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Trifluoroethanol and liquid-assisted grinding method: a green catalytic access for multicomponent synthesis

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Abstract An efficient and versatile mechanochemical route for the synthesis of chromene and isoindolo[2,1-*a*]quinazoline scaffolds has been developed via a simple mortar and pestle liquid-assisted grinding method using 2,2,2-trifluoroethanol (TFE) as an efficient catalyst. The present protocol is very efficient as it offers reaction in mild reaction condition, cleaner reaction profiles, effortless work-up step with excellent purity, and high yield of the desired products with short reaction time.

Keywords Liquid assisted grinding · Trifluoroethanol · Chromenes · Isoindolo[2,1-*a*]quinazolines

Introduction

Over the last few years, fluorinated compounds have attracted great interest in organic synthesis due to their favorable properties like low boiling points and high melting points compared with their non-fluorinated counterparts. In addition, they have high polarity and strong hydrogen bond donation which increase their ability to solvate water molecules [1]. Special attention has been paid to 2,2,2-trifluoroethanol (TFE) as its strong electron-withdrawing CF_3 group affects the course of reactions when it is used as a solvent. As TFE is acting as a Brønsted acid, the organic reactions in TFE are generally selective and carried out without using any catalysts.

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In addition, it works under mild conditions, allowing a facile isolation of the final product simply by distillation of TFE [2]. Nowadays, TFE has been used in various organic transformations as a catalytic platform for the synthesis of heterocyclic scaffolds [3, 4]. In addition, it has also been used in various catalytic processes such as oxidation [5], protection and deprotection of amine groups [6], cyclopropanation of alkenes, and oxirane ring-opening reactions [7, 8].

Green chemistry focuses on research that attempts to develop safe and environmentally friendly chemical operations for the design of biologically active and industrially important molecules in synthetic organic chemistry [9–12]. The green chemistry concept offers many alternative approaches to carry out organic transformations under environmental benign conditions including use of less solvents, high atom economy, and selectivity, elimination of toxic waste, use of ambient, more facile ways of product separation and purification and use of alternative energy sources [13–20]. One of the protocols belonging to this sustainable and "greener" concept is 'mechanosynthesis' by using "ball milling" or simple grinding methods [21, 22]. Manual grinding using a mortar and pestle is a very useful method at the laboratory scale, due to its simple experimental setup. This technique does not require external heating, hence is an energy efficient and more economical and ecologically favorable procedure in organic synthesis [23]. The work carried out by Toda and co-workers showed that many of the exothermic organic reactions can be performed in good yields using grinding by mortar and pestle [24, 25]. This technique is mostly used for condensation reactions including Schiff's base formation and oxime formation [26, 27]. Recently, it has been found to be equally effective for the construction of heteroaromatic compounds of biological interest [28–30].

Heterocyclic motiffs like chromenes and isoindolo[2,1-*a*]quinazolines are rapidly gaining importance in synthetic and natural product chemistry [31–33]. They have also been employed as an intermediate for the synthesis of heterocycles [34, 35] and selective organic oxidants under mild conditions [36]. A detailed literature survey indicated that the synthesis of chromene cores was reported by the condensation of salicylaldehyde and 1,3-dicarbonyl compounds using I_2 [37], $FeCl_3{\cdot}6H_2O$ [38] and sulfamic acid [39]. These known methods have their own merits and demerits from the environmental and economic points of view.

Isoindolo[2,1-*a*]quinazoline is a prevalent heterocycle formed by the fusion of indole and quinazoline nuclei. These compounds are most versatile building blocks for the synthesis of various pharmaceuticals such as Batracylin and natural products like Tryptanthrin, Luotonin A, and Rutaecarpine [40]. Structurally, these molecules contain quinazolinone and isoindolone cores which synchronize to have astonishing biological properties [41, 42]. Isoindolo[2,1-*a*]quinazoline derivatives are the strong inhibitors of tumor necrosis factor-alpha which is one of the key cytokine mediators used as a marker for many inflammatory disorders such as rheumatoid arthritis, Crohn's disease and ulcerative colitis [43]. The interesting structural pattern of isoindolo[2,1-*a*]quinazolines coupled with its multifaceted pharmacological potential has motivated chemists to explore this useful scaffold [44–47].

Inspired by the characteristic properties of TFE and in continuation of our efforts to develop green chemistry methodology [48, 49], we report herein an efficient

alternative energy input for the multicomponent synthesis of chromenes and isoindolo[2,1-*a*]quinazolines under the catalytic umbrella of trifluoroethanol (TFE) within short reaction times.

