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A Facile and Green Approach for One-Pot Synthesis of New Pyrrole Derivatives by Three-Component Reaction Between Pyrrole, Arylglyoxals and Meldrum's Acid

Z. Damanzan[a], M. Anary-Abbasinejad[a,*] and M. Masoudi[b,*]

[a]*Department of Chemistry, Faculty of Science, Vali-e-Asr University of Rafsanjan, Rafsanjan 7718897111, Iran*

[b]*Department of Chemistry, Islamic Azad University, Rafsanjan Branch, Rafsanjan, Iran*

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An efficient one-pot three-component reaction of pyrrole with aryl glyoxal derivatives and Meldrum's acid (MA) in water at room temperature afforded 2,2-dimethyl-5-(2-oxo-2-aryl-1-(1H-pyrrol-2-yl)ethyl)-1,3-dioxane-4,6-dione derivatives under catalyst-free conditions in high yields. Products were isolated and purified by simple filtration and washing with diethyl ether and their structures were established from their IR and NMR spectroscopic data.

Keywords: Pyrrole, Arylglyoxals, Meldrum's acid, green synthesis, Three-component reaction

INTRODUCTION

Pyrrole is one of the most important nitrogen-containing heterocycles [1]; it is the critical motif in many natural products such as bacteriochlorins, porphyrins, porphyrinogens, vitamin B12, chlorophyll, bile pigments like biliverdin and bilirubin, and alkaloids isolated from marine sources [2-7]. Many pyrrole derivatives are also found to possess various biological and pharmaceutical activities [1-16].

Multicomponent reactions (MCRs) have attracted much attention from chemists for the synthesis of complex molecules with large molecular diversity, atom economy, and high efficiency. MRCs, which are usually single-step reactions made from three or more reactants, produce beneficial chemical products. Their design may be regarded as a preeminent point in organic chemistry [17-19].

Meldrum's acid (MA) is an interesting acidic organic compound introduced for the first time by Meldrum in 1908 [20]. Frequently, it is easily synthesized by the condensation reaction of malonic acid and acetone in acetic anhydride in Not only the compound has high acidity (pK_a = 7.5) [22] but the presence of concentrated sulfuric acid as a catalyst [21]. also it is used in MCRs to synthesize natural products. Furthermore, it is reactive toward nucleophiles at C4 and C6 and electrophiles at C5 [23-26].

In continuation of our recent works on the preparation of heterocyclic compounds [27-29], here we report a one-pot three-component synthesis of mono-substituted pyrrole derivatives from the reaction of pyrrole, aryl glyoxal, and MA in water, as a green solvent, at room temperature under catalyst-free conditions in high yield.

EXPERIMENTAL PROCEDURES AND MATERIALS

According to the reported procedure [31], the target aryl glyoxal compounds 2 were synthesized by SeO_2-oxidation of related aryl methyl ketones. IR spectra were obtained on a Shimadzu IR-470 spectrometer. The proton and carbon-13 NMR spectra were recorded on the Varian model UNITY Inova 500 MHz at 500 and 125 MHz, respectively, with Me_4Si as an internal standard in $CDCl_3$. The chemicals were purchased from Fluka (Buchs, Switzerland) and were used as received without further purification. All the products were characterized using the NMR and IR spectroscopy as well as analytical data.

*Corresponding author. E-mail: masoudimozhgan225@yahoo.com

General Procedure for Synthesis of Compounds 4a-f and 6a-d

A mixture of pyrrole or indole (1 mmol), aryl glyoxal derivative (1 mmol), and MA (1 mmol) in water (5 ml) as a green solvent was stirred at room temperature for 2.5 h. The precipitated solid was filtered off and washed with diethyl ether (10 ml) to afford the pure product 4 or 6.

