

## Synthesis of spirooxindole derivatives catalyzed by Fe (III)@graphitic carbon nitride nanocomposite via one-pot multi-component reaction

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### ABSTRACT

Fe (III) supported graphitic carbon nitride nanocomposite was synthesized by impregnation of FeCl<sub>3</sub> with g- C<sub>3</sub>N<sub>4</sub> (Fe (III) @ g-C<sub>3</sub>N<sub>4</sub>). Then, the synthesis of spirooxindole derivatives was carried out in the presence of Fe (III) @ graphitic carbon nitride nanocomposite via the multi-component reaction of malononitrile, isatins, and 1,3-dicarbonyl compounds in green media (ethanol/water (1:1)) under reflux conditions and the corresponding products were prepared in good to excellent yields (70-98 %). Also, the heterogeneous nanocatalyst was characterized by different techniques including the transmission electron microscopy (TEM), Fourier transform infrared spectroscopy (FT-IR), thermal gravimetric analysis (TGA), atomic absorption spectroscopy (AAS) and X-ray diffraction (XRD). Furthermore, the nanocomposite is recoverable and reusable several times without a negligible decrease in its catalytic performance.

**Keywords:** Graphitic carbon-nitride, Multi-component reaction, Spirooxindole derivatives, Nanocomposite.

### 1. Introduction

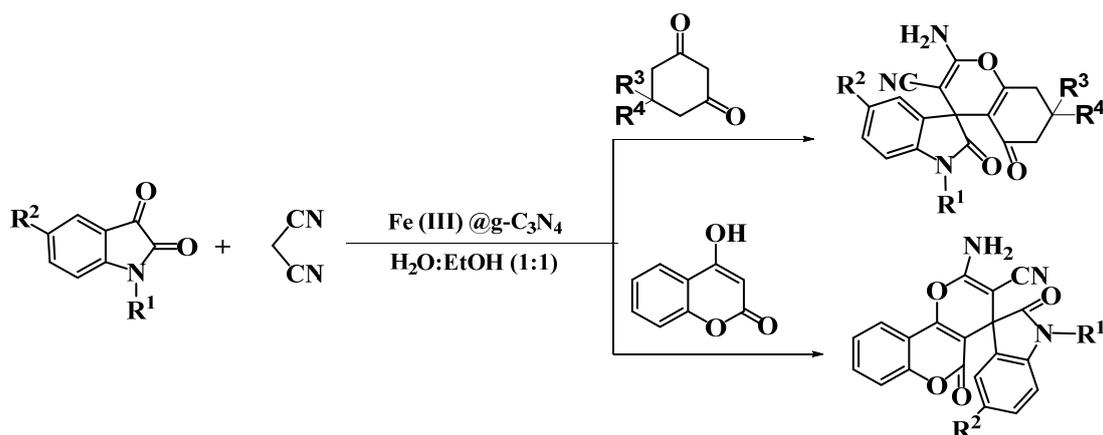
Considering the green chemistry point of view, green and benign solvents like water are the appropriate replacement for hazardous organic solvents polluting the environment [1-4]. Nanocomposites are also other aspects of green chemistry. Nanocomposites which are developed by the protocols in which multicomponent reactions were carried out in environmentally media with excellent yields might be one exciting and desirable direction [5]. These compounds are made from the matrix and layered. According to their matrix materials, nanocomposites can be classified into three categories including ceramic, polymer and metal matrix nanocomposites [6]. Nanocomposites are also synthesized by three methods which are “solution blending, in situ polymerization and the molten state [7]. In this context, graphitic carbon nitride as a 2-D graphene-like nanosheet is widely used as a special support for the synthesis of nanocomposites with multiple applications because of its extremely thermal and chemical stability. Initially, graphitic carbon nitride showed excellent catalytic activity toward alcohols,

amines and alkenes oxidation by oxygen under visible light in the absence of metal derivatives [8-10]. It also exhibits great potential in optical and electronic fields and the material science [11,12]. Furthermore, metal, metal oxide and organic compounds could be supported into the numerous anchoring sites on the surface of graphitic carbon nitride certainly promoting the catalytic activity of these nanocomposites [11,13-20].

