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Humic acid: A green acid catalyzed by acylation of amine, phenol, and thiols derivatives under solvent-free conditions

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Abstract:

Humic acid has been the catalyst in a novel process that effectively and environmentally acylates amine, phenol, and thiol derivatives. This method works well with aliphatic, aromatic, and heteroaromatic substrates, among other functionalized molecules. There is only one step involved in the simple method, which yields a high yield without any unintended side effects. In addition, the humic acid catalyst may be recycled at least four times, and the process is simpler, quicker, and greener than earlier research. The current work presented aniline (10 mmol, 0.93 mL), acetic anhydride (10 mmol, 1 mL), and humic acid catalyst (15 mg), which were stirred at room temperature for 10 minutes under solvent-free conditions to get a 97% yield.

Keywords: Acetylation; Humic acid; Solvent-free conditions; Green catalyst; Phenol

1. Introduction

Recently, attention has been focused on innovative approaches to the synthesis of organic compounds as well as non-traditional strategies for lowering the impact on the ecosystem [1]. Several elements, such as toxic solvents, chemicals, catalysts, and harsh reaction conditions, contribute to the production of environmentally hazardous substances during the synthesis of organic molecules [2]. Researchers said that such limitations in organic synthesis should be prevented due to this alert [3]. Green solvents are always a source of joy for chemists designing environmentally friendly procedures [4]. Eco-friendly procedures that don't use harmful solvents erase all of their negative consequences [5]. There are not many methods described in the literature for a more ecologically friendly synthesis of organic molecules without the use of solvents [6].

Due to their ease of separation, little impact on the environment, and aptitude for recycling, high molecular weight molecules have attracted more attention in recent years in organic synthesis [7]. Humic acid is a high molecular weight polymer with advantageous catalytic activity due to the carboxyl and phenolic hydroxyl groups present in its structure. Humic acid, which is mostly found in coal, peat soil, well water, and desolate lakes, is more inexpensive, biodegradable, and highly reusable (figure 1). It has also been shown to have outstanding catalytic activity [8]. Due to the minimal work on humic acid in the previous literature on the catalytic study, there have been several opportunities to study the various organic reactions [9, 10].

Green chemistry principles are motivating scientists to develop synthetic approaches to decrease the production of harmful substances [11–15]. Green catalysts are necessary for both environmentally friendly synthesis and organic manufacturing [16–20]. The requirement for hazardous volatile solvents, reagents, catalysts, risky operations, and the necessary minimum reaction time are all avoided by this method, making it the most effective one for chemical synthesis [21–23]. Additionally, it avoids creating harmful sludge [24–27].

According to the green chemistry viewpoint, pollutants may be reduced and eliminated from the reaction mixture to provide a cleaner process. The synthetic method for sustainable organic transformations uses a variety of solvents, including glycerol, polyethylene glycol, water, and biodiesel waste [28–32].

Because it plays a role in the multistep synthesis of chemicals in the chemical and pharmaceutical industries, the acylation reaction of amines, alcohols, phenols, and thiols with various catalysts is a well-known method in chemical synthesis [33–38]. The acylation of phenol and alcohols using acetyl chloride or acetic anhydride in the presence of various functionalized nanoparticles, [39–42] base [43–46] and acid [47–49] is reported in earlier literature for catalysts under different reaction conditions. Several disadvantages to each of the catalyzed processes include a slower reaction rate, a harsh solvent, challenging handling, etc. A green catalyst was used in a highly efficient reaction that takes place without the need for solvents [50–52].

The present work represents the acylation of amine, alcohol, phenol, and thiol derivatives using humic acid. Firstly, we studied the various solvents that were used in the presence of distinct amounts. After that, we studied various phenol, alcohol, thiol, and amine derivatives under solvent-free conditions. Finally, we study the recyclability of the humic acid for a minimum of 4 cycles (scheme 1).

