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ORIGINAL PAPER

Sonosynthesis of furan-2(5*H*)-ones using nanosilica-tethered polyhedral oligomeric silsesquioxanes

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Abstract

An easy and rapid method for the synthesis of 3,4,5-substituted furan-2 (5*H*)-ones has been achieved through a three-component reaction of aniline, dialkyl acetylenedicarboxylate, and aromatic aldehydes using nanosilica-tethered polyhedral oligomeric silsesquioxanes with eight branches of 3-aminopropyltriethoxysilane as catalyst under ultrasonic irradiations. We reported a class of organic–inorganic hybrid material based on nanosilica-tethered polyhedral oligomeric silsesquioxanes with eight branches of 3-aminopropyltriethoxysilane (nanosilica @APTPOSS). The catalyst has been characterized by proton nuclear magnetic resonance (^{1}H NMR), scanning electronic microscopy, energy dispersive X-ray, DLS (dynamic light scattering), and thermal gravimetric analysis. The high efficiency, short reaction times, simple workup, reusability of the catalyst up to six runs without considerable loss of activity, using the sonochemical procedure as green process, and an alternative energy source are some benefits of this method.

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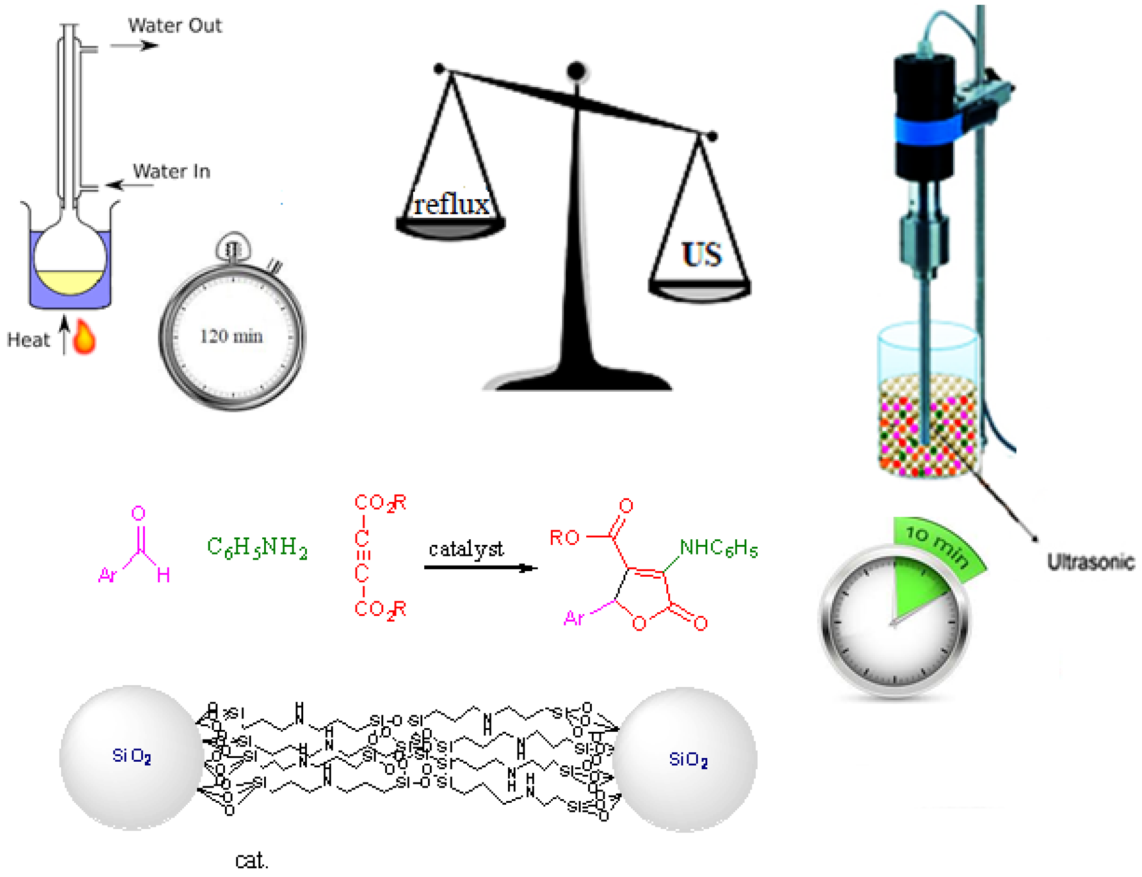
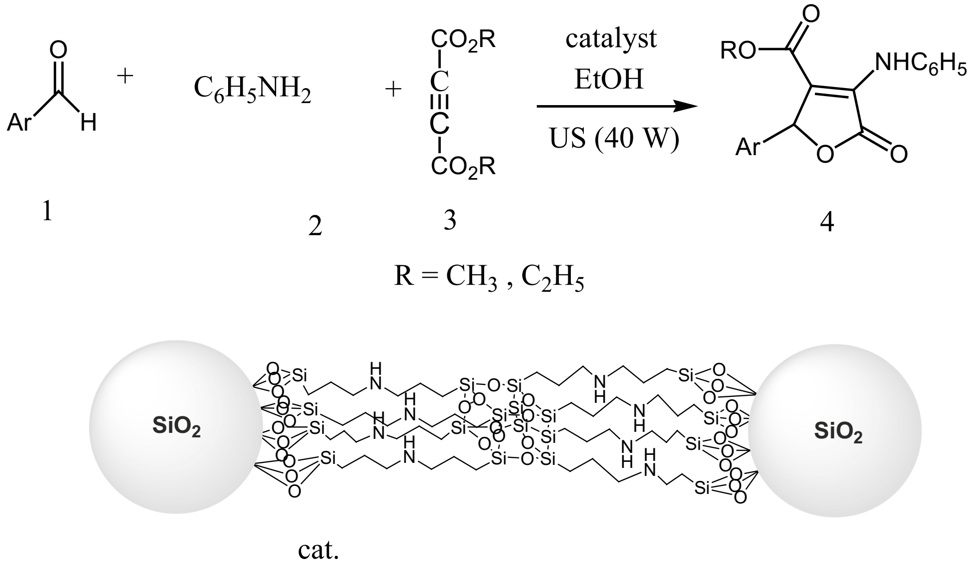
Graphic abstract



