



## A Rhodolite/ZnO nanocomposite as an amphiphilic catalyst for the hydrogenation of 3-substituted indoles

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### Abstract

Researchers in the fields of engineering, materials science, contemporary chemistry, and physics have recently shown a great deal of interest in graphene and other two-dimensional sp<sup>2</sup>-hybridized carbon nanomaterials. Graphene oxide (GO) and reduced graphene oxide (RGO) are two examples of graphene-based materials that have recently garnered a lot of interest due to their diverse range of potential uses as sensors, supercapacitors, pollutant adsorbents, catalysts, and other similar technologies.

**Keywords:** Reactants, stereoselectivity, chemistry, stereoisomer, ZNO

### Introduction

Taking a page out of K.B. Sharpless's book, catalysis is what really gets chemistry moving forward. Industrial applications of catalysis, rational design, serendipitous discovery or combinational identification of new ligands, catalysts, new solid supports, organic, inorganic, amorphous or mesoporous silica phases and metal organic frameworks, etc., are clearly the focus of great achievements in applied chemistry.

A perfect catalyst would be very productive and have selectivity close to 100%. There are three main types of selectivity in catalysts: chemo selectivity, regioselectivity, and stereoselectivity. Chemo selectivity is the ability of the catalyst to choose specific reactants from complex mixtures; regioselectivity is the selection of specific sites on the reactants; and stereoselectivity is the preferred formation of a single stereoisomer.

In catalysis, the process is often hypothesis driven. A chemist would draw on their knowledge to brainstorm potential catalysts, which will subsequently undergo testing, study, and optimisation using tools like library and high throughput technologies. New ways of thinking about rational design and catalysis research are becoming more important. There is an immediate need to address sustainability concerns in research and development, in

addition to the traditional economic issues of cost, yield, selectivity, time, resistance to poisoning and deactivation, and product contamination by catalyst residues. The usage of harmful and environmentally harmful substances, as well as energy consumption, are their primary areas of concern. Reducing energy usage, waste, and the use of harmful chemicals is their main goal.

### Materials and Methods

El-Nassan, H.B. Amberlyst (2021) <sup>[21]</sup>, When it comes to organic synthesis, amberlyst 15 (A-15) is a popular heterogeneous catalyst. A-15 has been an effective green catalyst in several acid catalytic organic processes since its invention in 1962. The use of A-15 as a catalyst in heterocyclic synthesis has been the subject of several publications published in the last twenty years. There are a lot of benefits to using A-15, such as the fact that it is cheap, non-toxic, easy to handle, and separates quickly from the reaction mixture. The recyclability and ease of product separation are two other benefits. Sustainable chemistry makes extensive use of the latter. This study compiles the literature on Amberlyst 15 and its eco-friendly catalyst uses in heterocycle synthesis from 2010 to 2019. The review is organised according on the ring size, with an emphasis on the reactions catalysed by A-15 and their mechanisms.

Ruiz, Diego M., Pasquale, Gustavo A., Martínez, José J., and Romanelli, Gustavo P. (2022) [2] Synthetic methods that are ecologically benign, cost-effective, selective, and have a high atom economy are often used to create bioactive compounds with high functionalization levels and high value additions. Using nonpolluting substrates, recoverable solid catalysts, or hazardous organic solvents in accordance with these standards substantially increases these needs. Due to their high Bronsted acidity, outstanding oxidizing capability under moderate circumstances, and different reuse cycles without substantial loss of catalytic activity, heteropoly acids (HPAs) and their derivatives have garnered tremendous interest as recyclable solid catalysts in the last thirty years. To make a process involving HPAs more sustainable, however, additional activation mechanisms need to be explored. To reduce the energy requirements of organic synthesis, we detail recent developments in the use of microwaves, ultrasonics, mechanochemistry, and photochemistry to synthesize compounds with bioactive potential. Biomass recovery, selective oxidation, heterocycle synthesis, and construction reactions are among the transformations investigated.

Izquierdo, S., Durán-Valle, C.J., and Silvero, G. (2023) [3] One of the difficulties and aims of chemical research in academia and industry is the development of novel chemical synthesis techniques that turn industrial chemical processes into more sustainable ones. One of the best ways to improve process efficiency is to employ heterogeneous catalysts, such as activated carbons. This will help with the reaction rate, yield, and work-up ease. Using nitric or sulfuric acid to treat three commercially available, inexpensive carbons, we were able to create six novel activated carbons. Nine different activated carbons have been thoroughly characterised using various techniques such as elemental organic analysis, scanning electron microscopy, porosimetry, X-ray photoelectron spectroscopy, and determination of the point of zero charge. Two of these carbons have also been examined using transmission electron microscopy. The acetylation reaction of hydroxyl groups, one of the most flexible and extensively utilised chemical transformations in both laboratory and commercial settings, was employed to investigate the catalytic activity of these nine catalysts. Based on the findings, a more effective catalyst may be created by simply and inexpensively adding sulfuric acid to commercial xerogel. Isolation of the product in quantitative yield is a breeze, and the best catalyst allows for acetylation to be finished in 90 minutes at room temperature. With little degradation in catalytic activity, the system may be reused for five cycles.

