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ZnO nanorods as an efficient catalyst for the green synthesis of indole derivatives using isatoic anhydride

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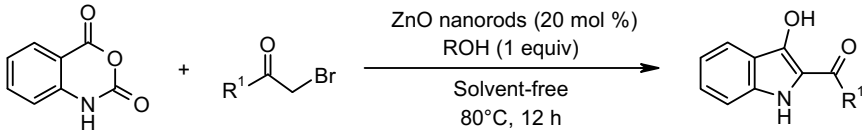


R = Me, Et; R^1 = Ar, CO_2Et

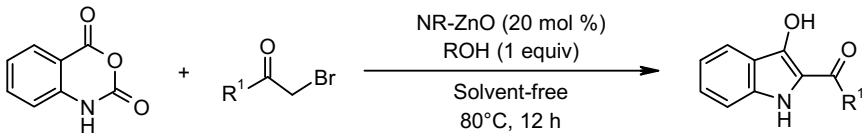
Indole derivatives have been synthesized in high yields by the reaction of isatoic anhydride and alkyl bromides in the presence of ZnO nanorods and an equimolar amount of an alcohol as a catalyst under solvent-free conditions at 80°C. The reaction work-up is simple, and the products can be easily separated from the reaction mixture. The ZnO nanocatalyst proved to be reusable and exhibited a significant yield enhancement in comparison with bulk ZnO and some other nanocatalysts.

Keywords: alkylbromide, indole, ZnO nanorods, green synthesis, solvent-free conditions.

Indole nucleus is important in medicinal chemistry and is considered as one of the so-called advantageous scaffolds.[1] Consequently, the synthesis of indoles derivatives with high selectivity have been the focus of active research in recent years.[2–8] Indole derivatives comprise an important class of compounds of interest in medicinal chemistry involving anticancer,[9] antioxidant,[9,10] antirheumatic, and antiHIV activities[11,12] and influence on the immune system.[13,14] Many indole derivatives are considered as effective scavengers of free radicals.[15] The avoidance of using hazardous organic solvents in organic syntheses is the most important goal in green chemistry.[16–18] Solvent-free organic reactions make syntheses simpler, save energy, and avoid environmental hazards due to toxic solvent waste. Also, in recent years, there has been an increased interest in catalysis by nanomaterials. These materials display better catalytic activity compared to the same substances consisting of bulk-size particles.[19,20] Nanocrystalline zinc oxide is a non-hygroscopic, cheap, and non-toxic material which has been used as a catalyst of various organic reactions.[21–26] Continuing our efforts directed towards the simple preparation of biologically active target molecules through one-pot reactions,[27–29] we present here the synthesis of some novel indoles *via* a one-pot reaction of isatoic anhydride, alcohols, and alkyl bromide in the presence of zinc oxide nanorods (NR-ZnO) at 80°C in a solvent-free reaction medium (Scheme 1).

Solvent-free stirred mixture of isatoic anhydride (**1**) and alkyl bromides **2a**–**f** in the presence of equimolar amount of alcohol and ZnO nanorods led to indole derivative **3a**–**f**

Scheme 1



1 **2a–f** **3a–f**

R = Me, Et;
2, **3 a** R^1 = 4-$MeOC_6H_4$, **b** R^1 = 4-BrC_6H_4, **c** R^1 = 4-MeC_6H_4,
d R^1 = 4-$O_2NOC_6H_4$, **e** R^1 = 3-$MeOC_6H_4$, **f** R^1 = CO_2Et

in 85–90% yields (Scheme 1). The alcohol used was methanol (for the synthesis of compounds **3a**,**c**,**e**,**f**) or ethanol (for the synthesis of compounds **3b**,**d**). The products were obtained as solids, and no impurities were observed by TLC. Structures of products were confirmed by IR, ^{1}H and ^{13}C NMR spectroscopy. For example, the ^{1}H NMR spectrum of compound **3a** exhibited singlets at 3.85, 11.23, and 11.74 ppm for OMe, NH, and OH protons, respectively. The ^{13}C NMR spectrum of compound **3a** featured the carbonyl group signal at 189.3 ppm and the signal of the methoxy group carbon at 55.6 ppm in agreement with the proposed structure.