5-(2-(4-Chlorophenyl)-2-oxo-1-(1*H*-pyrrol-2-yl) ethyl)-2,2-dimethyl-1,3-dioxane-4,6-dione (4a). Yield 333 mg (92%), gray powder, mp137-139 °C. IR spectrum (KBr), ῡ, cm^{-1}: 3421 (NH), 1768, 1731, 1675 (C=O). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 1.81 (3H, s, CH_3), 1.87 (3H, s, CH_3), 3.87 (1H, d, *J* = 2.4 Hz, CH), 5.67(1H, d, *J* = 2.4 Hz, CH), 6.19 (1H, m, Pyrrole -H), 6.35(1H, m, Pyrrole-H), 6.71 (1H, m, Pyrrole -H), 7.28 (2H, d, *J* = 8.7 Hz, Ar-H), 7.72 (2H, d, *J* = 8.7 Hz, ArH), 9.33 (1H, bs, NH), ^{13}C NMR spectrum, δ, ppm (*J*, Hz): 27.0, 28.2, 48.7, 49.4, 105.4, 108.7, 110.6, 119.7, 124.9, 128.7, 130.6, 133.5, 139.8, 164.8, 166.2, 194.4. Found, %: C 59.48; H 4.36; N 3.65. $C_{18}H_{16}ClNO_5$. Calculated, %: C 59.76; H 4.46; N 3.87.

5-(2-(4-Bromophenyl)-2-oxo-1-(1*H*-pyrrol-2-yl) ethyl)-2,2-dimethyl-1,3-dioxane-4,6-dione (4b). Yield 386 mg (95%), gray powder, mp 145-147 °C. IR spectrum (KBr), ῡ, cm^{-1}: 3417 (NH), 1769, 1732, 1676 (C=O). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 1.80 (3H, s, CH_3), 1.87 (3H, s, CH_3), 3.87 (1H, d, *J* = 2.4 Hz, CH), 5.66(1H, d, *J* = 2.4 Hz, CH), 6.18 (1H, m, Pyrrole-H), 6.35(1H, m, Pyrrole-H), 6.71 (1H, m, Pyrrole-H), 7.45 (2H, d, *J* = 8.8 Hz, Ar-H), 7.64 (2H, d, *J* = 8.8 Hz, Ar-H), 9.33 (1H, bs, NH). ^{13}C NMR spectrum, δ, ppm (*J*, Hz): 27.0, 28.2, 48.7, 49.4, 105.4, 108.8, 110.7, 119.7, 124.9, 128.6, 130.6, 131.7 164.8, 166.2, 194.6. Found, %: C 53.36; H 3.88; N 3.60. $C_{18}H_{16}BrNO_5$. Calculated, %: C 53.22; H 3.97; N 3.45.

2,2-Dimethyl-5-(2-(4-nitrophenyl)-2-oxo-1-(1*H*-pyrrol-2-yl)ethyl)-1,3-dioxane-4,6-dione(4c). Yield 361 mg (97%), gray powder, mp 136-139 °C. IR spectrum (KBr), ῡ, cm^{-1}: 3407 (NH), 1775, 1740, 1686 (C=O). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 1.83 (3H, s, CH_3), 1.89 (3H, s, CH_3), 3.94 (1H, d, *J* = 2.4 Hz, CH), 5.69 (1H, d, *J* = 2.4 Hz, CH), 6.18 (1H, m, Pyrrole -H), 6.36 (1H, m, Pyrrole -H), 6.71 (1H, m, Pyrrole -H), 7.88 (2H, d, *J* = 9.1 Hz, Ar-H), 8.14 (2H, d, *J* = 9.1 Hz, Ar-H), 9.36 (1H, bs, NH). ^{13}C NMR spectrum, δ, ppm (*J*, Hz): 26.9, 28.2, 48.9, 49.4, 105.6, 109.1, 111.3, 120.1, 123.4, 123.9, 130.0, 140.1, 150.1, 164.5, 166.0, 194.6. Found, %: C 58.16; H 3.98; N 7.26. $C_{18}H_{16}N_2O_7$. Calculated, %: C 58.07; H 4.33; N 7.52.

