

# Grafting of sulphamic acid on functionalized sawdust: A novel solid acid catalyst for the synthesis of 1,8-dioxo-octahydroxanthenes

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## Abstract

A solid acid catalyst bearing sulphamic acid on sawdust (SA@Sawdust) was designed as a novel heterogeneous catalyst. The structure of prepared catalyst was assessed by various spectroscopic techniques such as FT-IR, field emission scanning electron microscopy and energy dispersive X-ray, thermogravimetric analysis, solid-state CP/ MAS <sup>13</sup>C-NMR spectroscopy, and CHNS analysis. The efficiency of prepared catalyst was explored for synthesis of diverse 1,8-dioxo-octahydroxanthene derivatives. The mild reaction conditions are found to be more compatible with a wide range of functional groups. The obtained products could be conveniently separated from the reaction mixture by simple filtration, and the catalyst is recovered and reused for next cycle without significant loss in catalytic efficiency. The alluring features of this synthetic route are operational simplicity, shorter reaction time, high conversion, wide substrate scope, easy work-up procedure, and reusability of catalyst.

#### Graphic abstract



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### Introduction

Nowadays, the challenges for maintaining ecological balance of our planet are among the most complicated and daunting ever faced by society. To protect environment, the research community is highly engaged in facing these challenges fiercely through their systematic improvements and breakthrough innovations. Further, they are trying to create advanced product by generating the libraries of drug-like organic molecules involving complex framework [1]. In order to accelerate the innovations and adoption of green metrics, an implementation of green synthetic methodologies plays an important role in organic synthesis. Such methodologies are the perfect response to these constraints due to its remarkable performance in designing of recyclability process, reduce and valorize the wastes, exceptional synthetic efficiency, use of environmentally benign catalyst, high atom economy, and selection of eco-friendly solvents which are the fundamental aspects of green synthetic methodologies that ultimately come under the roof of green chemistry [2, 3]. Therefore, a concerted and pervasive effort is needed for the development and execution of these methodologies.

Presently, petroleum-derived monomers obtained from fossil resources as raw material are the main source of many polymeric substances. A recent quest towards the manufacture of polymers using renewable bioresources as a raw material is the subject of immense curiosity which elegantly attracted the research community [4]. In manufacture of polymer, researchers are engaged in exploring the use of renewable bioresources instead of fossil resources. Biomass; being renewable bioresource, is exploited as a new feedstock material for manufacturing the polymer is definitely benefited for both the sustainable development as well as environmental protection [5]. Recently, severe efforts have been focused on the use of cellulose as biomassbased polymeric support for designing a variety of heterogeneous catalysts which effectively catalyzes number of synthetic routes [6-8]. In this regards, effort has been made to utilize the sawdust as natural source of cellulose instead of refined cellulose for the catalytic support [9]. Sawdust is a renewable bioresource and easily available natural source of cellulose obtained from wood, owing to presence of number of active sites on its surface. It offers the exceptional opportunities for designing eco-friendly heterogeneous catalyst for mediating significant organic transformations [10, 11]. Our earlier experience with sawdust as catalytic support [12], enthuse us to investigate its compatibility as support material in preparation of heterogeneous catalyst in the synthesis of biologically privileged heterocyclic scaffolds.

Xanthenes are probably one of the most ubiquitous oxygen containing heterocyclic scaffold and also well-known versatile synthons due to the presence of reactive inbuilt pyran ring as core unit in the moiety and also exhibit a broad range of potential applications in the diverse fields [13–18]. Owing to such multifarious biological and pharmacological applications, the numerous synthetic procedures with plethora of catalysts have been investigated for the synthesis of 1,8-dioxo-octahydroxanthenes [22–36]. Among these, most common approach involves the reaction of dimedone and aromatic aldehydes. Nevertheless, most of the reported methods suffer from drawbacks such as tedious catalyst preparation, longer reaction time, high reaction temperature, low yields, excess use of reagents as well as toxic or expensive catalysts. Therefore, the synthesis of medicinally pertinent 1,8-dioxo-octahydroxanthenes using robust heterogeneous catalyst involving easily adaptable protocol is of prime importance. Herein, we report the successfully grafted sulphamic acid on functionalized sawdust (SA@Sawdust) as a novel heterogeneous solid acid catalyst for synthesis of medicinally acclaimed 1,8-dioxo-octahydroxanthenes.

