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SYNTHESIS, CHARACTERIZATION AND ANTIMICROBIAL EVALUATION OF SOME NEW 4(3H)-QUINAZOLINONE

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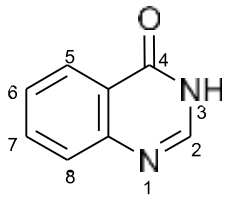
ABSTRACT
Heterocyclic systems comprising quinazolinones have been explored to a major extent in last few decades due to its chemotherapeutic and antimicrobial potential. Quinazolinone is a bicyclic compound containing benzene ring fused with pyrimidine ring. One of the most frequently encountered heterocyclic in medicinal chemistry is 4(3H)-quinazolinone occupies a distinct place in the field of medicinal chemistry due to its wide spectrum of biological activities such as analgesic, antioxidant, antimicrobial, anticancer, anti-inflammatory, antitubercular, antihyperlipidemic, antihistamine, antiviral, anticonvulsant, and antiparkinsonian.

KEY WORDS: *Quinazolines, Synthesis, Spectral data, Heterocycles, antimicrobial, chemotherapeutic.*

INTRODUCTION

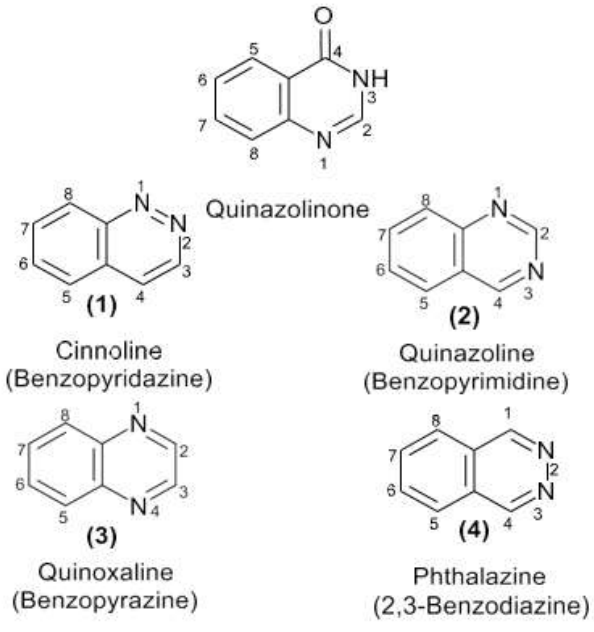
Heterocyclic compound can be aliphatic or aromatic in character depending upon the electronic constitution. The atoms of a simple heterocyclic ring are numbered from the heteroatom which is counted as one [5]. Substituent's are given the lowest possible numbers and then arranged in alphabetical order viz. quinazolinone the nucleus of present study is numbered as follows:



Quinazolinone

Quinazolin-4(3H)-one is a versatile pharmacophor which exhibits wide variety of biological activities like hypnotic, antitussive, analgesic, muscle relaxant, anticonvulsant, antiallergic, antibacterial, hypoglycemic and antispasmodic.

Quinazoline is a compound in which pyrimidine fused with benzene and is called benzopyrimidine. Since N+ and C are isoelectronic, the simplest and most direct hetero analogue of benzene is the pyrimidinium ion. There are several examples of benzene fused with six membered ring containing two nitrogen atoms. Some of the examples are benzodiazines (cinnoline, phthalazine, quinoxaline, quinazoline). Quinazoline or benzopyrimidine is the fused pyrimidine with benzene [12].



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depending upon the position of the keto or oxo group, Quinazolinone compounds may be classified into two types: 2-(1H)quinazolinone or 1,2-dihydro-2-oxoquinazolines and 4(3H)-quinazolines or 3,4-dihydro oxoquinazolines. These system exhibit lactam-lactimtautomerism and undergo hydroxy group replacement reactions.

Quinazoline derivatives, which belong to the nitrogen-containing heterocyclic compounds, have caused universal concerns due to their widely and distinct biopharmaceutical activities. Researchers have already determined many therapeutic activities of quinazoline derivatives, including anticancer1-4, antiinflammation5,6, antibacterial7-10, antivirus11, anti cytotoxin12, antispasm13, antituberculosis14, anti oxidation15, anti-malarial16, anti-hypertension17, anti-obesity18, antipsychotic19, anti diabetes20, etc.

Encouraged by the diverse biological activities of quinazoline heterocyclic compounds, it was decided to prepare a new series of quinazoline derivatives.

The structures of all synthesized compounds were assigned on the basis of IR, Mass, H NMR spectral data analysis. Further, these compounds were subjected for antifungal and antibacterial activity.

EXPERIMENTAL

Material

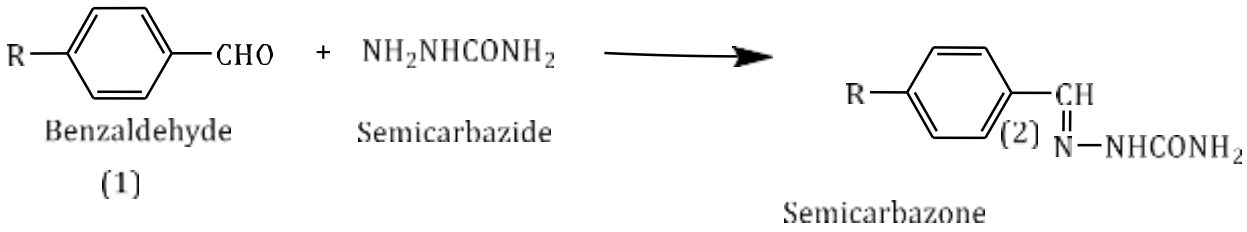
The 2-phenylbenzoxazinone was procured from Sigma Aldrich, Mumbai, India. Semicarbazide and sodium acetate was purchased from CDH, India. Different substituted aldehyde (Benzaldehyde, p-chloro benzaldehyde, o-chloro benzaldehyde, p-bromo benzaldehyde, o-bromo benzaldehyde, p-nitro benzaldehyde, o-nitro benzaldehyde, p- methyl benzaldehyde, p-ethyl benzaldehyde, p-methoxy benzaldehyde) were purchased from Hi-media, India. The chemicals used for the experimental work were of synthetic grade.

