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ARTICLE

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A rapid and quantitative synthesis of xanthene derivatives using sulfonated graphitic carbon nitride under ball-milling

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Abstract

A truly green, solventless, and fast protocol for the quantitative preparation of biologically active xanthene derivatives with 10 mol% sulfonated graphitic carbon nitride (Sg—CN) as a heterogeneous acidic catalyst was developed. Excellent yields, low cost, solvent-free conditions, high catalytic activity, reusability of the catalyst, simple workup, and heterogeneous carbon-based catalyst make the present method particularly attractive from a green chemistry perspective.

1 | INTRODUCTION

Today, the reinforcement of novel and greener approach for constructing heterocyclic compounds has continuously gained much attention in recent years [1]. Xanthene and its derivatives are the most promising biologically active compounds with great pharmaceutical applicability [2–6]. Furthermore, these compounds have been utilized in local dyes [7], laser technologies [8,9], and photodynamic therapy [10]. Structures of some bioactive xanthenes (rhodamine, amsacrine, and RO67-4853) [11] are depicted in Figure 1.

Due to the potential importance of xanthenes, various approaches for the symmetrical and unsymmetrical synthesis of related organic compounds, such as boric acid [12], PVSA [13], sulfamic acid [14], nano-TiO_2 [15], core/shell Fe_3O_4@GA@isinglass [16], [Et_3NSO_3H]Cl [17], and amberlyst-15 [18] reported. Some drawbacks of these reported procedures have been harsh reaction conditions, unsatisfactory yields, toxic solvents, tedious workup, and harmful catalysts. From the green chemistry perspective, it has become essential to develop sustainable methods for efficient and clean synthesis of xanthene derivatives as bioactive compounds. In this sense, the mechanochemistry approach for organic transformations has received significant attention. Ball-milling is a grinding system that is an excellent method for some organic reactions, such as oxidations, reductions, polymerization, and metal-catalyzed bond-forming reactions [19–23]. This mechanochemical technique has more advantages, such as solvent-free reaction, high yields, low reaction time, and considerably pure products.

Nanomaterial-based catalysts play a significant role in various chemical transformations [24–26], due to the selectivity, low toxicity, good stability, simple surface functionalization, and magnetic separation [27,28]. Carbon materials such as activated carbons, graphene, and graphitic carbon nitride are environmentally acceptable and cheap nanomaterials, combining efficient use of energy and resources in heterogeneous catalysis [29–33]. Sulfonated carbon nitride (Sg—CN) has recently emerged a new organo-sulfonated heterogeneous acid catalyst prepared via simple calcination of urea [34] and sulfonation of resulting carbon nitride, respectively. Sg—CN has been recently used for the synthesis of biodiesel in the efficient yield [35–37].

Engaged in the development of green chemistry and following our ongoing research in green organic synthesis [38–40] herein, we investigate the ball-milling approach in the presence of Sg—CN (10 mol%) as an environmentally friendly protocol for the multicomponent synthesis of xanthene derivatives under solvent-free conditions (Scheme 1).

