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Synthesis, Characterization and Microbial Evaluation of Novel Schiff base bearing Semicarbazone moiety

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Abstract:

The chemistry of compounds bearing Semicarbzone moiety of Schiff base have been an interesting field of research since long time. 1-(4-aminophenyl) ethan-1-one was taken with semicarbazide which gives 2-[1-(4-aminophenyl) ethylidene] hydrazine-1-carboxamide. Semicarbazone linkage containing 2-[1-(4-aminophenyl)ethylidene] hydrazine-1-carboxamide derivatives are further condensed with aromatic aldehyde derivatives which gives a series of newly synthesized Schiff base.

Synthesized derivatives of Schiff base are characterized by the physiochemical, IR and ^{13}CNMR spectral data. All synthesized compounds are screened for their antibacterial and antifungal activity by using the agar well diffusion method.

Key Words: Semicarbazone moiety, Physiochemical, Microbial evaluation.

1.Introduction:

Pathogens are micro-organisms that have the potential to cause various kinds of diseases. A few harmful microbes such as bacteria, viruses and fungus cause illness and responsible for the substantial morbidity and mortality.

The chemistry of compounds bearing Schiff base moiety have been an interesting field of research since long. Schiff Base Moiety (>C=N-) show various biological applications such as anti-cancer[01], anti-tuberculostatic[02], diuretic[03], anti-bacterial[03], anti-fungal[03] and anti-inflammatory[04], apart from their role in dye and agrochemical industries[05]. Literature survey exposed that work on Semicarbazone have been extensively studied for its spacious range for biological and clinical applications[(06-07)].

2. Material and Methods

All the chemical reagents and solvents were purchased commercially and used without any further purification. The melting point of compounds were recorded on a hot stage Gallen

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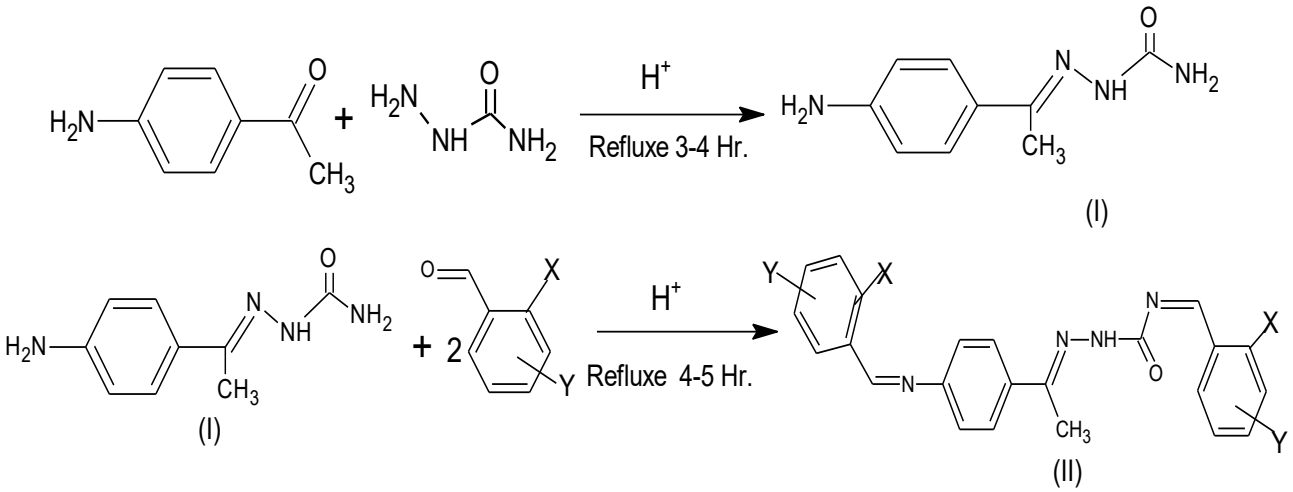
Kamp melting point apparatus in open capillary and was found uncorrected. The characterization of synthesized compounds was determined by physiological properties, IR and ^{13}CNMR spectral data.

2.1 Synthesis series of Schiff base:

4-aminoacetophenone (0.005 mole, 0.60gm) and Semicarbezide (0.005 mol, 0.37gm) were taken in a round bottom flask containing absolute ethanol (15ml). To this mixture, few drops of condensing reagent as glacial acetic acid was added and then refluxed on water bath for 3-4 hrs. Progress of the reaction was monitored by TLC. The mixture was cooled to room temperature and poured into crushed ice. Product separated was filtered and further crystallized from ethanol to give a yellow colored crystalline solid form of "2-[1-(4-aminophenyl) ethylidene] hydrazine-1-carbothioamide]".

A series of substituted aldehyde (IIA to IIE) was condensed with "2-[1-(4-aminophenyl)ethylidene] hydrazine-1-carbothioamide]" in 2:1 molar ratio in presence of 1,4-dioxane by adding few drops of sulphuric acid and placed on water bath for 4-5 hrs. Further ice cubes were added to it. The solid mass was filtered and recrystallized in ethanol.

2.2 Synthesis Scheme



Where	(IIa)	(IIb)	(IIc)	(IId)	(IIe)
X	H	OH	H	H	H
Y	H	H	(P)NO_2	(P)NH_2	(P)Cl

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Table [01]: Physical data of novel synthesized Schiff bases bearing Semicarbazone moiety.