The method that was used for the synthesis of NR-ZnO upon the interaction of NaOH and $Zn(OAc)_2$ in water has been already reported in literature.[29] The average crystal size for NR-ZnO is about 30 nm.[30,31] The nanoparticle morphology can be controlled using sodium dodecylsulfate (SDS). The morphology of the products was confirmed by SEM (Fig. 1). Formation of ZnO with different morphologies, like nanosheets and nanorods, involve the interaction between crystallization nucleus and SDS bilayers or micelles to control the growth rate in a certain direction, i.e., at various faces of the preformed nucleus.[32] It seems that the NR-ZnO morphology results under the influence of cylindrical inverse micelles in aqueous solutions of SDS.[33]

The X-ray diffraction (XRD) pattern of NR-ZnO is shown in Figure 2. All the characteristic peaks in the pattern correspond to the wurtzite structure of ZnO, which can be indexed on the basis of JCPDS file No. 36-1451. No other characteristic peaks of the impurities are observed, indicating the high purity of the NR-ZnO catalyst.

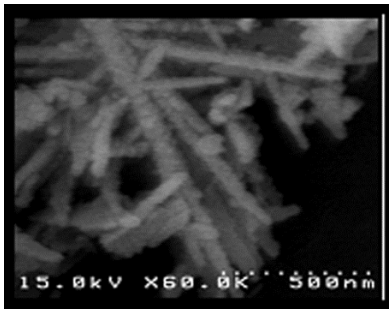


Figure 1. SEM image of NR-ZnO particles.

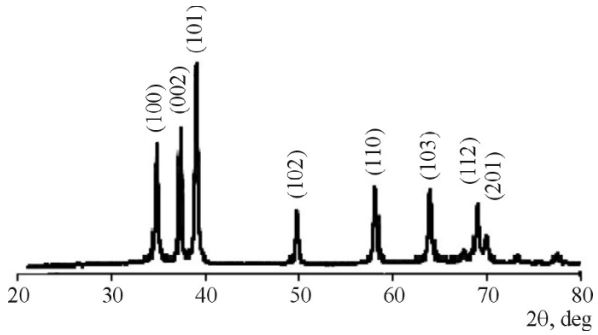


Figure 2. XRD pattern of NR-ZnO.

Table 1. Optimization of reaction conditions for the synthesis of compound **3a**

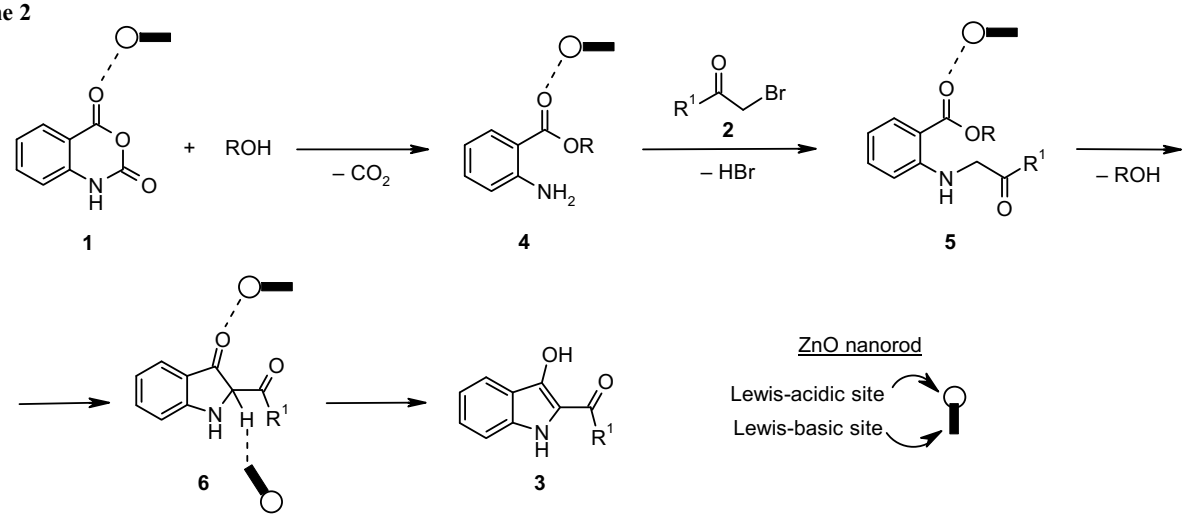
Entry	Catalyst	Catalyst load, mol %	Yield, %
1	None	None	0
2	CM-ZnO	10	65
3	CM-ZnO	15	70
4	NR-ZnO	10	75
5	NR-ZnO	15	78
6	**NR-ZnO**	**20**	**85**
7	NR-ZnO	25	80
8	NP–KF/clinoptilolite	10	55
9	NP–KF/clinoptilolite	15	67
10	Nanozeolite clinoptilolite	15	55
11	Nanozeolite clinoptilolite	20	55