Keywords Organic–inorganic hybrid · Nanocatalyst · Furan-2 (5*H*)-ones · Ultrasonic conditions · One-pot

Introduction

Furan-2(5*H*)-ones represent biological properties such as anti-cancer [1], antimicrobial [2], antifungal [3], anti-ulcer [4], and anti-HIV [5]. Hence, seeking easy and brief techniques for the preparation of Furan-2(5*H*)-ones is a significant subject. Recently, reports have been developed on synthesis of furan-2 (5*H*)-ones using the catalysts such as cyclodextrin [6], $Al(HSO_4)_3$ [7], $(CH_3CH_2CH_2CH_2)_4N(HSO_4)$ [8], HCOOH [9], Maltose [10], $SnCl_2$ [11], and Vitamin B12 [12]. Despite the use of these ways, there remains adequate purpose to offer a new way for an efficient method to achieve such systems. The use of highly efficient, economic, and retrievable catalysts, with low or nil toxicity, is required from the green chemistry viewpoint. The surface of nanoparticles (NPs) can be modified through loading by desirable functionalities such as polyhedral oligomeric silsesquioxanes (POSS). Silsesquioxane is an organosilicon compound with the chemical formula $[RSiO_{3/2}]_n$ (R = H, alkyl, vinyl, aryl, alkoxy), including of an inorganic core of oxygen and silicon has size ~0.45 nm in diameter [13, 14]. In continuation of our works on synthesis of nanoparticles [15–18], a series of polyhedral oligomeric silsesquioxanes with eight branches of 3-aminopropyltriethoxysilane (APTPOSS) has been anchored on the surface of colloidal silica nanoparticles. In the current study, we investigated an easy and rapid method for the synthesis of 3,4,5-substituted furan-2 (5*H*)-ones through a three-component reaction of aniline, dialkyl acetylenedicarboxylate, and aromatic aldehydes using nanosilica-tethered polyhedral oligomeric silsesquioxanes with eight branches of 3-aminopropyltriethoxysilane as catalyst under ultrasonic irradiations (Scheme 1). Recently, several nanocatalysts have been utilized for the preparation of organic compounds under ultrasonic conditions [19, 20]. The ultrasound approach decreases times and increases yields of products by creating the activation energy in microsurroundings [21, 22]. Cavitation process generates high temperature and pressure in the microsurroundings which causes a disturbed current in the liquid and elevated mass transfer [23, 24]. Compared



Scheme 1 Synthesis of 3,4,5-substituted furan-2(5*H*)-ones under ultrasound irradiations

to conventional heating which creates thermal energy in the macrosystem, ultrasound irradiation is able to activate numerous reactions by the activation energy in microenvironment [25].