## Experimental

## **General remarks**

Proton and <sup>13</sup>C NMR spectra were recorded on Bruker AC NMR Spectrometer using TMS as an internal standard in CDCl<sub>3</sub>. The chemical shifts ( $\delta$ ) are expressed in ppm. The size and morphology of catalyst were observed by using FESEM of MIRA3 TESCAN microscope with 10 kV of an accelerating voltage. Solid-state CP/MAS <sup>13</sup>C-NMR spectrum was recorded on *JEOL-ECX400* spectrometer under the prescribed operating conditions. FT-IR spectra were measured on Perkin-Elmer Spectrum one FT-IR spectrophotometer. Dimedone, (3-aminopropyl)trimethoxysilane, chlorosulphonic acid and aryl aldehydes were purchased from local supplier and used without further purification.

#### Preparation of sawdust-alumina composite (2)

Fresh and clean sawdust was obtained from local timber industry. It was further washed by excess of distilled water to remove the surface impurities and dried in an oven at 60 °C. The dried sawdust was then pulverized and allowed to pass through 1 mm sieve to get fine powder. The mixture of fine sawdust powder (**1**, 15 g) and  $AlCl_3 \cdot 6H_2O$  (15 g) in water (100 mL) was stirred for 12 h at room temperature. Thereafter, mixture was filtered and the residue was exposed to ammonia and washed frequently with water, dried in vacuum to get sawdust-alumina composite (**2**).

## Preparation of amino-functionalized sawdust (3)

The sawdust-alumina composite (**2**, 10.0 g), (7.0 mL, 40.11 mmol) (3-aminopropyl)trimethoxysilane was added and resulting mixture was refluxed in toluene (40 mL) for 24 h. The reaction mixture was cooled, filtered and washed with toluene (20 mL) and then dried under vacuum to get the amino-functionalized sawdust (**3**). CHN analysis observed: %C 37.62, %H 4.66, %N 2.31; Loading: 0.0824 mmol of amino group per gram of sawdust.

#### Preparation of SA@Sawdust (4)

In 25-mL RB flask, amino-functionalized sawdust (**3**, 5 g) in diethyl ether (20 mL), chlorosulfonic acid (2.33 g, 20 mmol) was added dropwise over 20 min via a syringe at 0 °C. The reaction mixture was stirred at room temperature for 30 min; brown powder was obtained which was again washed with diethyl ether (10 mL). The resultant solid was dried under vacuum at 60 °C for 4 h to get desired SA@Sawdust (**4**). FT-IR (KBr) (cm<sup>-1</sup>): 3347, 3245, 2885, 1228, 1025, 885, 837, 696, 632, 581; <sup>13</sup>C CP-MAS NMR (500 MHz):  $\delta$  105.64 (s, cellulose-C<sub>1</sub>), 89.48-84.27 (d, cellulose-C<sub>4</sub>), 75.56-65.64 (m, cellulose-C<sub>2</sub>, C<sub>3</sub>, C<sub>5</sub>), 56.84 (s, cellulose-C<sub>6</sub>), 43.68 (s, N–CH<sub>2</sub>), 22.32 (s, N–CH<sub>2</sub>–CH<sub>2</sub>–CH<sub>2</sub>), 11.12 (bs, CH<sub>2</sub>–Si); EDAX analysis: %C 19.03, %O 40.81, %A1 12.05, %Si 26.03, %S 2.08; CHNS analysis observed: %C 27.93, %H 5.09, %N 2.36, %S 5.71; Loading: 0.130 mmol of sulphonic group per gram of sawdust.

The covalent grafting of chlorosulphonic acid on amino-functionalized sawdust is shown in Fig. 1. Initially, aluminium oxide was highly dispersed on the surface of sawdust (1) with high degree of adhesion to form sawdust-alumina composite (2). This composite was then allowed to react with (3-aminopropyl)trimethoxysilane at reflux in toluene to form amino-functionalized sawdust (3) with good degree of organofunctionalization. Afterwards, chlorosulfonic acid was covalently grafted in the matrix of functionalized sawdust using free aminopropyl group resulting in the formation of desired catalyst acronymed as SA@Sawdust (4).

