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Eco-friendly multicomponent synthesis of spirooxindole catalyzed by *Acacia concinna* pod extract

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Abstract

This study describes an eco-friendly multicomponent protocol for the synthesis of spirooxindoles using hydrazine/phenylhydrazine in the presence of a biodegradable and low-cost natural catalyst derived from *Acacia concinna* pod extract. The reaction proceeds efficiently at room temperature within 10 minutes, affording spiro[indoline-3,4'-pyrano[2,3-c]pyrazole] derivatives in excellent yields of up to 95% without the need for chromatographic purification. Optimization studies revealed that 30 mol% of the extract was sufficient to achieve high catalytic efficiency. The developed method offers several advantages, including mild reaction conditions, operational simplicity, rapid reaction time, and environmentally benign synthesis. Compared with previously reported catalysts, the present protocol provides an efficient and sustainable approach for the synthesis of biologically important spirooxindole derivatives and may be useful for further substrate diversification and green synthetic applications.

Keywords: Spirooxindole derivatives, Multicomponent reaction, Natural catalyst, One-pot synthesis, *Acacia concinna* pod extract

Introduction

The synthesis of spirooxindole derivatives has attracted considerable attention because these heterocyclic frameworks are widely found in biologically active natural products and pharmaceutical compounds ^[1]. The oxindole core is widely recognized for its diverse pharmacological properties, including anticancer ^[2], antimicrobial ^[3], anti-inflammatory activities ^[4-5], anti-viral ^[6], anti-oxidant ^[7], anti-tumor ^[8], and anti-malarial effects ^[9-10]. In particular, the spirooxindole framework, with its quaternary spiro carbon center, provides a rigid 3D structure that enhances biological interactions and selectivity.

Spirooxindole derivatives have attracted considerable attention because of their diverse biological activities and broad applications in medicinal chemistry. Recent developments in green synthetic methodologies, particularly multicomponent reactions and recyclable catalytic systems, have significantly improved the sustainable construction of these heterocyclic frameworks ^[11]. Over the past five years, several efficient strategies for the synthesis of spirooxindole scaffolds have been developed. For example, spirooxindole γ -butyrolactones have been synthesized through a metal-free NHC-catalyzed cascade reaction involving 3-hydroxyoxindoles and α -bromo- α,β -unsaturated aldehydes with notable regio- and diastereoselectivity. Similarly, a three-component [3+2] cycloaddition approach using cyclic amines, aryl aldehydes, and olefinic oxindoles has enabled the efficient synthesis of rigid spirooxindole-pyrrolidine frameworks. Cycloaddition methods like [3+2], [4+1], and [4+2] effectively yield five- and six-membered heterocycle-fused spirooxindoles with commendable diastereo- and enantioselectivity ^[12-17]. Microwave irradiation has been used to promote the reaction of isatin, α -amino acids, and 1,4-dihydro-1,4-epoxynaphthalene, resulting in oxygenbridged spirooxindoles. This method is notable for its short reaction time and high efficiency, making it suitable for scale-up and further synthetic transformations ^[18]. Ammonium acetate facilitates a simple, rapid, and one-pot synthesis of spirooxindole derivatives through Knoevenagel condensation and Michael addition. This method is environmentally friendly, avoiding toxic chemicals and energy-intensive processes ^[19].