In order to find the best reaction conditions, the reaction of isatoic anhydride (**1**), methanol, and 4-methoxyphenacyl bromide (**2a**) was selected as a model reaction (Scheme 1) and commercial zinc oxide (CM-ZnO) (CAS Number: 1314-13-2) was used as a catalyst. It was found to give 70% yield of product at 80°C under solvent-free condition (Table 1, entry 2). Confident by this outcome, further optimization was performed by NR-ZnO as catalyst.

The result of our optimization studies in catalyst charging are showed in Table 1. The reaction did not take place without any catalyst (entry 1), and the yield of the product was increased when NR-ZnO was used (entries 4–7) in comparison to the use of bulk ZnO or some two other nanocatalysts. The yield increased along with the catalyst load up to 20%, but further increase in the amount of catalyst led to the decrease of product yield. The best reaction temperature when using NR-ZnO was 80°C. The catalyst can be separated by filtration and reused at least six times without significant loss of activity. The reusability of the catalyst was checked for the synthesis of compound **3a**. The catalyst was separated after each run and washed thoroughly with ethyl acetate. Then it was dried at room temperature for 24 h and used for the next catalytic cycle.

Although we have not established the mechanism of the reaction between the isatoic anhydride (**1**) and alkyl bromides **2** in the presence of alcohol and NR-ZnO, a possible explanation is suggested in Scheme 2. NR-ZnO has Lewis-acidic sites (Zn^{2+}) and Lewis-basic sites (O^{2-}).[17,34] It is possible that the reaction includes the initial formation of anthranilic ester **4** from the alcoholysis of isatoic anhydride (**2**) on the surface of NR-ZnO and elimination of carbon dioxide. Then alkyl bromide **2** reacts with the amine group of intermediate **4** producing adduct **5**. Possibly, the generation of enol form of the intermediate **5** and the subsequent intramolecular attack on the ester carbonyl group in the presence of NR-ZnO eliminates alcohol and generates diketone **6** which finally produces 3-hydroxyindole **3** by enolization.[23]

Scheme 2



In summary, the procedure described here provides an acceptable one-pot method for the preparation of indole derivatives from the reaction of isatoic anhydride and alkyl bromides in the presence of alcohol and NR-ZnO. Moreover, easy work-up, solvent-free conditions, and reusability of catalyst make this method as an interesting alternative to other approaches.

Experimental

IR spectra were recorded on a Nicollet Magna 550-FT spectrometer in KBr. ^{1}H and ^{13}C NMR spectra were recorded on a Bruker FT-500 spectrometer (500 and 100 MHz, respectively) in $CDCl_3$, and TMS was used as internal standard. Electron ionization mass spectra were recorded on a Finnigan Mat TSQ-70 spectrometer operating at an ionization potential 70 eV. Elemental analyses were carried out with a Perkin-Elmer model 240-C apparatus. Melting points were measured on an Electrothermal 9100 apparatus. The morphology of nanorods of ZnO was established by utilizing a Holland Philips XL30 scanning electron microscope. X-ray diffraction analysis was performed at room temperature on a Holland Philips Xpert X-ray powder diffractometer with CuKα radiation (λ 1.5406 Å), over the 2θ collection range of 20–80°.[23] Average crystal size of the nanoparticles was calculated using the Scherrer formula: $D = 0.9\lambda/(\beta\cos\theta)$,[23] where D is the diameter of the nanoparticles and β is the full-width at half-maximum of the diffraction lines.[17] All chemicals were purchased from Fluka or Merck and were used without further purification.

Preparation of ZnO nanorods. Sodium hydroxide (0.44 g, 1.1 mmol) was dissolved in distilled water (75 ml) under vigorous stirring at room temperature. Afterwards, sodium dodecylsulfate (1.57 g, 5 mmol) and $Zn(OAc)_2 \cdot 2H_2O$ (0.6 g, 3 mmol) were added, and the solution (pH 14) was refluxed for 1.5 h at 80°C. The product was filtered off and washed with distilled water and ethanol (96%) several times.[30] Yield 89%.