Rubab, Laila, Ayesha Anum, Ali Irfan, Sajjad Ahmad, Sami Ullah, Aamal A. Al-Mutairi, and Magdi E. A. Zaki (2022) [4] Chemical engineers, medicinal chemists, chemists, and ecologists may all find a home in green chemistry, which aims to promote long-term sustainability via the development of environmentally friendly products and practices. Catalysis and other environmentally friendly synthesis conditions provide the backbone of green chemistry. Synthetic chemists can solve the issues with traditional synthesis, like long reaction times, harmful solvents and catalysts, and slow reaction rates, by applying green chemistry principles. These principles include creating environmentally friendly catalysts and solvents,

using microwave and ultrasonic radiation, and developing solvent-free, grinding, and chemo-mechanical approaches. The nitrogen-sulfur heterocycle class includes the preferred structural motif 1,2,4-thiadiazole, which has several uses in medicine and the pharmaceutical industry. This extensive review organizes various green solvents and catalysts, ideal green organic synthesis characteristics, and green synthetic approaches used to build various 1,2,4-thiadiazoles scaffolds. The approaches include solvent-free, metal-free conditions, ionic liquids, microwave irradiation, ultrasound, green solvents, and heterogeneous catalysis.

Here are a few instances of recent breakthroughs in heterogeneous catalysis that showcase elegance: In 2016, Kranthi Kumar Gangu et al. detailed a new way to synthesize pyran pyrazoles using an iron-doped heterogeneous catalyst in a one-pot multicomponent mixture of malononitrile, hydrazine hydrate, different aldehydes, and dimethyl acetylene dicarboxylate in ethanol as solvent, all carried out at 50 °C. With this process, you may get a yield capacity of up to 90% in a short reaction time, and it's easy to prepare and catalytically recycle.

### An amphiphilic catalyst for the production of 3-substituted indoles in water: Rgo/Zno nanocomposite Significance and synthetic methods of 3-substituted indoles

A wide variety of compounds with agrochemical, medicinal, and physiologically active properties have indole frameworks. Ages 32–35 Particularly intriguing are the 3-substituted indoles, which have a variety of biological functions including acting as an inhibitor of HIV-1 integrase and as an anticancer agent (Figure 1).

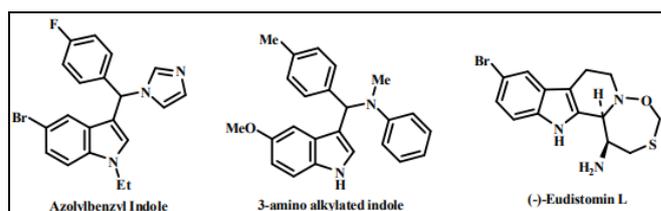
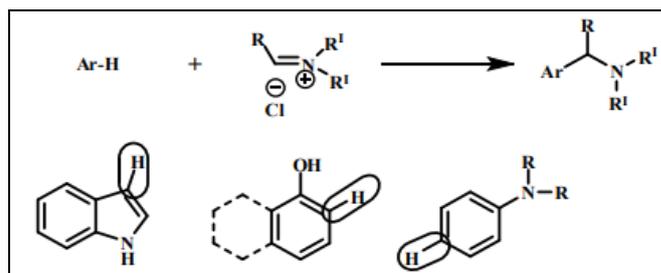


Fig 1: Trisubstituted indoles with biological activity

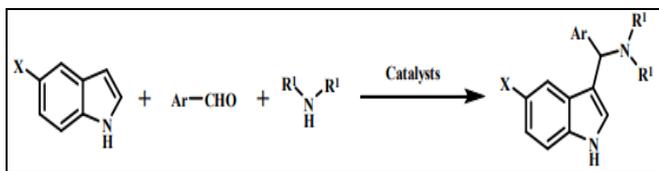
The amino alkylation of indoles, phenols, and N, N-dimethylaniline with aromatic aldehydes and iminium salts of a secondary aliphatic amine was investigated by Risch et al. in 1996 (Scheme 1).



Scheme 1: Amino alkylation of aromatic compounds with a high electron density

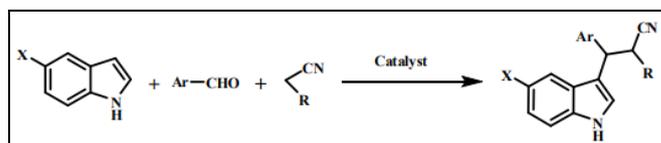
Recently, Kumar Three-amino alkylated indoles were synthesized in a single step by combining indoles with aromatic aldehydes and aliphatic/aromatic secondary

amines, as described by et. al. under homogeneous catalytic system such as either L-proline at room temperature or sodium dodecyl sulfate in water at 80 °C (Scheme 2). However, this methodology has not been well explored with aliphatic secondary amines, but various catalysts have employed in case of aromatic secondary amine i.e. N-methylaniline.



