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OPPI BRIEF

Synthesis and Antimicrobial Activities of Some 3-Phenanthryl Chalcones

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Chalcones are structurally interesting compounds with important biological activities. Because of their significance, numerous methods have been reported for the synthesis of these compounds. For example, recent work has shown the usefulness of potassium carbonate catalysis under microwave irradiation for synthesis of chalcones.[1] Chalcones have been reported to have antimicrobial,[2–4] anti-inflammatory,[5] antimalarial,[6,7] anti-leishmanial,[8] antioxidant,[9] and antitubercular activities.[10,11]

We now report our results on the preparation and biological activities of some novel 3-phenanthryl chalcones (*Scheme 1*). Our synthesis method uses microwave irradiation and $FeCl_3$-bentonite catalyst and is noteworthy for its convenience, short reaction times and high yields.

Our synthetic results are shown in *Tables 1 and 2*. Yields were uniformly 90% or higher, and the spectroscopic data were consistent with the expected structures.

The measured antibacterial activities of all styryl 3-phenanthryl ketones are presented in *Table 3*. All of the compounds displayed activities comparable to or slightly better than the standard ampicillin.

The observed antifungal activities of all styryl 3-phenanthryl ketones are presented in *Table 4*. All of the compounds showed activities comparable to or slightly better than the standard miconazole in the organisms tested.

In conclusion, we have demonstrated that novel 3-phenanthryl chalcones may be prepared by a convenient method in high yields using $FeCl_3$-bentonite catalyst under microwave irradiation. The synthesized compounds have useful antimicrobial activities. It is hoped that the ease of preparation will stimulate further research on these materials.

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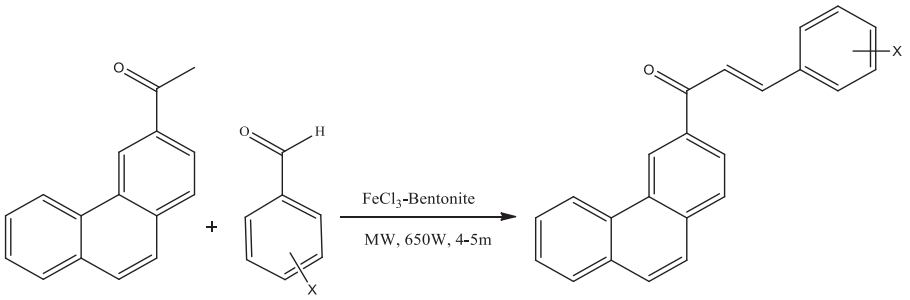

X= H, 3-Br, 4-Br, 2-Cl, 3-Cl, 4-F, 4-OCH_3, 3-CH_3, 2-NO_2, 4-NO_2

Scheme 1. Synthesis of 3-phenanthryl chalcones.

Experimental Section

All chemicals were procured from Sigma Aldrich. The melting points of all the chalcones were determined in open glass capillaries on a Mettler FP51 melting point apparatus and are uncorrected. Elemental analyses of all ketones were performed on a Perkin Elmer 240c analyzer. The IR spectra of all compounds were recorded using KBr discs on an Avatar-300 ThermoNikolet Fourier Transform infrared spectrophotometer with the frequency range of 4000–400 cm^{-1}. The NMR spectra of compounds were recorded on an INSTRUM AV300 spectrometer, operating at 500 MHz for ^1H spectra and 125.46 MHz for ^{13}C spectra in DMSO solvent using TMS as internal standard. Mass spectra of all compounds were recorded on a SHIMADZU GC-MS2010 spectrometer and VARIAN-SATURN 2200 GC-MS spectrometer using electron impact techniques. The $FeCl_3$-bentonite catalyst was prepared and its purity examined by a literature method.[13] 3-Acetylphenanthrene (2 mmol), substituted benzaldehyde (2 mmol), and $FeCl_3$-bentonite (0.3 g, 1.25 mmol) were taken in an accidental explosion proof pressure tube and flushed with argon and tightly capped. The reaction mixture was shielded and subjected to microwave irradiation for 4-5 minutes at 650W energy, applying 250V, 5 Amps AC in a microwave oven (LG Grill, Intellowave Microwave Oven, 160-800W) (*Scheme 1*) and then cooled to room temperature. The organic layer was extracted into dichloromethane which, on filtration and evaporation, gave the solid product. The insoluble catalyst was recycled by washing the solid remaining on the filter with ethyl acetate (8 mL), then drying in an oven at 100 °C for 1h. The crude chalcones were recrystallized from ethanol to obtain pale yellow glittering solids. The synthesized chalcones were fully characterized by their physical constants, elemental analysis and spectroscopic data, which are presented in *Tables 1 and 2*.