Instruments used:-The 1HNMR spectra were obtained at room temperature on a Bruker Avance 400-MHz spectrometer. DMSO-d6 used as solvent. FT-IR spectra were measured at room temperature with a Bruker alpha. Melting points are uncorrected and were determined using a MettlerFp5 apparatus.

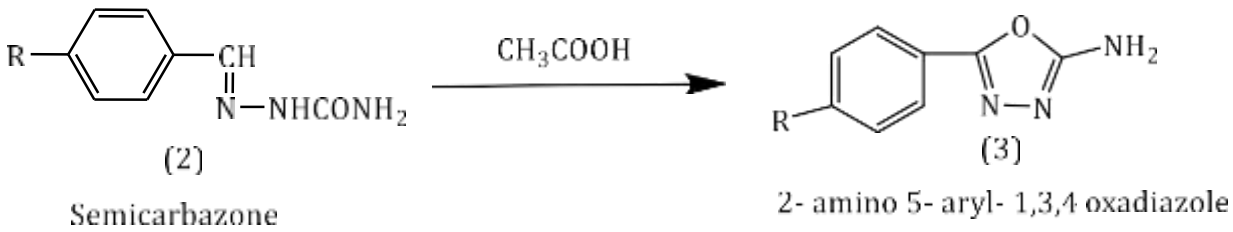
Synthesis-The synthesis comprises the two schemes, In first scheme, first step semicarbazone (2) was synthesized and second steps different 2-amino-5-arylsubstituted-1,3,4–oxadiazole (compound 3) was synthesized and in second scheme, compounds 3, reacted with the 2- phenylbenzoxazinone to obtained titled compound 2,3-disubstituted quinazoline.

(a) Scheme 1: Synthesis of 2-amino-5-arylsubstituted-1,3,4–oxadiazole

Step-1: Synthesis of Semicarbazone-



Step 2: Synthesis of 2-amino-5-arylsubstituted-1,3,4–oxadiazole



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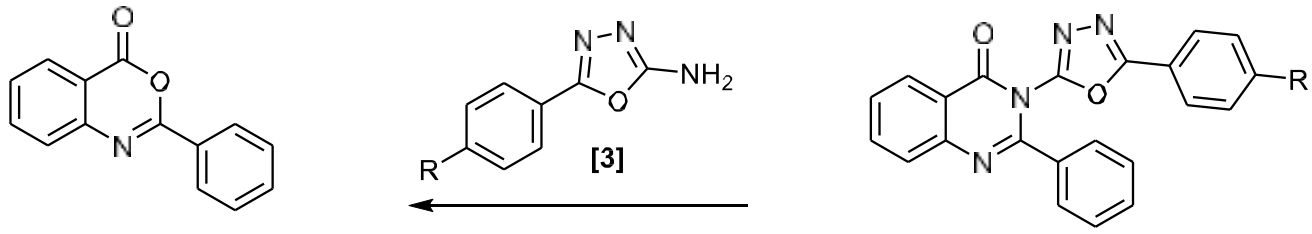
Scheme 2: Synthesis of title compounds (2,3-disubstituted quinazoline)

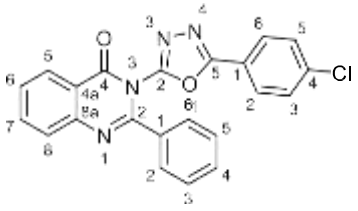


2-phenyl benzoxazinone

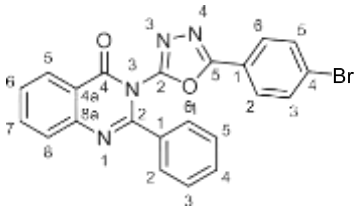
[4]

2,3-disubstituted quinazoline

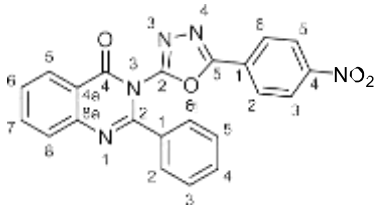
4.3 LIST OF FINAL SYNTHESIZED COMPOUNDS



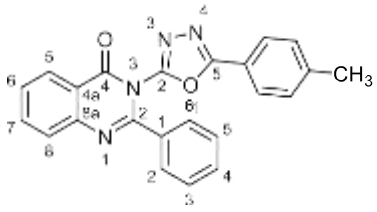
3-(5-(4-chlorophenyl)-1,3,4-oxadiazol-2-yl)-2-phenylquinazolin-4(3*H*)-one



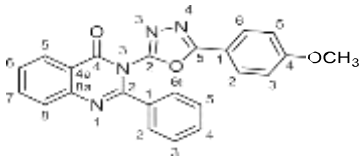
3-(5-(4-bromophenyl)-1,3,4-oxadiazol-2-yl)-2-phenylquinazolin-4(3*H*)-one



