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Original Article

Construction of a Novel Magnetic Nanomaterial, and its Utility as an Effectual Catalyst for the Fabrication of 1, 8-Dioxo-Octahydroxanthenes

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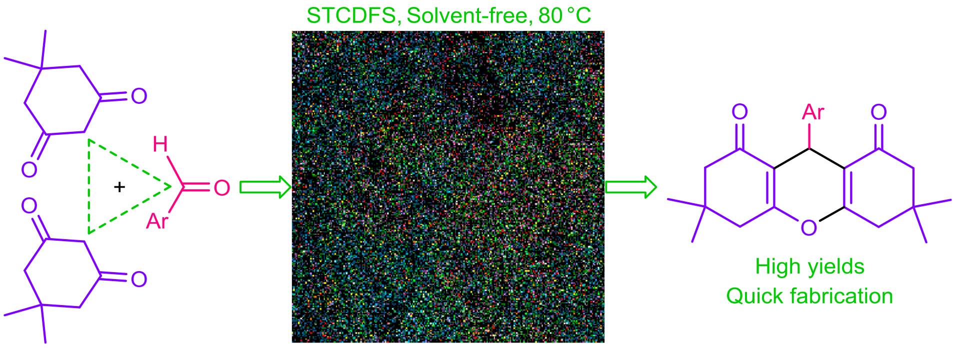
N^1-(Si-pr)-(N^1,N^1,N^4,N^4-tetramethylbenzene-1,4-diaminium) chloride dihydrogen phosphate grafted on $Fe_3O_4@SiO_2$ (STCDFS)
1,8-Dioxo-octahydroxanthene
Solvent-free

ABSTRACT

At first, construction and characterization of a novel magnetic nanomaterial titled N^1-(Si-pr)-(N^1,N^1,N^4,N^4-tetramethylbenzene-1,4-diaminium) chloride dihydrogen phosphate grafted on $Fe_3O_4@SiO_2$ (STCDFS) have been described. The characterization has been accomplished by EDX, elemental mapping, FE-SEM, FT-IR, XRD, and VSM analyses. In continue, effectual and quick fabrication of 1,8-dioxo-octahydroxanthenes form aryl aldehydes and dimedone using STCDFS in solvent-free conditions has been reported. The xanthenes were acquired in 92-98% in 5-10 min. Moreover, the catalyst was reusable for one time without remarkable decrement in its performance.

GRAPHICAL ABSTRACT



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Introduction

Magnetic nanomaterials have attracted much attention owing to their inimitable features and vast utilities in diverse industries. Some features of these substances comprise high performance, eco-friendly nature, thermic and chemical durability, effortless segregation from the process medium, and capacity for constructing numerous kinds of them. Magnetic nanomaterials have been exploited for selective sorption of proteins [1], wastewater treatment [2], tamoxifen determination [3], analysis of pesticides remainder in fruits [4], and degradation of dyes [5]. They have been also utilized in solar cells [6], sensors [7], theranostic nanomedicine [8], electroanalytical methods [9], and organic synthesis (as catalyst) [10-27]. "Solvent-free conditions" is an applicatory and highly efficacious approach which has been extensively exploited in organic synthesis; its privileges compared with "solution conditions" comprise easier workup and purification of products, eco-friendly nature, higher selectivity and yield, saving energy and time, and excluding or detracting waste and sub-products [28-35]. The materials bearing xanthene component are an attractive category of heterocycles, which have a wide range of applications. Antibacterial [36], COX inhibitory [37], anticancer [37], antioxidant [37], antifungal [38], antiproliferative [39], and antileishmanial [40] activities have been reported for xanthenes. Moreover, these materials have been applied in bioimaging [41], fluorophores [42], photothermal therapy [43], and photocatalytic processes [44]. A category of xanthene-bearing heterocycles is 1,8-dioxo-octahydroxanthenes, which can be fabricated form Equation (1) of aldehyde and Equation (2) of dimedone using a catalyst [45-55]. Herein, we have accomplished construction and characterization a novel magnetic nanomaterial titled N^1-(Si-pr)-(N^1,N^1,N^4,N^4-tetramethylbenzene-1,4-diaminium) chloride dihydrogen phosphate grafted on $Fe_3O_4@SiO_2$ (STCDFS), and then we have done the fabrication of 1,8-dioxo-octahydroxanthenes form aryl aldehydes and dimedone using STCDFS.