Heterocyclic compounds synthesis is still a challenge in the field of organic synthesis. These types of molecules show desirable properties in the fields of material chemistry, pharmaceutical industry and coordination chemistry [21]. Spirooxindoles as an important class of heterocyclic compounds have good biological properties [22]. Due to the importance of spirooxindole derivatives, introducing a benign and novel method for the synthesis of these materials from available and simple compounds is of great interest. Thus, continuing from our previous work to develop environmental synthetic protocols [23-25] and considering the above-mentioned points, herein, we report the production of spirooxindoles via the one-pot multi-component reaction of 1,3-dicarbonyl compounds, malononitrile and isatins in the presence of catalytic amount of Fe(III)@ graphitic carbon nitride at reflux conditions in a green solvent (Ethanol/Water: 1:1) (Scheme 1).

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**Scheme 1.** The synthetic procedure for the preparation of spirooxindole derivatives in the presence of Fe (III) @graphitic carbon nitride nanocomposite.

## 2. Experimental

### 2.1. Materials

Chemical materials were provided from Merck Company and used without further purification. The FT-IR spectrometer (Perkin Elmer, Spectrum 2) was applied for getting IR spectra in a KBr pellet. The Philips CM10 instrument was applied for the recording of TEM images. The XRD pattern was also recorded using the Bruker D8-Advance X-ray diffractometer (Cu  $K\alpha$  radiation,  $k = 1.5406 \text{ \AA}$ ). Finally,  $^{13}\text{C}$  &  $^1\text{H}$  NMR spectra were recorded at 62.9 MHz and 250 MHz in DMSO- $d_6$  using TMS as an internal standard and the Bruker DPX-250 Advance NMR instrument.

### 2.2. Synthesis of the graphitic carbon nitride nanosheet

To synthesize graphitic carbon nitride nanosheet, 10 g of melamine was placed in an alumina crucible with a cover and the crucible was heated at a rate of  $2.5 \text{ }^\circ\text{C}/\text{min}$  to  $550 \text{ }^\circ\text{C}$  in a muffle furnace. The crucible content was kept at  $550 \text{ }^\circ\text{C}$  for 4 h. All of the mentioned steps were done in an air atmosphere. Finally, the resulting product (yellow powder) was stored for next steps [26].

### 2.3. Fe(III)@graphitic carbon nitride synthesis

The Fe (III)@graphitic carbon nitride was synthesized according to the previous reported method with some modification [27]. Briefly, graphitic carbon nitride (1g) was dispersed in 30 ml EtOH for 30 min (Mixture 1). Solution 1 was also prepared by dissolving 5 mmol of  $\text{FeCl}_3$  in 25 mL of ethanol. The solution 1 was gradually added to the mixture 1 and stirred at  $40 \text{ }^\circ\text{C}$  for 6 h. Then, the powder was recovered by centrifuging and washed until neutralization. The nanocomposite was placed in an oven and dried at  $80 \text{ }^\circ\text{C}$  for 8 h.

### 2.4. The Synthesis of Spirooxindole Derivatives

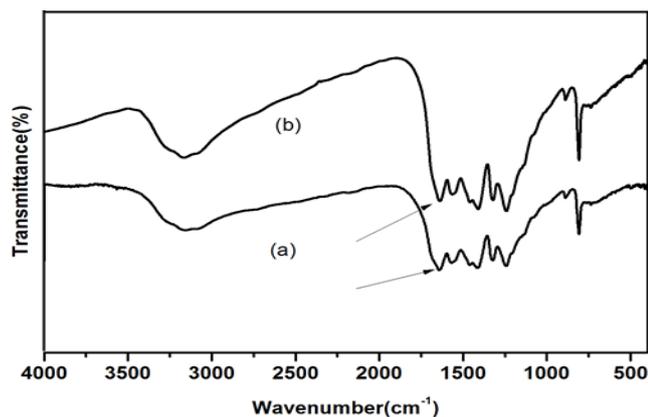
An appropriate amount of Fe (III)@graphitic carbon nitride (0.07 g) was added to the 3 ml of EtOH/ $\text{H}_2\text{O}$  (1:1) and dispersed for 30 min. Then, 1 mmol of each compound (isatin, dimedone, and malononitrile) was poured into the mixture followed by stirring the mixture under reflux conditions for a definite time. After completion of the reaction, the solvent was removed by centrifuging. Then, the crude product was extracted by EtOAc. The solvent was dried using sodium sulfate and evaporated. Finally, the product was recrystallized in hot ethanol to give the pure product. The recovered nanocomposite was reused for another run.