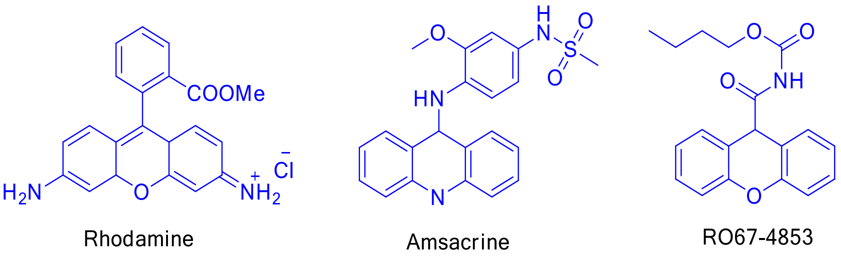
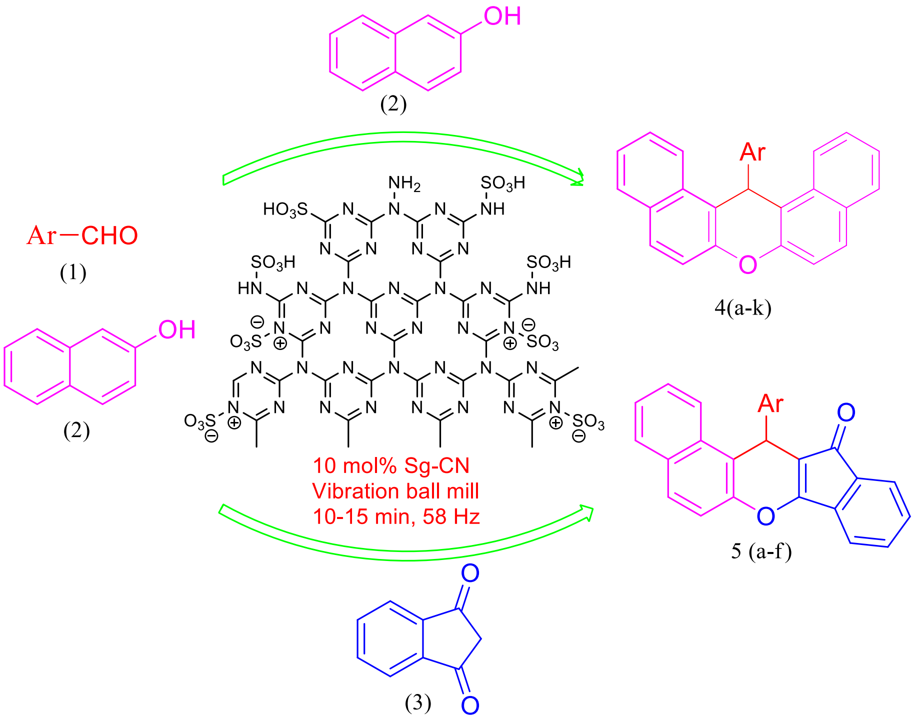


FIGURE 1 Structures of some bioactive xanthene derivatives



SCHEME 1 One-pot synthesis of xanthene derivatives using Sg—CN in a vibration ball-mill

2 | EXPERIMENTAL SECTION

2.1 | Reagents and instrumentation chemicals

Aldehydes, β-naphthol, 1,3-indandione, and other chemicals were purchased from Merck chemical company and used without further purification. The Retsch MM 400 ball mill instrument was used.

2.2 | Synthesis of 12-aryl-8,9,10,12-tetrahydrobenzo[a]xanthene-11-one derivatives (4)

β-Naphthol (2 mmol), aldehyde (1 mmol), and Sg—CN (10 mol%) were ball-milled according to Table 1 at r.t. After reaction completion, the appropriate solvents (ethanol or ethyl acetate, 10 ml) was added, and the nanocatalyst was filtered or centrifuged. The reused nanocatalyst was washed with ethanol and ethyl acetate, and it was dried at 60°C for 4 h and reused for neat runs.

2.3 | Some selected data

2.3.1 | 14-Phenyl-14*H*-dibenzo[a,j]xanthene (**4a**)

IR (KBr): ν = 3058, 2961, 1624, 1426, 1348 cm^{-1}. ^1H NMR (500 MHz, $CDCl_3$) δ = 5.74 (s, 1H, CH), 7.04–7.22 (m, 2H), 7.33–7.46 (m, 4H), 7.76–8.02 (m, 3H). ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 50.89, 117.70, 123.69, 124.91, 126.25, 127.01, 128.07, 128.24, 128.43, 128.84, 129.77, 131.40, 131.49, 144.74, 147.73, 163.97.

2.3.2 | 14-(4-Chlorophenyl)-14*H*-dibenzo[a,j]xanthene (**4b**)

IR (KBr): ν = 3059, 3022, 2954, 2869, 1594, 1466, 1373 cm^{-1}. ^1H NMR (500 MHz, $CDCl_3$) δ = 5.65 (s, 1H, CH), 6.62–6.68 (2H, m), 7.15–7.21 (2H, m), 7.31–7.47 (2H, m), 7.68–8.01 (m, 2H). ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 50.23, 116.06, 117.04, 123.15, 123.46, 125.25, 127.40, 128.67, 129.44, 129.66, 131.01, 131.57, 147.77, 152.13, 166.35.