Experimental

All organic materials were purchased commercially from Sigma-Aldrich and Merck and were used without further purification. A multiwave ultrasonic generator (Sonicator 3200; Bandelin, MS 73, Germany), equipped with a converter/transducer and titanium oscillator (horn), 12.5 mm in diameter, operating at 20 kHz with a maximum power output of 200 W, was used for the ultrasonic irradiation. The ultrasonic generator automatically adjusted the power level. CHN compositions were measured by Carlo ERBA Model EA 1108 analyzer. The thermogravimetric analysis (TGA) curves are recorded using a V5.1A DUPONT 2000. To investigate the morphology and particle size of the synthesis structures, NPs, FE-SEM images, and EDS spectrum of the products were visualized by a Sigma ZEISS, Oxford Instruments Field Emission Scanning Electron Microscope.

Preparation of octakis(3-chloropropyl)octasilsesquioxane (Cl-POSS)

3-Chloropropyltrimethoxysilane (80 g, 0.403 mol) was added to a stirred mixture of methanol (1800 mL) and concentrated hydrochloric acid (90 mL). The reaction mixture was stirred for 5 weeks at room temperature. Then, the resultant solution was filtered and dried to give a white solid in 42% yield. Anal. Calcd for $Si_8O_{12}C_{24}H_{48}Cl_8$ (1036.9): C, 27.80; H, 4.67. Found: C, 27.74; H, 4.60. IR (KBr; ν, cm^{-1}): 2953, 1439, 1104,810. ^{1}H NMR (400 MHz, $CDCl_3$; δ, ppm): 0.81 (m, 2H), 1.88 (m, 2H), 3.54 (m, 2H).

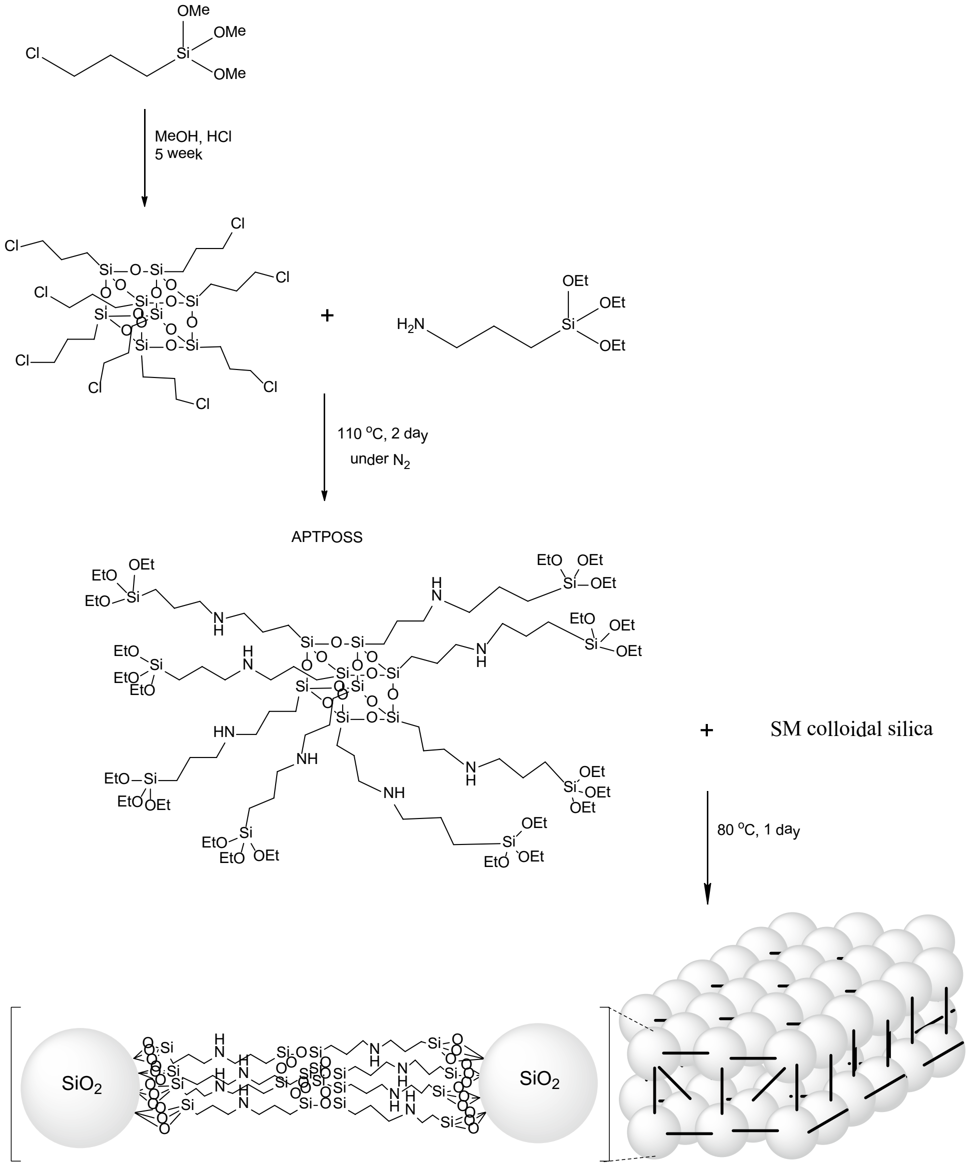
Preparation of octakis[3-(3-aminopropyltriethoxysilane)propyl]octasilsesquioxane (APTPOSS)