Experimental

Solvents and reagents were of AR grade commercially sourced from Sigma Aldrich and used without further purification. Melting points were determined in an open capillary and are uncorrected. Infrared spectra were measured on a Perkin Elmer FT-IR spectrophotometer. The samples were examined as KBr discs ~ 5% *w/w*. NMR spectra were recorded on a Bruker AC (300 MHz for ^{1}H NMR and 75 MHz for ^{13}C NMR) spectrometer using $CDCl_3$ and DMSO as solvents.

General procedure for the synthesis of chromene scaffolds (3a–3l)

A mixture of CH activated acids such as 1H-indene-1,3(2H)-dione or dimedone or barbituric acid (2 mmol), salicylaldehydes (1 mmol) and TFE (0.5 mL) was gently ground in a mortar and pestle. The reaction mixture turns to pasty mass after 5–7 min of grinding. The grinding was continued for the times mentioned in Table 3 (see "Results and discussion"). The progress of the reaction was monitored by thin layer chromatography (TLC). In some cases, 0.2 mL of TFE was added after 7 min to facilitate complete conversion. After the completion of the reaction, the reaction mixture was washed with cold water, and then filtered to obtain the pure products.

General procedure for the synthesis of isoindolo[2,1-a]quinazolines (4a–4l)

A mixture of an equimolar quantities of 2-aminobenzamides (1 mmol), 2-carboxybenzaldehyde (1 mmol) and TFE (0.5 mL) was ground together for 9–11 min using a mortar and pestle at room temperature. The progress of the reaction was monitored by TLC. After the completion of the reaction, the reaction mixture was washed with petroleum ether followed by $CHCl_3$, and then filtered to obtain the pure products.

Spectral data of compounds

2-(11-Oxo-10,11-dihydroindeno[1,2-b]chromen-10-yl)-1H-indene-1,3(2H)-dione ***(3a)*** *(Table 3, Entry 1)* ^{1}H NMR (300 MHz, $CDCl_3$): δ 3.72–3.73 (d, 1H, $J = 2.5$ MHz, CH), 4.88–4.89 (d, 1H, $J = 2.5$ MHz, CH), 6.95–6.99 (m, 1H), 7.11–7.13 (d, 1H, $J = 7.5$ MHz), 7.21–7.22 (m, 2H), 7.31–7.31 (m, 1H), 7.37–7.44 (m, 3H), 7.74–7.81 (m, 2H), 7.83–7.86 (m, 1H), 7.94–7.96 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 29.3, 57.4, 105.3, 117.1, 117.6, 120.3, 120.6, 122.0, 124.6, 127.8, 127.9, 129.3, 131.3, 134.3, 134.6, 135.8, 141.2, 141.9, 150.1, 169.7, 191.4, 197.0, 198.0.

*2-(8-Chloro-11-oxo-10,11-dihydroindeno[1,2-b]chromen-10-yl)-1H-indene-1, 3(2H)-dione (**3b**) (Table 3, Entry 2)* ^{1}H NMR (300 MHz, $CDCl_3$): δ 3.68–3.69 (d, 1H, $J = 2.5$ MHz, CH), 4.82–4.83 (d, 1H, $J = 2.5$ MHz, CH), 7.14 (s, 1H), 7.16–7.21 (m, 2H), 7.30–7.37 (m, 2H), 7.38–7.44 (m, 2H), 7.78–7.82 (m, 2H), 7.87–7.91 (m, 1H), 7.94–7.97 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 29.2, 57.5, 104.8, 117.6, 118.4, 120.8, 122.2, 122.6, 122.9, 123.8, 127.3, 127.8, 127.9, 128.5, 129.5, 129.7, 131.2, 131.4, 133.9, 134.5, 134.8, 135.5, 141.1, 141.8, 148.7, 169.4, 191.0, 196.6, 197.5.

