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Microwave-Assisted Synthesis and Antimicrobial Activity of Novel Pyrazole–Indanone Hybrid Analogs

N. Venkatesh[a], Sh. Sundergoud[a], M. Kumara Swamy[a], and P. Veerasomaiah[a],*

[a] *Department of Chemistry, University College of Science, Osmania University, Hyderabad, 500007 India*

**e-mail: veerasomaiahp@gmail.com*

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Abstract—A simple and efficient microwave-assisted protocol has been developed for the synthetic of a series of novel pyrazole–indanone hybrid analogs. The target compounds have been synthesized by the Claisen–Schmidt condensation of different 1,3-diphenyl-1*H*-pyrazole-4-carbaldehydes with 2,3-dihydro-1*H*-inden-1-one in the presence of potassium hydroxide. The compounds were characterized by IR, ^{1}H and ^{13}C NMR, and mass spectra and were found to exhibit potent antimicrobial activity in vitro.

Keywords: pyrazole, indan-1-one, chalcone, microwave irradiation, Vilsmeier–Haack reaction, Claisen–Schmidt condensation, antimicrobial activity

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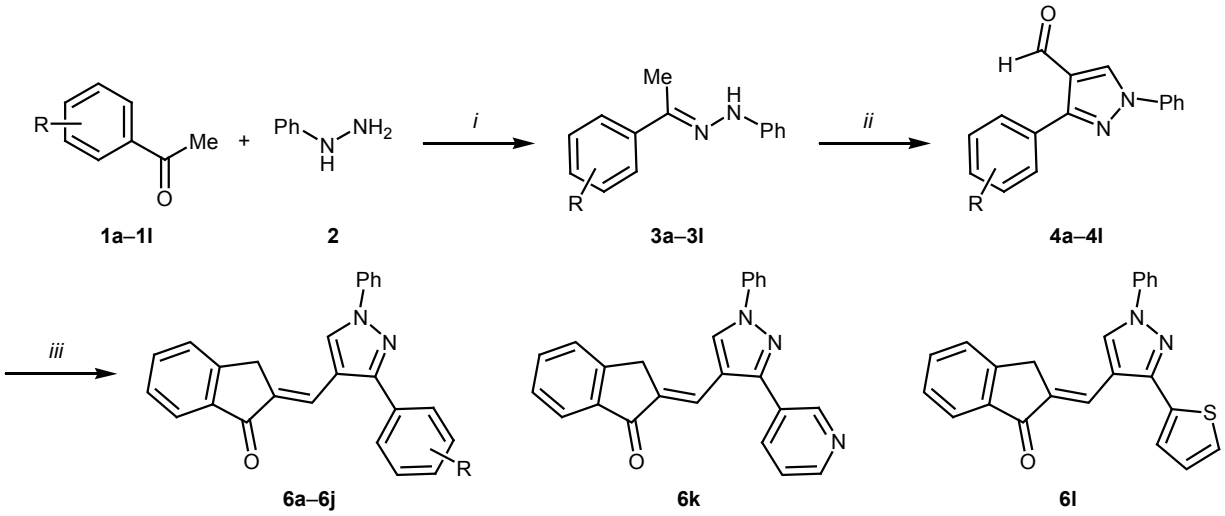
INTRODUCTION

Pyrazoles constitute one of the important classes of nitrogen heterocycles and occupy a unique position in medicinal chemistry due to their broad spectrum of biological activities such as antimicrobial [1], anti-inflammatory [2], anticancer [3], anticonvulsant [4], and antipyretic [5] activities. Among the pyrazole family, 1,3-diarylpyrazoles have gained much interest in recent pharmacological applications. Indanone derivatives are widely used in medicinal chemistry, natural product synthesis, and agricultural products. Indanone derivatives are associated with various biological activities such as anti-inflammatory [6], analgesic [7], antimicrobial [8], anticholinergic [9], dopaminergic [10], anticancer [11], and antimalarial [12] activities. Indanone derivatives are very useful synthons for various carbocyclic and heterocyclic molecules as synthetic intermediates for several drugs and natural products [13–15]. On the other hand, chalcones are natural biocides and are well known intermediates in the synthesis of various heterocyclic compounds. Chalcone derivatives possess a wide range of biological activities such as antimicrobial [16], anti-inflammatory [17], and anticancer [18] activities. Chalcones constitute an important class of natural products belonging to the flavonoid family [19] and are also key precursors in the synthesis of many biologically important heterocycles such as benzothiazepines [20], dihydropyrazoles [21], 1,4-diketones [22], and flavones [23]. On the other hand, microwave irradiation method has gained attention of chemists during the last decades due to its unique advantages such as shorter reaction times, cleaner reaction products, higher yields, and better selectivity, and provided a valuable alternative to accomplish more efficient syntheses of a variety of organic compounds with much simpler operation and milder reaction conditions. In addition, synthesis of hybrid heterocyclic compounds has attracted much attention in recent years. It is expected that combining features of more than one biologically active fragments in a single molecule may result in pronounced pharmacological activity while retaining high diversity and biological relevance. Owing to the above facts, we made an attempt to synthesize (*E*)-2-[(1,3-diphenyl-1*H*-pyrazol-4-yl)methylidene]-2,3-dihydro-1*H*-inden-1-one derivatives under microwave irradiation.