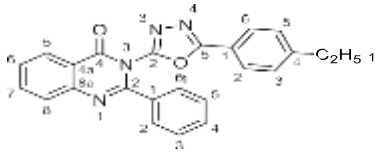
3-(5-(4-nitrophenyl)-1,3,4-oxadiazol-2-yl)-2-phenylquinazolin-4(3*H*)-one



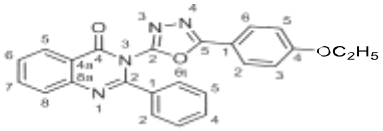
2-phenyl-3-(5-(*p*-tolyl)-1,3,4-oxadiazol-2-yl)quinazolin-4(3*H*)-one



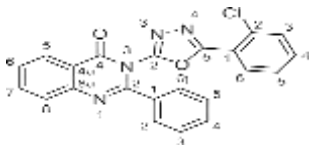
3-(5-(4-methoxyphenyl)-1,3,4-oxadiazol-2-yl)-2-phenylquinazolin-4(3*H*)-one



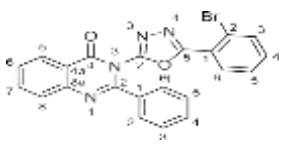
3-(5-(4-ethylphenyl)-1,3,4-oxadiazol-2-yl)-2-phenylquinazolin-4(3*H*)-one



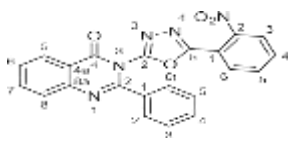
3-(5-(4-ethoxyphenyl)-1,3,4-oxadiazol-2-yl)-2-phenylquinazolin-4(3*H*)-one



3-(5-(2-chlorophenyl)-1,3,4-oxadiazol-2-yl)-2-phenylquinazolin-4(3*H*)-one



3-(5-(2-bromophenyl)-1,3,4-oxadiazol-2-yl)-2-phenylquinazolin-4(3*H*)-one



3-(5-(2-nitrophenyl)-1,3,4-oxadiazol-2-yl)-2-phenylquinazolin-4(3*H*)-one

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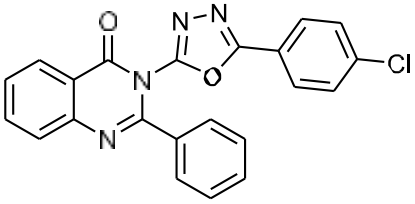
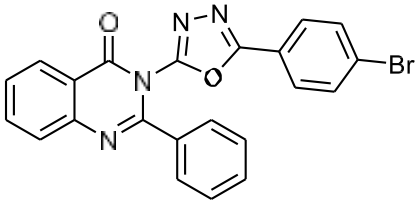
4.4 LIST OF THE SYNTHESIZED COMPOUNDS WITH CODE



[6A]



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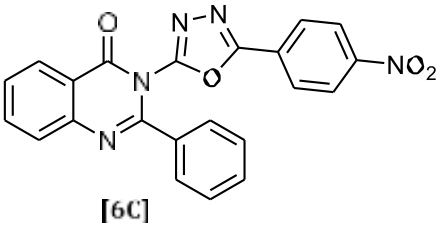

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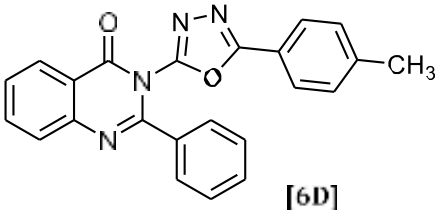

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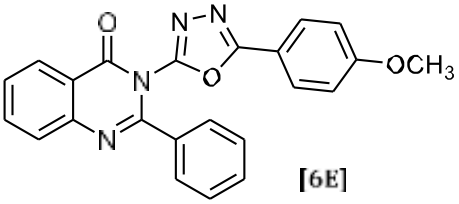
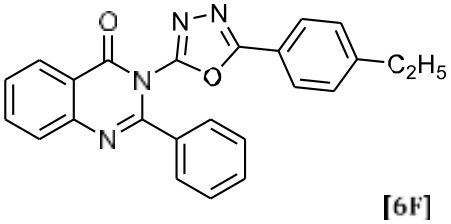
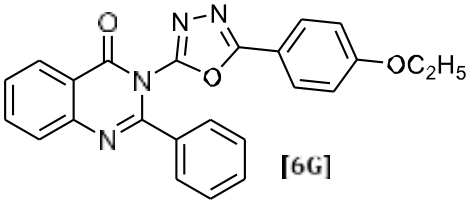


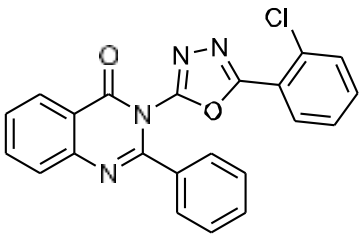
[6E]



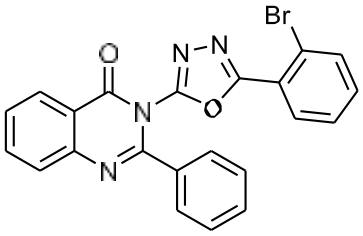
[6F]



[6G]



[6H]



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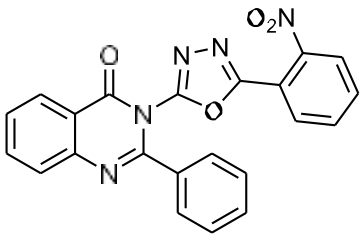

[6J]