TABLE 1 Optimization of the reaction condition for the synthesis of **5a**

Entry	Amount Sg—CN (Mol%)	Conditions/temperature (°C)	Time (min)	Yield (%)[a]
1	No catalyst	EtOH/reflux	60	0
2	No catalyst	H_2O/reflux	70	0
3	No catalyst	Ball-milling	60	0
4	Sg—CN (5 mol%)	EtOH/reflux	45	40
5	Sg—CN (10 mol%)	H_2O/reflux	40	35
6	Sg—CN (5 mol%)	CH_3CN/reflux	50	40
7	Sg—CN (5 mol%)	EtOH/Ball-milling/r.t	30	55
8	Sg—CN (5 mol%)	Ball-milling/r.t	12	75
9	Sg—CN (10 mol%)	Ball-milling/r.t	10	99
10	Sg—CN (15 mol%)	Ball-milling/r.t	10	99
11	g-C_3N_4 (10 mol%)	Ball-milling/r.t	10	21

[a] Isolated yields.

2.3.3 | 14-(*p*-tolyl)-14*H*-dibenzo[a,j]xanthene (**4c**)

IR (KBr): $\nu = 3063, 1595, 1369\ cm^{-1}$. ^1H NMR (500 MHz, $CDCl_3$) $\delta = 2.57$ (s, 3H, CH_3), 5.69 (s, 1H, CH), 6.62–6.69 (m, 2H), 7.15–7.21 (m, 2H), 7.32–7.47(m, 3H), 7.68–7.99 (m, 3H). ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 20.30, 51.06, 116.96, 117.83, 123.76, 124.90, 126.97, 128.38, 128.77, 129.53, 129.66, 131.36, 131.50, 137.30, 147.65, 154.04, 163.94.

2.3.4 | 14-(4-Nitrophenyltrophenyl)-14*H*-dibenzo[a,j]xanthene (**4d**)

IR (KBr): $\nu = 3063, 1596, 1300\ cm^{-1}$. ^1H NMR (500 MHz, $CDCl_3$) $\delta = 5.76$ (s, 1H, CH), 7.08–7.23 (m, 3H), 7.37–7.47 (m, 5H), 7.81–7.82 (d, J = 7.0 Hz, 2H), 7.97–8.01 (d, J = 7.0 Hz, 2H). ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 57.06, 11,698, 117.71, 123.71, 124.89, 126.26, 127.0, 128.26, 128.38, 128.50, 128.84, 131.39, 131.49, 145.09, 147.78, 165.63.

2.4 | General procedure for the synthesis of 5

β-Naphthol (1 mmol), aldehyde (1 mmol), 1,3-indandione (3, 1 mmol), and Sg—CN (10 mol %) was ball-milled for the appropriate time (Table 1) at room temperature. After reaction completion, the suitable solvents (ethanol or ethyl acetate, 10 ml) were added, and the nanocatalyst was filtered or centrifuged. The reused nanocatalyst was washed with ethanol and ethyl acetate, and it was dried at 60°C for 4 h and reused for neat runs. After that, the crude product was further purified by recrystallization from EtOH to afford the pure product.

2.5 | Selected compounds

2.5.1 | 13-Phenylbenzo[f]indeno[1,2-b]chromen-12(13*H*)-one (**5a**)

IR (KBr): ν = 3065, 2956, 2945, 1698 (C═O), 1608 cm^{-1}. ^1H NMR (500 MHz, $CDCl_3$) δ = 5.06 (s, 1H, CH), 7.02–7.05 (m, 1H), 7.11–7.19 (m, 1H), 7.46–7.60 (m, 2H), 7.84–7.93 (m, 3H), 8.02–8.08 (m, 3H), 8.48 (d, *J* = 6.8 Hz, 2H). ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 48.86, 102.89, 111.15, 117.39, 118.07, 121.35, 123.42, 124.82, 124.91, 128.48, 128.67, 128.76, 128.86, 129.65, 130.17, 130.27, 131.56, 132.11, 134.06, 134.13, 135.24, 154.25, 195.22.

2.5.2 | 13-(3-Chlorophenyl)benzo[f]indeno[1,2-b]chromen-12(13*H*)-one (**5b**)

IR (KBr): ν = 3058, 2951, 2901, 1686 (C═O), 1607 cm^{-1}. ^1H NMR (500 MHz, DMSO-d_6) δ = 5.12 (s, 1H, CH), 6.63–6.94 (t, *J* = 8.0 Hz, 4H), 6.93–6.97(d, *J* = 8 Hz, 1H), 7.13–7.16 (d, *J* = 7.0 Hz, 1H), 7.57–7.62 (t, *J* = 7.5 Hz, 3H), 7.73–7.75 (d, *J* = 8.0 Hz, 1H), 8.02–8.16 (d, *J* = 8 Hz, 2H), 8.10–8.12 (d, *J* = 7.2 Hz, 2H). ^{13}C NMR (DMSO-d_6, 125 MHz): δ = 48.85, 115.79, 116.79, 118.62, 123.60, 124.06, 125.82, 126.64, 130.02, 131.27, 133.48, 135.64, 139.17, 142.27, 144.90, 146.47, 147.50, 148.62, 149.81, 151.78, 152.94, 154.49, 156.33, 158.57, 159.53, 164.67, 188.30.