Firstly, 2 mmol (2.07 g) of Cl-POSS was added in 20 mmol (4.43 g) of 3-aminopropyltriethoxysilane and was transferred to a round-bottom flask under N_2 atmosphere. The mixture was heated in an oil bath at 110 °C for 2 days. After the reaction was complete, the mixture was cooled to room temperature and the mixture was filtered and washed with acetone and methanol to wash the additional reactants. Finally, the resultant pale brown precipitates were dried in a vacuum oven at 70 °C for 12 h (36% yield). Anal. Calcd for $C_{96}H_{224}N_8O_{36}Si_{16}$ (2516): C, 45.82; H, 8.97; N, 4.45. Found: C, 45.56; H, 8.67; N, 4.32. IR (KBr; ν, cm^{-1}): 2924, 1633, 1112, 1025.

Preparation of nanosilica @APTPOSS

In a typical procedure, 0.3 mL of colloidal silica nanoparticles (LUDOX SM colloidal silica 30 wt.% suspensions in H_2O) was diluted in 2 mL of deionized water. After that 0.6 g (0.24 mmol) of APTPOSS was dispersed in 3 mL of deionized water by ultrasonic vibration for 15 min. Then, the suspension was added slowly during 1 h to above solution. The mixture was kept at 80 °C for one day (Scheme 2). Finally, the nano-colloidal silica-attached APTPOSS was separated by centrifugation and washed with acetone and ethanol for several times, and then, the mixture was dried in vacuum at 50 °C (28% yield) [26, 27].

Scheme 2 Preparation of nano-colloidal silica @APTPOSS



General procedure for the synthesis of furan-2(5H)-ones

A mixture of aldehyde (2 mmol), aniline (2 mmol), dialkyl acetylenedicarboxylate (2 mmol), and 14 mg of nano-colloidal silica@APTPOSS in ethanol (15 mL) was sonicated at 40 W power for the appropriate times (monitored by TLC). After completion of the reaction, the reaction mixture was diluted in CH_2Cl_2; the catalyst was isolated by simple filtration, and the heterogeneous catalyst was recovered. The CH_2Cl_2 was evaporated, and the solid separated out was filtered and was washed with ethanol to get pure product.

Spectra data of new compounds

Ethyl 2-(4-chlorophenyl)-2,5-dihydro-5-oxo-4-(phenylamino)furan-3-carboxylate (4j)

White powder; m.p.: 181–184 °C; IR (KBr): $\bar{\nu}=3220$, 2922, 1687, 1596, 1239, 11368 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): $\delta=1.20$ (t, 3 H, $J=7.2$ Hz, CH_3), 4.05 (m, 2H, $J=7.2$ Hz, OCH_2), 5.73 (s, 1H, benzylic), 7.12–7.52 (m, 9H, ArH), 8.95 (br s, 1H, NH) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): $\delta=168.95$ ($\underline{C}OO$), and 165.04 ($\underline{C}OOCH_2$), 156.07 ($\underline{C}$-NH of aromatic), 137.85 ($\underline{C}$-NH alkene), 135.22 ($\underline{C}$ of aromatic), 134.96 ($\underline{C}$–Cl of aromatic), 133.22 ($\underline{C}$H of aromatic), 129.16

($\underline{C}$H of aromatic), 126.19 ($\underline{C}$H of aromatic), 123.22 ($\underline{C}$ of alkene), 122.54 ($\underline{C}$H of aromatic), 114.48 ($\underline{C}$H of aromatic), 60.92 (O$\underline{C}$H$_2$), 52.18 ($\underline{C}$H of benzylic), 14.32 ($\underline{C}$H$_3$) ppm; Anal. Calcd for $C_{19}H_{16}ClNO_4$: C, 63.78; H, 4.51; N, 3.91; Found: C, 63.70; H, 4.45; N, 3.84.