2,2-Dimethyl-5-(2-oxo-2-phenyl-1-(1*H*-pyrrol-2-yl)ethyl)-1,3-dioxane-4,6-dione (4d). Yield 311 mg (95%), gray powder, mp 146-148 °C. IR spectrum (KBr), ῡ, cm^{-1}: 3385 (NH), 1777, 1740, 1665 (C=O). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 1.81 (3H, s, CH_3), 1.88 (3H, s, CH_3), 3.87 (1H, d, *J* = 2.4 Hz, CH), 5.73 (1H, d, *J* = 2.4 Hz, CH), 6.18 (1H, m, Pyrrole-H), 6.3 6(1H, m, Pyrrole-H), 6.70 (1H, m, Pyrrole -H), 7.31 (2H, t, *J* = 7.7 Hz, Ar-H), 7.46 (1H, t, *J* = 7.6 Hz, Ar-H), 7.79 (2H, d, *J* = 8.0 Hz, Ar-H), 9.33 (1H, bs, NH) ppm, ^{13}C NMR spectrum, δ, ppm (*J*, Hz): 27.0, 28.2, 48.8, 49.5, 105.3, 108.6, 110.5, 119.5, 125.3, 128.3, 129.1, 133.3, 138.6, 164.6, 166.3, 195.4. Found, %: C 66.10; H 5.44; N 4.15. $C_{18}H_{17}NO_5$. Calculated, %: C 66.05; H 5.23; N 4.28.

5-(2-(4-Fluorophenyl)-2-oxo-1-(1*H*-pyrrol-2-yl) ethyl)-2,2-dimethyl-1,3-dioxane-4,6-dione (4e). Yield 335 mg (97%), gray powder, mp 134-137 °C. IR spectrum (KBr), ῡ, cm^{-1}: 3406 (NH), 1775, 1740, 1686(C=O). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 1.84 (3H, s, CH_3), 1.89 (3H, s, CH_3), 3.95(1H, d, *J* = 2.3 Hz, CH), 5.70 (1H, d, *J* = 2.3 Hz, CH), 6.19 (1H, m, Pyrrole-H), 6.37 (1H, m, Pyrrole-H), 6.72 (1H, m, Pyrrole-H), 7.88 (2H, dd, *J* = 8.7 Hz, *J* = 2.0 Hz, Ar-H), 8.15 (2H, m, Ar-H), 9.37 (1H, bs, NH). ^{13}C NMR spectrum, δ, ppm (*J*, Hz): 26.9, 28.2, 48.9, 49.4, 105.6, 109.1, 111.3, 120.1, 123.4, 130.0, 163.2 (d, j = 234 Hz), 164.6, 166.0, 195.4. Found, %: C 62.37; H 4.59; N 3.95. $C_{18}H_{16}FNO_5$. Calculated, %: C 62.61; H 4.67; N 4.06.

2,2-Dimethyl-5-(2-oxo-2-phenyl-1-(1*H*-pyrrol-2-yl)ethyl)-1,3-dioxane-4,6-dione (4f). Yield 307 mg (90%), gray powder, mp153-156 °C. IR spectrum (KBr), ῡ, cm^{-1}: 3363 (NH), 1775, 1743, 1667 (C=O) ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 1.80 (3H, s, CH_3), 1.87 (3H, s, CH_3), 2.32 (3H, s, CH_3), 3.85 (1H, d, *J* = 2.3 Hz, CH), 5.72 (1H, d, *J* = 2.3 Hz, CH) 6.18 (1H, m, Pyrrole -H), 6.35 (1H, m, Pyrrole-H), 6.70 (1H, m, Pyrrole -H), 7.11 (2H, d, *J* = 7.7 Hz, Ar-H), 7.71 (2H, d, *J* = 7.6 Hz, Ar-H), 9.33 (1H, bs, NH). ^{13}C NMR spectrum, δ, ppm (*J*, Hz): 21.9, 27.3, 28.5, 48.7, 49.5, 105.8, 108.2, 110.3, 119.1, 125.5, 129.00, 129.3, 164.5, 166.1, 194.5. Found, %: C 66.67; H 5.49; N 3.89. $C_{19}H_{19}NO_5$. Calculated, %: C 66.85; H 5.61; N 4.10.