#### General procedure for synthesis of 1, 8-dioxo-octahydroxanthenes

In 25-mL RB flask, the mixture of dimedone (2 mmol) and aryl aldehyde (1 mmol) in presence of SA@Sawdust (0.05 g, 0.0075 mmol) was refluxed in ethanol (5 mL). Upon the completion of reaction (TLC monitored), reaction mixture was diluted with hot ethanol (5 mL) and filtered to remove the catalyst. The evaporation of solvent in



Fig. 1 Preparation of heterogeneous solid acid catalyst SA@Sawdust

vacuo, afforded crude product which was recrystallized from ethanol to obtain the desired 1,8-dioxo-octahydroxanthenes.

#### **Result and discussion**

Fourier transform infrared (FT-IR), field emission scanning electron microscopy (FESEM), energy dispersive X-ray (EDX), thermogravimetric analysis (TGA), and cross-polarization magic angle spinning (CP-MAS) <sup>13</sup>C-NMR spectroscopy were employed to monitor the structure of SA@Sawdust.

The FT-IR spectrum of the catalyst was recorded on Perkin-Elmer Spectrum one FT-IR spectrophotometer and is depicted in Fig. 2. The FT-IR spectrum of SA@Sawdust displays O–H stretching of sulphonic group at 3375 cm<sup>-1</sup> while N–H stretching appears at 3242 cm<sup>-1</sup>. The peak obtained at 2854 cm<sup>-1</sup> is attributed to C–H stretching vibration of methylene groups of propyl unit present in the catalyst. The SO<sub>2</sub> symmetric and asymmetric stretching vibrations appeared at 1242, 1022 cm<sup>-1</sup>, respectively. The peaks at 888 and 752 cm<sup>-1</sup> belong to S–OH bending vibration, whereas S–O symmetric stretching bands appeared at 625 and 583 cm<sup>-1</sup> confirms the formation of desired catalyst.

The formation of SA@Sawdust was further corroborated by recording crosspolarization magic angle spinning (CP-MAS) <sup>13</sup>C-NMR spectroscopy (Fig. 3) which demonstrated the peaks at 105.64 (s, cellulose-C<sub>1</sub>), 89.48–84.27 (d, cellulose-C<sub>4</sub>), 75.56–65.64 (m, cellulose-C<sub>2</sub>, C<sub>3</sub>, C<sub>5</sub>), 56.84 (s, cellulose-C<sub>6</sub>), 43.68 (s, N–<u>C</u>H<sub>2</sub>), 22.32 (s, N–CH<sub>2</sub>–<u>C</u>H<sub>2</sub>–CH<sub>2</sub>), 11.12 (bs, <u>C</u>H<sub>2</sub>–Si) which is in well agreement with proposed structure.

The EDX analysis of desired catalyst is shown in Fig. 4. Figure 4a pristine sawdust revealed carbon and oxygen as the major elements where as Fig. 4b indicates the peaks for carbon, oxygen, aluminium and silicon for amino-functionalized sawdust. Further, SA@Sawdust recognized the peaks for carbon, oxygen, aluminium silicon and sulfur as shown in Fig. 4c. Presence of sulfur at its respective energy position at



Fig. 2 FT-IR Spectrum of heterogeneous solid acid catalyst SA@Sawdust





Fig. 4 EDX: a Pristine Sawdust, b amino-functionalized sawdust, c SA@Sawdust

2.30 keV also suggests the successful grafting of chlorosulfonic acid on amino-functionalized sawdust. The loading of sulphonic group as determined by EDX analysis was found to be 0.15 mmol/g of sawdust.

Field emission scanning electron microscopy (FESEM) is used for the studying the particle size and surface morphology of catalyst (Fig. 5). FESEM image clearly shows that the catalyst particles exhibit fibrous morphology having length ranging from couple of hundred nanometres to few micrometres.

The thermal stability of prepared catalyst was assessed using thermogravimetric analysis (TGA) in the temperature range 25–1000 °C under nitrogen atmosphere (Fig. 6). Initial weight loss of 18.62% displayed by the catalyst due to removal of physically absorbed water associated with catalyst through the hydrogen bonding. The second weight loss of 22.48% at 312 °C is attributed to the loss of combustible organic moieties [19]. Further, the combined weight loss of 24.22% and 21.11% is assigned to the pyrolytic decomposition of major constituents of



Fig. 5 FESEM of SA@Sawdust



Fig. 6 TGA analysis of SA@Sawdust

sawdust (cellulose, hemicelluloses and lignin), through the formation of levoglucosan and other volatile compounds [20]. Finally, the residual weight of 10.64% is due to formation of oxides of alumina.