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STRUCTURE CHARACTERIZATION-

PHYSICO-CHEMICAL CHARACTERIZATION

Comp.	Mol. formula	Mol. wt.	% yield	Physical appearance		Melting point (˚C)	Rf value
				Color	State		
6A	$C_{22}H_{13}ClN_4O_2$	400.82	76	Shiny white	**Solid**	270-272°C	
6B	$C_{22}H_{13}BrN_4O_2$	445.28	56	Off white	**Solid**	243-245°C	
6C	$C_{22}H_{13}N_5O_4$	411.38	78	Yellow	**Solid**	260-262°C	
6D	$C_{23}H_{16}N_4O_2$	380.41	70	Cream	**Solid**	176-178°C	
6E	$C_{23}H_{16}N_4O_3$	396.41	68	Cream	**Solid**	166-268°C	
6F	$C_{24}H_{18}N_4O_2$	394.43	65	Light brown	**Solid**	233-235°C	
6G	$C_{24}H_{18}N_4O_3$	410.43	62	Off grey	**Solid**	213-215°C	
6H	$C_{22}H_{13}ClN_4O_2$	400.82	55	Shiny white	**Solid**	255-257°C	
6I	$C_{22}H_{13}BrN_4O_2$	445.28	48	Yellow	**Solid**	248-250°C	
6J	$C_{22}H_{13}N_5O_4$	411.38	68	Off white	**Solid**	250-252°C	

Solubility studies of the synthesized compounds

Compound	Water	Alcohol	Acetone	Glacial Acetic Acid	Benzene	Dimethyl Sulfoxide
6A	-	+++	+	++	-	++
6B	-	+++	+	++	-	++
6C	-	+++	+	++	-	++
6D	-	+++	+	++	-	++
6E	-	+++	+	++	-	++
6F	-	+++	+	++	-	++
6G	-	+++	+	++	-	++
6H	-	+++	+	++	-	++
6I	-	+++	+	++	-	++
6J	-	+++	+	++	-	++

- Insoluble; + = Slightly soluble; ++ = soluble; +++ = Freely soluble

The synthesized compounds are

1) **Compound 6A:** 3-(5-(4-chlorophenyl)-1,3,4-oxadiazol-2-yl)-2-phenylquinazolin- 4(3H)-one
2) **Compound 6B:** 3-(5-(4-bromophenyl)-1,3,4-oxadiazol-2-yl)-2-phenylquinazolin- 4(3H)-one
3) **Compound 6C:** 3-(5-(4-nitrophenyl)-1,3,4-oxadiazol-2-yl)-2-phenylquinazolin- 4(3H)-one
4) **Compound 6D:** 2-phenyl-3-(5-(p-tolyl)-1,3,4-oxadiazol-2-yl)quinazolin-4(3H)-one
5) **Compound 6E:** 3-(5-(4-methoxyphenyl)-1,3,4-oxadiazol-2-yl)-2-phenylquinazolin- 4(3H)-one
6) **Compound 6F:** 3-(5-(4-ethylphenyl)-1,3,4-oxadiazol-2-yl)-2-phenylquinazolin- 4(3H)-one
7) **Compound 6G:** 3-(5-(4-ethoxyphenyl)-1,3,4-oxadiazol-2-yl)-2-phenylquinazolin- 4(3H)-one
8) **Compound 6H:** 3-(5-(2-chlorophenyl)-1,3,4-oxadiazol-2-yl)-2-phenylquinazolin- 4(3H)-one
9) **Compound 6I:** 3-(5-(2-bromophenyl)-1,3,4-oxadiazol-2-yl)-2-phenylquinazolin- 4(3H)-one
10) **Compound 6J:** 3-(5-(2-nitrophenyl)-1,3,4-oxadiazol-2-yl)-2-phenylquinazolin- 4(3H)-one

5.0 RESULT AND DISCUSSION

5.0.1 Antibacterial activity

In accordance with the data obtained from antibacterial activity, all the synthesized 2,3- disubstitued 4(3H)-quinazolinone (6A-6J) have showed activity against tested organisms. Antibacterial activity of the synthesized compounds has been carried out for gram-negative bacterial strain.

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5.6.1.2 Antibacterial activity against gram negative bacteria

Table Antibacterial activity of synthesized 2,3-disubstitued 4(3H) quinazolinone derivatives against gram negative bacteria.

S. No.	COMPOUND	Zone of inhibition in mm			
		Pseudomonas Aeruginosa		*Escherichia coli*	
		50µg	100µg	50µg	100µg
1.	6A	19.05±0.21	22.11±0.25	18.07±0.26	20.02±0.21
2.	6B	16.28±0.28	19.24±0.25	15.32±0.27	18.25±0.28
3.	6C	18.28±0.24	21.28±0.28	17.30±0.22	19.25±0.24
4.	6D	14.05±0.24	16.10±0.22	13.09±0.23	15.02±0.24
5.	6E	12.57±0.26	14.59±0.23	11.61±0.27	13.54±0.28
6.	6F	13.57±0.25	15.54±0.26	12.56±0.23	14.54±0.23
7.	6G	11.67±0.24	12.64±0.29	10.71±0.23	12.64±0.24
8.	6H	08.45±0.23	10.46±0.26	07.49±0.22	09.42±0.23
9.	6I	07.15±0.27	09.19±0.27	06.19±0.27	08.12±0.22
10.	6J	09.31±0.23	09.52±0.23	07.35±0.25	09.28±0.22
11.	Ciprofloxacin	19.28±0.36	23.45±0.23	19.70±0.65	22.65±0.26