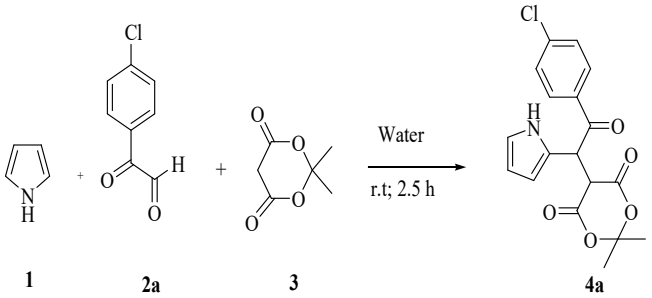
1-(Phenyl)-2,2- di(1H-indol-3-yl)ethanone (6a) [30]. Yield 377 mg (90%), brown solid; mp 205-206 °C. IR spectrum (KBr), ῡ, cm^{-1}: 3390, 1675, 1453, 1339, 1006,

^{1}H NMR spectrum, δ, ppm (*J*, Hz): 6.64 (s,1H), 6.93 (t, *J* = 7.2 Hz, 2H), 7.04 (t, *J* = 7.2 Hz, 2H), 7.17 (s, 2H), 7.33 (d, *J* = 7.6 Hz, 2H), 7.46(d, *J* = 7.6 Hz, 2H), 7.55 (d, *J* = 7.6 Hz, 3H), 8.16 (d, *J* = 7.6 Hz, 2H), 10.91 (2H, NH).

1-(4-Bromophenyl)-2,2-di(1H-indol-3-yl)ethanone (6b) [30]. Yield 410 mg (95%), brown solid; mp 229-231 °C. IR spectrum (KBr), ῡ, cm-1: 3400, 1676, 1583, 1452, 1004; ^{1}H NMR spectrum, δ, ppm (J, Hz): 6.32 (s, 1H), 6.83 (s, 2H), 6.94 (t, *J* = 7.2 Hz, 2H), 7.04 (t, *J* = 7.2 Hz, 2H), 7.25 (d, *J* = 8 Hz, 2H), 7.42 (d, 2H), 7.42 (d, *J* = 8.2 Hz, 2H), 7.86 (d, *J* = 8.2 Hz, 2H), 9.23 (2H, NH).

1-(4-Nitrophenyl)-2, 2-di(1H-indol-3-yl)ethanone (6c) [30]. Yield 397 mg (95%), brown solid; mp 225-227 °C. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 3400, 1676, 1527, 1342; ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 6.7 (s,1H), 6.94 (t, *J* = 7.2 Hz, 2H), 7.04 (t, *J* = 7.2 Hz, 2H), 7.18 (d, *J* = 2 Hz, 2H), 7.33 (d, *J* = 8 Hz, 2H), 7.55 (d, *J* = 8 Hz, 2H), 8.25 (d, *J* = 8.4 Hz, 2H), 8.35 (d, *J* = 8.4 Hz, 2H), 10.94 (2H, NH).

1-(4-Chlorophenyl)-2,2-di(1H-indol-3-yl)ethanone (6d) [30]. Yield 348 mg (90%), brown solid; mp 195-197 °C. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 3400, 1677, 1586, 1433, 1080; ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 6.58 (s, 1H), 6.9 (t, *J* = 7.2 Hz, 2H), 7.01 (t, *J* = 7.2 Hz, 2H), 7.15 (d, *J* = 2 Hz, 2H), 7.3 (d, *J* = 8 Hz, 2H), 7.52 (d, *J* = 8 Hz, 2H), 7.54 (d, *J* = 8.4 Hz, 2H), 8.15 (d, *J* = 8.4 Hz, 2H), 10.89 (2H, NH).



1 2a 3 4a

Scheme 1. Reaction between pyrrole, 4-chlorophenylglyoxal and MA for synthesis of substituted pyrrole derivative 4a

Table 1. Optimization of the Reaction Conditions

Entry	Solvent	Temp.[a]	Yield (%)[b]
1	DMF	r.t.	56
2	DCM	r.t.	43
3	EtOH	r.t.	67
4	CH_3CN	r.t.	73
5	H_2O	r.t.	92
6	H_2O	Reflux	45

[a]Reaction conditions: solvent was 5 ml, reaction time was 2.5 h. [b]Isolated yields

