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Solvent Free Synthesis of Chalcones and their Antibacterial Activities

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Abstract: The solvent free synthesis of six chalcones was carried out by grinding the piperanal and the acetophenone (unsubstituted, 4-methyl, 4-methoxy, 4-bromo, 4-nitro, 3-chloro) in the presence of solid sodium hydroxide with a mortar and pestle. In general, the chalcones were obtained in high yield and high purity. Minor quantities of Ketol and Michael addition product were easily removed by recrystallization. The result indicates a correlation between the success of the solvent-free synthesis and melting point of the chalcone. Chalcone with relatively high melting points (higher than 80°C) were obtained in high yields. The two chalcones that could not be produced in good yields were having relatively low melting points. They have been screened for their antibacterial activity against Gram positive and Gram negative bacteria.

Key words: Solvent free synthesis, Chalcone, Antibacterial activity

Introduction

Chalcones derivatives constitute an important class of compounds possessing diverse type of biological properties including Antiplasmodial¹, Antibacterial², Trypanocidal and Leishmanicidal Properties³, Antitumour^{4,5}, Antiinflammatory⁶, Antiviral activities⁷. Extensive work on synthesis of chalcones has been done by various routes. Condensation of the appropriate acetophenone and piperanal in the presence of sodium hydroxide (solid) via solvent free path⁸ yielded the corresponding 1, 3-diarylchalcones.

The chalcones possess an α,β -unsaturated grouping in the molecules. The structural assignment of these chalcones was based on elemental analysis, IR and NMR spectral data. The antibacterial activity of these chalcones has been studied by cup-plate method^{9, 10, 11}.

Experimental

Melting points are uncorrected. The IR spectra were recorded in KBr disc on a Nicolet Megna-FT-IR 550 spectrometer, ^1H NMR were recorded on model DRX 300 at 300.13 in CDCl_3 / $\text{DMSO}-d_6$ using TMS as internal standard. The purity of the newly synthesized compounds was checked by TLC.

Solvent free synthesis of chalcone

The solvent free synthesis of chalcones was carried out by grinding the piperanol (0.05 mol) and acetophenone derivative (0.05 mol) in presence of solid sodium hydroxide (0.05 mol); petroleum ether (1:1) yielded the crystalline product. Purity of the compounds was checked by TLC using CHCl_3 as mobile phase.

Spectral studies

The IR spectra showed a characteristic bands at 1645-1635 ($\text{C}=\text{O}$), 1580-1570 cm^{-1} ($\text{C}=\text{C}$), asymmetrical C-O-C stretching band around 1200-1275 cm^{-1} and symmetrical stretching at 1020-1075 cm^{-1} . In ^1H NMR, a singlet was observed at δ 5.98-6.01 due to dioxymethylene group. Doublet at 7.82 for α -proton and 8.02 for β -proton in chalcones.

A complicated pattern in the aromatic region at δ 6.9-8.2 showed the presence of seven aromatic protons. Elemental analysis and ^1H NMR data of the title compounds are given in **Table 1** and **2** respectively.

Figure-1

Antibacterial activity

The compounds were screened for antibacterial activity by cup-plate method at a concentration of 50 μml^{-1} against gram-positive and gram-negative bacteria viz. *Staphylococcus aureus*, *Escherichia coli*, *Streptococcus viridians* and *K. salmonella* using their dimethylformamide solution. The activity was compared with chloromycetin. The result (**Table 3**) showed that most of the chalcones possess moderate to good activity (zone of inhibition, 10-15 mm). Some of the compounds containing chloro / bromo substituents were active while other possessed moderate or feeble activity against the bacteria.

Table-1. Elemental analysis data of title compounds

Compd	M.F.	M.Wt	Elemental Analysis Calculated. and (Found)			M.P. (°c)
			C%	H%	N%	
1a	C ₁₆ O ₃ H ₁₂	252	76.19 (76.12)	4.76 (4.60)	----- -----	107
1b	C ₁₇ O ₃ H ₁₄	266	76.91 (76.85)	5.26 (4.23)	----- -----	33
1c	C ₁₇ O ₄ H ₁₄	282	72.34 (72.35)	4.96 (4.90)	----- -----	127
1d	C ₁₆ O ₃ H ₁₁ Br	331	58.06 (57.93)	3.32 (3.33)	----- -----	134
1e	C ₁₆ O ₅ H ₁₁ N	297	64.64 (64.58)	3.70 (3.68)	4.71 (4.67)	207
1f	C ₁₆ O ₃ H ₁₁ Cl	286.5	67.02 (67.01)	3.83 (3.77)	----- -----	28

Table-2. ¹H NMR Data of Title Compounds (in δ, ppm)

Compd.	Ar-X	-C-CH=CH O (α CH) 1H, (d)	-C-CH=CH O (β CH) 1H, (d)	OCH ₂ O 2H,(s)
1a	6.92-8.1, complex	7.82	8.02	6.00
1b	6.72-7.89, complex	7.81	8.04	5.98
1c	6.912-8.22, complex	7.77	8.03	6.00
1d	6.77-8.23, complex	7.84	8.01	6.00
1e	6.34-7.9, complex	7.83	8.02	6.01
1f	6.88-6.99, complex	7.82	8.03	6.00

Table-3 Zone of inhibition of compounds 1a-1f

S.No.	S. aure-us	E. coli.	S. viridi-aus	K. salm-onella
1a	++	+++	++	++
1b	++	++	+++	++
1c	++	++	++	+++
1d	+++	+++	++++	+++
1e	++	++	+++	++
1f	+++	++++	+++	++++
Chloromycetine	+++	++++	+++	----

++++= 11-13mm, +++= 8-10mm, ++= 5-7mm

Conclusion

Solvent free synthesis of chalcones was a novel method for synthesis of chalcones. The chalcones were obtained in high yield and high purity. The result indicates a correlation between the success of solvent free synthesis and melting point of the chalcones. Chalcone with relatively high melting point were obtained in high yields of these, the chalcones 1a-1f were tested for their antibacterial activity. The result shows that chalcones, containing chloro/bromo substituents were active while other possessed moderate activity against bacterias.

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