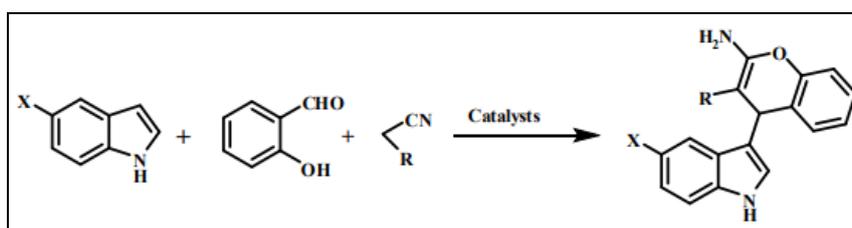
**Scheme 2:** 3-Amino alkylated indoles synthesized in a single step

A homogeneous catalyst, such as a Zn(salphen) complex, active methylene compounds, indoles, and benzaldehydes were used to produce 2-((1H-Indol-3-yl) (phenyl)methyl) Malo nitriles.,  $\text{KH}_2\text{PO}_4$ ,  $\text{Cu}(\text{OAc})_2$ , and TBAF (Scheme 3).



**Scheme 3:** 3-Substituted indole synthesis in a single reaction vessel

There are very few reports on 2-amino-4-(indol-3-yl)-4H-chromenes may be synthesized using catalysts in the Knoevenagel/Pinner/Friedel Crafts process involving indole, active methylene molecules, and 2-hydroxy benzaldehyde (Scheme 4). It was reported by Feng et al. (2011) [16] that 2-amino-4-(indol-3-yl)-4H-chromenes could be enantioselectively synthesized in one pot using an N, NII-dioxide Zn (II) complex. Recently, Singh *et al.* reported tetrabutylammonium fluoride (TBAF) as a homogeneous catalyst, and Das *et al.* studied the synthesis of functionalized 4H-chromenes in the presence of ZnO NPs as recyclable catalysts.

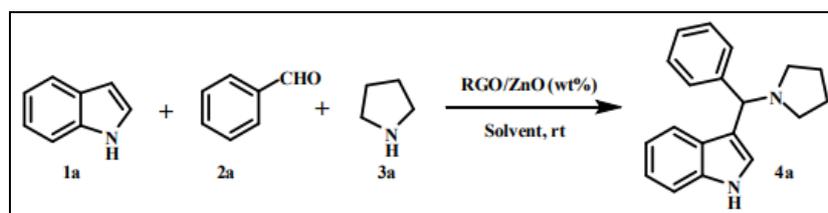


**Scheme 4:** Determination of 2-amino-4-(indol-3-yl)-4H-chromenes

## Results and Discussion

**RG0/ZnO nanocomposite catalyzed indole synthesis with three amino groups:** First, as indicated in Scheme 5, we studied the catalytic activity of RG0/ZnO in a model

reaction involving indole, benzaldehyde, and pyrrolidine to produce 3-amino alkylated indole (4a). The findings are shown in Table 1.