2. Experimental section

2.1 Material methods

All the chemicals were given from commercial chemical suppliers and used directly in the reaction. All the prepared compounds were purified by chromatographic techniques on a 60 − 120 mesh silica powder. The reaction progress was monitored on Merck plates (F254 coated with silica gel 60). The prepared compounds were characterized using ^{1}H-NMR and ^{13}C-NMR at a 450 MHz Bruker AV 400 NMR spectrometer.

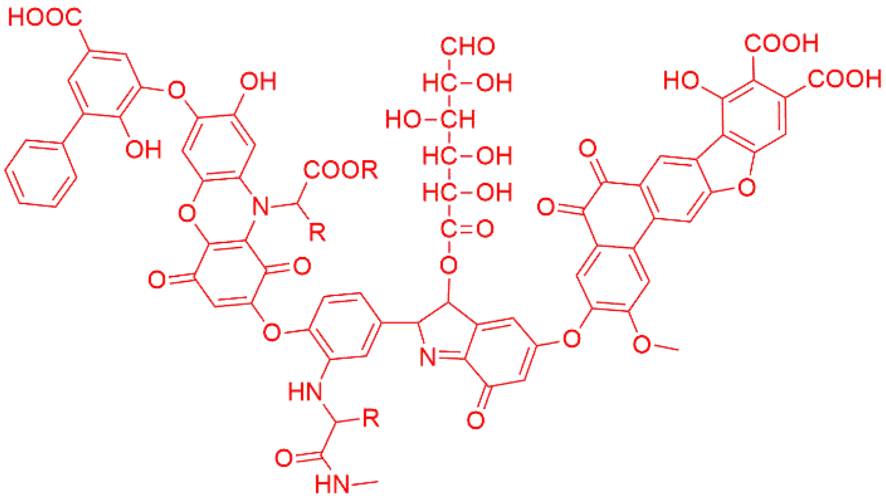
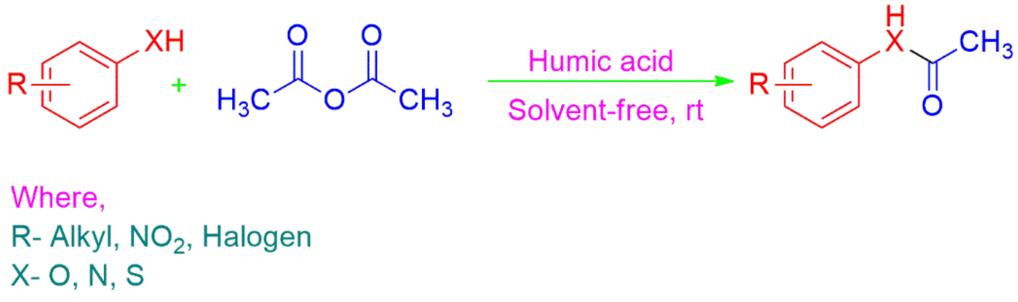


Figure 1. Humic acid structure.



Scheme 1. The acylation of amines, alcohols, phenols, and thiols catalyzed by humic acid.

2.2 General procedure for the acylation of aniline derivatives

A mixture of aniline (10 mmol, 0.93 mL), acetic anhydride (10 mmol, 1 mL), and humic acid catalyst (15 mg) was stirred at room temperature for 10 minutes under solvent-free conditions. The reaction was monitored on TLC in n-hexane and ethyl acetate (9:1) ratio. After the complete reaction, the mixture was extracted with diethyl ether (10 mL). Then filter the reaction mixture to separate humic acid. The ether layer was evaporated at room temperature to form a white solid crude product.

2.3 General procedure for the acylation of phenol derivatives

A mixture of phenol (10 mmol, 0.94 mL), acetic anhydride (10 mmol, 1 mL), and humic acid catalyst (15 mg) was stirred at room temperature for 10 minutes under solvent-free conditions. The reaction was monitored on TLC in n-hexane and ethyl acetate (9:1) ratio. After the complete reaction, the mixture was extracted with diethyl ether (10 mL). Then filter the reaction mixture to separate humic acid. The ether layer was evaporated at room temperature to form a colorless liquid product.