#### **Determination of acidic site**

Acidity of catalyst can be determined by using pH measurement. SA@Sawdust (0.5 g) was added to 25 mL of aq. 1 M sodium chloride (pH 5.4) and resultant mixture was stirred for 2 h at room temperature. The pH of solution decreased to 1.42 which equal to loading 1.86 mmol [H<sup>+</sup>] per gram of catalyst [21].

Our next task was to explore the catalytic potential of SA@Sawdust in the synthesis of 1, 8-dioxo-octahydroxanthenes. In this regards, the dimedone and benzaldehyde were chosen as a model substrates for optimization of reaction conditions. In order to optimize the solvent, the template reaction in the presence of SA@Sawdust (0.05 g, 0.0075 mmol) was carried out in different organic solvents at reflux condition (Table 1). The reaction marches sluggishly in solvents such as toluene, chloroform, dichloromethane, acetonitrile, EDC, THF, and acetone, affording corresponding 1,8-dioxo-octahydroxanthene (**7a**) in lower yields (Table 1, entries 1–7). The high yield was acquired in presence of ethanol under reflux condition (Table 1, entry 10). Further, in order to optimize the loading of catalyst, the model reaction was carried out using various quantities of catalyst (Table 1). It was observed that the optimal amount of catalyst was 0.05 g (0.0075 mmol) in order to obtain the best result (Table 1, entry 10). Further decrease in catalyst quantity lower than 0.05 g did not increase the yield of the product significantly (Table 1, entries 8 and 9).

Table 1	Optimization	of reaction	condition	for syı	nthesis o	of 1, 8	8-dioxo-octah	ydroxanthenes
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Entry	Catalyst quantity (g)	Reaction condition	Reaction time (min)	Isolated yield (%) <sup>a</sup>	
1	0.05	Toluene/reflux	50	32	
2	0.05	Chloroform/reflux	50	38	
3	0.05	DCM/reflux	50	42	
4	0.05	Acetonitrile/reflux	50	47	
5	0.05	EDC/reflux	50	48	
6	0.05	THF/reflux	50	51	
7	0.05	Acetone/reflux	50	53	
8	0.01	Ethanol/reflux	50	72	
9	0.03	Ethanol/reflux	50	84	
10	0.05	Ethanol/reflux	50	92	

Reaction conditions: Dimedone (2 mmol), benzaldehyde (1 mmol) and SA@Sawdust in solvent (5 mL) at given condition

<sup>a</sup> In all reaction 100% conversion were obtained

To explore the scope and limitations of this protocol, we focused our attempts on the synthesis of 1, 8-dioxo-octahydroxanthene derivatives (7) using dimedone (5) with variety of substituted aryl aldehydes (6) under the optimal condition and the results are summarized in Table 2. In all cases 1, 8-dioxo-octahydroxanthenes were obtained as the sole product. Aromatic aldehydes carrying the both electron-withdrawing and electron-donating (Table 2, entries 2–5 and 6–9) furnishes the corresponding 1, 8-dioxo-octahydroxanthenes in good to excellent yields. Surprisingly, the significant yields were obtained with the sterically crowded aldehydes, such as 2-nitrobenzaldehyde, 2,5-dimethoxybenzaldehyde and 3,4,5-trimethoxybenzaldehyde under optimized reaction condition highlighting the general applicability of present