## 3. Results and Discussion

The Fe(III)@graphitic carbon nitride catalyst was prepared as shown in Scheme 2. Initially, graphitic carbon nitride was produced by the hydrothermal procedure and dispersed in EtOH/water (1:1) and treated with  $\text{FeCl}_3$  to obtain the Fe(III)@graphitic carbon nitride nanocomposite. The corresponding Fe(III)@graphitic carbon nitride nanocomposite was characterized by different techniques.

### 3.1. FT-IR study

Fig. 1 shows the IR spectra of graphitic carbon nitride (a) and Fe(III)@graphitic carbon nitride (b). The peak which is ranged from  $3401$  to  $2801 \text{ cm}^{-1}$  belongs to the stretching vibration bond of N-H and the peak appeared at  $1646 \text{ cm}^{-1}$  belongs to the stretching vibration of the C=N bond. The stretching vibration bond of C-N triazine is observed at  $1237$ ,  $1318$ ,  $1406$  and  $1544 \text{ cm}^{-1}$ . Also, the vibration of triazine (out-of-plane) cycle is indexed at a peak appeared at  $807 \text{ cm}^{-1}$  (Fig. 1a). The peaks which are shown at  $1238$ ,  $1405$  and  $1543 \text{ cm}^{-1}$  are shifted to the lower positions demonstrating the Fe (III) ion onto the graphitic carbon nitride (Fig. 1b).



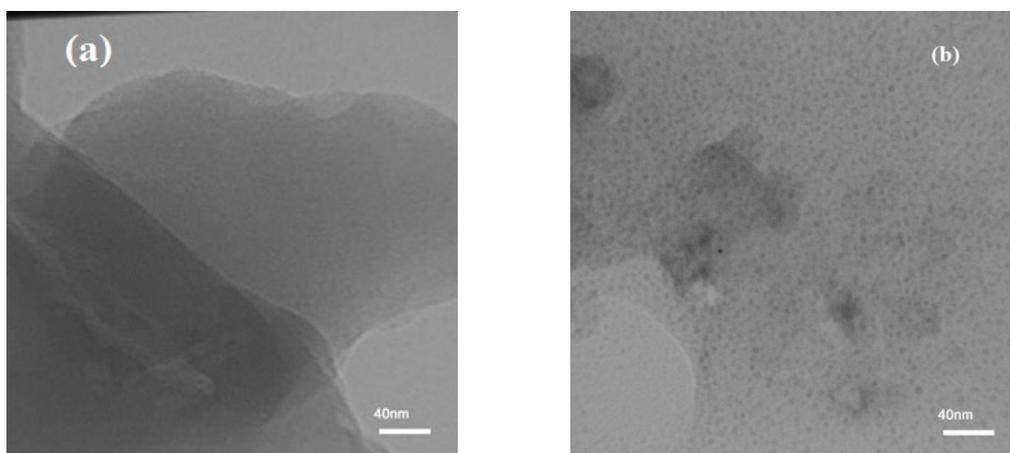
**Fig. 1.** FT-IR spectra of graphitic carbon nitride (a), Fe(III)@graphitic carbon nitride (b).