2.4 General procedure for the acylation of thiol derivative

A mixture of thiophenol (10 mmol, 1.10 mL), acetic anhydride (10 mmol, 1 mL), and humic acid catalyst (15 mg) was stirred at room temperature for 10 minutes under solvent-free conditions. The reaction was monitored on TLC in n-hexane and ethyl acetate (9:1) ratio. After the complete reaction, the mixture was extracted with diethyl ether (10 mL). Then filter the reaction mixture to separate humic acid. The ether layer was evaporated at room temperature to form a yellow liquid product.

2.5 General procedure for the recycling of humic acid catalyst

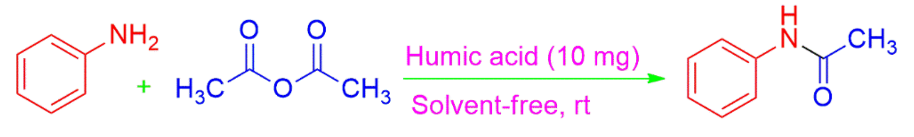
After the completion of the reaction, the mixture was extracted with diethyl ether (10 mL). Then filter the reaction mixture to separate the humic acid catalyst. After that, the catalyst was dried at room temperature and used directly in the next reaction.

3. Results and discussion

Using humic acid as a green catalyst, we thoroughly investigated the diverse acylation of amines, alcohols, phenols, and thiols in the presence of acetic anhydride (scheme 1). A similar type of acylation reaction has also been proposed by K. Datta et al. [24]. The reaction between aniline and acetic anhydride was performed in the presence of humic acid under various reaction conditions. The experiment showed that the acylation reaction functions effectively under solvent-free conditions. The acylation reaction was performed by adding 5 mg of humic acid catalyst with stirring at room temperature to give 80% yield within 10 minutes. Additionally, increasing the catalyst concentration from 10 to 15 mg raises the reaction yield by about 98%. The yield remained unaffected when the catalyst was increased to 20 mg. Several conditions were carried out in the presence of solvents like aprotic polar (Tetrahydrofuran and acetonitrile), protic polar (Methyl alcohol and ethyl alcohol), non-polar (Toluene), and light polar (dichloromethane), but the results were not satisfactory. In the absence of a solvent and a catalyst, the reaction gives a trace yield (Table 1).

The reaction conditions for acylating amine, phenol, alcohol, and thiol derivatives were optimized to achieve the desired acetate product with moderate to good yields under solvent-free conditions. (Table 2) The study analyzed the different types of amines, considering both electron-donating and electron-withdrawing substituents. Substituents with

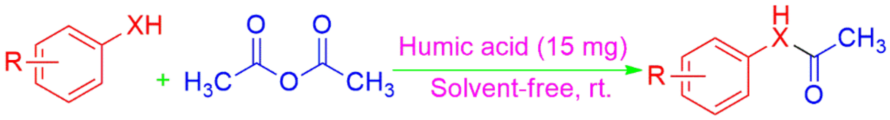
Table 1. Optimization of various reaction conditions for the acylation of aniline catalyzed under Humic acid[a].



Entry	Solvent	Catalyst (mg)	Time (min)	Yield[b] %
1	None	None	1h	Trace
2	None	5	10	80
3	**None**	**10**	**2**	**97**
4	None	20	2	97
5	CH_3CN	10	10	78
6	THF	10	10	80
7	EtOH	10	10	78
8	DCM	10	10	68
9	Methanol	10	10	74
10	Toluene	10	10	82

[a] Reaction conditions: aniline (10 mmol), acetic anhydride (1.5 mmol), room temperature, and solvent (3 mL).
[b] Isolated yield.

Table 2. Acylation of phenol, amine, thiol, and alcohol derivatives using humic acid as a green catalyst[a].