*5-(2,4-Dioxo-2,3,4,5-tetrahydro-1H-chromeno[2,3-d]pyrimidin-5-yl)pyrimidine-2,4, 6(1H,3H,5H)-trione (**3g**):(Table 3, Entry 7)* ^{1}H NMR (300 MHz, DMSO-d_6): δ 3.85–3.86 (d, 1H, $J = 2.5$ MHz, CH), 4.71–4.72 (d, 1H, $J = 2.0$ MHz, CH), 7.08–7.10 (d, 1H, $J = 8.0$ MHz), 7.14–7.15 (d, 1H, $J = 7.5$ MHz), 7.20–7.23 (m, 1H), 7.32–7.36 (m, 1H), 10.98 (s, 1H, NH), 11.17 (s, 1H, NH), 11.28 (s, 1H, NH), 11.97 (s, 1H, NH); ^{13}C NMR (75 MHz, DMSO-d_6): δ 33.0, 52.7, 84.6, 115.9, 120.3, 125.0, 127.5, 128.6, 148.6, 149.0, 149.9, 154.8, 162.9, 168.3, 169.0.

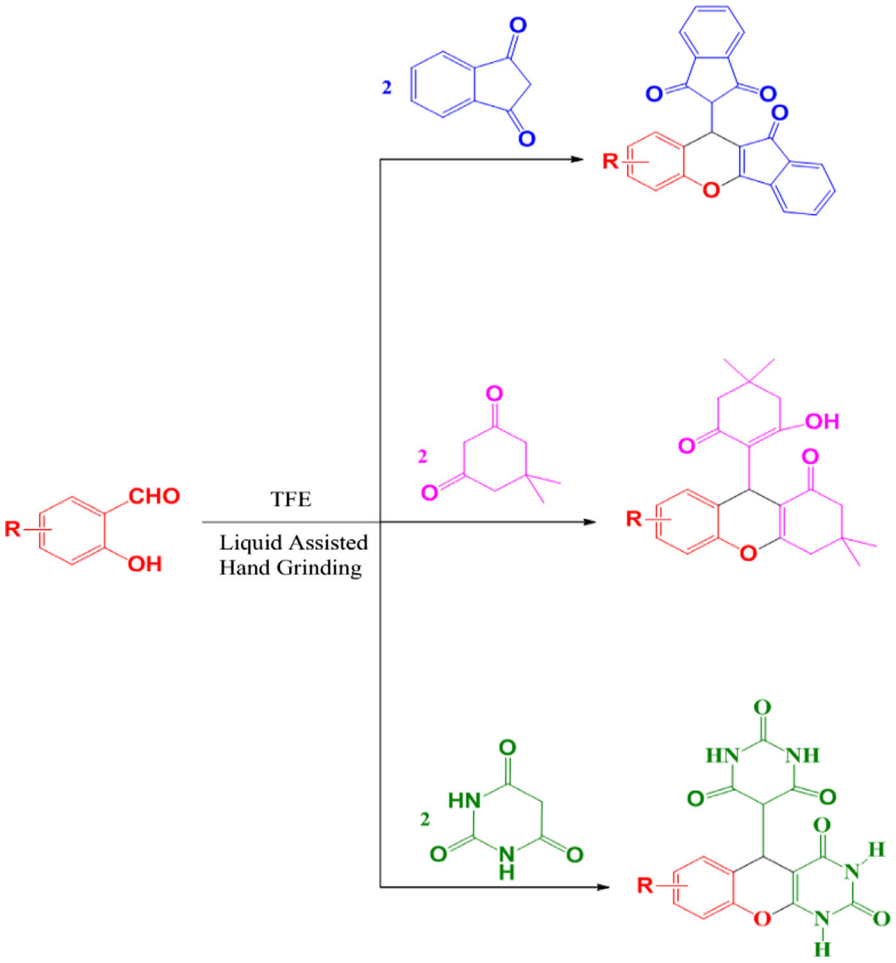
*6-Phenyl-6,6a-dihydroisoindolo[2,1-a]quinazoline-5,11-dione (**4a**) (Table 6, Entry 1)* ^{1}H NMR (300 MHz, $CDCl_3$): δ 6.13 (s, 1H), 7.28–8.33 (m, 13 H); ^{13}C NMR (75 MHz $CDCl_3$) δ 69.3, 119.2, 120.1, 120.9, 121.8, 125.8, 123.6, 126.7, 128.9, 129.5, 133.4, 134.6, 138.2, 139.8, 163.3, 166.4 ppm.