Preparation of indoles 3a–f (General method). NR-ZnO (20 mol %) was added to a stirred mixture of isatoic anhydride (**1**) (0.32 g, 2 mmol) and methanol or ethanol (2 mmol). After 40 min, alkyl bromide **2a**–**f** (2 mmol) was added at 80°C. The reaction mixture was stirred for about 12 h. After completion of reaction (TLC monitoring), the viscous residue was purified by column chromatography on silica gel (Merck 230–400 mesh) using *n*-hexane–EtOAc, 7:1, as eluent.

(3-Hydroxy-1*H*-indol-2-yl)(4-methoxyphenyl)methanone (4a). Yield 0.46 g (87%), yellow powder, mp 152–154°C (mp 157–158°C[35]). IR spectrum, ν, cm^{-1}: 3345, 1728, 1725, 1696, 1478, 1225. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 3.85 (3H, s, CH_3O); 6.94 (2H, d, ^3J = 7.4, H Ar); 7.18 (1H, t, ^3J = 7.5, H Ar); 7.48 (1H, t, ^3J = 7.5, H Ar); 7.58 (1H, d, ^3J = 7.6, H Ar); 8.12 (2H, d, ^3J = 7.4, H Ar); 8.32 (1H, d, ^3J = 7.6, H Ar); 11.23 (1H, s, NH); 11.74 (1H, s, OH). ^{13}C NMR spectrum, δ, ppm: 55.6 (CH_3O); 97.3 (C); 113.2 (CH); 114.5 (2CH); 116.4 (CH); 117.6 (CH); 120.4 (C); 123.2 (CH); 129.7 (C); 132.4 (2CH); 135.4 (C); 159.5 (C–OH); 161.7 (C); 189.3 (C=O). Mass spectrum, *m/z* (I_{rel}, %): 267 [M]$^+$ (15), 135 (100), 132 (45), 31 (100). Found, %: C 71.83; H 4.82; N 5.14. $C_{16}H_{13}NO_3$. Calculated, %: C 71.90; H 4.90; N 5.24.

(4-Bromophenyl)(3-hydroxy-1*H*-indol-2-yl)methanone (4b). Yield 0.54 g (85%), yellow powder, mp 178–180°C. IR spectrum, ν, cm^{-1}: 3365, 1732, 1727, 1695, 1462, 1275. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 7.18 (1H, t, ^3J = 7.4, H Ar); 7.62 (1H, t, ^3J = 7.4, H Ar); 7.75 (1H, d, ^3J = 7.5, H Ar); 7.87 (2H, d, ^3J = 7.8, H Ar); 7.92 (2H, d, ^3J = 7.8, H Ar); 8.42 (1H, d, ^3J = 7.4, H Ar); 11.27 (1H, s, NH); 11.85 (1H, s, OH). ^{13}C NMR spectrum, δ, ppm: 97.5 (C); 113.5 (CH); 118.7 (CH); 119.5 (CH); 120.4 (C); 123.6 (CH); 129.4 (C); 130.4 (2 CH); 131.6 (2CH); 134.2 (C); 135.6 (C); 160.3 (C–OH); 188.6 (C=O). Mass spectrum, *m/z* (I_{rel}, %): 315 [M]$^+$ (6), 184 (100), 132 (65). Found, %: C 56.87; H 3.12; N 4.35. $C_{15}H_{10}BrNO_2$. Calculated, %: C 56.99; H 3.19; N 4.43.

(3-Hydroxy-1*H*-indol-2-yl)(4-methylphenyl)methanone (4c). Yield 0.43 g (85%), pale-yellow powder, mp 148–150°C (mp 154°C[35]). IR spectrum, ν, cm^{-1}: 3342, 1725, 1720, 1687, 1464, 1247. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 2.37 (3H, s, CH_3); 7.16 (1H, t, ^3J = 7.5, H Ar); 7.38 (2H, d, ^3J = 7.6, H Ar); 7.56 (1H, t, ^3J = 7.5, H Ar); 7.72 (1H, d, ^3J = 7.5, H Ar); 8.14 (2H, d, ^3J = 7.6, H Ar); 8.52 (1H, d, ^3J = 7.5, H Ar); 11.15 (1H, s, NH); 11.62 (1H, s, OH). ^{13}C NMR spectrum, δ, ppm: 21.3 (CH_3); 96.4 (C); 112.8 (CH); 118.2 (CH); 119.4 (CH); 120.5 (C); 123.4 (CH); 129.4 (2CH); 130.4 (2CH); 133.7 (C); 135.2 (C);

142.4 (C); 158.9 (C–OH); 188.6 (C=O). Mass spectrum, *m*/*z* (I_{rel}, %): 251 $[M]^+$ (10), 132 (85), 119 (100). Found, %: C 76.34; H 5.12; N 5.48. $C_{16}H_{13}NO_3$. Calculated, %: C 76.48; H 5.21; N 5.57.

