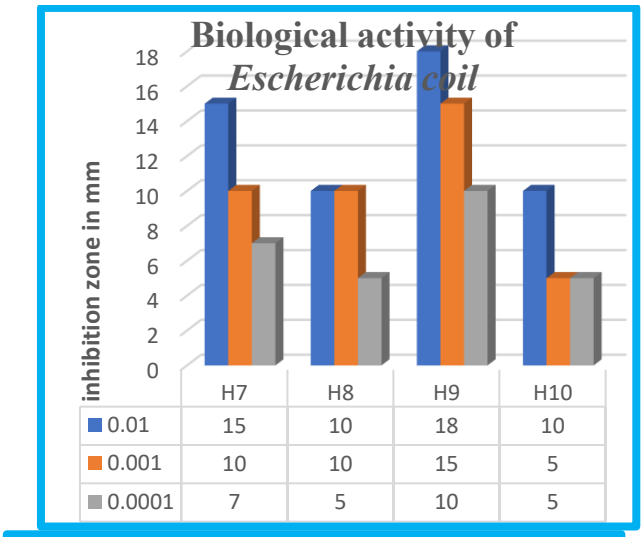
Figure 6. ^{13}C.MNR for (H_9).

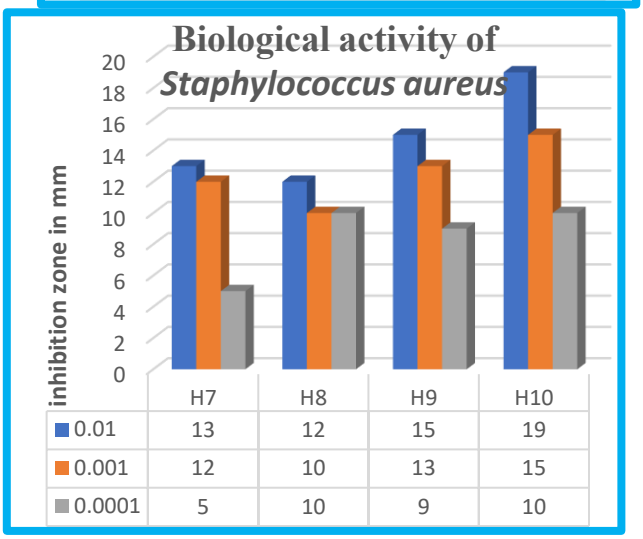
Biological efficacy evaluation

The antibacterial activities of the compounds H7–H10 were measured for Escherichia coli and Staphylococcus aureus at three different concentrations of 0.0001, 0.001, and 0.01 mg/mL. The findings indicated the effects of varying concentrations on the inhibition zones and different levels of bacterial susceptibility. Compound H7 displayed a moderate antibacterial activity towards E. coli with the zone sizes measuring 5, 10, and 10 mm respectively [13,14]. The activity of the compound H7 against S. aureus was enhanced slightly from 10 mm to 12 mm with increasing concentrations. Compound H8 displayed the highest antibacterial activity compared to other compounds, especially against E. coli where there was an increase in the zone sizes from 10 to 18 mm. Against S. aureus, the zone sizes varied between 9 to 15 mm [15-18]. Compound H9 was less active towards E. coli in the lower concentrations (5 and 5 mm), with its activity being increased to 10 mm in 0.01 mg/mL, compared to its higher activity towards S. aureus, where its activity was increased from 10 to 19 mm. The overall observation in the experiment is that an increase in the concentration leads to an increase in the antibacterial activity, with H8 having the highest antibacterial activity towards E. coli and H9 having the highest activity towards S. aureus. This difference can be attributed to the difference in the molecular structures of the synthesized diazepine derivatives [19-22].

Table 3. The biological activity in millimeters around the agar well of chemical substances synthesized for S. aureus and E.coli.

	Staphylococcus aureus			***Escherichia coli***		
Comp. No.	**0.01**	**0.001**	**0.0001**	**0.01**	**0.001**	**0.0001**
H_7	13	12	5	15	10	7
H_8	12	10	10	10	10	5
H_9	15	13	9	18	15	10
H_{10}	19	15	10	10	5	5





Scheme 2: Antibiotic effect of the synthesized compounds on E. coli and Staph. Aureus