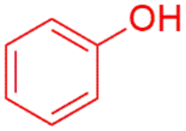
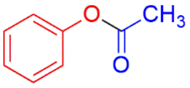
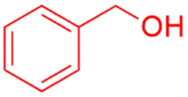
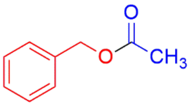
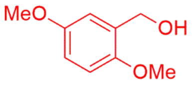
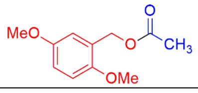
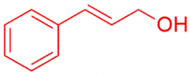
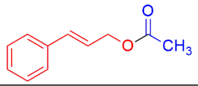
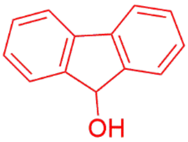
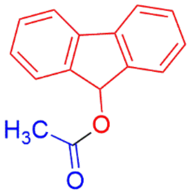
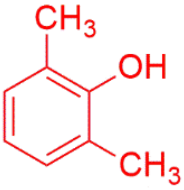
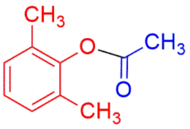
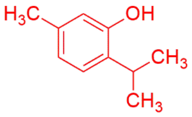
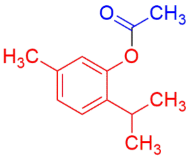
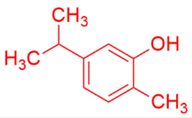
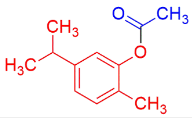
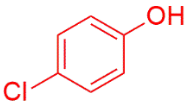
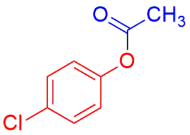
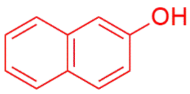
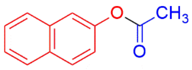
Entry	Substrate (R)	Product	Time (min)	Yield[b] (%)	TON	TOF (h^{-1})
1			15	97	87.33	21.75
2			15	98	90.66	22.66
3			15	97	81.33	20.33
4			15	94	82.66	20.66
5			20	95	78.01	25.74
6			15	91	81.33	20.33
7			15	91	78.01	19.50
8			12	92	78.66	15.73
9			10	94	83.33	13.88
10			10	92	79.33	12.69

Table 2. Acylation of phenol, amine, thiol, and alcohol derivatives using humic acid as a green catalyst[a].