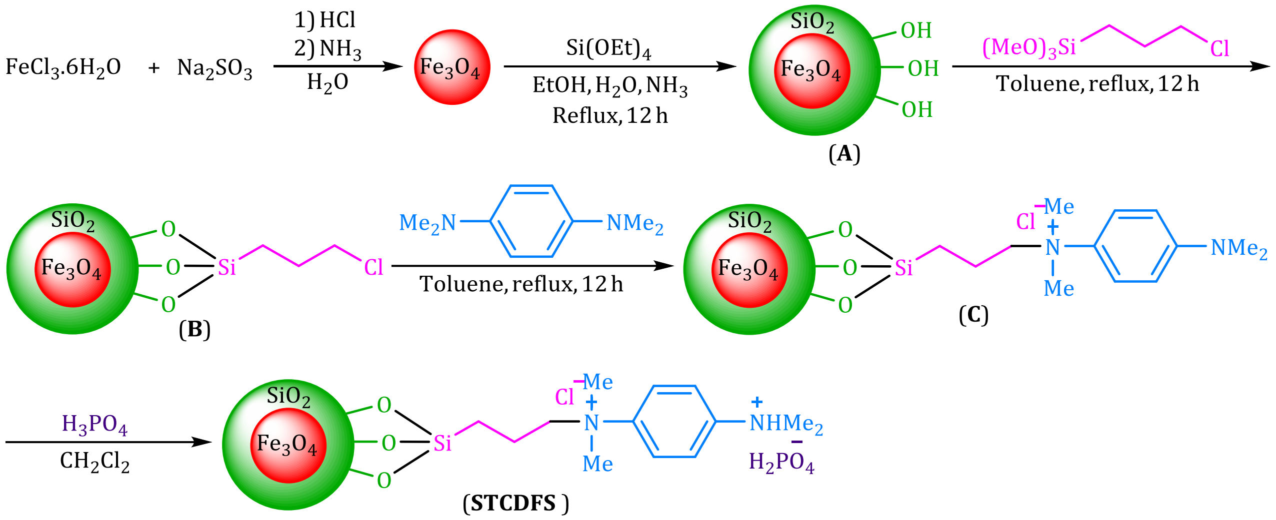
Experimental

Materials and Apparatuses

The materials and solvents were supplied from Sigma-Aldrich Chemical Company. TLC (silica gel SIL G/UV 254 plates) was exploited to see the reactions progress. Measuring melting points was accomplished by a Thermo Scientific 9200 apparatus. A Bruker Avance DPX FT-NMR device was utilized to run NMR spectra. EDX and elemental mapping analyses were carried out using a TESCAN apparatus (model MIRA II). FE-SEM pictures were recorded by a TESCAN device (model MIRA III). For recording the FT-IR spectra, a Thermo device (model AVATAR) was applied. XRD analysis was done by a PHILIPS device (Cu Kα radiation, λ=1.54056 Å, model PW1730). VSM analysis was performed using a MDK (Meghnatis Daghigh Kavir, Iran) apparatus at ambient temperature.