3 | RESULTS AND DISCUSSION

The Sg—CN nanocatalysts was synthesized using our previous procedure [24]. The morphology and elemental analysis of the Sg—CN characterized by SEM and EDX mapping. As shown in Figure 2A, Sg—CN displayed an approximate sheet-like porous structure with slight agglomeration, and the sheets are stacked on each other. Compositional analysis of Sg—CN using EDX spectroscopic studies and elemental analyses confirmed the presence of C, O, N, and S elements (Figure 2B). The result indicated that the SO_3H groups were successfully supported on the surface of g-C_3N_4.

The effect of SO_3H groups on the crystalline properties of g-C_3N_4 was studied using large-angle XRD analysis (Figure 3). The g-C_3N_4 is a crystalline carbon nitride polymer, which supports two crystalline peaks at 27.4° with high intensity and 13.1° with low intensity due to the in-plane ordering of s-triazine units and the periodic stacking of layers of aromatic heterocyclic units, respectively. The XRD patterns of g-C_3N_4 and g-C_3N_4-SO_3H (Sg—CN) were nearly similar, which means that the structure of g-C_3N_4 was not destroyed during the functionalization process.

Boehm titration is used to quantify the amount of sulfonic acid groups on the g-C_3N_4 before and after the

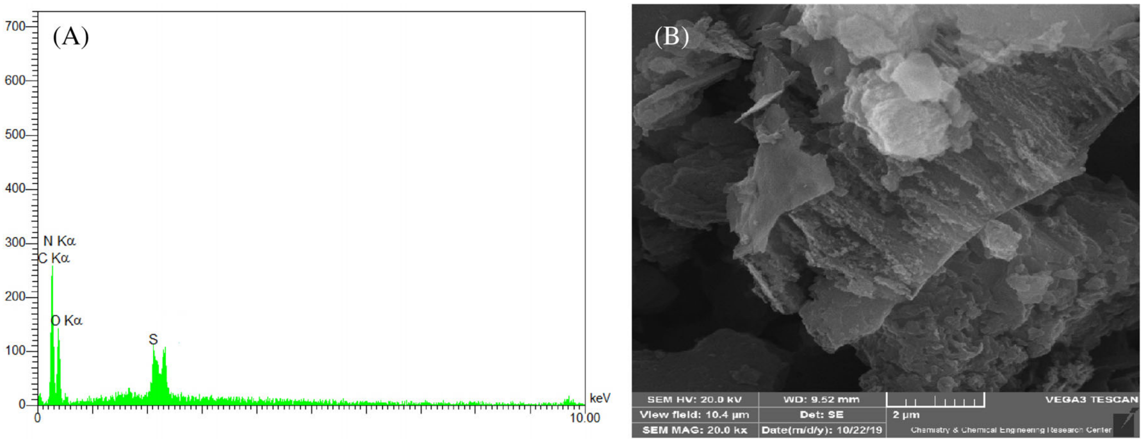


FIGURE 2 (A) EDX mapping of Sg—CN and (B) SEM images Sg—CN

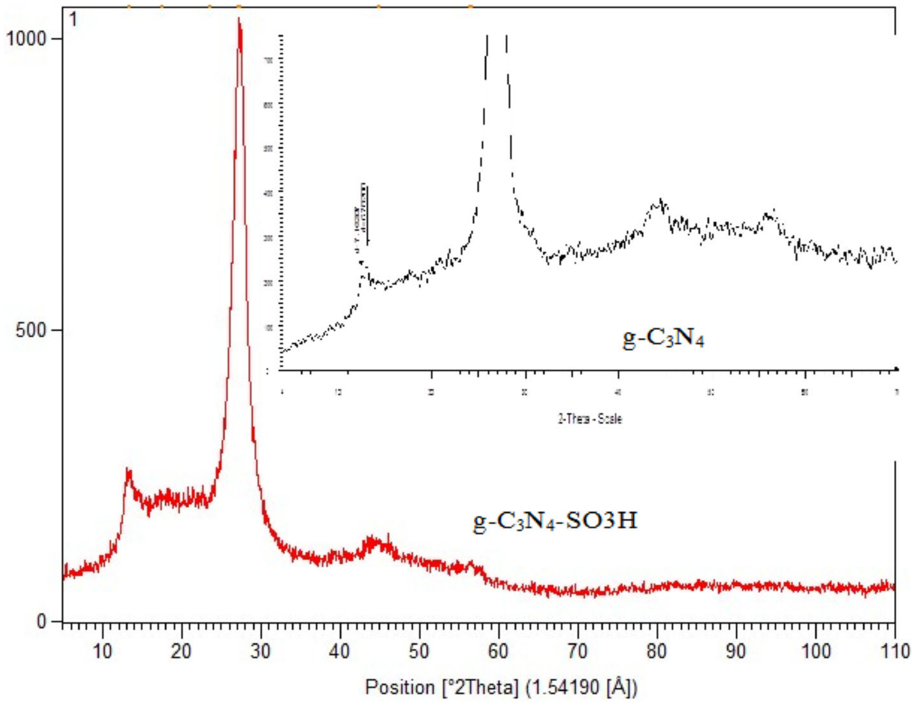
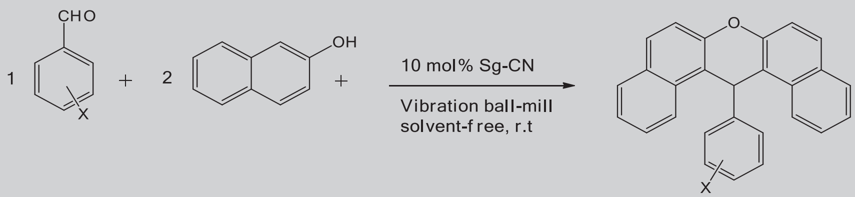
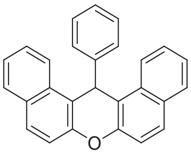
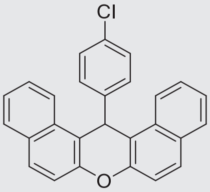
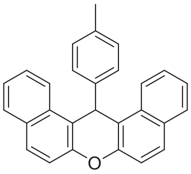
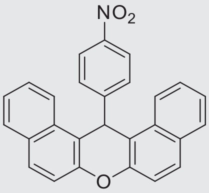
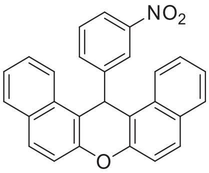
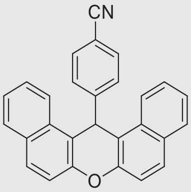
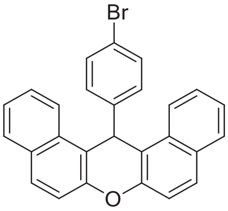


FIGURE 3 XRD patterns for g—C_3N_4 and g—C_3N_4-SO_3H

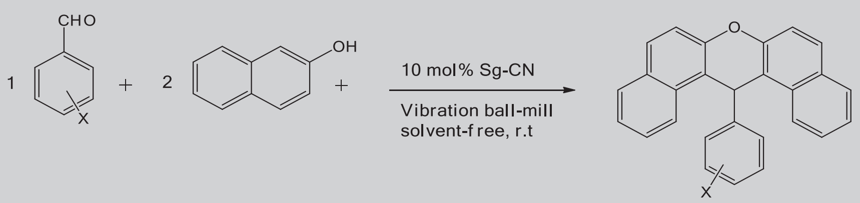
TABLE 2 One-pot preparation 14-aryl-14*H*-dibenzo[a,j]xanthenes

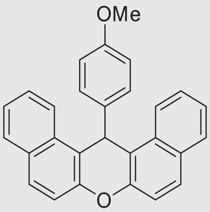
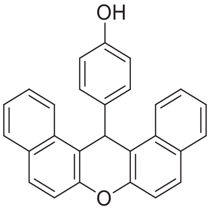
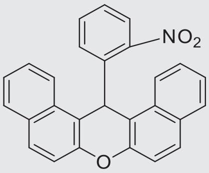
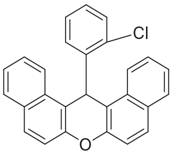