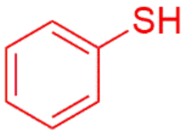
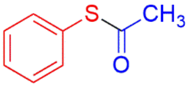
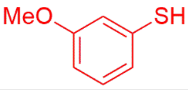
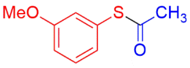
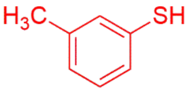
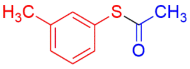
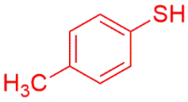
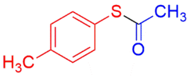
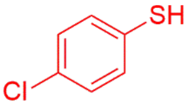
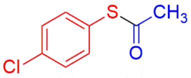
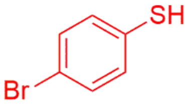
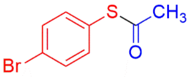
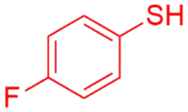
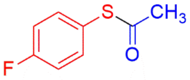
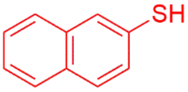
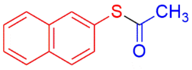
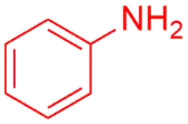
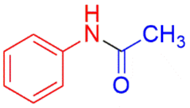
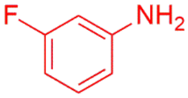
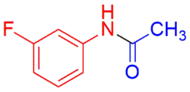
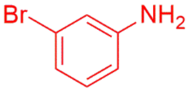
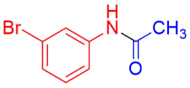
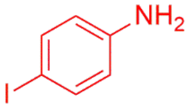
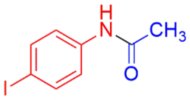
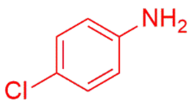
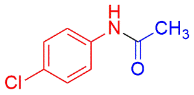
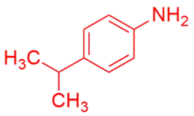
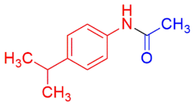
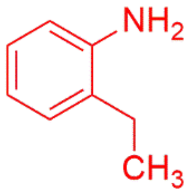
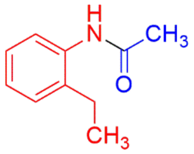
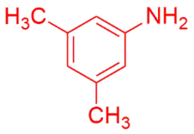
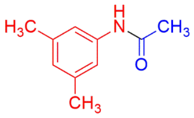
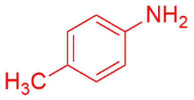
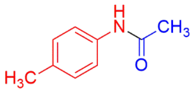
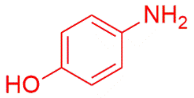
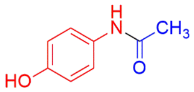
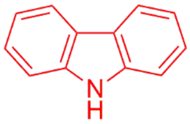
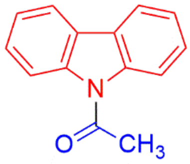
Entry	Substrate (R)	Product	Time (min)	Yield[b] (%)	TON	TOF (h^{-1})
11			30	90	83.33	41.66
12			40	93	80.66	53.23
13			30	92	81.33	40.66
14			30	91	80.66	40.33
15			40	88	74.66	49.77
16			40	89	72.00	47.52
17			35	87	88.00	51.04
18			25	95	84.00	34.80
19			10	97	96.66	15.46
20			15	94	91.89	22.97
21			20	92	82.66	27.28
22			20	93	79.45	26.21

Table 2. Acylation of phenol, amine, thiol, and alcohol derivatives using humic acid as a green catalyst[a].

Entry	Substrate (R)	Product	Time (min)	Yield[b] (%)	TON	TOF (h^{-1})
23			15	94	88.66	22.16
24			15	95	87.40	21.85
25			12	96	89.33	17.86
26			10	96	89.33	14.29
27			10	97	92.83	14.85
28			10	96	92.35	14.77
29			20	95	87.40	28.84

[a] Reaction conditions: Reagent (10 mmol), Humic acid (15 mg), and acetic anhydride (15 mmol), stirring at room temperature under solvent-free conditions. [b] Isolated Yield.

electron-donating properties tend to be more reactive than those with electron-withdrawing properties. In the acylation of phenol, the substituent that donates electrons needs to react at a faster rate.

Significant quantities of compounds can be obtained through this reaction with various alcohol derivatives, encompassing electron-donating substituents. (Table 2, entry 1-3) Favorable outcomes can also be yielded through the acylation of unsaturated alcohols. (Table 2, entry 4) Acylating secondary alcohols are an effective way to achieve improved yield variations in products (Table 2, entry 5). For optimum product yield during the acylation process, it is essential to use aromatic phenols that have electron-donating or electron-withdrawing derivatives (Table 2, entries 6-9). Interestingly, bicyclic compound β-naphthol also gave a much better yield (Table 2, entry 10). We also examine aromatic thiol compounds, such as electron-donating, electron-withdrawing, and halogens, that also gave high yields in a shorter reaction time (Table 2, Sr. entries 11-18). Finally, we studied the halogen-containing substituent exhibiting a slower reaction rate as compared with the aniline reaction (Table 2, entries 19-23). In amine acylation, the substituent that donates electrons needs to react at a faster rate (Table 2, entries 24-28). After extensive research, we have discovered a highly effective acylation reaction for the secondary cyclic amine of 9H-carbazole. (Table 2, entries 29).