Antibacterial Sensitivity Assay

Antibacterial sensitivity assays were performed using the Kirby-Bauer[14] disc diffusion technique. In each Petri plate about 0.5 ml of the test bacterial sample (10 mg of the bacterial sample dissolved in 100 ml of sterile distilled water) was spread uniformly

Table 1
Preparation of 3-Phenanthryl Chalcones

Compound	X	MF	MW	Time (m)	Yield (%)	m.p. (° C)	Microanalysis (%)			Mass (m/z)
							C (calcd)	H (calcd)	N (calcd)	
1	H	$C_{23}H_{16}O$	308	4	96	106–107 (108–110)[12]	89.59 (89.58)	5.18 (5.23)	—	308[M^+]
2	3-Br	$C_{23}H_{15}BrO$	386	4.5	91	123–124	71.36 (71.33)	3.86 (3.90)	—	386[M^+], 338[M^{2+}]
3	4-Br	$C_{23}H_{15}BrO$	386	4.5	93	117–118	71.29 (71.33)	3.88 (3.90)	—	386[M^+], 338[M^{2+}]
4	2-Cl	$C_{23}H_{15}ClO$	342	4	90	132–133	80.59 (80.58)	4.38 (4.41)	—	342[M^+], 344[M^{2+}]
5	3-Cl	$C_{23}H_{15}ClO$	342	4	90	119–120	80.61 (80.58)	4.39 (4.41)	—	342[M^+], 344[M^{2+}]
6	4-F	$C_{25}H_{15}FO$	326	5	90	132–133	84.68 (84.64)	4.59 (4.63)	—	326[M^+], 328[M^{2+}]
7	4-OCH_3	$C_{24}H_{18}O_2$	338	4	95	106–107 (108–110)[12]	85.15 (85.18)	5.33 (5.36)	—	338[M^+]
8	3-CH_3	$C_{24}H_{18}O$	322	4	95	116–117 (115–118)[12]	89.45 (89.41)	5.59 (5.63)	—	322[M^+]
9	2-NO_2	$C_{23}H_{15}NO_3$	353	6	91	122–123	78.19 (78.17)	4.24 (4.28)	3.92 (3.96)	353[M^+]
10	4-NO_2	$C_{23}H_{15}NO_3$	353	6	94	117–118 (115–118)[12]	78.22 (78.17)	4.26 (4.28)	3.89 (3.96)	353[M^+]

Table 2
The Infrared Carbonyl Stretches (ν, cm^{-1}), NMR Chemical Shifts (δ, ppm) of H_α, H_β, Protons, CO, C_α and C_β Carbons of Synthesized 3-Phenanthryl Chalcones

Compound	X	IR (ν, cm^{-1})		^{1}HNMR (δ, ppm)		^{13}C NMR (δ, ppm)		
		$CO_{s\text{-}cis}$	$CO_{s\text{-}trans}$	H_α	H_β	CO	C_α	C_β
1	H	1651	1604	7.486	7.671	197.35	125.25	138.53
2	3-Br	1633	1600	7.492	7.645	197.10	124.83	133.40
3	4-Br	1631	1602	7.505	7.637	197.11	125.66	133.39
4	2-Cl	1639	1600	7.466	7.591	197.10	125.65	138.32
5	3-Cl	1643	1587	7.447	7.637	196.03	125.66	134.73
6	4-F	1683	1597	7.490	7.668	197.06	126.30	138.22
7	4-OCH_3	1685	1594	7.487	7.654	197.01	127.28	138.23
8	3-CH_3	1629	1600	7.434	7.705	197.45	124.35	138.67
9	2-NO_2	1691	1610	7.525	7.774	196.66	124.66	137.93
10	4-NO_2	1687	1608	7.551	7.787	196.67	124.55	136.26

Table 3
The Zone of Inhibition (mm) Values of Antibacterial Activity of Substituted Styryl 3-Phenanthryl Chalcones

Compound	X	Zone of Inhibition (mm)				
		Gram positive Bacteria			Gram negative Bacteria	
		B. subtilis	*M. luteus*	*S. aureus*	*E. coli*	*P. aeruginosa*
1	H	7	7	8	7	9
2	3-Br	9	10	11	9	7
3	4-Br	9	11	9	11	11
4	2-Cl	10	10	11	12	13
5	3-Cl	11	11	10	12	12
6	4-F	8	9	7	10	10
7	2-OCH_3	7	8	10	8	9
8	3-CH_3	8	9	8	9	10
9	2-NO_2	8	8	9	9	9
10	4-NO_2	7	7	7	9	10
Standard	Ampicillin	12	13	12	13	15
Control	DMSO	–	–	–	–	–