RESULTS AND DISCUSSION

Intermediate 1,3-diaryl-1*H*-pyrazole-4-carbaldehydes **4a**–**4l** were prepared starting from commercially available acetophenones **1a**–**1l** and phenylhydrazine (**2**) through intermediate hydrazones **3a**–**3l** [24, 25]. 1,3-Diaryl-1*H*-pyrazole-4-carbaldehydes **4a**–**4l** were condensed with 2,3-dihydro-1*H*-inden-1-one (**5**) in the presence of potassium hydroxide in methanol under microwave irradiation to yield the corresponding pyrazole–indanone conjugates **6a**–**6l** (Scheme 1). Initially,

Scheme 1.



R = H (**a**), 4-Me (**b**), 4-MeO (**c**), 4-Br (**d**), 4-Cl (**e**), 4-F (**f**), 4-HO (**g**), 4-O_2N (**h**), 3-MeO (**i**), 2,4-Cl_2 (**j**).
i: Glacial acetic acid, MeOH, heating; *ii*: DMF (3 equiv), $POCl_3$ (7 equiv), 0–5°C, 90°C, 8 h;
iii: indan-1-one (**5**), KOH, MeOH, MW, 320 W, 4–5 min.

the model condensation of 1,3-diphenyl-1*H*-pyrazole-4-carbaldehyde (**1a**) with indanone **6** was carried out in the presence of piperidine at room temperature for 8 h to obtain 58% of **6a**. The yield did not increase to an appreciable extent when pyrrolidine was used instead of piperidine, other conditions being equal. Using sodium hydroxide as a base, the product yield was improved to 67%, but the best result (72% yield) was obtained in the presence of potassium hydroxide. The reaction was then carried out under different temperature conditions, but the yield did not vary significantly with temperature. Finally, the reaction was conducted under microwave irradiation, which significantly increased the yield up to 92% with shorter reaction time. The structure of compounds **6a**–**6l** was established by different spectroscopic methods such as IR, ^{1}H and ^{13}C NMR and mass spectrometry.

Compounds **6a**–**6l** were tested for their in vitro antibacterial activity against four bacterial strains, namely *B. faecalis*, *S. aureus*, *K. pneumoniae*, and *E. coli*, using ampicillin as standard drug. The activity was determined using cup plate agar diffusion method by measuring the zone of inhibition in mm at a concentration of 100μg/mL in DMSO. Compounds **6e**, **6f**, and **6k** showed a good activity against the tested bacterial organisms and the remaining compounds showed low to moderate activity compared with the standard drug ampicillin.

The antifungal activity of compounds **6a**–**6l** was evaluated in vitro against *Aspergillus niger* and *Candida metapsilosis* using griseofulvin as standard drug. The activity was determined using cup plate agar diffusion method by measuring the zone of inhibition in mm at a concentration of 500 μg/mL in DMSO. Compounds **6f**, **6j**, and **6l** showed a good activity against the tested fungi, and the remaining compounds showed low to moderate antifungal activity.

EXPERIMENTAL

The purity of the compounds was checked by TLC using precoated silica gel 60 F_{254} plates (Merck). The IR spectra were recorded in KBr on a Shimadzu FT-IR-8400s spectrometer. The ^{1}H NMR and ^{13}C NMR spectra were recorded on a Bruker Avance II 400 MHz spectrometer using tetramethylsilane as an internal standard. The mass spectra were recorded on a Shimadzu GCMS-QP 1000 EX mass spectrometer.