RESULTS AND DISCUSSION

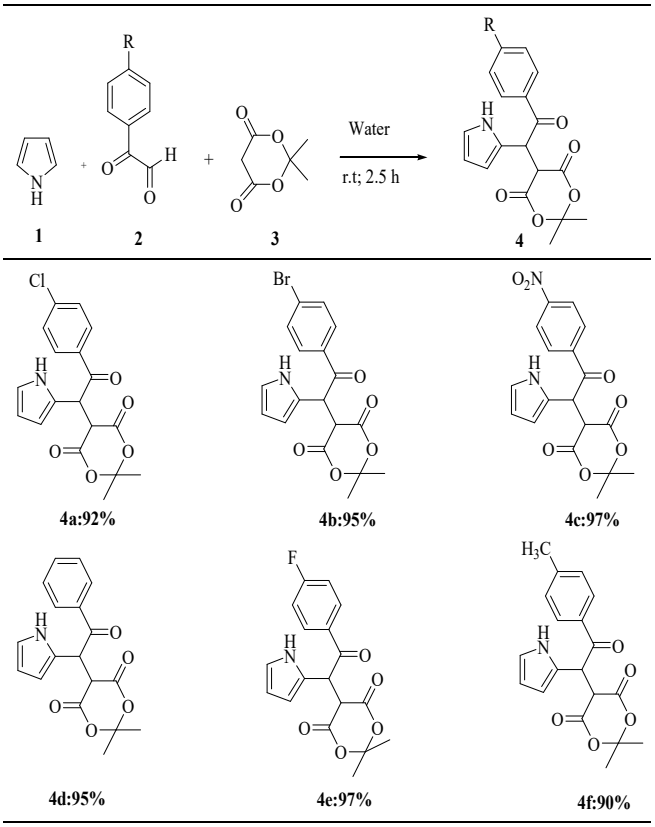
In a typical reaction, pyrrole, 4-chlorophenyl glyoxal, and MA were mixed in water at room temperature. The overall progress of the reaction was monitored by TLC. After 2.5 h stirring at room temperature, the starting materials disappeared on TLC and one pot that was identified to be 5-(2-(4-chlorophenyl)-2-oxo-1-(1H-pyrrol-2-yl)ethyl)-2,2-dimethyl-1,3-dioxane-4,6-dione 4a appeared. Compound 4a was isolated by simple filtration and washing by diethyl ether as a gray solid in 92% yield to offer a highly pure product (Scheme 1).

To find the optimal conditions, the cases such as the reaction solvent and the reaction temperature were investigated. For *optimization* of the *reaction solvent,* different solvents were examined. As shown in Table 1, the most product yield was obtained when water was selected as the solvent for this reaction. When the reaction was carried out in water under reflux conditions, the yield of product decreased (Table 1, entry 6) and due to the formation of by-products, isolation and purification of the product was very difficult.

In order to investigate the generality of the reaction, the different aryl glyoxals were treated with pyrrole and MA in the water; noticeably, the related pyrrole derivatives 4a-f were obtained in high yields (Table 2). This process has advantages such as easy separation and purification of products, high efficiency, and green reaction conditions, in addition to the mild reaction conditions.

All the products shown in Table 2 are stable solids and their structure assignments have been established on the basis of the elemental analysis, IR spectra, and NMR analysis. For instance, the ^{1}H NMR spectrum of 4a consisted of two singlets at 1.81 and 1.87 ppm for two methyl groups of Meldrum's acid. Two saturated CH protons that coupled to each other were discernible as two doublets at δ = 3.87 and