Ethyl 2,5-dihydro-2-(4-nitrophenyl)-5-oxo-4-(phenylamino)furan-3-carboxylate (4k)

White powder; m.p.: 250–252 °C; IR (KBr): $\bar{\nu} = 3305$, 2927, 2843, 1722, 1665, 1508, 1339, 1225 cm^{-1}; ^{1}H NMR (400 MHz, CDCl$_3$): $\delta = 1.22$ (t, 3 H, $J = 7.2$ Hz, CH$_3$), 4.07 (m, 2H, $J = 7.2$ Hz, OCH$_2$), 5.82 (s, 1H, benzylic), 7.22–8.32 (m, 9H, ArH), 9.12 (br s, 1H, NH) ppm; ^{13}C NMR (100 MHz, CDCl$_3$): $\delta = 165.2$ ($\underline{C}$OO), and 163.2 ($\underline{C}$OOCH$_3$), 155.4 ($\underline{C}$-NH of aromatic), 144.2 ($\underline{C}$-NO$_2$ of aromatic), 142.1 ($\underline{C}$-NH alkene), 134.1 ($\underline{C}$ of aromatic), 130.2 ($\underline{C}$H of aromatic), 129.0 ($\underline{C}$H of aromatic), 127.10 ($\underline{C}$H of aromatic), 124.75 ($\underline{C}$H of aromatic), 120.72 ($\underline{C}$ of alkene), 113.9 ($\underline{C}$H of aromatic), 61.3 (OCH$_2$), 52.1 (benzylic), 14.32 ($\underline{C}$H$_3$) ppm; Anal. Calcd for $C_{19}H_{16}N_2O_6$: C, 61.95; H, 4.38; N, 7.61; Found: C, 61.82; H, 4.20; N, 7.50.

Results and discussion

The preparation steps of nanosilica-tethered polyhedral oligomeric silsesquioxanes with eight branches of 3-aminopropyltriethoxysilane have been described in Scheme 2. In the first step, Cl-POSS was synthesized by the hydrolysis of 3-chloropropyltrimethoxysilane under acidic conditions. Afterward, reaction of 3-aminopropyltriethoxysilane (20 mmol) with Cl-POSS (2 mmol) yields APTPOSS (36% yield). Then, reaction of nanosilica with APTPOSS afforded nanosilica @APTPOSS.

Figure 1 shows the ^{1}H NMR spectra for the octakis(3-chloropropyl)octasilsesquioxane (Cl-POSS) in CDCl$_3$. The NMR spectra of Cl-POSS are consistent with expected results. The ^{1}H NMR spectra of Cl-POSS display a signal at δ 0.81 ppm due to the methylene protons (–Si–$\mathbf{\underline{CH_2}}CH_2CH_2$Cl) and signals at δ 1.88 ppm and 3.54 are, respectively, assigned to the resonance of the methylene protons (–Si–CH$_2$C$\mathbf{\underline{H_2}}$CH$_2$Cl) and (–Si–CH$_2$CH$_2$C$\mathbf{\underline{H_2}}$Cl). The ^{1}H NMR reveals that Cl-POSS was successfully prepared.

Figure 2 shows FE-SEM image of nano-colloidal silica @ APTPOSS (nanocatalyst). It is observed that the particles are strongly aggregated and glued with very large and continuous aggregates easily observed.