General procedure for the synthesis of (*E*)-2-[(1,3-diphenyl-1*H*-pyrazol-4-yl)methylidene]-2,3-dihydro-1*H*-inden-1-ones 6a–6l. A quartz tube inserted into a screw-capped Teflon vial was charged with a mixture of 1,3-diphenyl-1*H*-pyrazole-4-carbaldehyde **4a**–**4l** (1 mmol), 2,3-dihydro-1*H*-inden-1-one (**5**, 1 mmol), and potassium hydroxide (2 mmol) in methanol (10 mL). The mixture was subjected to microwave irradiation at 320 W for 4–5 min, and the progress of the reaction was monitored by TLC. After completion of the reaction, the mixture was poured into ice-cold water and neutralized with dilute hydrochloric acid to obtain solid product. The product was washed with water, dried, and purified by column chromatography using *n*-hexane–ethyl acetate as eluent.

(*E*)-2-[(1,3-Diphenyl-1*H*-pyrazol-4-yl)methylidene]-2,3-dihydro-1*H*-inden-1-one (6a). Yield 90%. ^{1}H NMR spectrum, δ, ppm: 4.12 s (2H, CH_2), 7.40–7.44 t (1H, H_{arom}), 7.46–7.51 m (3H, H_{arom}), 7.53–7.60 m (4H, H_{arom}), 7.66–7.74 m (4H, H_{arom}), 7.76 d (1H, H_{arom}), 8.06 d (2H, H_{arom}), 8.98 s (1H, H_{arom}). ^{13}C NMR spectrum, δ_C, ppm: 31.8, 116.6, 119.1, 122.2, 123.5, 126.3, 127.2, 127.5, 129.0, 129.5, 130.5, 131.0, 131.8, 134.2, 134.7, 137.7, 138.8, 149.3, 152.6, 192.5. Mass spectrum: *m*/*z* 363 $[M + H]^+$.

(*E*)-2-{[3-(4-Methylphenyl)-1-phenyl-1*H*-pyrazol-4-yl]methylidene}-2,3-dihydro-1*H*-inden-1-one (6b). Yield 92%. ^{1}H NMR spectrum, δ, ppm: 2.45 s (3H, OCH_3), 4.11 s (2H, CH_2), 7.26 d (2H, H_{arom}), 7.41–7.51 m (3H, H_{arom}), 7.56–7.61 m (3H, H_{arom}), 7.66–7.78 m (4H, H_{arom}), 8.05 d (2H, H_{arom}), 8.96 s (1H, H_{arom}). ^{13}C NMR spectrum, δ_C, ppm: 31.8, 24.5, 115.9, 121.3, 123.4, 125.9, 127.1, 127.4, 128.9, 129.4, 130.2, 130.9, 132.0, 133.9, 134.5, 137.5, 138.6, 149.2, 152.5, 192.4. Mass spectrum: *m*/*z* 377 $[M + H]^+$.

(*E*)-2-{[3-(4-Methoxyphenyl)-1-phenyl-1*H*-pyrazol-4-yl]methylidene}-2,3-dihydro-1*H*-inden-1-one (6c). Yield 92%. ^{1}H NMR spectrum, δ, ppm: 3.85 s (3H, OCH_3), 4.13 s (2H, CH_2), 7.14 d (2H, H_{arom}), 7.39–7.50 m (3H, H_{arom}), 7.57–7.60 m (3H, H_{arom}), 7.67–7.78 m (4H, H_{arom}), 8.05 d (2H, H_{arom}), 8.97 s (1H, H_{arom}). ^{13}C NMR spectrum, δ_C, ppm: 31.8, 55.6, 108.5, 116.5, 118.8, 122.4, 123.1, 125.9, 126.8, 127.3, 128.4, 128.9, 129.5, 131.3, 133.9, 134.5, 136.8, 149.1, 152.5, 153.5, 192.3. Mass spectrum: *m*/*z* 393 $[M + H]^+$.