### 3.2. XRD study

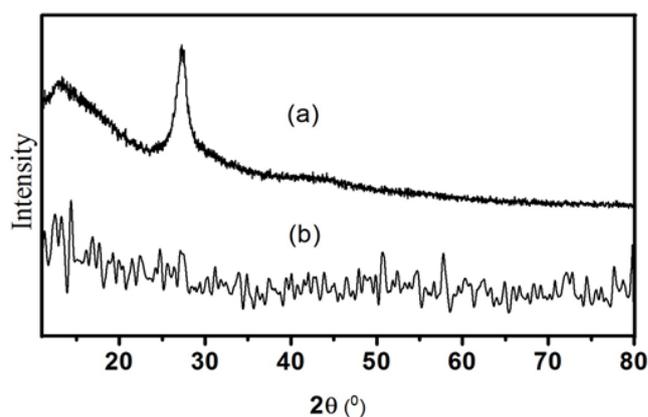
Fig. 2a shows the XRD patterns of graphitic carbon nitride. The  $2\theta = 13.1^\circ$  and  $27.4^\circ$  belonged respectively to (100) and (002) according to the JCPDS 087-1526 and demonstrate that an interlayer is the stacking peak with in-plane repeated units and the hexagonal phase of the graphitic carbon nitride. In Fig. 2b, the presence of Fe (III) caused destruction of the in-plane repeated units, an interlayer stacking peak and also the hexagonal phase of the nanosheet of graphitic carbon nitride.

### 3.3. TEM study

The TEM images of graphitic carbon nitride and Fe(III)@graphitic carbon nitride are displayed in Fig. 3a & 3b, respectively. The agglomeration of the lamellar structure of pure graphitic carbon nitride nanosheets is shown in the TEM image (Fig. 3a). The TEM image of Fe (III)@graphitic carbon nitride demonstrates the presence of Fe (III) on the surface of graphitic carbon nitride nanosheets (Fig. 3 b).



**Fig. 3.** The TEM images of graphitic carbon nitride (a) Fe(III)@graphitic carbon nitride(b).



**Fig. 2.** XRD pattern of graphitic carbon nitride (a), Fe(III)@graphitic carbon nitride (b).

To investigate the efficiency of nanocomposite, a model reaction consists of 1,3-cyclohexadione, isatin, malononitrile was conducted in the presence of Fe(III)@graphitic carbon nitride (0.05 g, 0.14 mmol based on Fe metal) in EtOH/H<sub>2</sub>O (1:1) and the prepared product was obtained in 30 % yield under the room temperature at 1 h (Table 1). These results showed that nanocomposite is essential for the synthesis of spirooxindole derivatives. Then, the reaction was carried out at 40, 60, and 80 °C and the corresponding products were isolated in 40, 51 and 65 % yields, respectively. To improve the further yield, the effects of various reaction parameters such as the amount of catalyst, and the solvent were evaluated systematically. The model reaction was investigated in the different amounts of catalyst (0.05-0.09 g), and the results showed that 0.07 g of catalyst (0.195 mmole based on Fe metal) gave the best yield (98 %) (Table 1, entries 4-6). No negligible increase in the yield of product was observed when the consuming of the catalyst increased (Table 1, entries 7 & 8).

**Table 1.** The effect of the different amounts of composite on the model reaction.<sup>a</sup>

Entry	gr of catalyst (mmol Fe III)@graphitic carbon nitride (g)	Time (min)	Yield (%) <sup>b</sup>
1	---	60	trace
2 <sup>c</sup>	0.07	60	trace
3 <sup>d</sup>	0.05 (0.14)	60	30
4	0.05 (0.14)	2	70
5	0.06 (0.167)	2	80
6	0.07 (0.195)	2	98
7	0.08 (0.22)	2	98
8	0.09 (0.25)	2	98
9 <sup>e</sup>	0.05 mmol	2	98

<sup>a</sup>Reaction conditions: Malononitrile (1mmol), isatin (1 mmol), 1,3-cyclohexadione (1 mmol), in EtOH/H<sub>2</sub>O (1:1) (3 mL) as the solvent at reflux conditions.

<sup>b</sup>Isolated yields.

<sup>c</sup>graphitic carbon nitride as a catalyst.

<sup>d</sup>Room temperature.

<sup>e</sup>FeCl<sub>3</sub> as the catalyst.

Also, the control experiment confirmed that in the absence of the catalyst only the trace amount of the corresponding product was detected (Table 1, entry 1). Furthermore, the model reaction was studied in the presence of graphitic carbon nitride (0.07 g) and no obvious product was detected (Table 1, entry 2). Moreover, the effect of Fe (III) was investigated on the progress of the reaction and despite the reaction proceeded well with 0.05 mmole of the Fe (III) the recovery of the product was a major challenge (Table 1, entry 11).