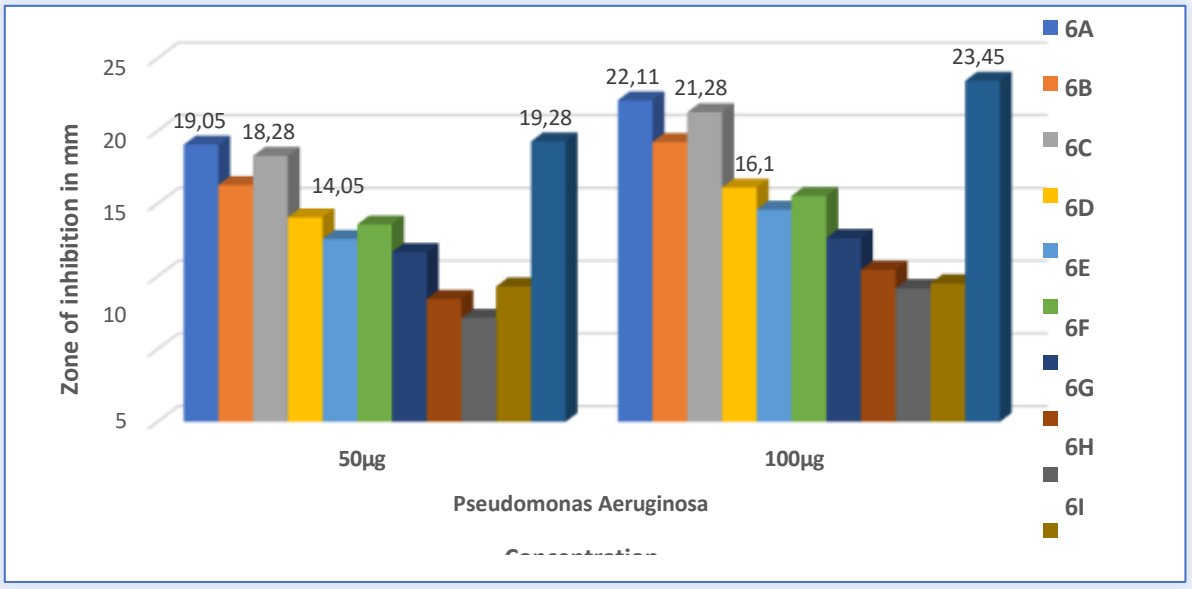

Figure: Graph showing Zone of inhibition of the synthesized derivatives against gram negative bacteria.

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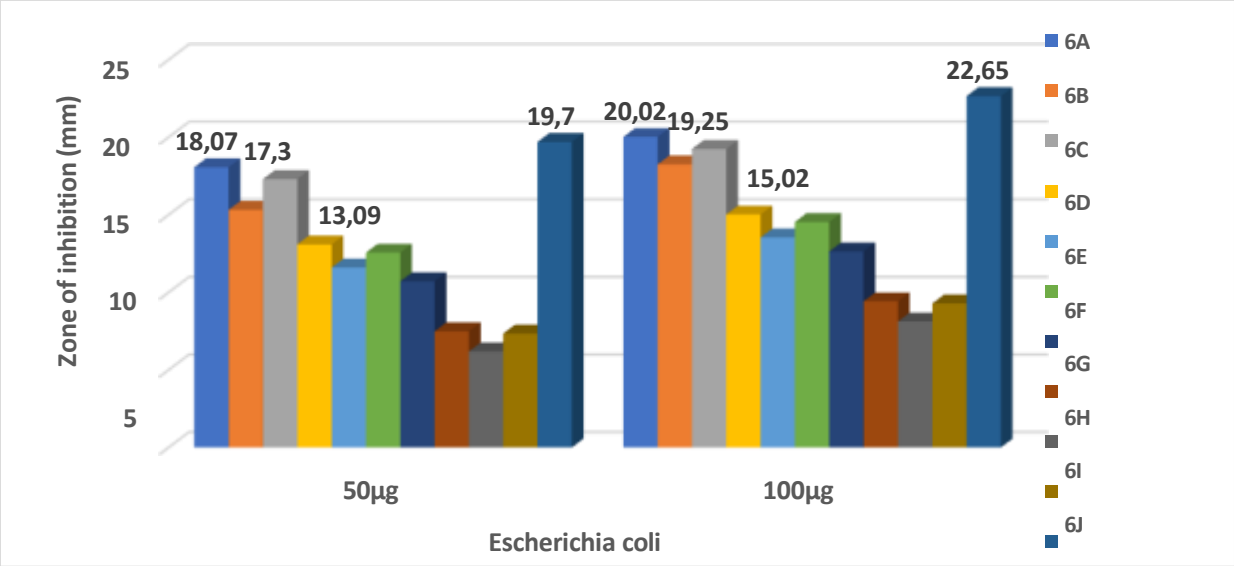

Figure: Graph showing Zone of inhibition of the synthesized derivatives against gram negative bacteria.