An efficient and facile one-pot, five-component reaction for the synthesis of 2,6-diamino-1-alkyl-2-oxospiro[indoline-3,4'-thiopyran]-3,5-dicarbonitrile derivatives was reported by a reaction between primary amines, carbon disulfide, malononitrile, and isatin derivatives [20]. Rare-earth metal salts, particularly $\text{Sc}(\text{OTf})_3$, enable the synthesis of spirocyclopropyl oxindoles with high diastereoselectivity. The metal's coordinating capabilities and oxophilicity are crucial for the selectivity observed in these reactions [21]. Transition metals like palladium are extensively used for spirooxindole synthesis. Palladium-catalyzed reactions involve mechanisms such as carbocyclization and C-H functionalization, which allow for diastereoselective synthesis of spirooxindoles, emphasizing their biological importance and classifying the reactions into intramolecular and intermolecular types [22]. A water-mediated synthetic route was developed for the preparation of spirooxindole derivatives, yielding a diverse range of compounds with promising antifungal activity. The synthesized molecules exhibited significant effectiveness against several fungal pathogens, including *Cercospora arachidicola*, *Phylospora piricola*, *Rhizoctonia cerealis*, and *Fusarium moniliforme* [23]. In recent years, green chemistry approaches have emerged as sustainable alternatives for the synthesis of heterocyclic compounds. The use of natural catalysts and biocatalytic systems derived from plant materials, fruit juices, enzymes, *Zingiber zerumbet*, and organic acids such as citric acid has attracted increasing interest because of their eco-friendly nature, low cost, biodegradability, and operational simplicity [24-27]. Plant-based catalytic and bioreduction systems have been extensively explored as greener alternatives for organic transformations and asymmetric synthesis under mild reaction conditions [28-29]. Spirooxindole motifs are widely found in numerous natural products and biologically active synthetic compounds. The four-component condensation of isatin, malononitrile, ethyl acetoacetate, and hydrazine is a useful method for producing derivatives of spiro[indoline-3,4'-pyrano[2,3-c]pyrazole]. Its substrate flexibility, bond economy, and convenience of usage are all achieved in a single one-pot process [30-33]. Among these, multicomponent reactions (MCRs) have gained considerable attention due to their operational simplicity, atom economy, and ability to generate structural diversity in a single step. However, many of the reported methods suffer from drawbacks such as the use of toxic solvents, expensive catalysts, prolonged reaction times, and moderate yields.

Despite these advantages, the development of efficient green protocols for the synthesis of spirooxindole derivatives remains a challenging task. Therefore, in the present work, we report a novel green approach for the synthesis of substituted spirooxindoles using an extract of *Acacia concinna* pods. This method utilizes a biodegradable catalyst to afford the desired products in excellent yields (up to 95%).

Materials and Methods

Experimental

General procedure for the preparation of green catalyst:

Take a fine *Acacia concinna* pods powder (30 g) in 100 mL water was stirred in a 250 mL conical flask at room temperature for 40 min. The solid material was filtered and the aqueous extract was collected. The prepared extract has

concentration 30% w/v.

General procedure for the synthesis of spirooxindole derivatives (5a-b)

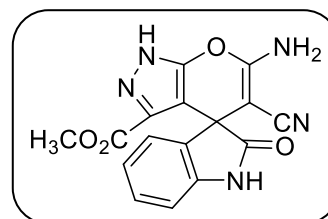
Using extract of *Acacia concinna* pods (20 mL), a mixture of isatin 1 (1 mmol), malononitrile 2 (1 mmol), dimethyl acetylenedicarboxylate 3a (1 mmol) and hydrazine/phenylhydrazine (4a-b) (1 mmol) was stirred at room temperature for 10 min or until the completion of reaction as indicated by TLC. After completion of the reaction, there is no column chromatography for purification.

General procedure for the synthesis of spirooxindole derivatives (5c-d)

Using extract of *Acacia concinna* pods (20 mL), a mixture of isatin 1 (1 mmol), malononitrile 2 (1 mmol), ethyl acetoacetate 3b (1 mmol) and hydrazine/phenylhydrazine (4a-b) (1 mmol) was stirred at room temperature for 10 min or until the completion of reaction as indicated by TLC. After completion of the reaction, there is no column chromatography for purification.

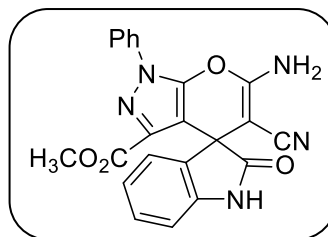
Spectral Data

Methyl 6'-amino-5'-cyano-2-oxo-1'H-spiro[indoline-3,4'-pyrano[2,3-c]pyrazole]-3'-carboxylate (5a).



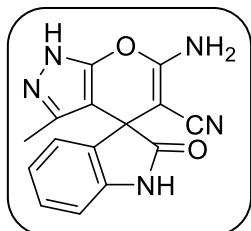
Pale orange-brown solid; Yield 95%; mp 270 °C; IR(KBr): 3328, 3127, 2175, 1699, 1579, 1309, 1200, 867; ^1H NMR (500 MHz, DMSO) δ 13.97 (s, 1H), 10.57 (s, 1H), 7.29 (s, 2H), 7.20 (td, J = 7.4, 1.4 Hz, 1H), 6.95 (d, J = 6.6 Hz, 1H), 6.91 (d, J = 7.4 Hz, 1H), 6.88 (d, J = 7.8 Hz, 1H), 3.45 (s, 3H); ^{13}C NMR (126 MHz, DMSO) δ 177.42, 161.01, 157.65, 155.96, 142.10, 133.79, 128.49, 123.63, 122.00, 117.97, 109.13, 100.69, 56.62, 51.49, 47.36.