*6-(4-Methyl)-6,6a-dihydro-isoindolo[2,1-a]quinazoline-5,11-dione (**4c**) (Table 6, Entry 3)* ^{1}H NMR (300 MHz, $CDCl_3$):δ 2.40 (s, 3H), 6.17 (d, $J = 7.6$ Hz, 1H), 6.28 (s, 1H), 7.20–7.28 (m, 4H), 7.40–7.58 (m, 4H), 7.91 (d, $J = 7.6$ Hz, 1H), 8.07–8.18 (m, 2H) ppm; ^{13}C NMR (75 MHz $CDCl_3$): δ 21.0, 71.7, 120.0, 124.1, 124.9, 125.2, 129.1, 129.4, 130.0, 130.1, 131.8, 132.2, 133.4, 133.9, 134.9, 136.7, 138.2, 138.9, 164.0, 164.9 ppm.

Results and discussion

Initially, we performed multicomponent synthesis of chromenes by the reaction of salicylaldehyde and 1,3 dicarbonyl compounds using TFE via a liquid-assisted grinding (LAG) method (Scheme 1).

In preliminary optimization experiments, we examined whether grinding under neat or under LAG condition is more effective by reacting salicylaldehyde (1 mmol) and 1H-indene-1,3(2*H*)-dione. Initially, the reactants were placed in an agate mortar and ground gently with a pestle under neat condition at ambient temperature (25–28 °C) (Table 1, entry 1). It was observed that the reaction produces a sticky solid mass after 10 min, which involves intermediate adduct, unreacted starting materials and a trace amount of the desired product, as observed by TLC. The reason behind the incomplete reaction was that, as some unreacted starting materials were trapped inside the solid mass, further reaction became sluggish under neat condition. Fortunately, an addition of a small amount of TFE into the same reaction mixture accelerated the rate of formation of the desired product to a great extent. It was observed that the added TFE could dissolve a major part of the solid



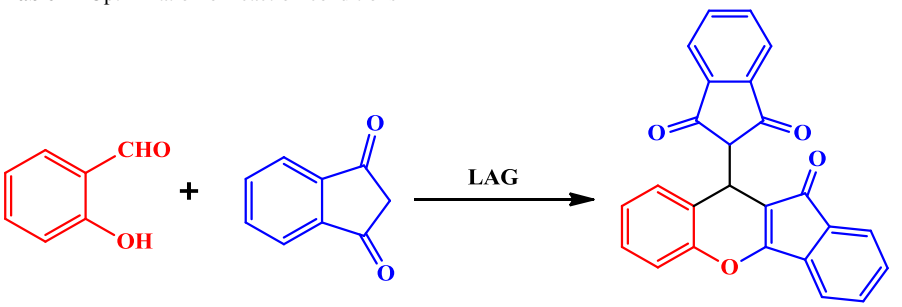
Scheme 1 Synthesis of chromene scaffolds using TFE under LAG method

intermediates and, thereby, release the trapped starting materials to facilitate their complete conversion to the desired product.

It is well known that liquid-assisted grinding often brings out better results than "dry" grinding [50]. Therefore, we used various common organic solvents for LAG, and the percentage of isolated yields of the desired product was analyzed to find out the solvent of choice (Table 1, entries 2–7). In this regard, 0.5 mL of TFE was found to be the most suitable in terms of yield and rate of the reaction (Table 1, entry 8).

In order to further show the efficiency of our protocol, a comparison with available reported protocols in the literature was carried out (Table 2). The data showed that the superior efficiency, environment compatibility and practical applicability of our protocol in comparison to reported procedures.

Table 1 Optimization of reaction conditions



Entry	Solvent	Time (min)	Yield[a] %
1	Neat	10	20
2	CH_3CN	10	20
3	EtOAc	10	20
4	H_2O	10	25
5	Methanol	10	30
6	Ethanol	10	32
7	2-Propanol	10	32
8	**TFE**	**08**	**88**

Reaction conditions: salicylaldehyde (1 mmol) and 1H-indene-1,3(2*H*)-dione, TFE (0.5 mL) at R.T. (25–28 °C)