Com Code	Mole. Formula	Mole. Weight	IUPAC Name	Color	MP (^{o}C)	Yeild (%)
IIA	$C_{23}H_{20}N_4O$	384	2-(1-{4-[-benzylideneamino]phenyl}ethylidene)-*N*-[(phenylmethylidene]hydrazine-1-carboxamide	Yellow	192	37.61
IIB	$C_{23}H_{20}N_4O_3$	416	*N*-[(2-hydroxyphenyl)methylidene]-2-[1-(4-{[(2-hydroxyphenyl)methylidene]amino}phenyl)ethylidene]hydrazine-1-carbothioamide	Purple	182	25.12
IIC	$C_{23}H_{18}N_6O5$	506	*N*-(1-(4-nitrophenyl)methylidene)-2-(1-(4-(1-(4-nitrophenyl) methylidene)amino)phenyl) methylidene)hydrazinecarboxamide	Black	181	41.12
IID	$C_{23}H_{18}N_4OCl_2$	437	(*N*-[((4-chlorophenyl)methylidene]-2-[1-(4-{[(2-chlorophenyl)methylidene]amino}phenyl)ethylidene]hydrazine-1-carboxamide	Brown	182	29.92
IIE	$C_{23}H_{22}N_6O$		*N*-(4-aminobenzylidene)-2-(1-(4-(4-aminobenzylidene)amino)phenyl)ethylidene)hydrazinecarboxamide	Muddy Yellow	172	23.12

3. Results and Discussion

All novel synthesized Schiff bases bearing Semicarbazone moiety were found to be stable in air at room temperature. They are soluble in Chloroform and DMSO, but insoluble in water. The physical properties of all synthesized compounds tabulated in table [01]. The approximate yield recorded was 42-23 %. All compounds are Solid and colored and they gave satisfactory results for IR and ^{13}CNMR.

3.1 Infrared spectra

The FTIR spectra data provide valuable information on the nature of the functional group. The infrared spectra of the Schiff base showed a band at 1646 to 1565 cm^{-1} which was attributed to C=N band and 1691-1720 cm^{-1} is assigned C=O band. The frequencies that

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appeared at 2802 and 3032 cm^{-1} regions are assigned (-CH_3) and (C-H)) aro. are respectively and few other stretching are tabulated in table [02].

Table [02]: IR data of newly synthesized Schiff bases bearing Semicarbazone moiety.

Group	OH	NO_2	NH_2	C-Cl
υcm^{-1}	3382	1356	3182	689

3.2 ^{13}C-NMR spectra

The ^{13}C-NMR spectrum of the Schiff base was measured using Chloroform as solvent. Spectral analysis of Schiff base show multiple peaks at δ 130.94-122.86 ppm attributed to the phenyl ring. The single peaks at δ =1.28 ppm and 11.40 ppm due to the C=N moiety and δ 197.93 ppm is assigned C=O bond of Semicarbazone moiety.

4. Antimicrobial activities

The results obtained from microbial activity for the prepared Schiff bases are given in Table [04]. Diameter of inhibition zone (mm) including the disk diameter was measured for each treatment. The filter no. 1 disc of 6mm diameter was impregnated with different concentrations (25%, 50%, 75% and 100%) of IIA, IIB, IIC, IID, IIE and IIF. The disc was placed in the center of pre-inoculated culture plates and incubated for 24 hrs at 37 ºC. After incubation, plates were observed for the sensitivity of the test extract against microorganisms in terms of zone of inhibition (ZOI). The zone of inhibition was measured by using transparent plastic ruler scale

Table: [4] Antimicrobial activity of compound code II[A-D] against pathogens.

Com Code	Bacterial Pathogens	Bacterial culture-zone of inhibition concentration in % (mm)				Fungal Pathogens	Fungal culture-zone of inhibition concentration in % (mm)			
		25	50	75	100		25	50	75	100
IIA	*S.aureus*	-	-	-	+	*Candida albicans*	-	-	-	+
	P. aerugenosa	-	-	+	+	*A. flavus*	-	+	+	+
	E. coli	+	+	+	+	*A. fumigates*	-	+	+	+
IIB	*S.aureus*	+	+	+	+	*Candida albicans*	+	+	+	+
	P. aerugenosa	-	-	+	+	*A. flavus*	-	+	+	+
	E. coli	+	+	+	+	*A. fumigates*	-	+	+	+
IIC	*S.aureus*	-	+	+	+	*Candida albicans*	+	+	+	+
	P. aerugenosa	+	+	+	+	*A. flavus*	+	+	+	+
	E. coli	-	+	+	+	*A. fumigates*	-	-	+	+
IID	*S.aureus*	-	-	-	+	*Candida albicans*	-	+	+	+
	P. aerugenosa	-	+	+	+	*A. flavus*	-	-	+	+

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	E. coli	+	+	+	+	*A. fumigates*	-	-	-	-

5. Conclusion

In the present research study, we synthesized novel Schiff bases series bearing Semicarbazone moiety. These compounds are characterized by physicochemical and spectral analysis which gives sastifactory result. The antimicrobial screening of these compounds shows that inhibition depends on strain and structure. The anti-bacterial activity of newly synthesized novel Schiff bases with Semicarbazone moiety were effectively screened against *S. aureus, P. aeruginosa* and *E. coli* bacterial strains. Compound IIA, IIB and IID show excellent activity against *E. coli and mordent show against P. aerugenosaand and S.aureus.* Compound IIB show excellent against *S.aureus.*

To assess the antifungal activity of synthesized compounds, they were checked against *Candida albicans, A. flavus* and *A. fumigates.* All compounds show moderate anti-fungal activity against *A. flavus and A. fumigates* and show excellent against *Candida albicans*.

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