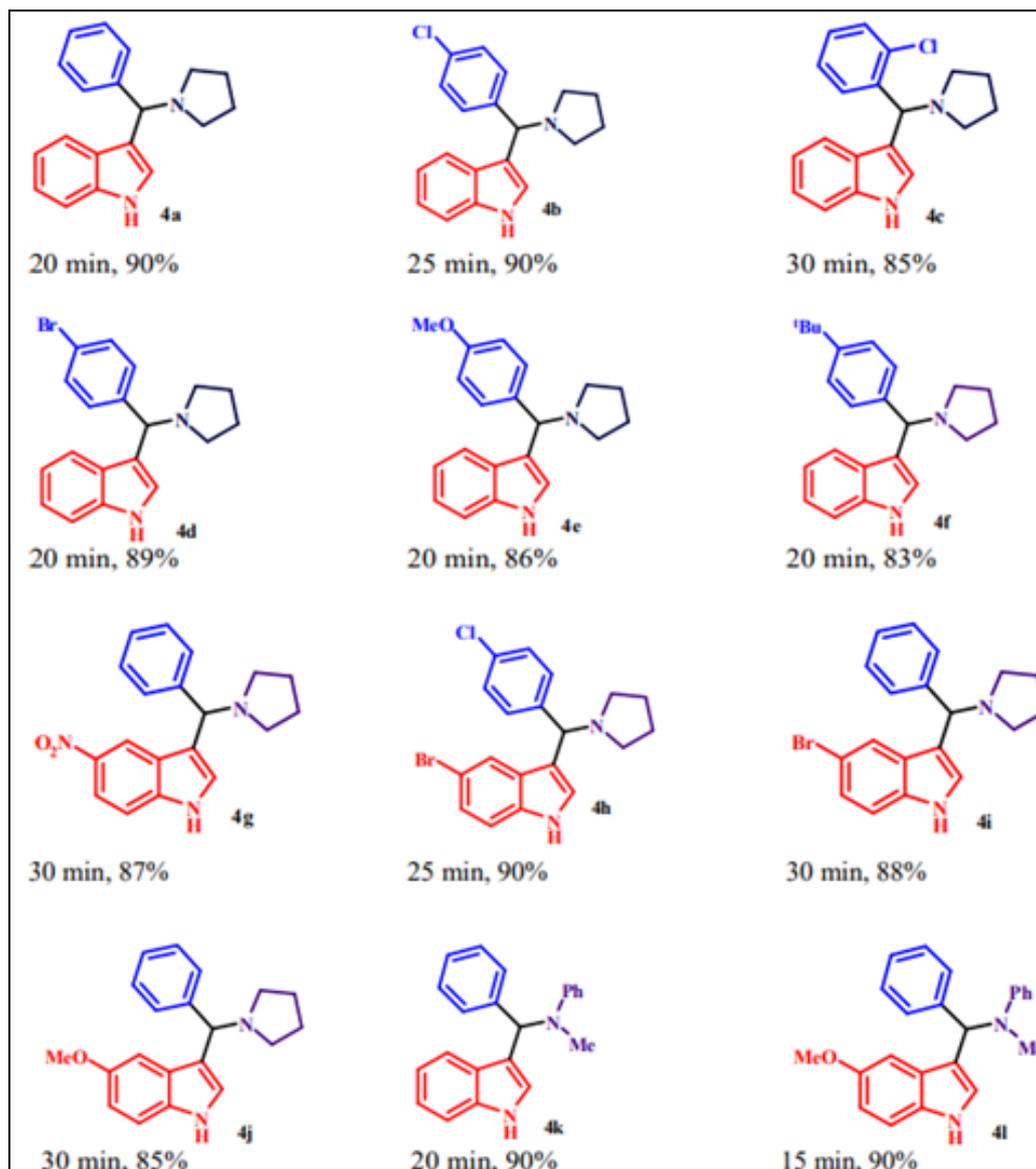


**Scheme 5:** Optimization of synthesized using an RG0/ZnO catalyst of (4a)

**Table 1:** Research on optimising the synthesis of (4a) using RG0/ZnO as a catalyst. <sup>A</sup>

Entry	Solvent	Catalyst	Time (min)	Yield of 4a (%) <sup>c</sup>
1	DMSO	RG0/ZnO	120	40
2	$\text{CH}_3\text{CN}$	RG0/ZnO	120	45
3	Acetone	RG0/ZnO	120	30
4	THF	RG0/ZnO	120	40
5	Water	RG0/ZnO	20	90
6	Water	GO	120	Trace
7	Water	ZnO NPs <sup>b</sup>	120	70
8	Water	No catalyst	240	No reaction

<sup>a</sup>Reaction conditions: The following ingredients were mixed at room temperature: 1 mole of indole, 1 mole of benzaldehyde, and 1 mole of pyrrolidine; 5 weight percent catalyst; and 1.5 millilitres of solvent. B Nano ZnO that is commercially accessible. C Yield that is isolated.

**Table 2:** 3-amino alkylated indoles synthesized using RGO/ZnO catalysis.

The 3-amino alkylated indoles were afforded by using a range of indole substrates, aromatic aldehydes, and secondary amines. This allowed us to explore the generality of the RGO/ZnO nano catalytic system. Table 2 provides a summary of the findings. Notably, all reactions were carried out without a hitch, and within 15-30 minutes of reaction time, 3-amino alkylated indoles (4a-4l) were obtained in high yields (83-92%). All synthesized compounds (4a-4l) were characterized from various techniques such as Methods such as  $^1\text{H}$  NMR,  $^{13}\text{C}$  NMR, FT-IR, mass analysis, and so on. An examination of the compound (4e) is described as below.

In  $^1\text{H}$  NMR spectrum, a characteristic proton of 3-amino alkylated indoles (4e) appeared as a singlet at  $\delta = 4.59$  ppm (Figure 2). A singlet at  $\delta = 3.76$  ppm corresponds to three protons of OMe group. Eight protons of pyrrolidine moiety appeared as multiplet of four protons at  $\delta = 4.59$  ppm and singlet of four protons at  $\delta = 1.79$  ppm. A broad singlet of one proton at  $\delta = 8.18$  ppm corresponds to NH proton of indole. The remaining nine aromatic protons appeared in the range of  $\delta = 7.82$  ppm to 6.82 ppm (Figure 2).

$^{13}\text{C}$  NMR spectrum of compound (4e) showed the presence

of characteristic carbon atom at  $\delta = 55.49$  ppm (Figure 3). Four carbon atoms correspond to pyrrolidine moiety appeared as two peaks at  $\delta = 53.94$  and 23.81 ppm. A carbon atom of OMe group appeared at  $\delta = 67.55$  ppm. Aromatic carbon attached to OMe group appeared at  $\delta = 158.49$  ppm and the remaining aromatic carbon atoms at  $\delta = 137.01, 136.44, 129.02, 126.79, 122.08, 120.08, 120.02, 119.56, 113.79$  and 111.30 ppm (Figure 3).