(3-Hydroxy-1*H*-indol-2-yl)(4-nitrophenyl)methanone (4d). Yield 0.48 g (85%), yellow powder, mp 185–187°C. IR spectrum, ν, cm^{-1}: 3352, 1734, 1730, 1685, 1482, 1263. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 7.32 (1H, t, ^3J = 7.6, H Ar); 7.42 (2H, d, ^3J = 7.8, H Ar); 7.58 (1H, t, ^3J = 7.5, H Ar); 7.72 (1H, d, ^3J = 7.5, H Ar); 8.38 (2H, d, ^3J = 7.8, H Ar); 8.53 (1H, d, ^3J = 7.6, H Ar); 11.16 (1H, s, NH); 11.68 (1H, s, OH). ^{13}C NMR spectrum, δ, ppm: 89.6 (C); 112.8 (CH); 118.4 (CH); 119.2 (CH); 120.7 (C); 123.4 (CH); 124.7 (2CH); 131.6 (2CH); 135.6 (C); 138.9 (C); 149.3 (C); 158.7 (C–OH); 189.5 (C=O). Mass spectrum, *m*/*z* (I_{rel}, %): 282 $[M]^+$ (15), 150 (100), 132 (58). Found, %: C 63.92; H 3.63; N 9.98. $C_{15}H_{10}N_2O_4$. Calculated, %: C 63.83; H 3.57; N 9.93.

(3-Hydroxy-1*H*-indol-2-yl)(3-methoxyphenyl)methanone (4e). Yield 0.46 g (87%), yellow powder, mp 156–158°C. IR spectrum, ν, cm^{-1}: 3344, 1728, 1726, 1695, 1478, 1227. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 3.87 (3H, s, CH_3O), 6.97 (1H, d, ^3J = 7.3, H Ar); 7.22 (1H, t, ^3J = 7.5, H Ar); 7.38 (1H, t, ^3J = 7.4, H Ar); 7.52 (1H, s, H Ar); 7.62 (2H, t, ^3J = 7.4, H Ar); 7.75 (1H, d, ^3J = 7.5, H Ar); 8.52 (1H, d, ^3J = 7.4, H Ar); 11.25 (1H, s, NH); 11.63 (1H, s, OH). ^{13}C NMR spectrum, δ, ppm: 54.2 (CH_3O); 96.5 (C); 113.5 (CH); 118.2 (CH); 119.4 (CH); 119.7 (CH); 120.8 (C); 122.4 (CH); 123.7 (CH); 125.3 (CH); 130.2 (CH); 135.2 (C); 136.3 (C); 158.7 (C–OH); 159.6 (C); 188.7 (C=O). Mass spectrum, *m*/*z* (I_{rel}, %): 267 $[M]^+$ (15), 135 (100), 132 (45), 108 (65), 31 (100).

Ethyl 2-(3-hydroxy-1*H*-indol-2-yl)-2-oxoacetate (4f). Yield 0.42 g (90%), white powder, mp 123–125°C. IR spectrum, ν, cm^{-1}: 3352, 1748, 1725, 1587, 1432, 1259. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 1.32 (3H, t, ^3J = 7.4, CH_3); 4.25 (2H, q, ^3J = 7.4, CH_2O); 7.32 (1H, d, ^3J = 7.6, H Ar); 7.65 (1H, t, ^3J = 7.5, H Ar); 7.78 (1H, t, ^3J = 7.5, H Ar); 8.63 (1H, t, ^3J = 7.5, H Ar); 10.34 (1H, s, NH), 10.67 (1H, s, OH). ^{13}C NMR spectrum, δ, ppm: 13.4 (CH_3); 62.2 (CH_2O); 102.7 (C); 112.4 (CH); 116.8 (CH); 119.2 (CH); 122.3 (C); 123.7 (CH); 137.4 (C); 159.6 (C–OH); 163.6 (C=O); 178.6 (C=O). Mass spectrum, *m*/*z* (I_{rel}, %): 233 $[M]^+$ (20), 188 (68), 45 (100). Found, %: C 61.92; H 4.83; N 6.15. $C_{12}H_{11}NO_4$. Calculated, %: C 61.80; H 4.75; N 6.01

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