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SYNTHESIS OF SOME NEW BIS-ISOXAZOLINES AND BIS-PYRAZOLINES AND THEIR ANTIMICROBIAL ACTIVITIES

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Abstract: Substituted bis-isoxazoline and bis-pyrazolines are known for their clinical importance. Bis-isoxazoline and Bis-pyrazoline have been synthesized by the condensation of Bis-chalcone with hydroxylamine hydrochloride and phenyl hydrazine hydrochloride in alc. KOH medium respectively structure of these compounds have been established by chemical properties and speetral analysis.

Keywords: Antimicrobial Activities, Synthesis



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INTRODUCTION

The title compounds have been characterized by a number of different activities. In the present work we report the reaction of bis-ketone with substituted aromatic aldehydes to yield the corresponding bis-chalcones which on treatment with hyohoxylamine hydrochloride and phenyl hydrazine hydrochloride in alcoholic KOH medium gives corresponding bis-isoxazolines and bis-pyrazolines respectively.

The prepared compounds showed no-coloration with FeCl_3 solution due to strongly hydrogen bounded $-(\text{OH})$ group. It is soluble in NaOH giving yellow coloration indicating the presence of phenolic $-(\text{OH})$ group. The bis-isoxazoline compounds showed yellow coloration with conc. H_2SO_4 indicating isoxazoline nucleus and the bis-pyrazolines compounds showed (green) coloration with conc. H_2SO_4 indicating pyrazoline nucleus not having $-\text{CO}-\text{CH}=\text{CH}-$ grouping.

The methylene dioxo test was performed by heating bis-isoxazoline and bis-pyrazoline compounds with gallic acid and conc. H_2SO_4 when embraled green and blue coloration respectively was observed which indicate the presence of $-\text{O}-\text{CH}_2-\text{O}-$ linkage in the compounds.

The structures of these compounds established by chemical properties and spectral analysis (IR-NMR-Spectra).

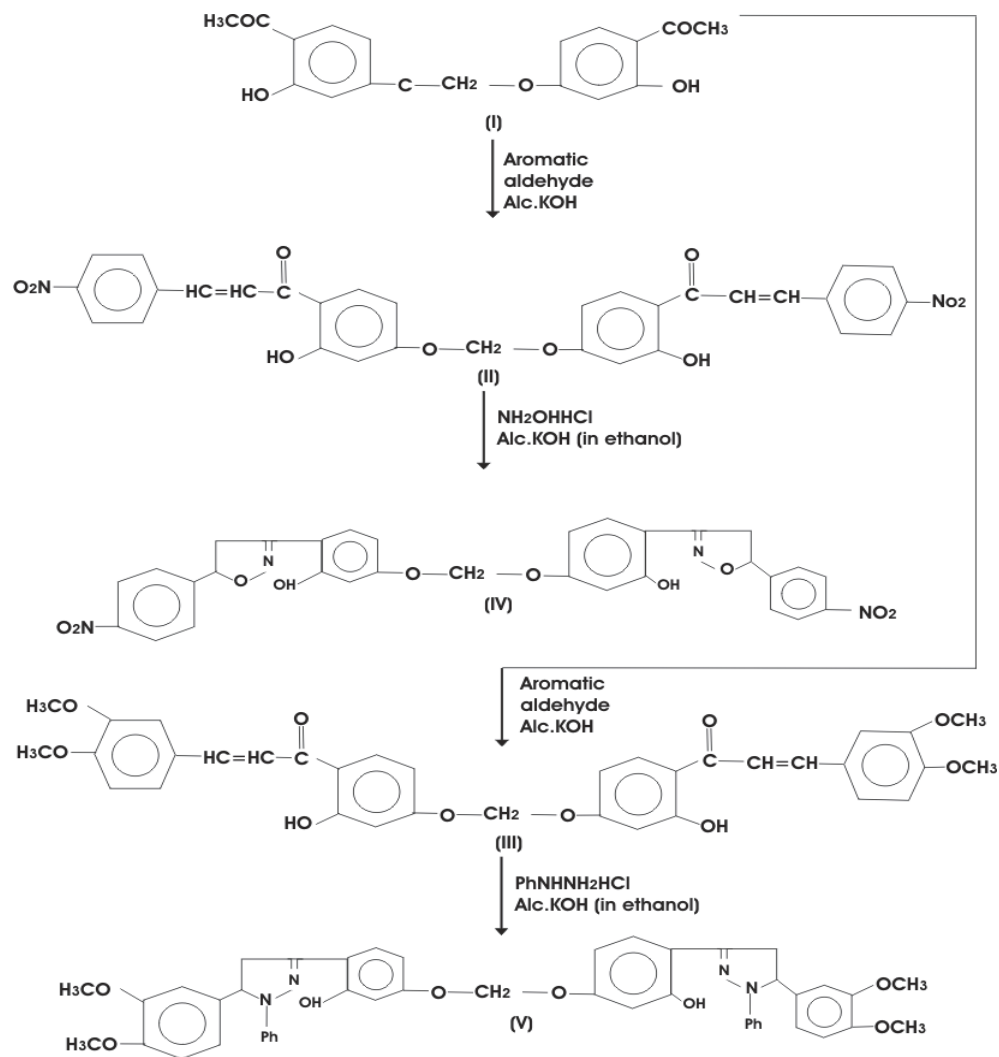
The IR- spectrum of compound (IV) clearly indicate presence of bond due $-\text{C}=\text{N}-$ (1631.78-cm^{-1}) $-\text{O}-\text{C}-\text{O}-$ (1120.64 cm^{-1}) and $-\text{N}-\text{O}-$ (968 cm^{-1}) and the NMR-Spectrum distinctly displayed the singals due to (Ar-H) protons at δ (6.61-7.91) (m) $-\text{O}-\text{CH}_2-\text{O}-$ protons at δ (5.84) (s) CH_2 protons of isoxazoline ring at δ (3.2-3.9) (d) and $-(\text{CH})$ protons of isoxazoline ring at δ (5.37) (t) .