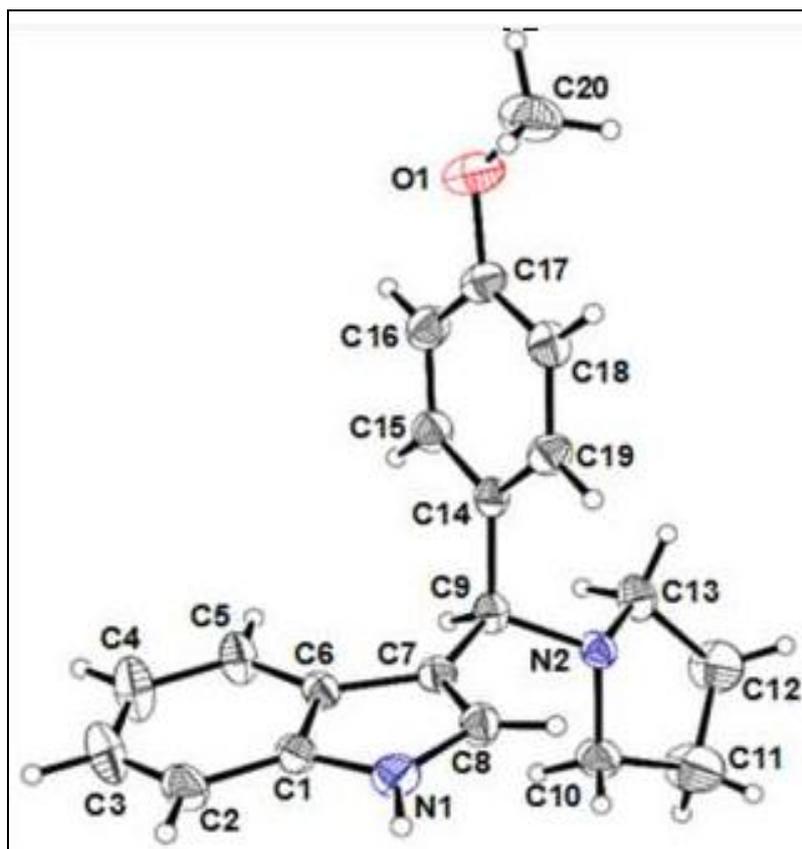
The structure further verified the presence of compound (4e) using a single X-ray crystallographic information as shown in Figure 4 and Table 3. 18 (CCDC 965703). The intensity data for compound (4e) was collected on an Oxford Xcalibur CCD diffractometer equipped with graphite monochromatic Mo- $K\alpha$  radiation ( $\lambda = 0.71073 \text{ \AA}$ ) at 293(2) K. A multi-scan correction was applied and the structure was processed by full-matrix least-squares refinement methods after being solved directly with SIR-92 on  $F^2$  using SHELXL97. The hydrogen atoms were placed into the calculated positions and included in the last cycles of the refinement. All calculations were done using Wingx software package.



**Table 3:** Structure refinement and crystal data for compound (4e)

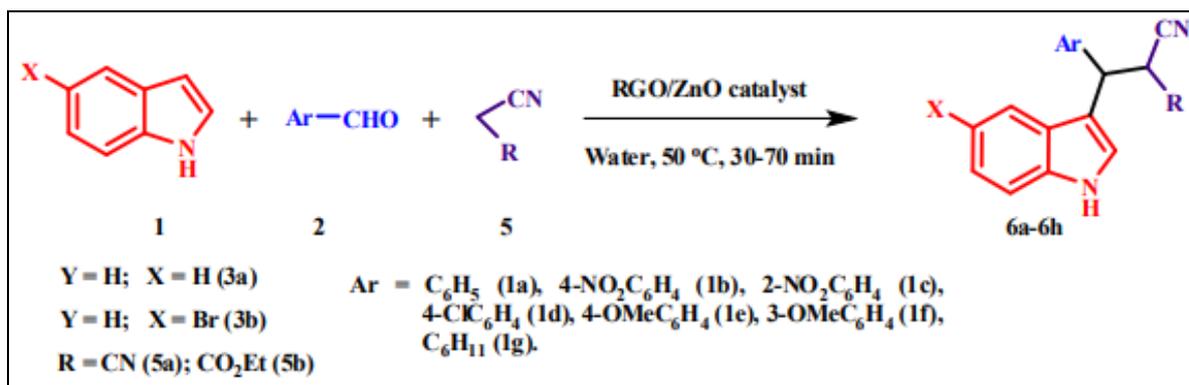
Empirical formula	C <sub>20</sub> H <sub>22</sub> N <sub>2</sub> O	
Formula weight	306.40	
Temperature	293(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P -1	
Unit cell dimensions	a = 10.3687(9) Å	$\alpha = 86.554(10)^\circ$
	b = 10.9616(13) Å	$\beta = 89.581(8)^\circ$
	c = 15.3932(17) Å	$\gamma = 74.889(9)^\circ$
Volume	1685.9(3) Å <sup>3</sup>	
Z	4	
Density (calculated)	1.207 Mg/m <sup>3</sup>	
Absorption coefficient	0.075 mm <sup>-1</sup>	
F(000)	656	
Crystal size	0.20 x 0.18 x 0.17 mm <sup>3</sup>	
Theta range for data collection	3.15 to 25.00°	
Index ranges	-12 ≤ h ≤ 12, -13 ≤ k ≤ 13, -18 ≤ l ≤ 18	
Reflections collected	14185	
Independent reflections	5872 [R(int) = 0.0558]	
Completeness to theta = 25.00°	98.9%	
Absorption correction	Multi-scan	
Max. and min. transmission	0.9874 and 0.9852	
Refinement method	Full-matrix least-squares on F <sup>2</sup>	
Data / restraints / parameters	5872 / 0 / 431	
Goodness-of-fit on F <sup>2</sup>	0.970	
Final R indices [I > 2σ(I)] <sup>a,b</sup>	R <sub>1</sub> = 0.0710, wR <sub>2</sub> = 0.1203	
R indices (all data)	R <sub>1</sub> = 0.1546, wR <sub>2</sub> = 0.1539	
Largest diff. peak and hole	0.208 and -0.204 e.Å <sup>-3</sup>	
Completeness to theta = 25.00°	98.9%	

$$^aR = \sum (|F_o| - |F_c|) / \sum |F_o| ; ^b wR = \{ \sum [w(F_o^2 - F_c^2)^2] / \sum [w(F_o^2)^2] \}^{1/2}$$

**Fig 4:** A three-amino alkylated indole molecule (4e)

Next, we further extended the RGO/ZnO catalytic system has several practical uses, one of which is the synthesis of 3-substituted indoles (6a-6h). This process involves reacting

indoles with active methylene compounds and different aromatic aldehydes via the Knoevenagel condensation and Michael addition reactions, as seen in the Scheme 6.