Table 4
Zone of Inhibition (mm) Values of Antifungal Activity of Substituted 3-Phenanthryl Chalcones

Compound	X	Zone of Inhibition (mm)		
		A. niger	*P. scup*	*T. viride*
1	H	7	8	10
2	3-Br	6	7	10
3	4-Br	8	7	10
4	2-Cl	8	8	10
5	3-Cl	6	7	9
6	4-F	8	7	11
7	2-OCH_3	7	10	10
8	3-CH_3	9	9	9
9	2-NO_2	9	10	10
10	4-NO_2	8	9	11
Standard	Miconazole	10	10	10
Control	DMSO	–	–	–

over the solidified Mueller Hinton agar using a sterile glass spreader. Then 5 mm diameter discs of Whatman No.1 filter paper, impregnated with the solution of the compound (250 μg/ml of each dissolved in 1 ml of DMSO solvent), were placed on the medium using sterile foreceps. The plates were incubated for 24 hours at 37°C. After

24 hrs, the plates were visually examined and the diameter values of the zones of inhibition were measured. Triplicate results were recorded by repeating the same procedure. Controls showed luxuriant growth.

Antifungal Sensitivity Assay

Antifungal sensitivity assays were performed using the Kirby-Bauer[14] disc diffusion technique. Potato dextrose agar medium was prepared and sterilized. It was poured into the plates already containing 1 mL of the fungal species (10 mg of the fungal sample dissolved in 100 ml of sterile distilled water). Whatman No.1 5 mm discs were impregnated with the test solution, prepared by dissolving 15 mg of the enone in 1 mL of DMSO solvent. The medium was allowed to solidify and kept for 24 hrs. Then the plates were visually examined and the diameter values of the zones of inhibition were measured. Triplicate results were recorded by repeating the same procedure. Controls showed luxuriant growth.

References

1. Y. K. Srivastava, *Rasayan J. Chem*., **1**, 884 (2008).
2. Y. Rajendra Prasad, A. Lakshmana Rao and R. Rambabu, *E-J. Chem*., **5**, 461 (2008).
3. S. N. López, M. V. Castelli, S. A. Zacchino, J. N. Domínguez, G. Lobo, J. C. Charris, J. C. G. Cortés, J. C. Ribas, C. Devia, A. M. Rodríguez and R. D. Enriz, *Bioorg. Med. Chem.,* **9**, 1999 (2001).
4. B. Baviskar, S. Patel, B. Baviskar, S. S. Khadabadi and M. Shiradkar, *Asian J. Res. Chem*., **1**, 67 (2008).
5. F. Herencia, M. L. Ferrandiz, A. Ubeda, J. N. Domínguez, J. E. Charris, G. M. Lobo and M. J. Alcaraz, *Bioorg. Med. Chem. Lett*., **8**, 1169 (1998).
6. X. Wu, P. Wilairat and M. L. Go, *Bioorg. Med. Chem. Lett*., **12**, 2299 (2002).
7. A. Agarwal, K. Srivastava, S. K. Puri and P. M. S. Chauhan, *Bioorg. Med. Chem.,* **13**, 6226 (2005).
8. T. Narender, T. Khaliq, Shweta, Nishi, N. Goyal and S. Gupta, *Bioorg. Med. Chem.,* **13**, 6543 (2005).
9. J. H. Cheng, C. F. Hung, S. C. Yang, J. P. Wang, S. J. Won and C. N. Lin, *Bioorg. Med. Chem*., **16**, 7270 (2008).
10. Y. M. Lin, Y. Zhou, M. T. Flavin, L. M. Zhou, W. Nie, F. C. Chen, *Bioorg. Med. Chem*., **10**, 2795 (2002).
11. P. M. Sivakumar, S. P. Seenivasan, V. Kumar and M. Doble, *Bioorg. Med. Chem. Lett*., **17**, 1695 (2007).
12. S. S. Misra, R.S. Tiwari, B. Nath, *Indian J. Appl. Chem*., **34**, 260 (1971).
13. I. Muthuvel, S. Dineshkumar, K. Thirumurthy, R. Rajasri and G. Thirunarayanan, *Indian J. Chem*., **55B**, 252 (2016).
14. A. W. Bauer, W. M. M. Kirby, J. C. Sherris and M. Truck, *Am. J. Clin. Pathol*., **45**, 493 (1966).