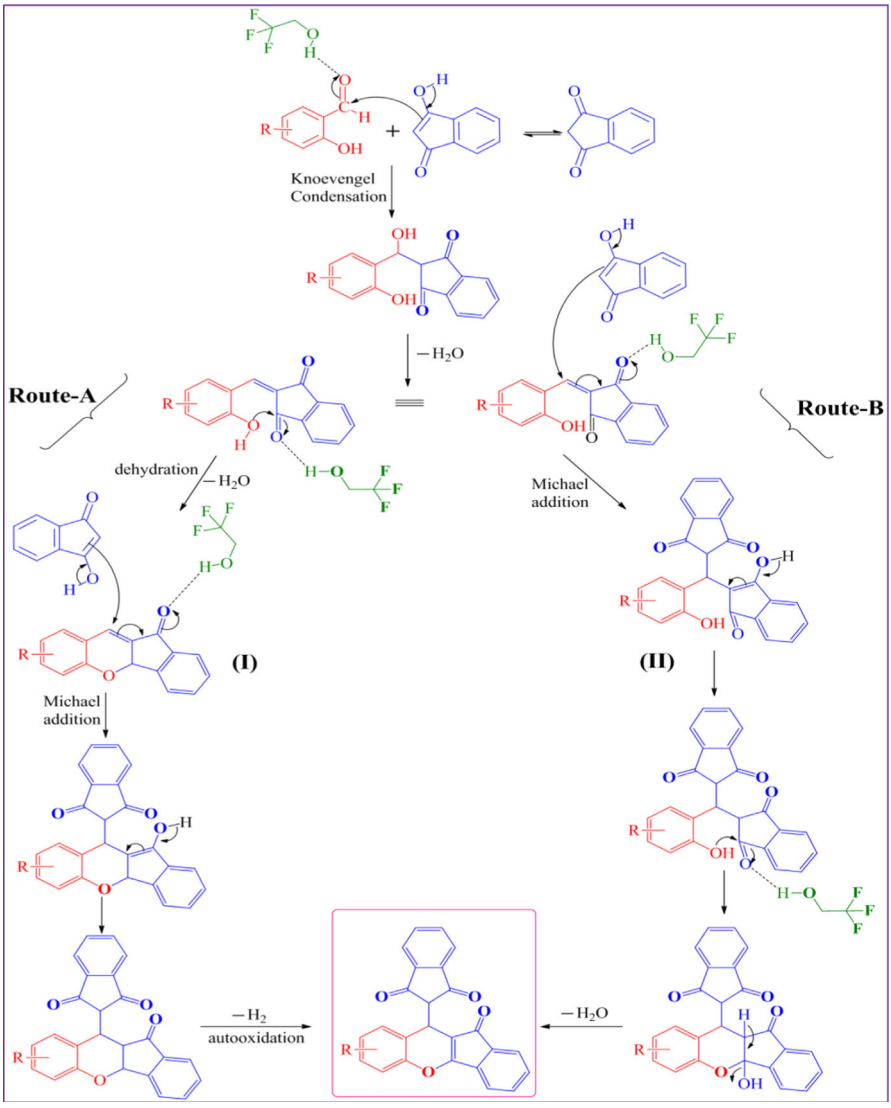
[a]Isolated yields

Table 2 Comparison of the efficiency of TFE/LAG method with reported procedures

Entries	Catalyst	Reaction conditions	Time (min)	Yield (%)	References
1	I_2	Ethanol/reflux	30	85	[37]
2	Sulfamic acid	Ethanol/water	60	92	[39]
3	TFE	Grinding/RT	08	88	This work

The plausible mechanism for the formation of chromenes is depicted in Scheme 2. The mechanism involves two plausible reaction pathways (Route A and Route B). TFE acts as a Brønsted acid and plays a significant role in increasing the electrophilic character of the salicylaldehyde. In addition, the polar transition state of the reaction could be well stabilized by the high ionizing solvent TFE. The first step involves the formation of a Knoevengel adduct via Knoevengel condensation of salicylaldehyde and 1H-indene-1,3(2*H*)-dione. The adduct undergoes cyclization via dehydration to form intermediate-I, which further undergoes Michael-type attack by a second molecule of 1H-indene-1,3(2*H*)-dione followed by auto-oxidation to furnish the final product.

The proposed mechanism is also supported by ^{1}H NMR analysis of intermediate-I (see ESI, Fig. 1,). In the ^{1}H NMR spectrum, a remarkable singlet at δ 2.40 ppm for methine proton confirms the cyclization of the product. Also, an olefinic proton (> C=CH–Ar) appears at δ 9.68 ppm. The eight protons of the aromatic ring



Scheme 2 Plausible mechanism for the synthesis of chromenes

appears as multiplets between δ 7.02 and δ 8.10 ppm. From NMR analysis, it is concluded that synthesis of chromenes follows the **Route A type** mechanism.