Methyl 6'-amino-5'-cyano-2-oxo-1'-phenyl-1'H-spiro[indoline-3,4'-pyrano[2,3-c]pyrazole]-3'-carboxylate (5b).

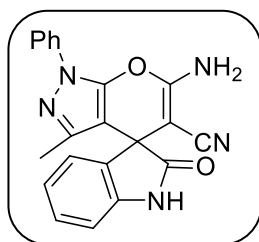


Yellowish-orange solid; yield 95%; mp >300 °C; IR(KBr): 3401, 3210, 2195, 1714, 1579, 1310, 1199, 742; ^1H NMR (500 MHz, DMSO) δ 13.97 (s, 1H), 10.58 (s, 1H), 7.29 (s, 2H), 7.20 (td, J = 7.5, 1.4 Hz, 1H), 6.96 (d, J = 7.2 Hz, 1H), 6.92 - 6.88(m, 2H), 3.45 (s, 3H); ^{13}C NMR (126 MHz, DMSO) δ 177.44, 161.03, 157.66, 155.98, 142.11, 133.81, 128.51, 123.64, 122.02, 117.98, 109.15, 100.70, 56.64, 51.51, 47.38.

6'-amino-3'-methyl-2-oxo-1'H-spiro[indoline-3,4'-pyrano[2,3-c]pyrazole]-5'-carbonitrile (5c).



Light brownish-orange solid; Yield 95%; mp 210 °C; IR(KBr): 3336, 3134, 2355, 1698, 1587, 1314, 1050; ¹H NMR (500 MHz, DMSO) δ 12.27 (s, 1H), 10.58 (s, 1H), 7.24 (td, J = 7.6, 1.5 Hz, 1H), 7.20 (s, 2H), 7.03 (m, 1H), 6.99 (td, J = 7.4, 1.0 Hz, 1H), 6.91 (d, J = 7.8 Hz, 1H), 1.54 (s, 3H); ¹³C NMR (126 MHz, DMSO) δ 177.92, 162.39, 155.17, 141.43, 134.63, 132.60, 128.80, 124.44, 122.40, 118.63, 109.57, 95.31, 55.12, 47.21, 8.87. 6'-amino-3'-methyl-2-oxo-1'-phenyl-1'H-spiro[indoline-3,4'-pyrano[2,3-c]pyrazole]-5'carbonitrile (5d).

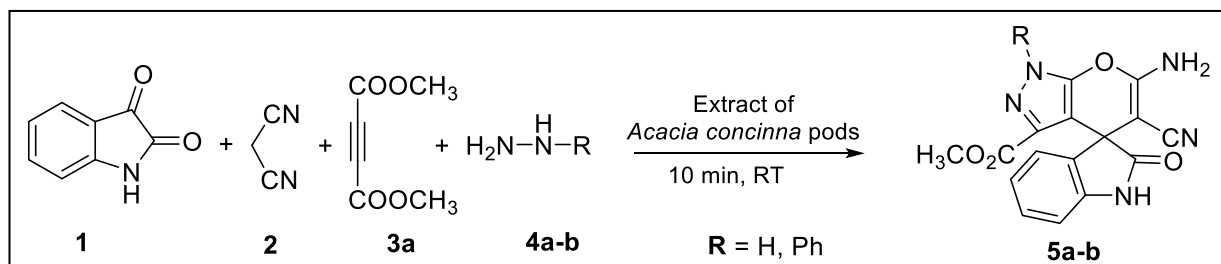


Light brownish-orange solid; Yield 95%; mp 250 °C; IR(KBr): 3218, 3114, 1698, 1593, 1387, 1217, 749; ¹H