Solvents influence on the yield of the reactions [28]. Therefore, the effects of different polar and non-polar solvents and also solvent-free conditions on the progress of multicomponent reaction were investigated; as the results show, EtOH/water(1:1) gave the maximum yield (98 %) in the short time and proved to be the best solvent (Table 2, entry 12).

Under optimized reaction conditions, the generality of this method was explored for the synthesis of spirooxindole derivatives (Table 3). First, 1,3-cyclohexadione and malononitrile were used as the substrates to investigate isatin substituents. The results showed that isatins with different substituent groups on benzene ring produced the desired products in high yields (Table 3, entries 1-9, 90-98 %). By using dimedone instead of 1,3-cyclohexadione in the reactions and a slight increase in the time of the reactions was observed (Table 3, entries 10-13). When 4-hydroxycoumarin was applied as the substrate, the reactions proceeded in longer times and lower yields

(Table 3, entries 14-22). These results may be due to the lower activity of carbonyl group of 4-hydroxycoumarin. To demonstrate the efficiency of the catalyst, the catalytic activities of Fe (III) @graphitic carbon nitride nanocomposite were compared with some other reports [25, 29-35]. As can be seen in Table 4, in comparison to the other reports, this study exhibits different advantages including facile preparation of the catalyst, short time of the reaction, and easy separation of the products.

**Table 2.** Solvents effect on the yield of spirooxindoles.<sup>a</sup>

Entry	Solvent	Time (min)	Yield (%)
1	n-Hexane	30	15
2	CH <sub>2</sub> Cl <sub>2</sub>	30	16
3	THF	30	47
4	DMF	30	58
5	CHCl <sub>3</sub>	30	46
6	CH <sub>3</sub> CN	30	60
7	EtOAc	30	59
8	DMSO	30	65
9	MeOH	30	77
10	EtOH	30	75
11	H <sub>2</sub> O	8	90
12	EtOH/water (1:1)	2	98
13	Solvent-free	30	40

<sup>a</sup>Reaction conditions: malononitrile (1 mmol), isatin (1 mmol), 1,3-cyclohexadione (1 mmol), Fe (III) @graphitic carbon nitride (0.0195 mmol based on Fe) at reflux conditions.

**Table 3.** The synthesis of spirooxindole derivatives.

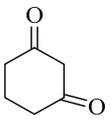
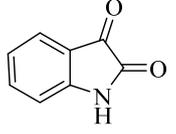
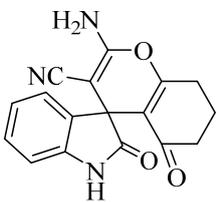
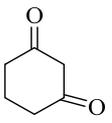
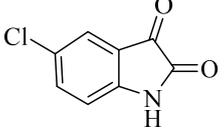
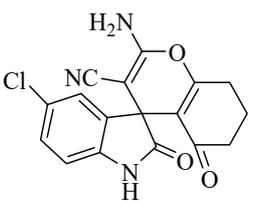
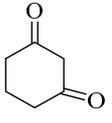
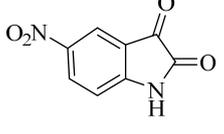
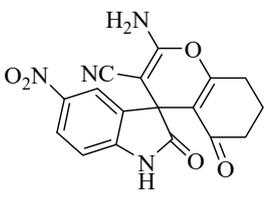
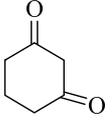
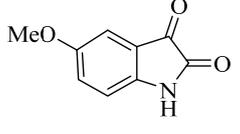
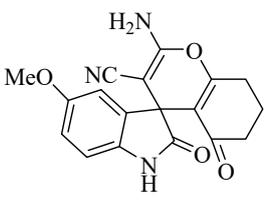
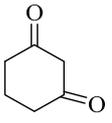
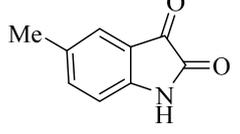
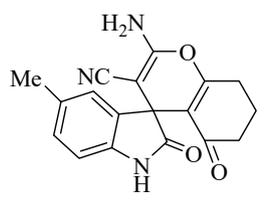
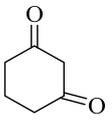
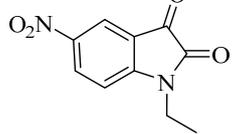
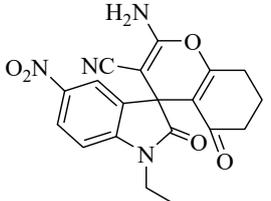
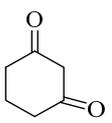
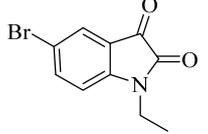
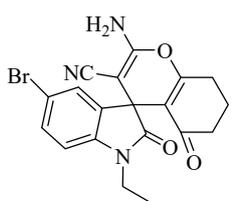
Entry	Substrate 1	Substrate 2	Product	Time (min)	Yield (%)	TON	TOF (min <sup>-1</sup> )
1				2	98	363	181.5
2				3	92	340.8	113.6
3				3	95	351.9	117.3
4				4	90	333.3	83.33
5				2	97	359.3	179.6
6				2	95	351.9	175.3
7				3	95	351.9	117.3