Construction of STCDFS

Nano-Fe_3O_4 (nano-magnetite) was constructed pursuant to the published method [56,57]. Nano-magnetite (0.75 g), tetraethyl orthosilicate (2.25 mL), water (15 mL), ethanol (60 mL), and ammonia (2.40 mL) were stirred and refluxed for 12 h to provide material **A** [23,57-58]. Thereupon, a mixture of **A** and (3-chloropropyl)trimethoxysilane (0.60 mL, 3.26 mmol) in dry toluene (20 mL) was stirred under reflux conditions for 12 h to synthesize **B** [23,57]. In continue, N^1,N^1,N^4,N^4-tetramethylbenzene-1,4-diamine (0.535 g, 3.26 mmol) was added to **B** in toluene (20 mL), and stirred under reflux and nitrogen atmosphere for 12 h to fabricate **C**. Lastly, H_3PO_4 (3.26 mmol) was gently added to **C** in CH_2Cl_2 (10 mL) at ambient temperature, and stirred under nitrogen atmosphere for 12 h at ambient temperature and 2 h under reflux conditions to construct STCDFS. Before each stage, the reaction mixture was sonicated for 20 min to scatter the particles. The fabricated material in each stage was magnetically separated, washed by the applied solvent in that stage, and dried (Scheme 1).



Scheme 1: The STCDFS fabrication

The Fabrication of 1,8-Dioxo-octahydroxanthenes

Aldehyde (0.25 mmol), dimedone (0.5 mmol, 0.070 g), and STCDFS (0.035 g) were added in a reaction vessel, and the mixture was stirred by a rod at 80 ºC. When TLC showed consuming aldehyde and dimedone, the reaction mixture was cooled to ambient temperature, EtOAc (10 mL) was added, and stirred under reflux conditions for 2 min, followed by magnetically isolation of STCDFS; then, STCDFS was washed by EtOAc (2×3 mL), dried, and utilized for next run. The attained solution after the catalyst isolation was distilled, and the remainder solid was recrystallized from EtOH (95%) to fabricate the pure xanthene.

Selected NMR Data of the Fabricated Xanthenes

*3,3,6,6-Tetramethyl-9-phenyl-3,4,6,7-tetrahydro-2H-xanthene-1,8(5H,9H)-dione (**a**)*

^{1}H NMR (500 MHz, DMSO-d_6): δ (ppm) 0.88 (s, 6H, $2CH_3$), 1.01 (s, 6H, $2CH_3$), 2.05 (d, J = 16.1 Hz, 2H, CH_2–C=C), 2.24 (d, J = 16.1 Hz, 2H, CH_2–C=C), 2.49 (d, J = 17.7 Hz, 2H, CH_2–C=O), 2.54 (d, J = 17.7 Hz, 2H, CH_2–C=O), 4.52 (s, 1H, methine CH), 7.05-7.08 (m, 1H, Ar), and 7.15-7.20 (m, 4H, Ar); ^{13}C-NMR (125 MHz, DMSO-d$_6$): δ (ppm) 26.6 (CH_3), 28.8 (*C*–CH_3), 31.3 (*C*–C=C), 31.9 (methine C), 50.2 (*C*H_2–C=O), 114.5 (*C*–C=O), 126.3 (Ar), 127.9 (Ar), 128.2 (Ar), 144.4 (Ar), 162.9 (C–O), and 196.1 (C=O).

*9-(2-Chlorophenyl)-3,3,6,6-tetramethyl-3,4,6,7-tetrahydro-2H-xanthene-1,8(5H,9H)-dione (**h**)*

^{1}H-NMR (500 MHz, DMSO-d_6): δ (ppm) 0.97 (s, 6H, $2CH_3$), 1.09 (s, 6H, $2CH_3$), 2.09 (d, J = 16.1 Hz, 2H, CH_2–C=C), 2.31 (d, J = 16.1 Hz, 2H, CH_2–C=C), 2.53 (d, J = 17.6 Hz, 2H, CH_2–C=O), 2.64 (d, J = 17.6 Hz, 2H, CH_2–C=O), 4.87 (s, 1H, methine CH), 7.16-7.20 (m, 1H, Ar), and 7.25-7.33 (m, 4H, Ar); ^{13}C-NMR (125 MHz, DMSO-d$_6$): δ (ppm) 26.5 (CH_3), 28.8 (*C*–CH_3), 31.8 (*C*–C=C), 33.0 (methine C), 50.2 (*C*H_2–C=O), 113.2 (*C*–C=O), 126.6 (Ar), 127.9 (Ar), 129.6 (Ar), 132.2 (Ar), 132.9 (Ar), 142.4 (Ar), 163.3 (C–O), and 196.0 (C=O).