Finally, efficiency assessments of turnover number (TON) and turnover frequency (TOF) were computed for all experiments conducted at a catalyst dose of 15 mg/mol in order to better facilitate comparison across the various tested circumstances. The quantity of molecules produced at a single catalytic site prior to deactivation is known as the turnover number.

$$\mathrm{TON} = \frac{N}{n_a} \quad (1)$$

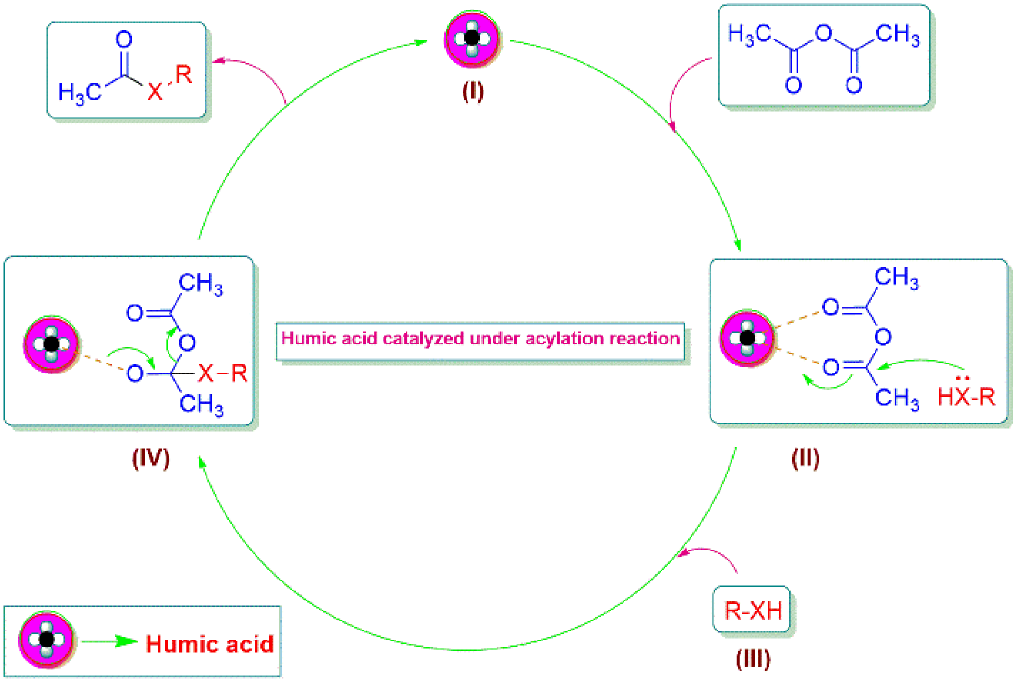
where N is the number of reactants converted or products produced. n_a-the number of moles of catalyst.

$$\mathrm{TOF} = \frac{\mathrm{TON}}{t} \quad (2)$$

where TON is the turnover number and t-reaction time. Scheme 2 depicts the acylation process in the presence of a mechanical route using humic acid as a green catalyst. The humic acid catalyst (I) reacted with acetic anhydride containing a carbonyl oxygen, creating a more electrophilic center (II) that allowed amines (R-X) (III) to undergo nucleophilic attack and form an intermediate (IV). Step (IV) was modified to produce both the acylated product and acetic acid as a by-product, similar to a method proposed by K. Datta et al. [24].

The recyclability study of humic acid shows crucial properties. That catalyst was used for the acylation of aniline in the presence of acetic anhydride as a model reaction for this investigation. When the reaction was completed, the catalyst was recycled using a simple filtration method, and the catalyst was again used in the reaction mixture at least 4 times. The reaction gave excellent results after 4 cycles. (figure 2) The humic acid catalyst was recycled 4th time, catalyst was both (before use and after use) characterized using FT-IR analysis. The data is attached in the supporting information. (S2, figure 1 and figure 2) The peak at 3339 cm^{-1} indicates that after the 4th cycle, acid is present in the catalyst.