Table 2. The Reaction between Pyrrole, aryl Glyoxal, and MA

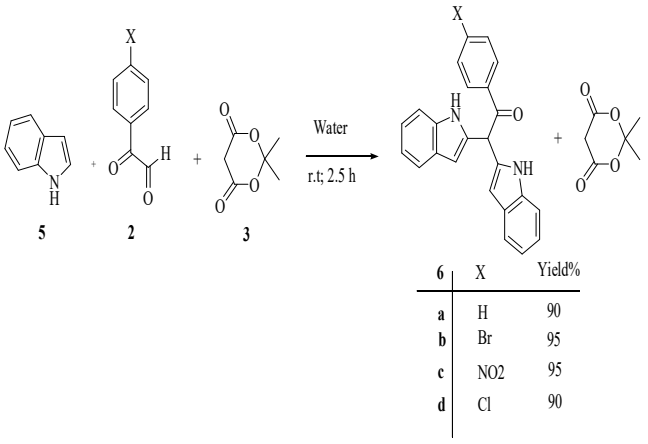


5.67 ppm with the same vicinal coupling constant ^{3}J of 2.4 Hz. Three signals were observed at 6.19, 6.35, and 6.71 ppm for the three protons of pyrrole ring in the form of three multiple signals. The aromatic protons resonated as two doublets at δ = 7.28 and 7.72 ppm. The last is a broad singlet integrated for one hydrogen that was observed at δ = 9.33 ppm for the NH proton. The ^{1}H-decoupled ^{13}C NMR spectrum of 4a showed characteristic signals at 27.0 and 28.2 ppm for two methyl groups of Meldrum's acid moiety and two signals of two CH groups were observed at 48.7 and 49.4 ppm. Also, 11 distinguishing signals were observed at 105.3-195.4 ppm, arising from the aromatic carbons and carbonyl of the desired product. The structure of compound 4a was also confirmed by its IR spectrum. An absorption band at 3421 cm^{-1} was assigned to NH stretching; in addition, three absorption bands at 1768, 1731, and 1675 cm^{-1} were assigned to carbonyl groups.

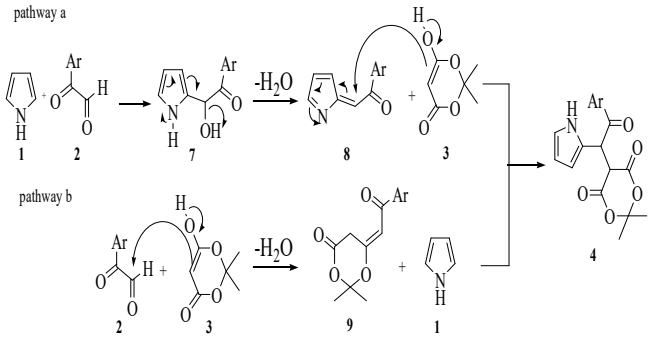
The reaction was also examined with linear 1,3-dicarbonyl compounds such as acetylacetone or methyl acetoacetate instead of MD. However, the product could not be isolated from the complex reaction mixture. We also could not isolate any product from the reaction of dimedone or barbituric acid with arylglyoxals and pyrrole.

When indole reacts with arylglyoxals and MA in reaction conditions mentioned for pyrrole, the only isolated product was bisindolylarylethanone derivatives 6 and MA played the role of an acidic catalyst (Scheme 2). The reaction of indole with arylglyoxals in the presence of acid catalysts has been previously reported for the synthesis of bisindolylarylethanones [30]. The structure of compounds 6a-6d was proved by the comparison of their IR and NMR data with the previously reported data [30].

Scheme 3 displays two plausible mechanisms for the formation of 5-(2-aryl)-2-oxo-1-(1H-pyrrol-2-yl)ethyl)-2,2-dimethyl-1,3-dioxane-4,6-dione derivatives 4a-4f. In pathway *a*, pyrrole first undergoes electrophilic aromatic substitution with aryl glyoxal derivatives to produce 2-[aroyl(hydroxyl)methyl]pyrrole intermediate **7**. The elimination of a water molecule from intermediate **7** leads to the formation of intermediate 8. Then, the nucleophilic addition of Meldrum's acid 3 to intermediate 8 afforded the desired products 4. In pathway *b*, arylglyoxals undergo the Knovenagel condensation with Meldrum's acid in aqueous media leading to the formation of adduct 9. Then the Michael addition of pyrrole to intermediate 9 furnishes the desired products 4.



Scheme 2. Synthesis of bisindolylarylethanone derivative catalyzed by Meldrum's acid



Scheme 3. The suggested mechanism for the formation of monosubstituted pyrrole derivatives 4

In summary, we found a simple and efficient method to synthesize a new class of monosubstituted pyrrole derivatives by a three-component reaction between pyrrole, arylglyoxals, and Meldrum's acid in aqueous media. Similar reactions between indole, arylglyoxals, and Meldrum's acid afforded bisindolylarylethanone derivatives with acceptable yields. The advantages of this method are the availability of the starting materials, neutral reaction conditions, and the use of water as an environmentally green solvent. The products were isolated and purified by simple filtration and washing by diethyl ether and obtained in excellent yields.

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