Table 3. (Continued).

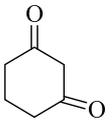
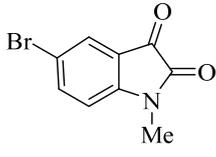
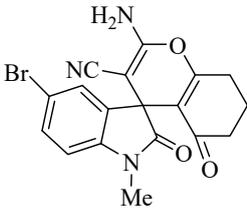
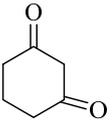
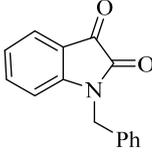
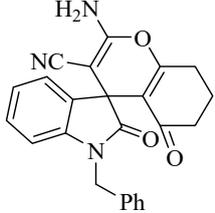
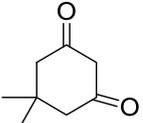
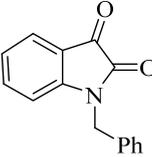
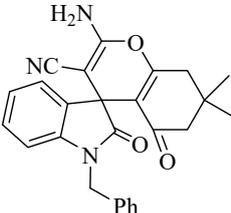
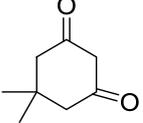
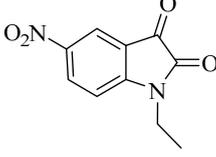
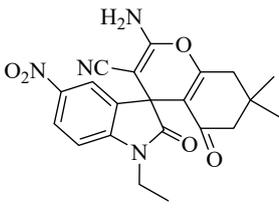
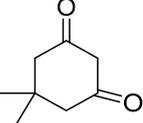
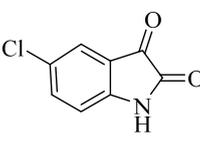
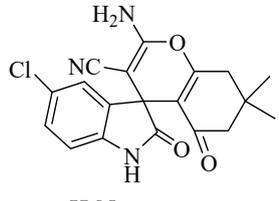
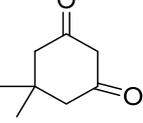
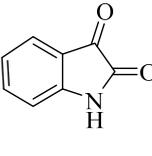
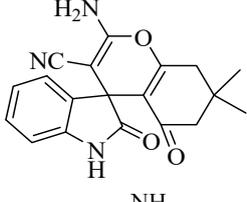
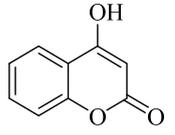
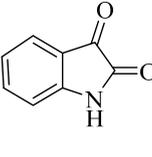
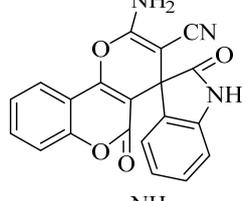
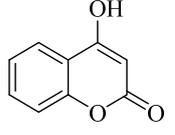
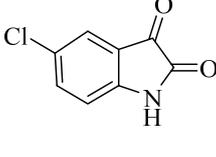
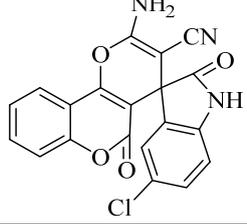
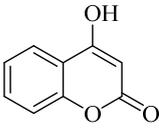
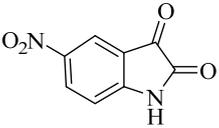
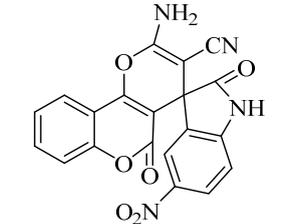
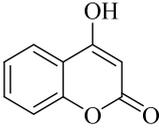
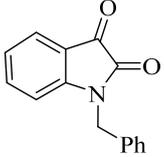