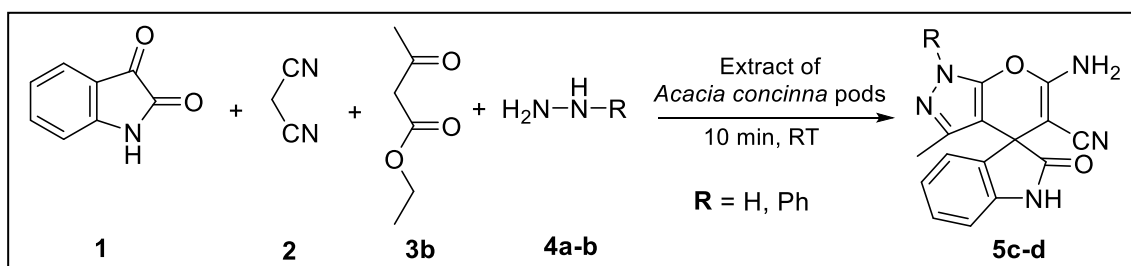
NMR (500 MHz, DMSO) δ 10.73 (s, 1H), 7.79 - 7.77 (m, 2H), 7.55 - 7.53 (m, 2H), 7.52 - 7.50 (m, 2H), 7.37 - 7.34 (m, 1H), 7.30 - 7.27 (m, 1H), 7.18 (d, J = 7.2 Hz, 1H), 7.04 - 7.01 (m, 1H), 6.95 (d, J = 7.8 Hz, 1H), 1.54 (s, 3H); ¹³C NMR (126 MHz, DMSO) δ 177.38, 160.91, 144.82, 143.83, 141.49, 137.13, 132.02, 129.34, 129.16, 128.83, 126.45, 124.78, 122.52, 120.50, 120.02, 117.81, 109.73, 96.24, 56.07, 47.67, 11.57.

Results and Discussions

In the first part of the study, different conditions were tested to create spirooxindole compounds. Experiments showed that reaction mixing isatin, malononitrile, DMAD or EAA, and hydrazine/phenylhydrazine together with a natural catalyst made from *Acacia concinna* pods could produce these compounds (5a-d) in good amounts and at a low cost. Firstly, the testing of different conditions before the explains, the best results were achieved by running the reaction for 10 minutes at room temperature using 20 mL of *Acacia concinna* pods extract. In this optimized process, hydrazine/phenylhydrazine (4a-4b) were mixed in equal amounts with isatin, malononitrile, and either dimethyl acetylenedicarboxylate (DMAD, 3a) or ethyl acetoacetate (EAA, 3b) in extract at room temperature. The reaction progress was monitored by TLC, and after the reaction finished, the solid products were filtered, washed with water, and obtained as pure substituted spirooxindoles (5a-d), as shown in Scheme 1, Scheme 2 and Table 1.



Scheme 1: Synthesis of spirooxindoles (5a-b) from DMAD (3a).



Scheme 2: Synthesis of spirooxindoles (5c-d) from EAA (3b).

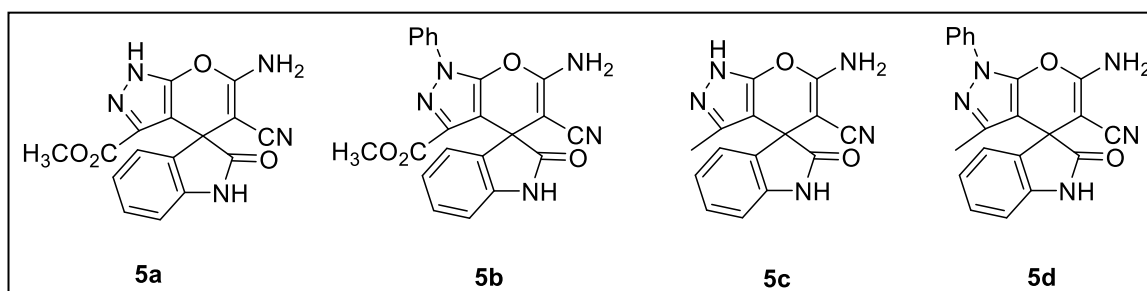


Fig 1: Structure of Spirooxindole derivatives.

The spiro-compounds (5a-d) were first produced in a 95% yield at room temperature by reacting isatin 1, malononitrile

2, and hydrazine/phenylhydrazine with DMAD (3a) or EAA (3b) in the presence of *Acacia concinna* pod extract for 10

min. The reaction can be catalyzed with just 30 mol% of extract (table 2, entry 5); larger mol% of extract (table 2, entry 6) did not significantly alter the yield of the products.