(*E*)-2-{[3-(4-Bromophenyl)-1-phenyl-1*H*-pyrazol-4-yl]methylidene}-2,3-dihydro-1*H*-inden-1-one (6d). Yield 90%. ^{1}H NMR spectrum, δ, ppm: 4.11 s (2H, CH_2), 7.42–7.47 m (3H, H_{arom}), 7.66–7.62 m (3H, H_{arom}), 7.67–7.71 m (3H, H_{arom}), 7.75–7.78 m (3H, H_{arom}), 8.04 d (2H, H_{arom}), 8.97 s (1H, H_{arom}). ^{13}C NMR spectrum, δ_C, ppm: 31.8, 116.6, 119.0, 122.6, 123.4, 126.4, 127.1, 127.5, 128.7, 128.8, 128.9, 129.5, 131.8, 134.0, 134.7, 137.8, 138.9, 149.4, 153.9, 192.6. Mass spectrum: *m*/*z* 441 $[M + H]^+$.

(*E*)-2-{[3-(4-Chlorophenyl)-1-phenyl-1*H*-pyrazol-4-yl]methylidene}-2,3-dihydro-1*H*-inden-1-one (6e). Yield 89%. ^{1}H NMR spectrum, δ, ppm: 4.11 s (2H, CH_2), 7.40–7.47 m (3H, H_{arom}), 7.66–7.63 m (2H, H_{arom}), 7.65–7.72 m (6H, H_{arom}), 7.76 d (1H, H_{arom}), 8.04 d (2H, H_{arom}), 8.97 s (1H, H_{arom}). ^{13}C NMR spectrum, δ_C, ppm: 31.8, 116.5, 119.0, 122.5, 123.3, 126.5, 126.9, 127.4, 128.8, 129.0, 129.3, 129.7, 131.5, 134.2, 134.8, 137.5, 138.7, 149.2, 153.5, 192.4. Mass spectrum: *m*/*z* 397 $[M + H]^+$.

(*E*)-2-{[3-(4-Fluorophenyl)-1-phenyl-1*H*-pyrazol-4-yl]methylidene}-2,3-dihydro-1*H*-inden-1-one (6f). Yield 90%. ^{1}H NMR spectrum, δ, ppm: 4.11 s (2H, CH_2), 7.28 d (2H, H_{arom}), 7.42–7.45 m (1H, H_{arom}), 7.65–7.61 m (4H, H_{arom}), 7.69–7.75 m (3H, H_{arom}), 7.76–7.80 m (2H, H_{arom}), 8.05 d (2H, H_{arom}), 8.96 s (1H, H_{arom}). ^{13}C NMR spectrum, δ_C, ppm: 31.8, 114.6, 118.9, 121.5, 123.4, 125.9, 126.9, 127.5, 128.6, 128.9, 129.3, 131.7, 133.8, 134.5, 137.5, 138.5, 149.2, 153.9, 192.4. Mass spectrum: *m*/*z* 381 $[M + H]^+$.

(*E*)-2-{[3-(4-Hydroxyphenyl)-1-phenyl-1*H*-pyrazol-4-yl]methylidene}-2,3-dihydro-1*H*-inden-1-one (6g). Yield 87%. ^{1}H NMR spectrum, δ, ppm: 4.11 s (2H, CH_2), 7.09 d (2H, H_{arom}), 7.38–7.47 m (3H, H_{arom}), 7.55–7.59 m (3H, H_{arom}), 7.65–7.75 m (5H, H_{arom}), 8.05 d (2H, H_{arom}), 8.96 s (1H, H_{arom}). ^{13}C NMR spectrum, δ_C, ppm: 31.8, 104.3, 116.3, 118.5, 122.0, 122.5, 125.7, 126.0, 126.5, 127.3, 127.6, 128.2, 128.5, 129.4, 131.1, 133.5, 134.2, 136.3, 149.0, 153.0, 192.2. Mass spectrum: *m*/*z* 379 $[M + H]^+$.

(*E*)-2-{[3-(4-Nitrophenyl)-1-phenyl-1*H*-pyrazol-4-yl]methylidene]-2,3-dihydro-1*H*-inden-1-one (6h). Yield 85%. ^{1}H NMR spectrum, δ, ppm: 4.17 s (2H, CH_2), 7.42–7.47 m (3H, H_{arom}), 7.61–7.90 m (5H, H_{arom}), 8.09 d (2H, H_{arom}), 8.13–8.15 m (3H, H_{arom}), 8.38 d (2H, H_{arom}), 9.06 s (1H, H_{arom}). ^{13}C NMR spectrum, δ_C, ppm: 31.8, 117.1, 119.2, 122.5, 123.3, 125.9, 126.9, 127.4, 127.9, 129.0, 129.4, 129.9, 130.2, 130.6, 136.9, 138.9, 142.3, 145.3, 149.5, 152.3, 192.4. Mass spectrum: *m*/*z* 408 $[M + H]^+$.