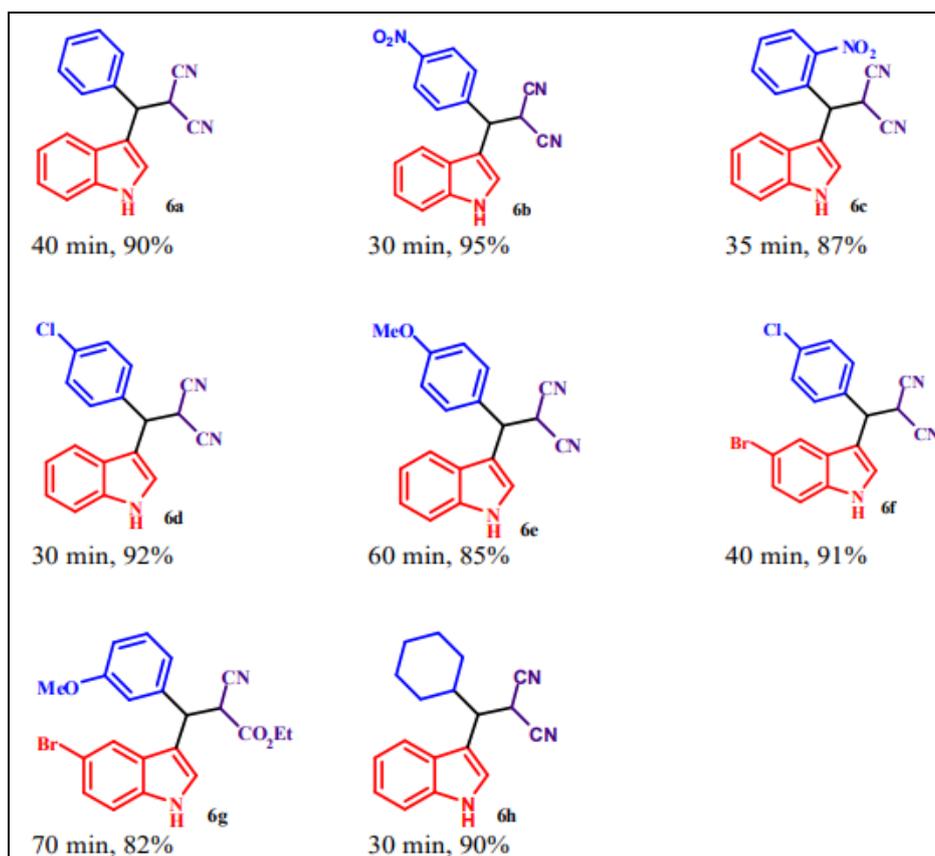


**Scheme 6:** The synthetic process of 3-substituted indoles catalyzed by RGO/ZnO (6a-6h)

3-Substituted indole (6a) was afforded under optimal circumstances by conducting a model reaction involving indole, benzaldehyde, and malononitrile in the presence of an RGO/ZnO catalyst. However, at 50 °C, the reaction ran smoothly, yielding product (6a) in 90% (Table 4). The reaction was tested with various aliphatic and aromatic aldehydes that had electron-withdrawing and electron-donating substituents at ortho, meta, and para positions. The corresponding 3-substituted indole derivatives (6a-6h) were