In compound (V) – The NMR-spectrum distinctly displayed signals due Ar-H-Protons $-\delta$ (6.67-7.41) (m) $-\text{O}-\text{CH}_2-\text{O}-$ protons at δ (5.96) (s), $-(\text{CH}_2)-$ protons of pylazoline ring (3.7) (d) $-(\text{CH})$ protons of pyrazoline ring at δ (5.8) (t) .

On the basis of above facts, the above compound (IV) with m. p. (240°C) assigned the structure and compound (IV) with m.p. (220°C) assigned the structure.

Table No.1 : Yield and Melting points of prepared compounds.

Sr. No.	Compounds	Yield	M. P.
1	Bisketone	50%	146°C
2	Chalcone	60%	190°C
3	Isoxazoline	58%	252°C
4	Pyrazoline	59%	220°C



Chemical Reaction:

I) 1,1-Bis-{3-hydroxy-4-acetyl-oxyphenyl}-methane

II) 1,1{Bis-[3-hydroxy-4-3-(4^{nitro}-nitrophenyl) prop-2-ene-1-one] -oxyphenyl}-methane.

III) 1,1{Bis-[3-hydroxy-4-3-(3^{dimethoxy}-4^{dimethoxy}-dimethoxyphenyl) prop-2-ene-1-one] -oxyphenyl}-methane.

IV) 1, 1 {Bis-[3-hydroxy-4 (-4-nitrophenyl-isoxazolin-3-yl)- oxyphenyl] methane.

V) 1,1{Bis-[3-hydroxy-4 (1-phenyl-5^{dimethoxy}-(3^{dimethoxy}-4^{dimethoxy}-dimethoxy phenyl)-pyrazolin-3-yl] Oxyphenyl} methane.

RESULT AND DISCUSSION:

The compounds are screened for antimicrobial activities against various pathogenic bacteria's such as *S. aureus*, *E-coli*, *P. vulgaris* by using disc plate method. The medium used for this is HI media. The antibacterial activities of compound (IV) was found higher than compound (V)

EXPERIMENTAL :

I) General Procedure for Preparation of bis-chalcone :

Compound I (0.01 mole) and aldehyde (0.02 mole) dissolved in 40 ml of Ethanol. The reaction mixture was warmed up to 50°C. KOH (0.04 mole) was added to reaction mixture with constant stirring. The reaction mixture was kept overnight. The crude product obtained was decomposed by 50% Ac-OH.

II) Preparation of bis-isoxazoline:

A mixture of bis-chalcone (0.0025 mole) alc(KOH) and NH₂OHHCl (0.005 mole) was refluxed in (20ml) in 40 ml ethanol round bottom flask for about 5 hours. The reaction mixture was poured into water and acidified with HCl. A white crude product obtained was crystallized from dilute ethanol.

III) Preparation of Bis-pyrazoline :

A mixture of bis chalcone (0.0075 mole) and phenyl hydrazine hydrochloride (0.005 mole) was refluxed in alc.KOH(10 ml) in 40 ml ethanol for 4 hours. The reaction mixture was cooled and poured in to water. It was acidified with 50% Acetic acid.

A yellowish crude product obtained was crystallized from ethanol.

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