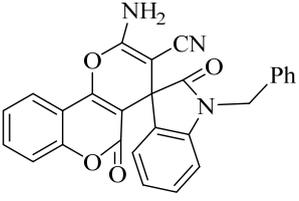
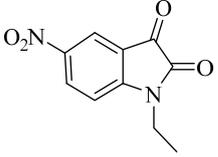
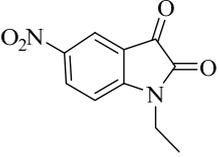
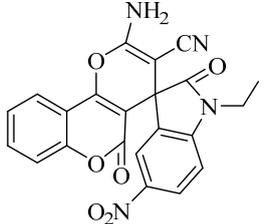
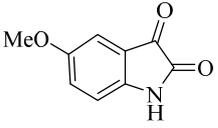
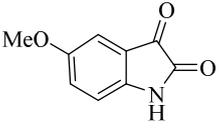
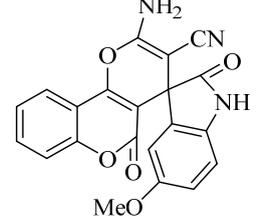
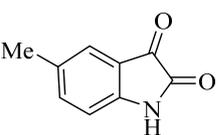
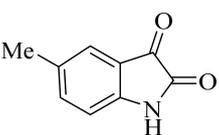
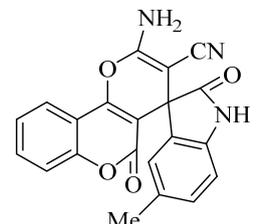
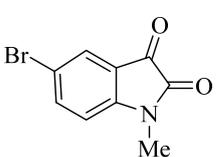
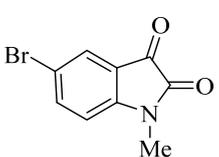
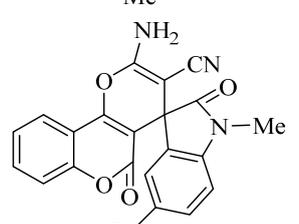
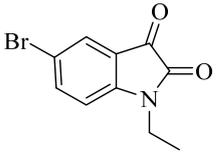
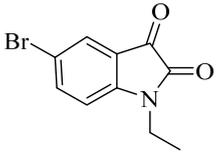
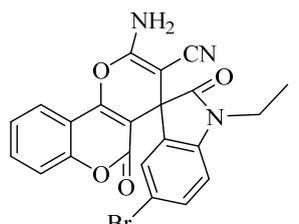
8				4	95	351.9	88
9				5	91	337	67.4
10				5	95	351.9	70.4
11				7	93	344.4	49.2
12				5	91	337	67.4
13				3	95	351.9	117.3
14				15	85	314.8	21
15				15	86	318.5	21.23

Table 3. (Continued).

16				15	75	277.8	18.5
17				15	88	326	21.72
18				15	70	259.3	17.3
19				15	85	314.8	21
20				15	85	314.8	21
21				15	75	277.8	18.5
22				15	83	307.4	20.5

<sup>a</sup>Reaction conditions: malononitrile (1 mmol), isatin (1 mmol), 1,3-cyclohexadione or 4-hydroxy coumarin (1 mmol), composite (0.0195 mmol based on Fe) and the solvent (3 mL), reflux conditions.