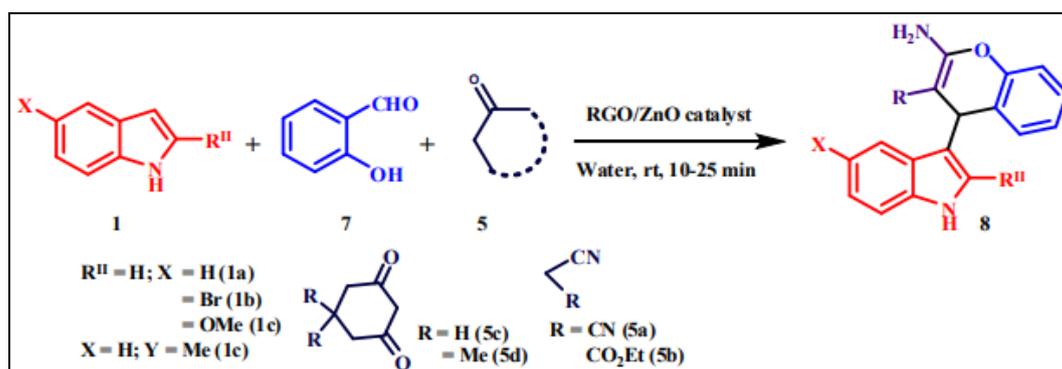
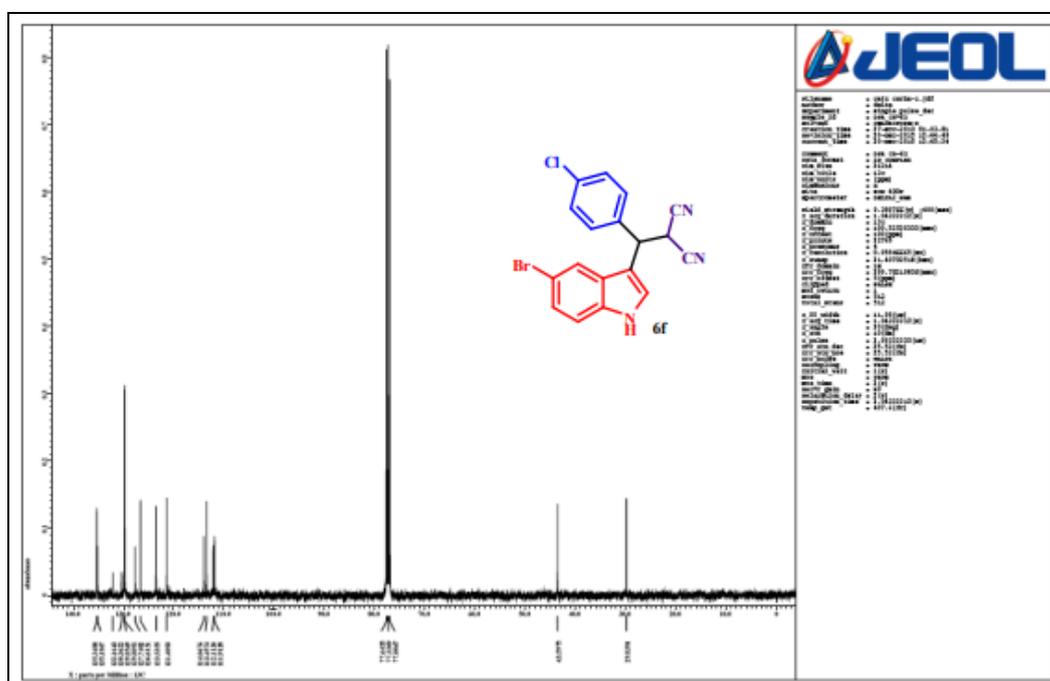
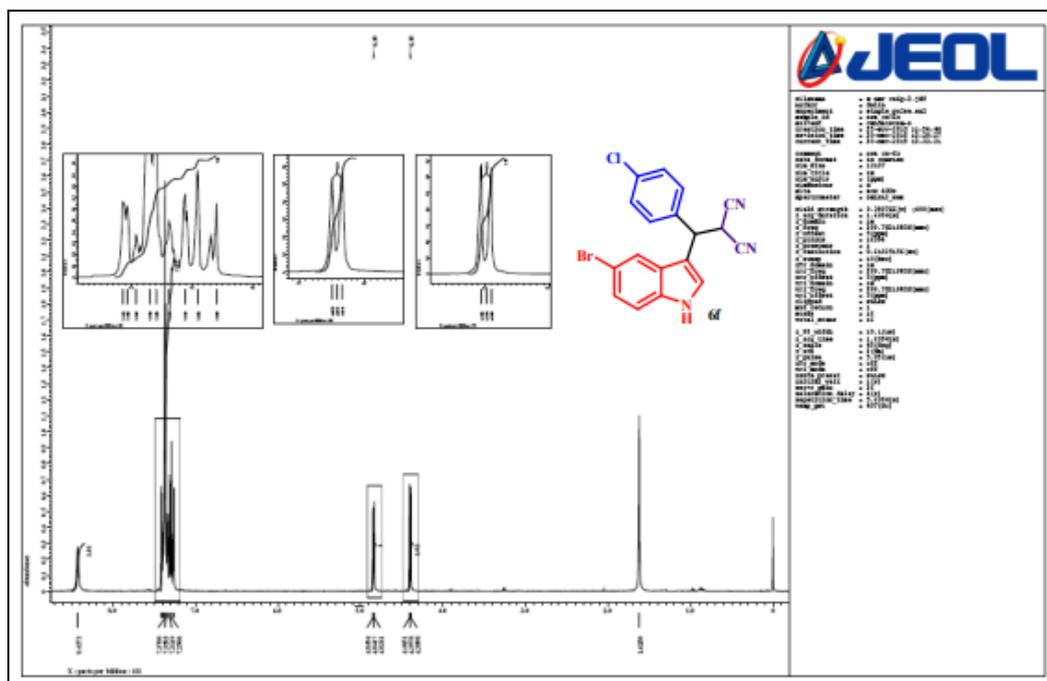
obtained using malononitrile/ethyl 2-cyano acetate, as shown in Table 4. All the reactions went off without a hitch, yielding high quality 3-substituted indoles (Table 4). In comparison to aromatic aldehydes with electron-withdrawing substituents like -NO<sub>2</sub> and chloro at ortho and para locations, those with electron-donating substituents like -OMe and -OH at meta and para positions exhibited lower reactivity (Table 4).