(*E*)-2-{[3-(3-Methoxyphenyl)-1-phenyl-1*H*-pyrazol-4-yl]methylidene]-2,3-dihydro-1*H*-inden-1-one (6i). Yield 88%. ^{1}H NMR spectrum, δ, ppm: 3.84 s (3H, OCH_3), 4.11 s (2H, CH_2), 7.11 d (1H, H_{arom}), 7.28 d (1H, H_{arom}), 7.42–7.49 m (3H, H_{arom}), 7.56–7.62 m (4H, H_{arom}), 7.67–7.75 m (3H, H_{arom}), 8.05 d (2H, H_{arom}), 8.96 s (1H, H_{arom}). ^{13}C NMR spectrum, δ_C, ppm: 31.7, 111.7, 113.2, 116.5, 119.1, 123.1, 126.6, 127.5, 128.5, 129.1, 129.5, 130.0, 131.3, 133.9, 137.5, 138.5, 149.5, 152.4, 153.6, 192.5. Mass spectrum: *m*/*z* 393 $[M + H]^+$.

(*E*)-2-{[3-(2,4-Dichlorophenyl)-1-phenyl-1*H*-pyrazol-4-yl]methylidene}-2,3-dihydro-1*H*-inden-1-one (6j). Yield 87%. ^{1}H NMR spectrum, δ, ppm: 4.11 s (2H, CH_2), 7.38–7.45 m (2H, H_{arom}), 7.47–7.50 m (1H, H_{arom}), 7.61–7.66 m (3H, H_{arom}), 7.65–7.71 m (4H, H_{arom}), 7.75 d (1H, H_{arom}), 8.04 d (2H, H_{arom}), 8.96 s (1H, H_{arom}). ^{13}C NMR spectrum, δ_C, ppm: 31.8, 116.3, 118.8, 121.5, 123.3, 125.2, 126.5, 1296.9, 127.3, 128.6, 129.5, 130.2, 130.5, 133.4, 134.6, 136.7, 137.4, 139.1, 149.5, 153.2, 192.4. Mass spectrum: *m*/*z* 431 $[M + H]^+$.

(*E*)-2-{[1-Phenyl-3-(pyridin-3-yl)-1*H*-pyrazol-4-yl]methylidene}-2,3-dihydro-1*H*-inden-1-one (6k). Yield 85%. ^{1}H NMR spectrum, δ, ppm: 4.10 s (2H, CH_2), 7.40–7.42 m (2H, H_{arom}), 7.62–7.77 m (9H, H_{arom}), 7.96 d (1H, H_{arom}), 8.06 d (1H, H_{arom}), 8.11 d (1H, H_{arom}), 8.96 s (1H, H_{arom}). ^{13}C NMR spectrum, δ_C, ppm: 31.7, 116.5, 118.9, 122.2, 123.2, 1245.5, 126.6, 127.3, 128.5, 129.5, 129.9, 130.5, 131.4, 133.6, 134.2, 135.7, 137.8, 140.5, 149.5, 153.1, 192.4. Mass spectrum: *m*/*z* 364 $[M + H]^+$.

(*E*)-2-{[1-Phenyl-3-(thiophen-2-yl)-1*H*-pyrazol-4-yl]methylidene}-2,3-dihydro-1*H*-inden-1-one (6l). Yield 90%. ^{1}H NMR spectrum, δ, ppm: 4.11 s (2H, CH_2), 7.10–7.12 m (1H, H_{arom}), 7.38–7.42 m (2H, H_{arom}), 7.66–7.63 m (2H, H_{arom}), 7.65–7.72 m (6H, H_{arom}), 7.76 d (1H, H_{arom}), 7.91 d (1H, H_{arom}), 8.96 s (1H, H_{arom}). ^{13}C NMR spectrum, δ_C, ppm: 31.7, 115.5, 116.3, 118.2, 122.5, 123.1, 125.7, 126.8, 128.5, 129.3, 129.6, 129.9, 131.2, 133.8, 136.9, 138.5, 140.2, 149.1, 152.2, 192.3. Mass spectrum: *m*/*z* 369 $[M + H]^+$.

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CONFLICT OF INTEREST

The authors declare that they have no conflict of interest.

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