However, even after five hours, the reaction produced no product in the absence of *Acacia concinna* pods extract.

Tables 1: Synthesis of spirooxindole in hydrazine derivatives^a (5a-d).

Entry	R	Reagents (3)	Products	Time (min)	Isolated yield (%)	MP (°C)
1	H	3a	5a	10	95	270
2	Ph	3a	5b	10	95	>300
3	H	3b	5c	10	95	210
4	Ph	3b	5d	10	95	250

a. Reaction Conditions: Isatin (1 mmol), malononitrile (1 mmol), dimethyl acetylenedicarboxylate or ethyl acetoacetate (1 mmol) and hydrazine derivatives (1 mmol) were stirred in a 20 mL *Acacia concinna* pods extract at room temperature.

Table 2: Optimization of catalyst amount^a

Entry	Catalyst amount (mol %)	Reaction time (min)	Isolated yield (%)
1	No Catalyst	300	NR
2	5	50	73
3	10	35	79
4	20	20	88
5	30	10	95
6	40	10	95

a. Reaction Conditions: Isatin (1 mmol), malononitrile (1 mmol), dimethyl acetylenedicarboxylate or ethyl acetoacetate (1 mmol) and hydrazine derivatives (1 mmol) were stirred in a 20 mL *Acacia concinna* pods extract at room temperature.

Table 3: Comparison of the efficiency of various catalysts in the synthesis of 5a-d^a

Entry	Catalyst and Conditions	Reaction time	Isolated yield (%)	Ref.
1	Citric acid, H ₂ O/EtOH (2:1), 80 °C	10 min	81	[26]
2	Cu/ZnO-graphene oxide nanocomposite, 80W	20 min	93	[4]
3	Zn(OAc) ₂ ·2H ₂ O (10% mol), Toluene, Reflux	2-4.5 h	93	[16]
4	Electrolysis, 48mA, EtOH, NaBr, 30 °C	55 min	85	[30]
5	<i>Acacia concinna</i> pods extract, RT	10 min	95	This work

a. Reaction Conditions: Isatin (1 mmol), malononitrile (1 mmol), dimethyl acetylenedicarboxylate or ethyl acetoacetate (1 mmol) and hydrazine derivatives (1 mmol) were stirred in a 20 mL *Acacia concinna* pods extract at room temperature.

The successful synthesis of spirooxindole derivatives (5a-d) using hydrazine derivatives under mild conditions. All reactions proceeded efficiently at room temperature within 10 minutes, yielding products with a consistent 95% isolated yield. The presence of hydrazine/phenylhydrazine and reagents (DMAD and EAA) did not affect the high yield or purity, confirming the broad applicability of the method. Focuses on optimizing the catalyst amount, revealing that 30 mol% of *Acacia concinna* pod extract is optimal, delivering the highest yield (95%) in the shortest time (table 2, entry 5). Lower catalyst loadings resulted in longer reaction times and reduced yields, indicating the importance of sufficient catalyst concentration for reaction efficiency. Compares the *Acacia concinna* pod extract catalyst with other reported catalysts (table 3, entry 6), highlighting its superior performance in terms of reaction time and yield under ambient conditions. Unlike other methods requiring elevated temperatures, longer durations, or toxic solvents, this green catalyst achieves excellent yields rapidly and eco-friendly, underscoring its potential as a sustainable alternative in spirooxindole synthesis. Additionally, this catalyst is eco-friendly, non-toxic, cheap, and easy to use.

Conclusion

The present study demonstrates an efficient and eco-friendly multicomponent strategy for the synthesis of spirooxindole derivatives using *Acacia concinna* pod extract as a biodegradable natural catalyst. The protocol affords excellent yields of up to 95% under mild reaction conditions

within short reaction times and eliminates the need for chromatographic purification. The use of a low-cost, non-toxic, and sustainable catalyst highlights the practical value of this method in green synthetic chemistry. Compared with previously reported catalytic systems, the developed approach offers operational simplicity, rapid synthesis, and environmentally benign reaction conditions. Furthermore, the methodology provides a promising foundation for future investigations involving substrate scope expansion, asymmetric synthesis, flow chemistry applications, and biological evaluation of newly synthesized derivatives.

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