The laser activity

To study the impact of the laser irradiation on the synthesized compounds, H7, H8, H9, and H10 were exposed to laser radiation for varied periods of time such as 30, 40, 50, and 60 s. Laser irradiation was carried out using varied parameters, namely distance between the source of the laser and the compound sample, which was equal to 10 cm; power of the laser was 1 mW; wavelength of the laser was 808 nm. Physical properties of the synthesized compounds such as color, melting point, and Rf values obtained by TLC analysis were studied after irradiation for varied periods of time. As can be seen from Table 4, significant differences were noted after 30 s of laser irradiation. For instance, the color of H7 was changed from white with a melting point of 147-149 °C and Rf value of 0.67 to green with a melting point of 152-154 °C and an Rf value of 0.41. In the same way, the changes of the color, Rf values, and melting points of H8, H9, and H10 after irradiation were noticed. As the time of irradiation was increased from 40 to 60 s, the Rf values, colors, and melting points did not change significantly, indicating that the major changes occur within the first 30 seconds of irradiation. It can be stated that exposure of the synthesized compounds to the laser beam can cause some structural or photochemical changes in them due to the possible rupture and rearrangement of chemical bonds and production of new products. It is evident that changes in TLC Rf values and melting points are the results of exposure of the compounds to the laser beam for a long enough period or with a sufficient energy [23-25].

Table 4. Illustrates the laser performance of the synthesized materials.

Comp No.	30 S			40 S			50 S			60 S		
	Color	M.P (^{0}C)	R*f*	Color	M.P (^{0}C)	R*f*	Color	M.P (^{0}C)	R*f*	Color	M.P (^{0}C)	R*f*
H_7	grand	200-202	0.49	grand	200-202	0.49	grand	200-202	0.49	white	147-149	0.67
H_8	Yellowish	218-220	0.44	Yellowish	218-220	0.43	Yellowish	218-220	0.44	greenish	152-154	0.41
H_9	White	224-226	0.59	White	224-226	0.57	White	224-226	0.59	Light white	138-140	0.44
H_{10}	White	174-176	0.57	White	174-176	0.57	White	174-176	0.57	Light white	122-124	0.63

4. Discussion

The combined spectroscopic, antibacterial, and laser-response findings demonstrate that the synthesized imidazolidin-4-one derivatives possess structure-dependent physicochemical and biological properties. The FT-IR bands assigned to N–H, C=O, aromatic C=C, and C–N groups, together with the characteristic ^{1}H-NMR and ^{13}C-NMR signals recorded for H8 and H9, support the formation of the intended heterocyclic framework and are consistent with previously reported imidazolidin-4-one derivatives [10]–[12]. Antibacterial activity generally increased with concentration, indicating a dose-dependent response, although the magnitude of inhibition varied according to both the substituent and bacterial strain. At 0.01 mg/mL, H9 produced the largest inhibition zone against *E. coli* (18 mm), whereas H10 was the most active against *S. aureus* (19 mm). This selectivity suggests that the bromo and dimethylamino substituents may differently influence lipophilicity, electronic distribution, and interactions with the distinct cell-envelope structures of Gram-negative and Gram-positive bacteria, in agreement with the variable antimicrobial behavior reported for related heterocyclic derivatives [13]–[22]. Exposure to an 808 nm laser also altered the color, melting point, and TLC Rf values, with the most pronounced changes occurring during the initial irradiation interval and comparatively limited variation thereafter. These observations indicate a rapid photochemical or structural response followed by relative stabilization, possibly involving bond rearrangement, changes in intermolecular organization, or the formation of photoproducts, as observed in comparable laser-responsive heterocyclic systems [23]–[25]. Nevertheless, because the structural changes following irradiation were inferred from physical properties rather than confirmed by post-irradiation spectroscopy, further FT-IR, NMR, mass-spectrometric, and quantitative microbiological analyses are required to establish the underlying reaction mechanism and therapeutic relevance.

5. Conclusion

Antibacterial activity of the synthesized imidazole derivatives, H7-H10, was demonstrated in a wide range against E. coli and Staphylococcus aureus; however, the antibacterial activity increased with increase in concentration of the derivative used. From the antibacterial test results, H8 exhibited the most remarkable activity against E. coli, where the inhibition zone was 18 mm at a concentration of 0.01 mg/mL while H9 exhibited the most remarkable activity against S. aureus, where the inhibition zone was 19 mm at the same concentration.Moreover, studies with laser irradiation showed that synthesized compounds were influenced by laser radiation, as there were certain changes observed in color, melting point, and Rf values on TLC plates after irradiation of samples. Most noticeable changes were recorded during the first 30 seconds of exposure, while at the further time points the studied parameters showed relatively stable values. Such changes

might be caused by some structural modifications, photochemical reaction, or bond cleavage. All in all, the results of antimicrobial activity and laser influence prove that synthesized imidazole derivatives are biologically active compounds and responsive to controlled laser irradiation.

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