Results and Discussion

Characterization of STCDFS

EDX, elemental mapping, FE-SEM, FT-IR, XRD, and VSM analyses were applied to characterize N^1-(Si-pr)-(N^1,N^1,N^4,N^4-tetramethylbenzene-1,4-diaminium) chloride dihydrogen phosphate grafted on $Fe_3O_4@SiO_2$ (STCDFS). The presence of Fe, O, Si, C, N, Cl, and P in STCDFS structure was corroborated by EDX and elemental mapping analyses (Figures 1 and 2). In addition, suitable distribution of the elements in STCDFS surface was confirmed by the elemental mapping pictures.

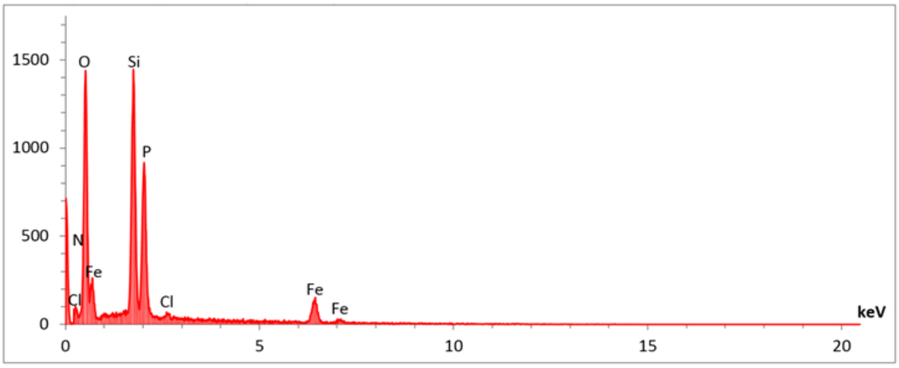


Figure 1: The EDX analysis of N^1-(Si-pr)-(N^1,N^1,N^4,N^4-tetramethylbenzene-1,4-diaminium) chloride dihydrogen phosphate grafted on $Fe_3O_4@SiO_2$