Entry	X	Product	Time	Yield[a]	M.P	
					Found	Reported
4a	H		15	99<	186–184	186–184[41]
4b	4-Cl		10	99<	290–288	288–290[41]
4c	H		20	99<	230–228	227–229[41]
4d	4-NO_2		10	99<	328–326	324–327[41]
4e	3-NO_2		15	99<	222–220	222–220[41]
4f	4-CN		10	99<	338–340	339–337[41]
4g	4-Br		10	99<	303–304	303–305 [41]

(Continues)

TABLE 2 (Continued)



Entry	X	Product	Time	Yield[a]	M.P	
					Found	Reported
4h	4-OMe		20	99<	307–308	308–310[41]
4i	4-OH		20	99<	261–262	262–260[41]
4j	2-NO2		15	99<	293–294	294–292[41]
4f	2-Cl		15	99<	216–218	216–218[41]

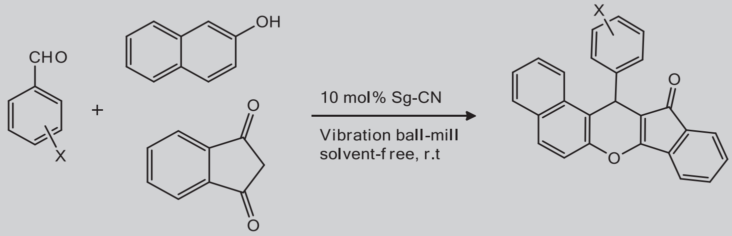
[a] Isolated yields.

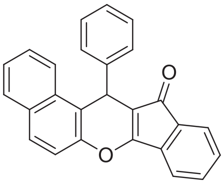
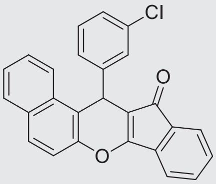
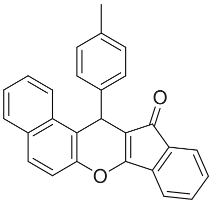
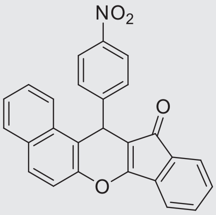
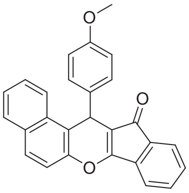
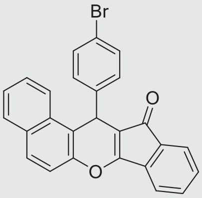
reaction. The Sg—CN (0.50 g) was added into standard NaOH solutions (0.01 M, 50 ml) and stirred at room temperature for 4 h. Then, it was back titrated with HCl (0.01 mol/L). Based on the titration experiments, the percentage loading of —SO3H groups over the Sg—CN surface is about 5.1 mmol/g which corresponds to about 14.7% of loading, in good agreement with the atomic percentage of sulfur (15.9%) determined from EDX analysis and elemental analysis (14.1%) for Sg—CN. Furthermore, reused Sg—CN were subject to elemental analysis, and the percentage of sulfur was nearly the same as fresh Sg—CN (13.9%), confirming the stability of our system.

After preparation and characterization of Sg—CN, to optimize the reaction parameter, the model condensation reaction of β-naphthol (2, 2 mmol) with 4-chlorobenzaldehyde (1 mmol) under various conditions including, catalyst-free, in the presence of Sg—CN catalyst, in different solvents under solvent-free condition. Additionally, the temperature and amount of Sg—CN in terms of yield and time period (Table 1) were optimized.

Notably, without catalyst, no desired product was obtained (Table 1, entries 1–3), while the yield of the model products improved by employing Sg—CN as a catalyst (Table 1, entries 4–10). Additionally, we also optimized the amount of Sg—CN (5.0, 10.0, and 15.0 mol%) in the model reaction, and the maximum efficiency in the presence of 10 mol% of the Sg—CN was obtained (Table 1, entry 9). It was found that when the amount of Sg—CN was further increased to 15 mol%, no further increase in yield was detected. Furthermore, the temperature parameters were investigated, and results were shown in Table 1 (entries 4–10).