Table 2 SA@Sawdust catalyzed reaction between dimedone and aryl aldehydes



Entries	Aryl aldehyde (6)	Product (7)	Time (min)	Yield (%) <sup>a</sup>	Mp (°C) [lit.]	References
1	Benzaldehyde	7a	50	92	200–202 [201–203]	[23]
2	4-Cyanobenzaldehyde	7b	35	93	218–222 [217–218]	[23]
3	4-Chlorobenzalde- hyde	7c	40	85	230–233 [230–232]	[23]
4	4-Bromobenzalde- hyde	7d	50	86	238–240 [240–242]	[23]
5	4-Nitrobenzaldehyde	7e	40	94	226–230 [228–230]	[23]
6	4-Hydroxybenzalde- hyde	7f	55	88	249–252 [250–251]	[24]
7	4-Methylbenzalde- hyde	7g	50	86	217–220 [219–221]	[23]
8	4-Methoxybenzalde- hyde	7h	45	85	246–248 [248–250]	[25]
9	3-Methoxybenzalde- hyde	7i	50	80	160–162 [161–162]	[24]
10	2-Nitrobenzaldehyde	7j	45	84	248–251 [249–250]	[25]
11	2,5-Dimethoxybenza- ldehyde	7k	50	80	171–173 [172–174]	[26]
12	3,4,5-Trimethoxyben- zaldehyde	71	55	82	206–208 [205–208]	[27]
13	Furfural	7m	60	80	162–165 [164–165]	[24]

Reaction conditions: dimedone (2 mmol), aryl aldehyde (1 mmol), and SA@Sawdust in ethanol (5 mL) were stirred at reflux condition

<sup>a</sup>Isolated yield of purified products



Scheme 1 Plausible mechanistic pathway for formation of 1,8-dioxo-foctahydroxanthenes





protocol (Table 2, entries 10 and 12). Fascinatingly, heteroaromatic thiophene-2-carbaldehyde (Table 2, entries 13) was examined and found to be well tolerated providing comparatively moderate yields of the anticipated product.

Mechanistic pathway for the synthesis of 1,8-dioxo-octahydroxanthenes catalyzed by SA@Sawdust is depicted in Scheme 1. We propose that one molecule of dimedone was firstly underwent knoenvengel condensation with an activated aldehyde (I) to afford intermediate (II). Then, another molecule of dimedone reacted with intermediate (II) via michael addition to give the intermediate (III) which finally on cyclodehydration furnishes the expected 1,8-dioxo-octahydroxanthenes.

The fundamental aspect of this study has to establish a reusability procedure for the synthesis of 1,8-dioxo-octahydroxanthenes (7a) in presence of SA@Sawdust under optimized reaction condition as shown in Fig. 7. The catalyst was easily recovered from reaction mixture by simple filtration, washed, dried and reused for next

Entry	Catalyst used	Reaction condition	Time	Yield (%)	References	
1	SA@Sawdust (0.05 g)	Ethanol/reflux	50 min	92	This work	
2	L-Proline (40 mol%)	Dichloroethane/reflux	6 h	83	[28]	
3	CaCl <sub>2</sub> (20 mol%)	DMSO/90 °C	4 h	85	[29]	
4	Montmorillonite K-10 (0.3 g)	Solvent-free/100 °C	2 h	82	[30]	
5	[Bmim]HSO <sub>4</sub> (0.1 g)	Solvent-free/80 °C	3 h	85	[31]	
6	[Hmim]TFA (0.1 g)	Solvent-free/80 °C	3 h	85	[32]	
7	SmCl <sub>3</sub> (20 mol%)	Solvent-free/120 °C	9 h	98	[33]	
8	[Cmmim][BF <sub>4</sub> ] (200 mg)	Methanol/ultrasound	50 min	87	[34]	
9	PMA-SiO <sub>2</sub> (0.1 mol%)	Acetonitrile/reflux	4 h	95	[35]	
10	InCl <sub>3</sub> ·4H <sub>2</sub> O (10 mol%)	[Bmim][BF <sub>4</sub> ]/80 °C	4 h	87	[36]	

<sup>a</sup>Based on benzaldehyde

cycle. It was observed that catalyst could be reused for three times without significant decrease in the yield of desired product.

In order to show the advantages of SA@Sawdust in comparison with other catalysts, we have summarized several results for the synthesis of 1,8-dioxo-octahydroxanthenes (Table 3). A comparison of the results reveals that SA@Sawdust is well efficient catalyst in terms of reaction time and product yield despite of other reported catalyst.

## Conclusions

In summary, we have reported highly efficient and cost-effective approach towards the synthesis of 1,8-dioxo-octahydroxanthenes in the presence of SA@Sawdust as an eco-friendly catalyst. The advantages of present procedure is short reaction time, cleaner reaction profile, high yields, eco-friendly solvent, operational simplicity, easy preparation of catalyst and its reusability.

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