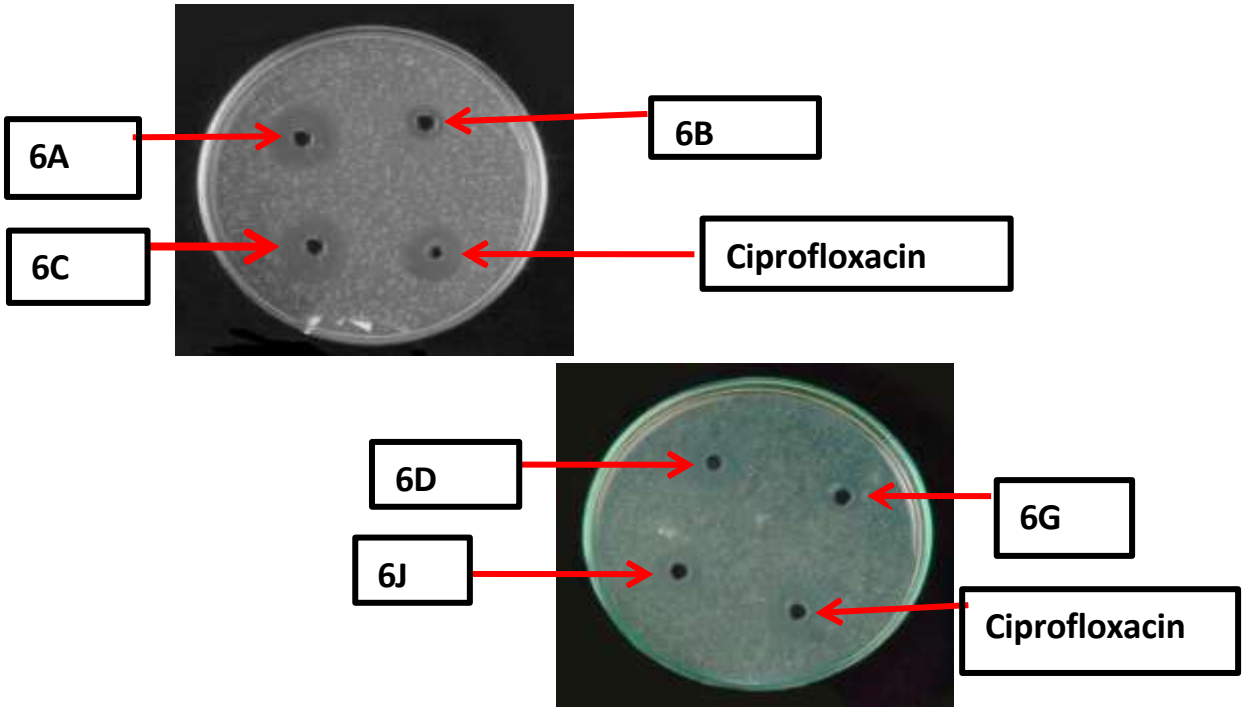

Figure: Zone of inhibition of synthesized derivatives against *Pseudomonas Aeruginosa*

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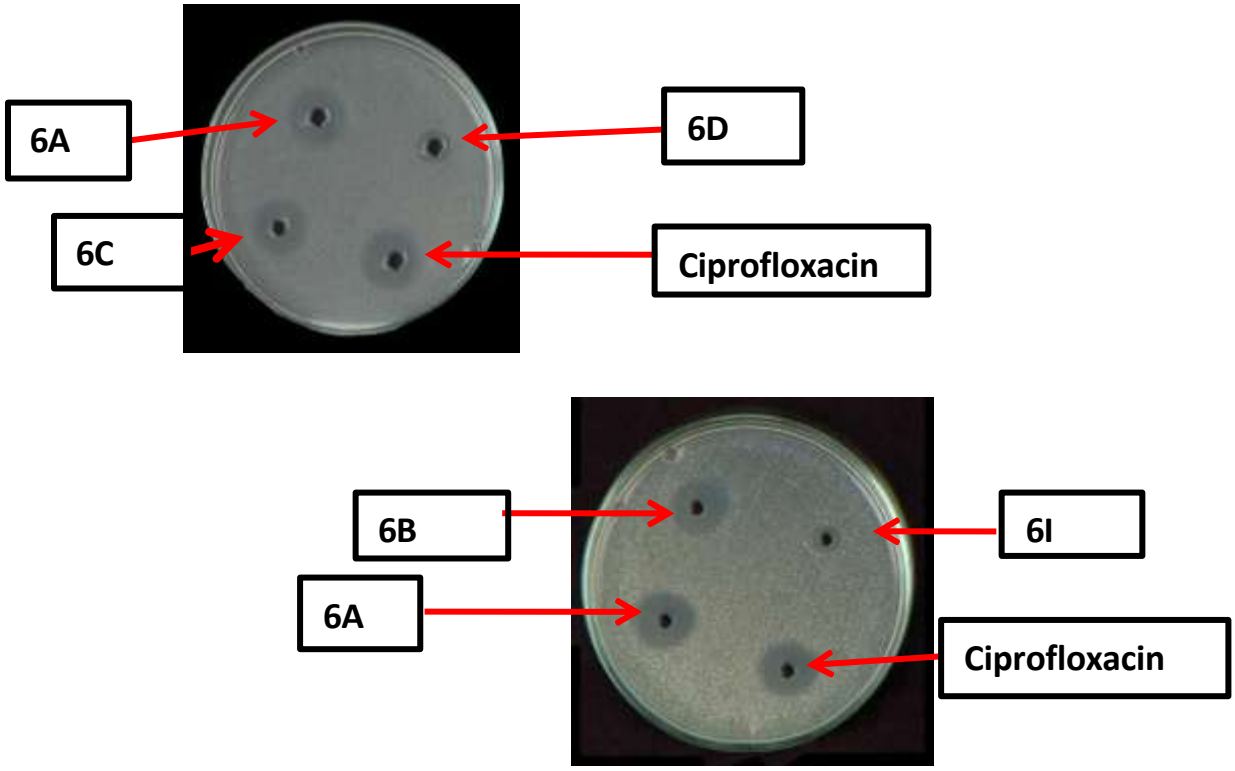

Figure.: Zone of inhibition of synthesized derivatives against *Escherichia Coli*

(16.42±0.67), 6F (15.32±0.32) 6G (14.62±0.72), 6H (8.32±0.33), 6I (6.32±0.84) and 6J (10.32±0.88) has shown zone of inhibition (mm) as compared to standard drug (Fluconazole, 25.30±0.32) has shown good activity against C. Albicans (Fungi strains) at 50µg concentration.