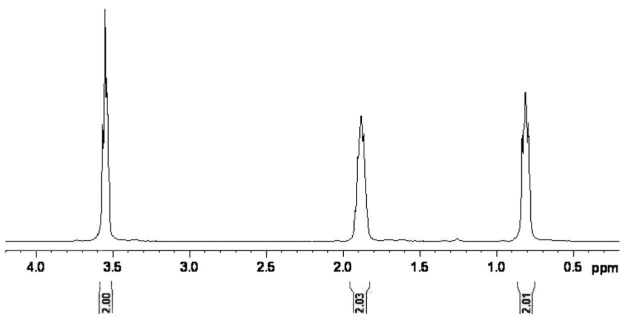


Fig. 1 ^{1}H NMR spectrum of Cl-POSS

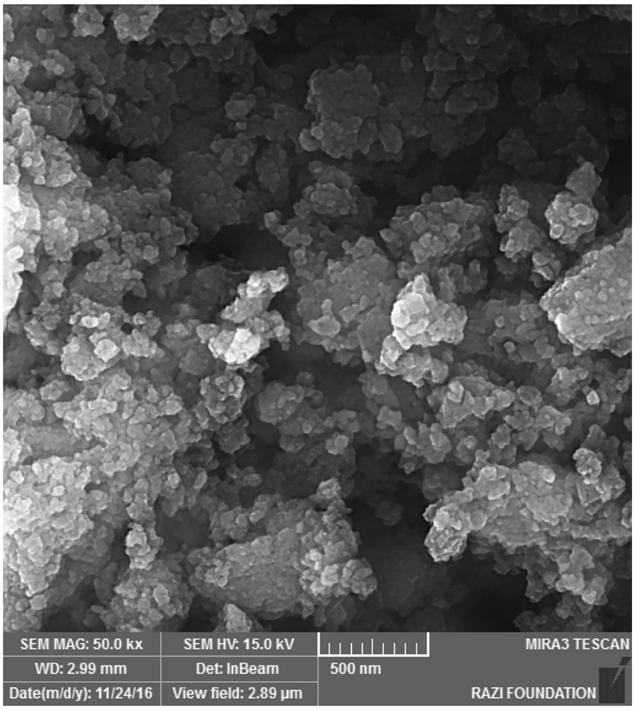


Fig. 2 FE-SEM image of nano-colloidal silica @APTPOSS

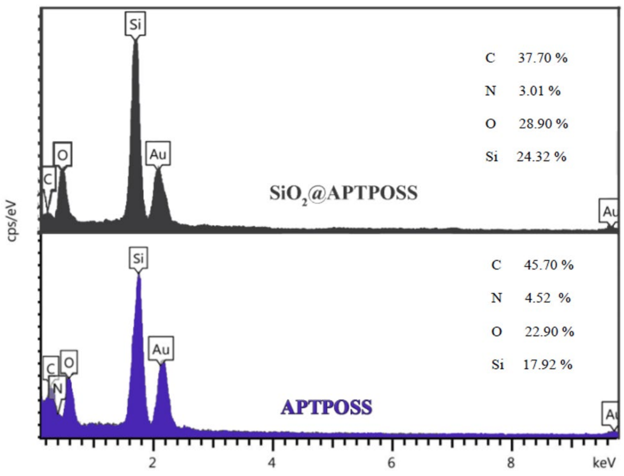


Fig. 3 Energy dispersive spectroscopy for APTPOSS and nano-colloidal silica @APTPOSS

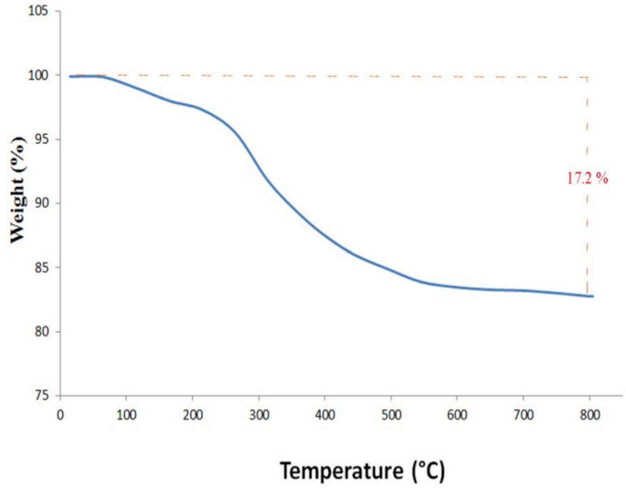


Fig. 4 TGA of nano-colloidal silica @APTPOSS

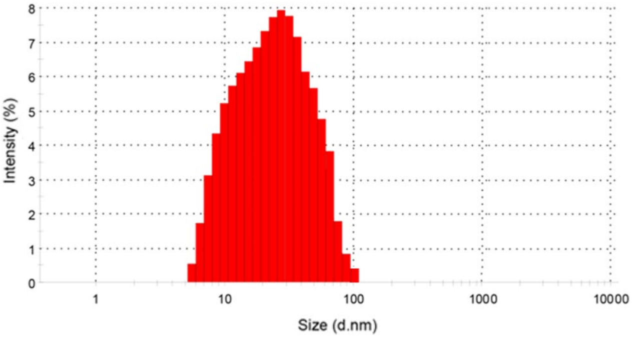


Fig. 5 DLS (dynamic light scattering) of nano-colloidal silica @ APTPOSS

The components of the APTPOSS and silica @APTPOSS were analyzed using EDS (Fig. 3). EDS confirmed the presence of C (45.70%), N (4.52%), O (22.90%), and Si (17.92%) in APTPOSS, and the higher intensity of Si peak compared with C peak in the nanocatalyst indicates SiO_2 is loaded with APTPOSS.

Thermogravimetric analysis (TGA) evaluates the thermal stability of the nano-colloidal silica-tethered APTPOSS. The curve shows a weight loss lower than 210 °C related to desorption of physically adsorbed water, while a weight loss at the temperature from 210 to 560 °C could be due to the decomposition of organic spacer attaching to the silica nanoparticles. Thus, the nanocatalyst was stable up to 210 °C (Fig. 4).