To test the generality of this method, a series of substituted salicylaldehydes were treated with 1,3-dicarbonyl compounds such as 1H-indene-1,3(2H)-dione, dimedone and barbituric acid under optimized reaction conditions. It was observed that the salicylaldehydes containing electron-donating (Table 3, entries 3 and 6) as well as electron-withdrawing groups (Table 3, Entries 2, 5 and 9) participated in the reaction uniformly in terms of the product yields. The method was also found to be

Table 3 Synthesis of chromene scaffolds using TFE under LAG method

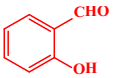
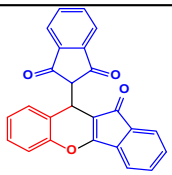
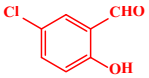
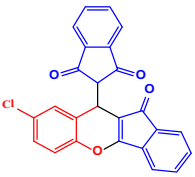
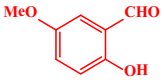
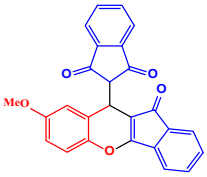
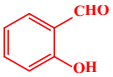
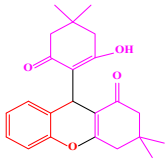
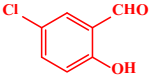
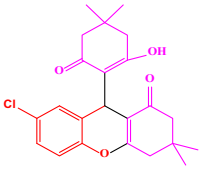
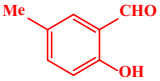
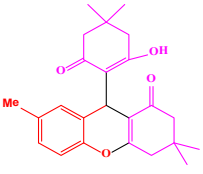
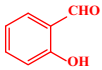
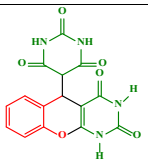
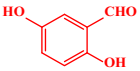
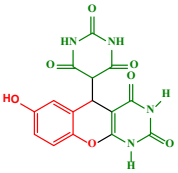
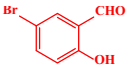
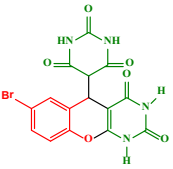
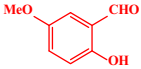
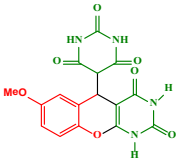
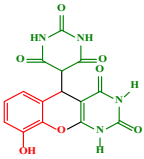
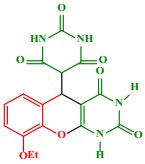
Sr. No.	Salicylaldehydes	Product	Time (min)	Yield[a] (%)	M.P. (°C) [L][b]
1.		 **3a**	11	88	176-178 [178-180][37]
2.		 **3b**	12	87	177-178 [176-178][37]
3.		 **3c**	11	87	198-200 [199-201][37]
4.		 **3d**	12	88	203-205 [205-206][38]
5.		 **3e**	12	87	229-231 [230-232][38]
6.		 **3f**	13	85	209-210 [210-211][38]

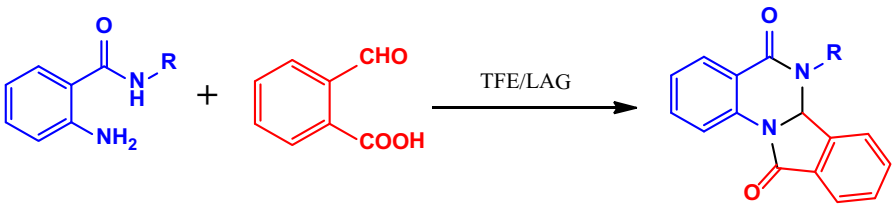
Table 3 continued

Sr. No.	Salicylaldehydes	Product	Time (min)	Yield[a] (%)	M.P. (°C) [L][b]
7.		 **3g**	11	86	220-221 [220-222][39]
8.		 **3h**	12	85	232-234 [234-235][39]
9.		 **3i**	13	85	240-242 [240-241][39]
10.		 **3j**	12	86	> 300 [> 300][39]
11.		 **3k**	14	84	240-241 [244-245][39]
12.		 **3l**	12	80	282-284 [282-284][39]

Reaction conditions: salicylaldehydes (1 mmol), dicarbonyl compounds (2 mmol), TFE (0.5 mL)

[a]Isolated yield

[b]Reference



Scheme 3 Synthesis of isoindolo[2,1-*a*]quinazolines

equally suitable for stearically hindered salicylaldehydes (Table 3, Entries 11 and 12).

With these satisfactory and encouraging results in hand, we extended synthetic applicability of the TFE-LAG method for one-pot three-component synthesis of bioactive isoindolo[2,1-*a*]quinazolines (Scheme 3). The isoindolo[2,1-*a*]quinazolines have been synthesized using I_2 in ionic liquid [51], Zr $(DS)_4$ in water [52], nano-CuO powder [53], *β*-cyclodextrin [54], Baker's yeast [55], montmorillonite K-10 [56], and acetic acid [57].