**Table 4.** The Comparison of efficiency of Fe (III) @graphitic carbon nitride with different catalysts.<sup>a</sup>

Entry	Catalyst	Solvent/Temp.(°C)	Time (min)	Yield (%)	Ref.
1	Piperidine	US, EtOH, r.t.	60	92	[29]
2	Carbon-SO <sub>3</sub> H	EtOH/ ref.	180	81	[30]
3	Nano-NiO	H <sub>2</sub> O/ r.t.	5	98	[31]
4	TBA acetate	Solvent-free/ 100 °C	10	92	[32]
5	(SB-DBU)Cl	EtOH/ r.t	150	97	[33]
6	Sodium Citrate	H <sub>2</sub> O, 60 °C	180	95	[34]
7	NaOAc	H <sub>2</sub> O, r.t	15	95	[35]
8	SiO <sub>2</sub> @graphitic carbon nitride	H <sub>2</sub> O/ r.t.	3	98	[25]
9	Fe (III) @graphitic carbon nitride	EtOH/water (1:1), ref.	2	98	This work

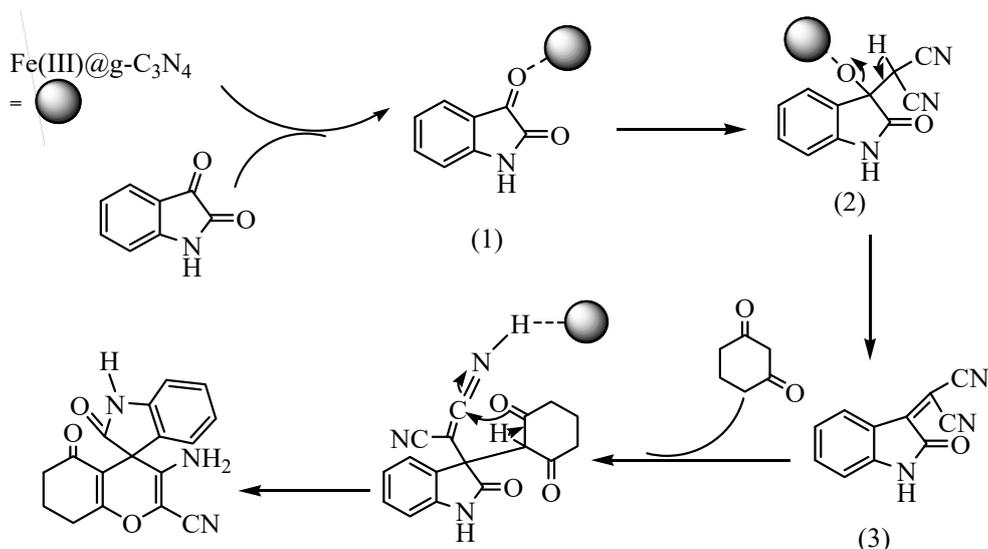
<sup>a</sup>Reaction conditions: Isatin (1 mmol), Malononitrile (1 mmol), Dimedone (1 mmol).

Also, the reusability of the nanocomposite was investigated in the model reaction. After each run, the model reaction was proceeded for the next run and analyzed the solution for Fe (III) to detect leaching. The results showed that the yield was not decreased and also leaching was negligible in the model reaction. These findings indicated the efficiency and stability of nanocomposite under reaction conditions.

We proposed a mechanism for the reaction (Scheme 2). As can be seen, Fe (III) @graphitic carbon nitride activated the carbonyl group of isatin [37] and this carbonyl group was then attacked via nucleophilic attack of malononitrile to produce intermediate 2. After that, the compound 2 undergoes elimination via Knoevenagel condensation to generate intermediate 3. Finally, dimedone was added to intermediate 3 to generate the corresponding products.

#### 4. Conclusions

Fe(III)@graphitic carbon nitride nanocomposite was synthesized and characterized by different techniques. The efficiency of nanocomposite was investigated in the synthesis of spirooxindole derivatives via the one-pot, three-component reaction of isatins, malononitrile and 1,3-dicarbonyl compounds in the presence of synthesized nanocomposite in ethanol/water (1:1) at reflux conditions, and the excellent yields of products were obtained in a short time. Compared with 1,3-dicarbonyl compounds, the reactions were performed slowly using 4-hydroxycoumarin as a substrate and the yields were slightly decreased. Finally, the advantage of this system includes the easy recovery and reusability of catalytic system, ecofriendly and green media, excellent yields and short reaction time.



**Scheme 2.** Proposed mechanism for the preparing oxindoles by Fe (III) @graphitic carbon nitride catalyst.

## Acknowledgments

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