In order to study the size distribution of nanocatalyst, DLS (dynamic light scattering) measurements of the nanoparticles are shown in Fig. 5. The dispersion for DLS analysis (2.5 g nanocatalyst at 50 mL ethanol) was prepared using an ultrasonic bath (60 W) for 30 min.

We commenced our investigation by testing the reaction of dimethyl acetylene dicarboxylate, aniline, and 4-chlorobenzaldehyde as model reaction for the synthesis of furan-2 (5H)-ones. To obtain the ideal reaction conditions for the synthesis of compound **4a**, we studied the diverse catalysts and solvents which are shown in Table 1. Screening of different catalysts such as $ZrOCl_2·8H_2O$, $BF_3·Et_2O$, nano-CeO_2, nano-Fe_3O_4, APTPOSS, and nano-colloidal silica@APTPOSS revealed nano-colloidal silica@APTPOSS (14 mg) as the most effective catalyst to perform this reaction under ultrasonic irradiation (40 W) (Table 1). In this research, ultrasound irradiation is used as green and complementary techniques for preparation of furan-2 (5H)-ones. When the catalysis was performed under ultrasound irradiations, the reaction rate increased considerably. Longer reaction times were required under conventional heating and assistance of ultrasound irradiation did improve the yields of the reactions and shortening the reaction times. The possible explanation for the positive association of irradiation is that the ultrasonic irradiation could increase the number of active cavitation bubbles and the size of the individual bubbles, both of which are predictable to result in higher maximum collapse temperature and accelerated respective reaction. We explored the feasibility of the reaction by choosing some representative substrates (Table 2). It has been considered that better yields are achieved with substrates having electron-withdrawing groups. In addition, we examined isatin instead of benzaldehydes in the reaction, but we could not find considerable amount of the title product from isatin. This reaction could not be observed when the isatins were used as a substrate.

We also checked reusability of nano-colloidal silica @APTPOSS as an efficient catalyst; its reusability was achieved by the reaction of dimethyl acetylene dicarboxylate, aniline, and 4-chlorobenzaldehyde and 14 mg of nano-colloidal silica@APTPOSS under optimized conditions. After completion of the reaction, the catalyst was washed with water and acetone and was used with new substrates under the same conditions. The results showed that the nano-colloidal silica @APTPOSS can be reused several times (yields 95 to 93%) in Fig. 6.

A proposed mechanism for the formation of **4** is shown in Scheme 3. An initial condensation of dimethyl acetylenedicarboxylate and aromatic amine generated intermediate **I**. Subsequently, the reaction of aromatic aldehyde and intermediate **I** and then cyclodehydration produced furan-2(5H)-ones. In this mechanism, nano-colloidal silica @APTPOSS acts as a highly efficient and green catalyst activating the C=O, COO groups for better reaction with nucleophiles. It is assumed that catalytically active site of catalyst contains SiO_2 that acts as a Lewis acid and APTPOSS (−NH−) that acts as a Lewis basic.

Table 1 Optimization of reaction conditions using different catalysts under different conditions[a]

Entry	Solvent (conditions)	Catalyst	Time (min)	Yield[d] %
1	EtOH (reflux)	No catalyst	800	Trace
2	EtOH (reflux)	$ZrOCl_2 \cdot 8H_2O$ (2 mol%)	400	40
3	CH_3CN (reflux)	$BF_3 \cdot Et_2O$ (2 mol%)	600	35
4	DMF (reflux)	Nano-CeO_2 (4 mol%)	350	40
5	EtOH (reflux)	Nano-Fe_3O_4 (4 mol%)	350	28
6	CH_3CN (reflux)	APTPOSS (10 mol%)	120	45
7	DMF (reflux)	Nano-colloidal silica@APTPOSS (14 mg)	120	48
8	H_2O (reflux)	Nano-colloidal silica@APTPOSS (14 mg)	120	54
9	CH_3CN (reflux)	Nano-colloidal silica@APTPOSS (14 mg)	120	59
10	EtOH (reflux)	Nano-colloidal silica@APTPOSS (14 mg)	120	76
11	DMF (US: 40 W)[b]	Nano-colloidal silica@APTPOSS[c] (14 mg)	15	66
12	H_2O (US: 40 W)	Nano-colloidal silica@APTPOSS (14 mg)	15	72
13	CH_3CN (US: 40 W)	Nano-colloidal silica@APTPOSS (14 mg)	10	75
14	EtOH (US: 30 W)	Nano-colloidal silica@APTPOSS (14 mg)	10	85
15	EtOH (US: 40 W)	Nano-colloidal silica@APTPOSS (14 mg)	10	95
16	EtOH (US: 50 W)	Nano-colloidal silica@APTPOSS (14 mg)	10	95
17	EtOH (US: 40 W)	Nano-colloidal silica@APTPOSS (12 mg)	10	89
18	EtOH (US: 40 W)	Nano-colloidal silica@APTPOSS (16 mg)	10	95
19	EtOH (US: 40 W)	–	15	40
20	EtOH (US: 40 W)	Nano-colloidal silica	10	52