**Table 4:** Three-substituted indoles in water: a one-pot synthesis catalyzed by RGO/ZnO.



All synthesized compounds (6a-6h) analysis using mass spectrometry, <sup>1</sup>H nuclear magnetic resonance, and fluorescence transfer spectra analysis etc. The structural

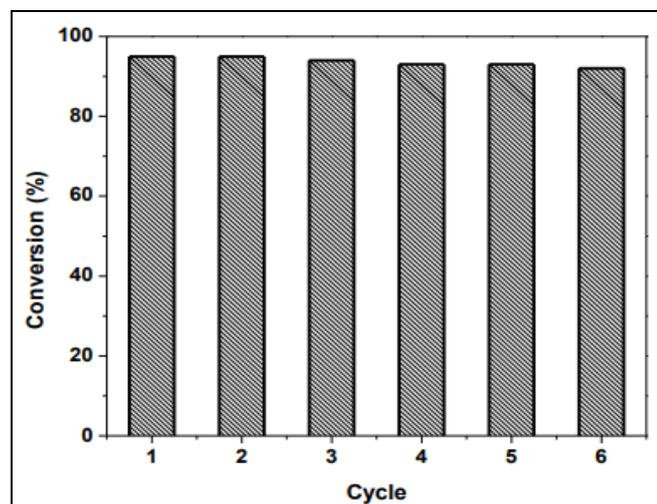
analysis of pale yellow solid compound (6f) is described as follows.





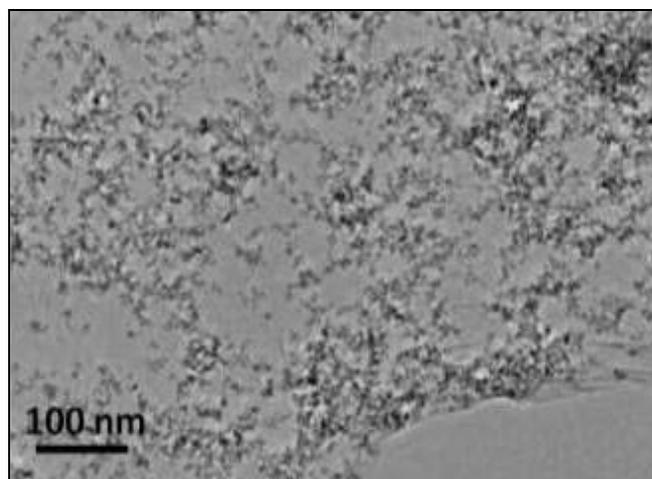


After that, we optimised a model reaction using indole, benzaldehyde, and pyrrolidine to produce 3-amino alkylated indole (4a) and investigated the recyclability of the RGO/ZnO catalyst. Centrifugation was used to separate the RGO/ZnO solid catalyst once the reaction was finished, and water was removed from the reaction mixture. EtOH was then added to the reaction mixture.



**Fig 10:** Analysed the RGO/ZnO catalyst's recycling potential for the synthesis of (4a).

The 3–4 washes with ethanol were used to remove catalyst the residual product and dried at 70 °C for the use of next cycles. The After six repetitions of the technique, the data showed that the catalytic activity remained unchanged as shown in Figure 10. The internal morphology of recovered RGO/ZnO catalysts was characterized by TEM technique. According to the findings, there was no change in their morphologies as shown in Figure 11.



**Fig 11:** Micrograph of repurposed RGO/ZnO nano composite

**Table 6:** Comparison using the previously documented procedures for the synthesis of (4a), (6a), and (8a) with an RGO/ZnO catalyst.

Entry	Product	Catalyst	Time (h)	Temp. (°C)	Yield (%)	[Ref.]	Recycle
1	(4a)	L-Proline	5.5	rt	87	[26a]	☐
2	(4a)	SDS	2	80	78	[26b]	☐
3	(4a)	RGO/ZnO	0.3	rt	90	Present	☐
4	(6a)	TBAF.3H <sub>2</sub> O	2	60	85	[32]	☐
5	(6a)	Cu-L salen complex	6	60	90	[28]	☐
6	(6a)	RGO/ZnO	0.6	50	90	Present	☐
7	(8a)	TBAF.3H <sub>2</sub> O	3	60	88	[32]	☐
8	(8a)	Zn (ClO <sub>4</sub> ).6H <sub>2</sub> O	26	rt	87	[33]	☐
9	(8a)	RGO/ZnO	0.25	rt	93	Present	☐

Next, we compared Results for the synthesis of 3-substituted indoles (4a), (6a), and (8a) using RGO/ZnO catalysts are summarised in Table 6. Table 6 shows that compared to the described approaches, the RGO/ZnO nano catalyst has better catalytic activity, uses moderate reaction conditions, and is recyclable.

### Conclusion

Using water as a green solvent is only one of the many benefits of an RGO/ZnO catalytic system, which also boasts excellent catalytic efficiency, rapid reaction times, high yields, and adaptability to a variety of indole-based chemical processes. With little degradation in catalytic activity, the RGO/ZnO catalyst may be recycled at least six times from reaction mixtures. In addition, we have created a sustainable process for the green synthesis of several 3-substituted indoles, which have a reduced E-factor and a greater atom economy, making them more environmentally friendly.

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