Table 5.4: Antifungal activity of synthesized 2,3-disubstitued 4(3H) quinazolinone derivatives.

COMPOUND	Zone of inhibition (mm)			
	C. Albicans		*A. Niger*	
Concentration	**50**	**100**	**50**	**100**
6A	24.75±0.53	27.65±0.43	23.34±0.28	28.25±0.32
6B	21.72±0.34	22.20±0.22	19.62±0.23	26.72±0.18
6C	22.45±0.28	26.32±0.26	22.23±0.43	26.35±0.25
6D	21.22±0.65	25.25±0.34	18.42±0.46	22.55±0.12
6E	16.42±0.67	22.72±0.27	18.52±0.25	24.32±0.14
6F	15.32±0.32	18.25±0.45	20.38±0.14	24.42±0.25
6G	14.62±0.72	17.62±0.23	17.24±0.13	22.52±0.22
6H	8.32±0.33	12.72±0.34	14.52±0.26	17.64±0.32
6I	6.32±0.84	11.12±0.25	15.65±0.12	19.22±0.14
6J	10.32±0.88	15.32±0.44	16.65±0.16	20.52±0.12
DMSO (Control)	-	-	-	-
Fluconazole	25.30 ±0.32	30.25±0.26	24.25±0.32	29.20±0.23

The order of the activity: 6A>6B>6C>6D>6E>6F>6G>6J>6H>6I

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Figure: Graph showing Zone of inhibition of the synthesized derivatives against C. Albicans fungi.

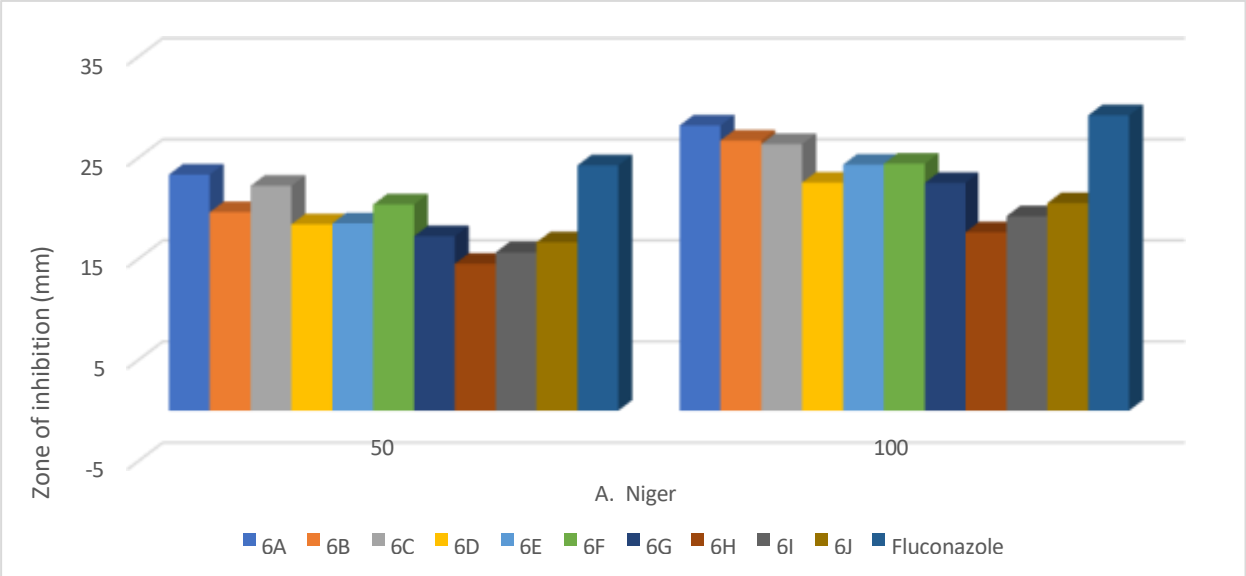

Figure: Graph showing Zone of inhibition of the synthesized derivatives against *A. Niger* fungi.