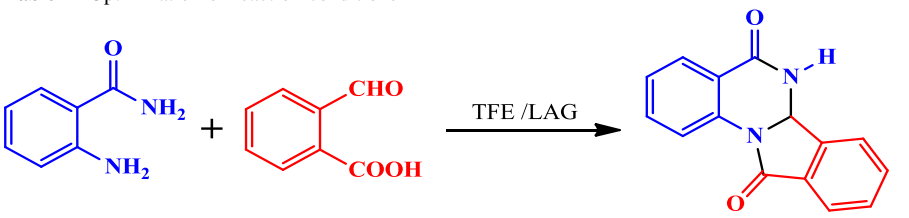
In an initial endeavor, we performed the reaction between an equimolar mixture of 2-aminobenzamide and 2-carboxybenzaldehyde under the TFE-LAG method at ambient temperature. The progress of the reaction was monitored by TLC. The best results were obtained when 0.5 mL of TFE was used as catalyst (Table 4).

To have a better understanding of the performance of our catalytic system, the effectiveness of the TFE-LAG method was also compared to that of catalysts reported previously. The results are listed in Table 5. Overall, the yields obtained from the TFE-LAG method are quite close to those from other reported methods.

As excellent results were obtained by the TFE-LAG method, we focused the scope of this protocol towards the synthesis of different isoindolo[2,1-*a*]quinazolines by applying various substituted 2-aminobenzamides (Table 6). The reaction displayed high functional group tolerance and proved to be the best method for the synthesis of isoindolo[2,1-*a*]quinazolines. It was observed that 2-aminobenzamide with methyl and methoxy substituents gave higher yields of the products due to higher nucleophilicity (Table 6, Entries 3 and 4), whereas halogen substituents offer slightly lower yield of the corresponding products (Table 6, Entries 5 and 6). The reactions with electron-withdrawing nitro groups gave slightly lower yields under the optimized conditions (Table 6, Entries 7 and 8). Sterically hindered 2-aminobenzamides well tolerate the reaction and offer good product yield (Table 6, Entry 9). Cyclic saturated 2-aminobenzamide offered excellent yield due to high nucleophilicity (Table 6, Entry 10). Moreover, other substituted 2-aminobenzamides were the most suitable substrates for the reaction (Table 6, Entries 11 and 12).

As the product is separated from the reaction mixture, workup involves simple filtration for the isolation of the product (Table 6, Entry 4a), the structure of which was confirmed by spectral techniques such as IR, ^{1}H NMR, ^{13}C NMR and mass analysis (see ESM, Figs. 2–5).

Table 4 Optimization of reaction conditions



Entries	Solvent	Conditions	Time (min)	Yield[a] (%)
1	TFE	25 °C	60	60
2	TFE	80 °C	80	82
3	TFE	Grinding	10	90
4	Ethanol	80 °C	60	20

Reaction conditions: 2-aminobenzamide (1 mmol), 2-carboxybenzaldehyde (1 mmol), TFE (0.5 mL)

[a]Isolated yields

Table 5 Reported catalytic systems for the synthesis of isoindolo[2,1-*a*]quinazolines

Entries	Catalyst	Reaction conditions	Time (min)	Yield[a] (%)	References
1	I_2	Ionic liquid/80 °C	60	90	[51]
2	Zr $(DS)_4$	Water/RT	60	90	[52]
3	Nano CuO powder	Water/reflux	600	86	[53]
4	*β*-cyclodextrin	Water/microwave	10	70	[54]
5	Baker's yeast	THF/ultrasound	120	84	[55]
6	Montmorillonite K-10	Ethanol/reflux	900	72	[56]
7	Acetic acid	110 °C	45	90	[57]
8	**TFE**	**Grinding**	**10**	**90**	**This work**

Reaction conditions: 2-aminobenzamide (1 mmol), 2-carboxybenzaldehyde (1 mmol)