The humic acid is well desired for the acylation of amines, alcohols, phenols, and thiol substrates as compared to other catalysts and methodologies. According to Table 3, the catalyst and reaction conditions described in the literature had several limitations, including a slow response time, a low product yield, and the fact that the catalysts were recycled in the reaction mixture. Moreover, the amount of humic acid, 15 mg, is sufficient for the complete conversion of the



Scheme 2. Plausible reaction mechanism of humic acid catalyzed under acylation of N, O, and S derivatives.

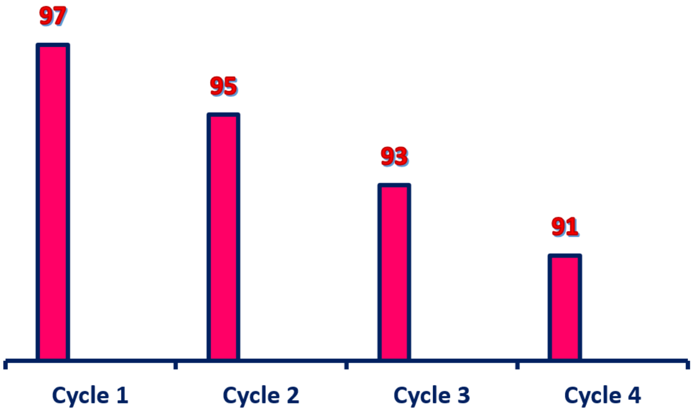


figure 2. Recyclable study of the humic acid catalyst.

Table 3. Comparison of the activity of the present system with literature reported for the acylation of amine.

Entry	Reaction condition	Time (min)	Yield (%)	Ref.
1	$Fe_3O_4@SiO_2@Co_3O_4$, r. t, solvent-free	10	97	[25]
2	$Ni(COD)_2$, 100 °C, 12h	12h	86	[26]
3	$Fe_3O_4@SiO_2$@Pd-complex, 60 °C, 12h	12h	92	[27]
4	Copper zirconium phosphate NPs, 60 °C, solvent-free	30	91	[28]
5	Yttria-zirconia, 110 °C, solvent-free	6 h	94	[29]
6	$ZnCl_2$, 80 °C, CH_3CN	3 h	90	[30]
7	CuO-ZnO, 80 °C, CH_2Cl_2	15	92	[31]
8	Nano γ-Fe_2O_3, sonication, r. t.	1 h	64	[32]
9	Silica-sulfamic acid, r. t, solvent-free	15	93	[33]
10	SiO_2-$ZnCl_2$, 80 °C, CH_3CN	3 h	90	[30]
11	Borated zirconia, 110 °C, toluene	15 h	25	[34]
12	Fe_3O_4@PDA-SO_3H, r. t, solvent-free	25	96	[35]
13	$Fe_3O_4@SiO_2@K_{10}$, r.t, solvent-free	20	96	[36]
14	Humic acid, solvent-free, r. t.	10	97	Present work

reaction. Interestingly, the humic acid can be recovered and reused at least 4 times.

4. Conclusion

We develop a simple and green methodology using humic acid-catalyzed acylation of amine, phenol, alcohol, and thiol derivatives. We also investigated the characteristics of prepared acylated compounds using ^{1}H-NMR and ^{13}C-NMR techniques. The current work presented aniline (10 mmol, 0.93 mL), acetic anhydride (10 mmol, 1 mL), and humic acid catalyst (15 mg), which were stirred at room temperature for 10 minutes under solvent-free conditions to get a 97% yield. The catalyst was recycled and reused 4 times. Humic acid is a green acid, that is easily available, cheap, and less toxic, the reaction proceeded at room temperature under solvent-free conditions.

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Authors contributions
Authors have contributed equally in preparing and writing the manuscript.

Availability of data and materials
The data that support the findings of this study are available from the corresponding author, upon reasonable request.

Conflict of interests
The author declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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