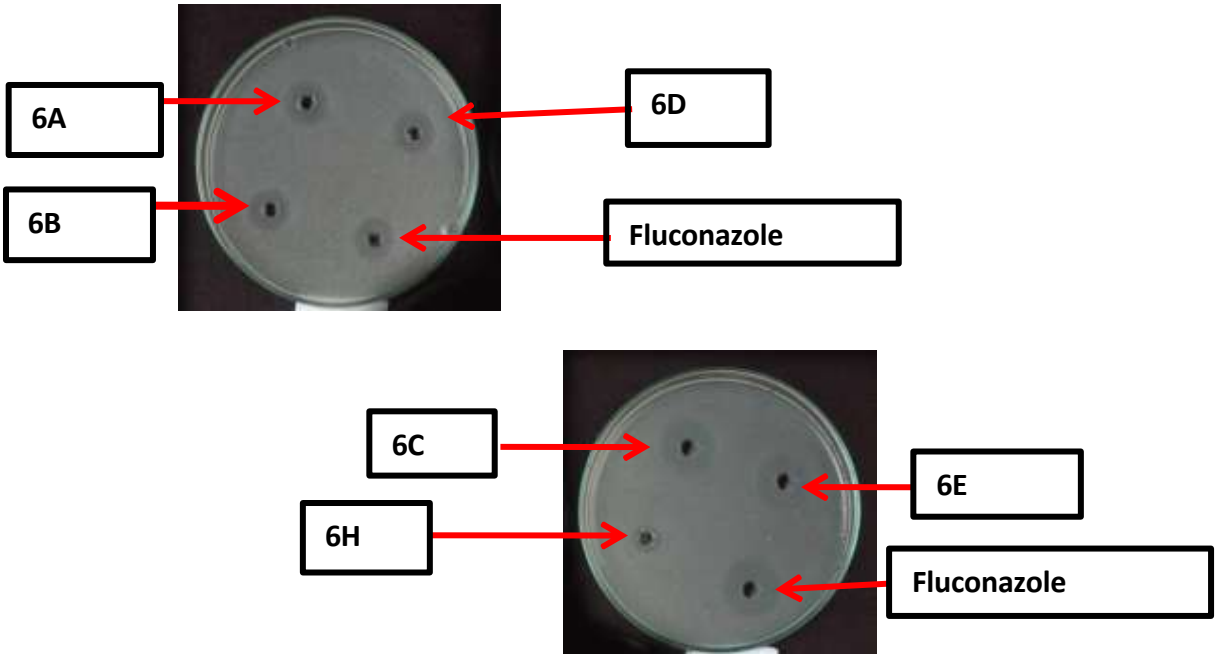

Figure 5.9: Zone of inhibition of synthesized derivatives against *C. Albicans*

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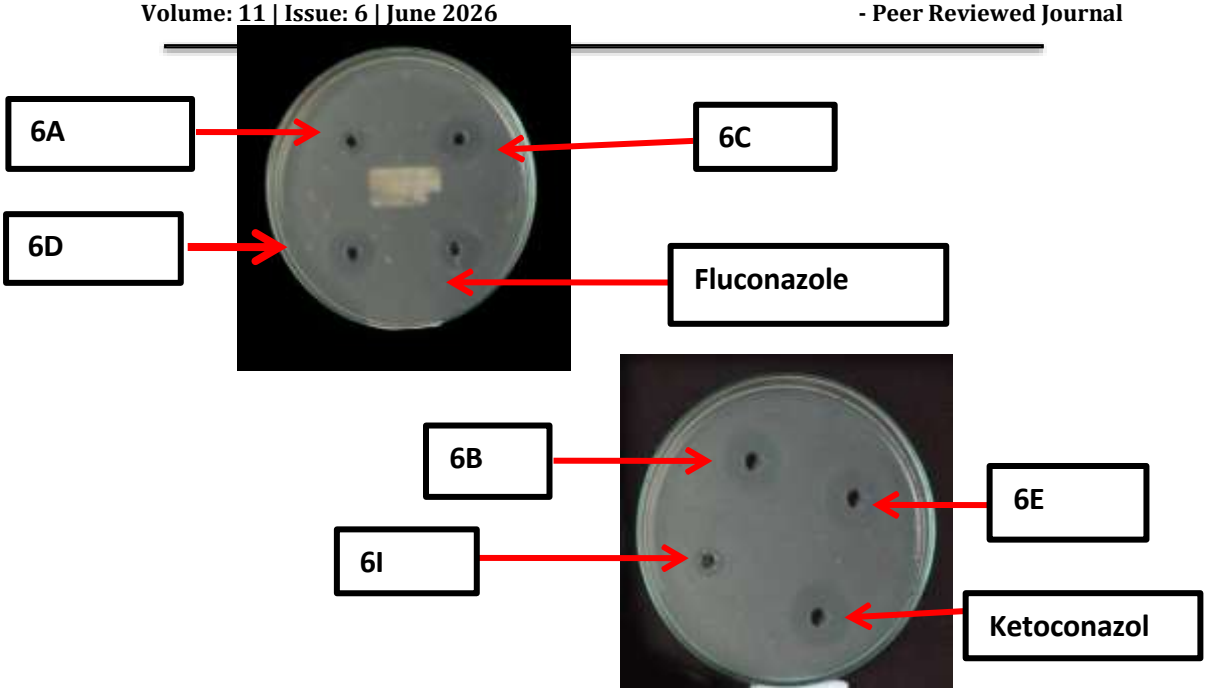

Figure 5.10: Zone of inhibition of synthesized derivatives against *A. Niger*

5.1 DISCUSSION

The ten new derivatives of 2,3-disubstituted 4(3H)-quinazolinone has been prepared and evaluated for their antibacterial activity against Gram-negative bacterial strains and two fungal strains are used to carried out antifungal activity using Agar diffusion method. The Ciprofloxacin used as standard for antibacterial activity and Fluconazole was used as standard to compare the antifungal potential of the synthesized compounds.

The antibacterial and antifungal activity data of 2,3-disubstituted 4(3H)- quinazolinone derivatives (6A-6J) indicated that the compounds have significant inhibitory activity on all the bacterial and fungal strains at both 50 µg (0.05 ml) and 100 µg (0.1 ml) dose levels when compared with standard. Among all the compounds tested, compounds 6A, 6B, 6C and 6D possessed maximum activity in both fungal as well as bacterial strains.

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Presence of electronegative group (Br, Cl, and NO2) with para substitution either at 5th position along with the 1,3,5-oxadiazole at 3 position in the quinazolinone is essential for the antifungal and antibacterial activity but in case of ortho substitution may diminish the activity. The research work established the fact that 2, 3, - disubstituted quinazolinone-4-(3H) can be further studied for their variety of pharmacological activities.

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