[a] 4-Chlorobenzaldehyde (2 mmol), aniline (2 mmol), dimethyl acetylenedicarboxylate (2 mmol)

[b] Ultrasonic irradiation

[c] Nano-colloidal silica@APTPOSS (4 mol% of APTPOSS)

[d] Isolated yield

Table 2 Synthesis of 3,4,5-substituted furan-2(5H)-ones using nano-colloidal silica@APTPOSS (14 mg) under ultrasonic irradiation (40 W) in ethanol

Entry	Aldehyde	R	Product	Time (min)	Yield/[a] %	m.p °C
1	4-Cl-C_6H_4	CH_3	**4a**	10	95	148–151 [11]
2	C_6H_5	CH_3	**4b**	10	92	160–163 [11]
3	4-O-Me-C_6H_4	CH_3	**4c**	15	86	240–243 [11]
4	2-Cl-C_6H_4	CH_3	**4d**	10	92	274–276 [11]
5	4-Br-C_6H_4	CH_3	**4e**	10	95	180–181 [12]
6	4-NO_2-C_6H_4	CH_3	**4f**	10	96	176–179 [10]
7	2-NO_2-C_6H_4	CH_3	**4g**	10	93	200–201 [12]
8	3-NO_2-C_6H_4	CH_3	**4h**	10	91	199–202 [12]
9	4-$CH(CH_3)_2$-C_6H_4	CH_3	**4i**	15	88	202–204 [12]
10	4-Cl-C_6H_4	CH_3CH_2	**4j**	10	92	181–184 [11]
12	4-NO_2-C_6H_5	CH_3CH_2	**4k**	10	92	250–252 [12]

[a] Isolated yield

Conclusion

In conclusion, we demonstrated an efficient method for the synthesis of 3,4,5-substituted furan-2 (5*H*)-ones through a three-component reaction of aniline, dialkyl acetylenedicarboxylate, and aromatic aldehydes using nanosilica-tethered polyhedral oligomeric silsesquioxanes with eight branches of 3-aminopropyltriethoxysilane as catalyst under ultrasound irradiations. The remarkable advantages of this methodology are easy workup, short reaction times, high to excellent product yields, operational simplicity, little catalyst loading, and use of ultrasonic irradiations as a valuable and powerful technology.

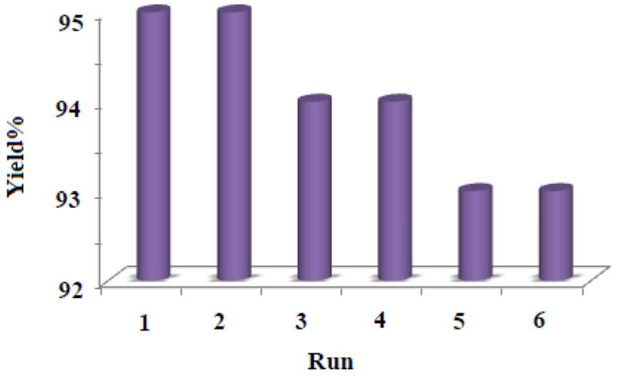
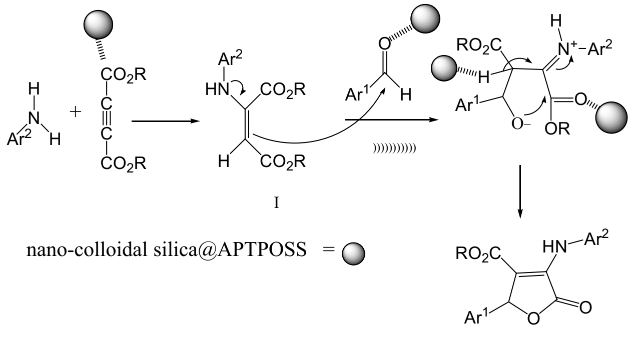


Fig. 6 Reusability of nano-colloidal silica@APTPOSS for the preparation of **4a**



Scheme 3 Possible mechanism for the synthesis of 3,4,5-substituted furan-2(5*H*)-ones in the presence of nano-colloidal silica@APTPOSS

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