TABLE 3 One-pot preparation 13-phenylbenzo[f]indeno[1,2-*b*]chromen-12(13*H*)-ones



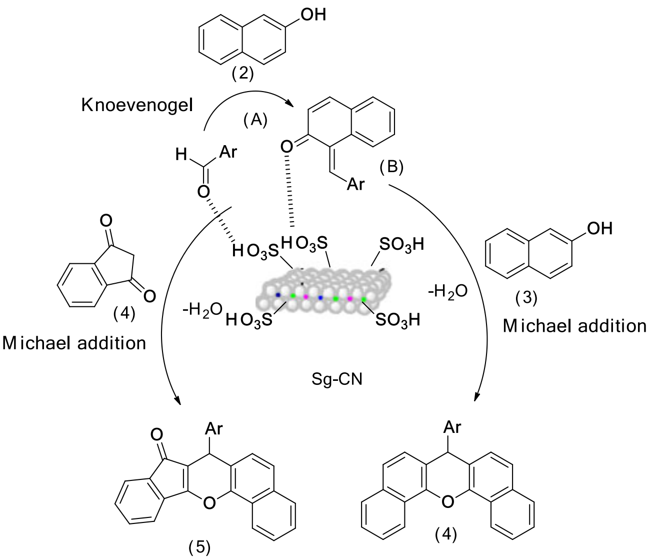
Entry	X	Product	Time	Yield[a]	M.P	
					Found	Reported
5a	H		10	>99	230–229	229–231[42]
5b	3─Cl		20	>99	211–210	210–212[42]
5c	4─Me		20	>99	196–197	169–198[43]
5d	4─NO_2		10	>99	265–267	265–267[43]
5e	4─OCH_3		20	>99	299–230	299–230[42]
5f	4─Br		10	>99 > 99	254–255	254–256[42]

[a]Isolated yields.

Among various conditions, kneading ball-milling in the presence of 10 mol% Sg─CN at r.t under solvent-free conditions was the most efficient route for the greener synthesis of **5b** in quantitive yield at 10 min (Table 1, entry 9). Further reactions were performed under optimized conditions to synthesize two series of xanthene

TABLE 4 Comparative study of the catalytic efficiency of Sg—CN

Catalyst	Catalyst load (Mol%)	Solvent	Temp. (°C)	Time (min)	Yield (%)
TMSCl[1]	100	MeCN	Reflux	420	95[41]
DBSA[2*]	10	H_2O, ultrasound	30	60	89[40]
$HCl0_4$—SiO_2	10	Solvent-free	140	180	32[42]
$SbCl_3$—SiO_2	10	Solvent-free	120	50	93[43]
TBAHS	10	Dioxane, H_2O	Reflux	210	88[44]
Sg—CN	10	Solvent-free	r.t	10	99 this work



SCHEME 2 The proposed mechanism in the presence of Sg—CN

TABLE 5 Reusability of Sg—CN

No of runs	1	2	3	4
Yield	99	99	99	98

derivatives in good to excellent yields (Tables 2). The isolated products were fully characterized by FT-IR, ^{1}HNMR, and ^{13}CNMR spectroscopy. All the compounds had physical and spectroscopic data identical to those known xanthenes (Table 3)[41-44].

A comparative study of the catalytic ability of sulfonated graphitic carbon nitride (Sg—CN) for catalytic synthesis of **4a** with the previously reported procedure is illustrated in Table 4. Ball milling with Sg—CN acts as an effective and green procedure concerning reaction time and yield (Table 4).

A plausible mechanism for the formation of pyran-annulated heterocycles is depicted in Scheme 2. Reaction initiates the activation of a carbonyl group of the aromatic aldehyde by Sg—CN, which generated the intermediate (A). Next, the nucleophilic attack of 2-naphthol on the activated carbonyl group due to the acidic nature of Sg—CN, resulting in Knoevenagel intermediate B formation. Michael addition by the second 2-naphthol or 1,3-indandione molecule resulting from the desired xanthene derivatives after water elimination.

In continuation, the recycling and reuse of the Sg—CN were evaluated in the model reaction (**5b**), and the results are shown in Table 5. When the reaction was completed, hot ethanol was added, and the catalyst was recovered with centrifuge or filtrations. Then filtered catalyst was washed with ethanol (10 ml) and ethyl acetate (10 ml) and dried at 60°C. The recovered Sg—CN was further used for three consecutive cycles, and the obtained yields are presented in Table 5.

4 | CONCLUSION

In summary, a green and straightforward procedure for the synthesis of various xanthene derivatives was reported. High yields of products, easy workup procedure, short reaction time, no need for organic solvents, and high atom economy are the key features of this green ball-milling procedure. Additionally, the Sg—CN nanocatalyst is a separable and stable heterogeneous catalyst in this system without activity loss for four runs.

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DATA AVAILABILITY STATEMENT

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

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