[a]Isolated yields

The infrared spectrum of the product depicted a broad signal at 3448 cm^{-1} for N–H stretching vibrations of the cyclic amide group, whereas the amide carbonyl stretching vibrations of the pentacyclic and hexacyclic rings were observed at 1725 and 1678 cm^{-1}, respectively. The weak signals at 2925 and 3061 cm^{-1} due to C–H stretching vibrations of the aromatic group were observed. In the ^{1}H NMR spectrum, a remarkable sharp singlet for methine carbon was observed at δ 6.25 ppm. The remaining protons of the aromatic ring resonated between δ 7.20 and δ 8.06 ppm. The N–H proton shows a broad peak at δ 9.24 ppm. In ^{13}C-NMR spectrum, characteristic methine carbon in the cyclized product appeared at δ 67.5 ppm. All aromatic carbon atoms observed at δ 119.9 to 140.6 ppm. Two carbonyl groups noticed at δ 164.3 and 165.0 ppm. The molecular ion peak at *m/z* 250 (M+) corresponds to the molecular weight of the compound.

Table 6 Synthesis of isoindolo[2,1-*a*]quinazolines

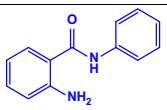
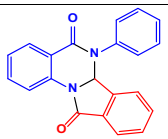
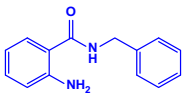
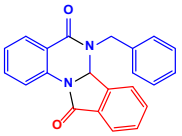
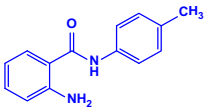
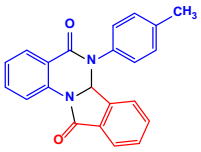
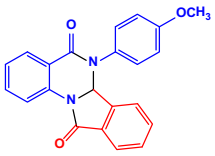
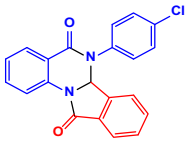
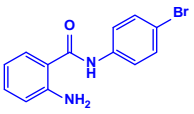
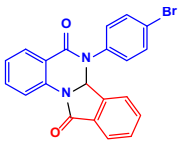
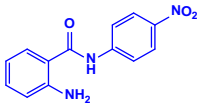
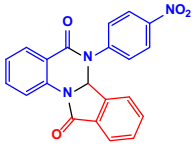
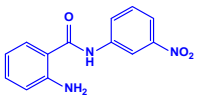
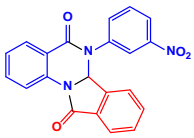
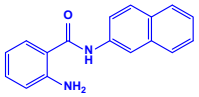
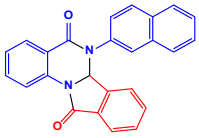
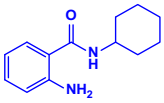
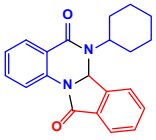
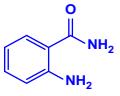
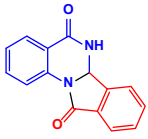
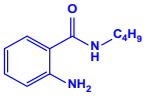
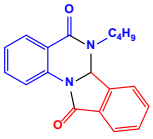
Sr. No.	2-aminobenzamides	Product	Time (min)	Yield[a] (%)	MP. (°C) [L][b]
1.		 **4a**	07	90	180-182 [184-185][53]
2.		 **4b**	08	88	155-157 [155-157][53]
3.		 **4c**	08	91	199-201 [199-200][54]
4.		 **4d**	09	91	137-139 [135-137][53]
5.		 **4e**	10	86	184-185 [186-187][53]
6.		 **4f**	10	86	115-117 [117-119][54]

Table 6 continued

Sr. No.	2-aminobenzamides	Product	Time (min)	Yield[a] (%)	MP. (°C) [L][b]
7.		 **4g**	09	85	235-237 [241-243][57]
8.		 **4h**	10	86	216-218 [217-219][57]
9.		 **4i**	10	85	166-168 [169-170][54]
10.		 **4j**	09	85	151-153 [152-154][53]
11.		 **4k**	07	88	278-280 [280-281][51]
12.		 **4l**	09	87	139-141 [141-143][51]

Reaction conditions: 2-aminobenzamides (1 mmol), 2-carboxybenzaldehyde, (1 mmol), TFE (0.5 mL)

[a]Isolated yield

[b]Reference

Conclusion

In conclusion, we have developed green and efficient synthesis of chromenes and isoindolo[2,1-a]quinazolines under the TFE-LAG method. Here, TFE acts as Brønsted acid and plays a significant role in increasing the electrophilic character of the aldehyde. The reaction proceeds via Knoevengel condensation followed by Michael-type addition. The TFE-LAG method brings better results than dry grinding. In addition to this, the method has notable outcomes such as conversion under mild reaction conditions and operational simplicity with good yields.

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