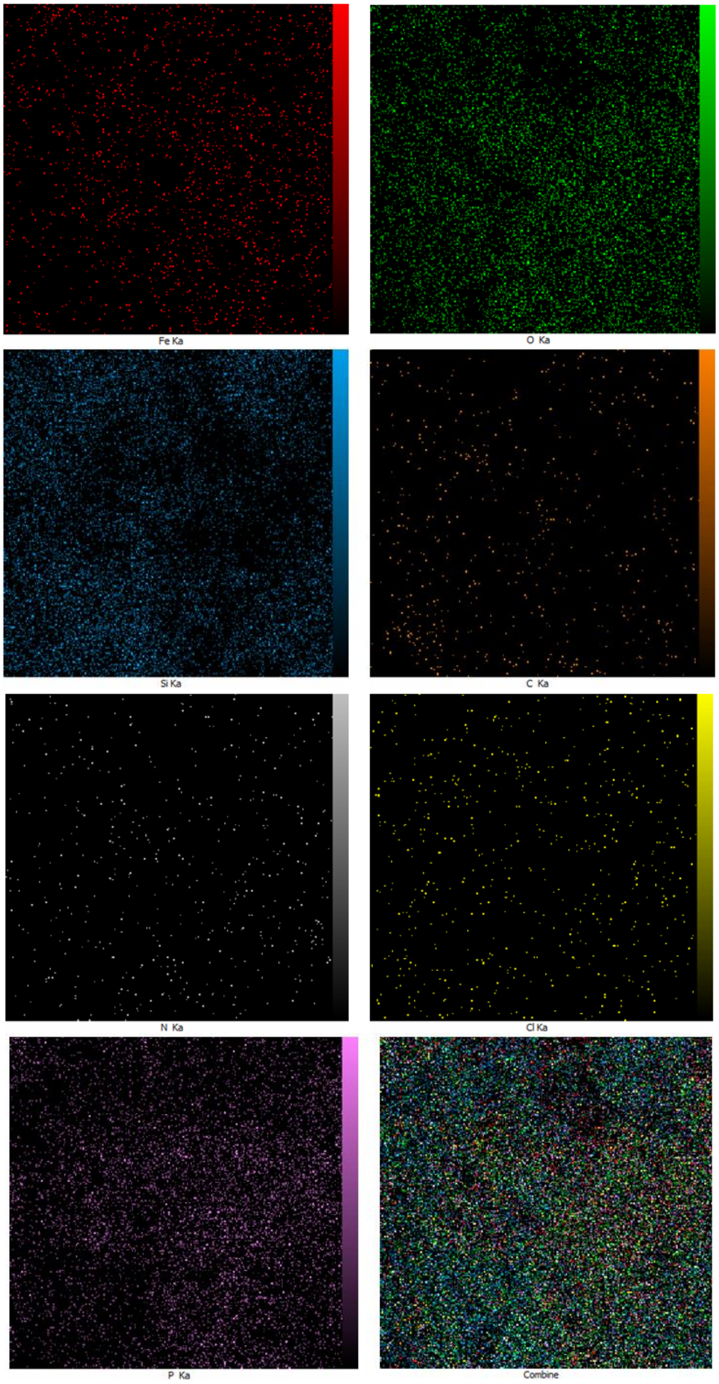


Figure 2: The elemental mapping pictures of STCDFS

According to the FE-SEM picture (Figure 3), STCDFS is a porous material. Furthermore, the particles have diverse shapes and sizes, and are in nano-scale, e.g., 27.0, 47.2, and 57.7 nm. The spectrum and the information of FT-IR analysis are illustrated in Figure 4 and Table 1, correspondingly. Appearing the peaks pertinent to all bonds in STCDFS structure corroborated its successful fabrication.

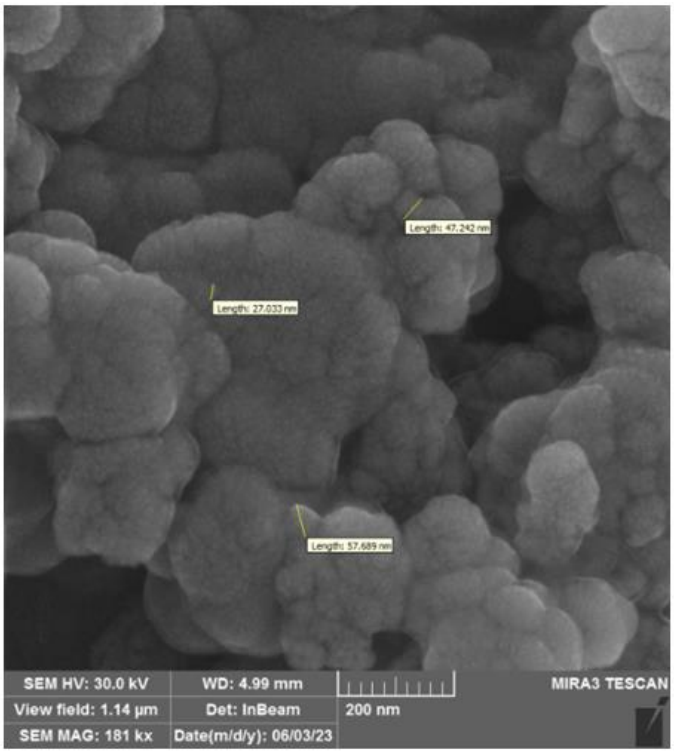


Figure 3: The FE-SEM picture of STCDFS

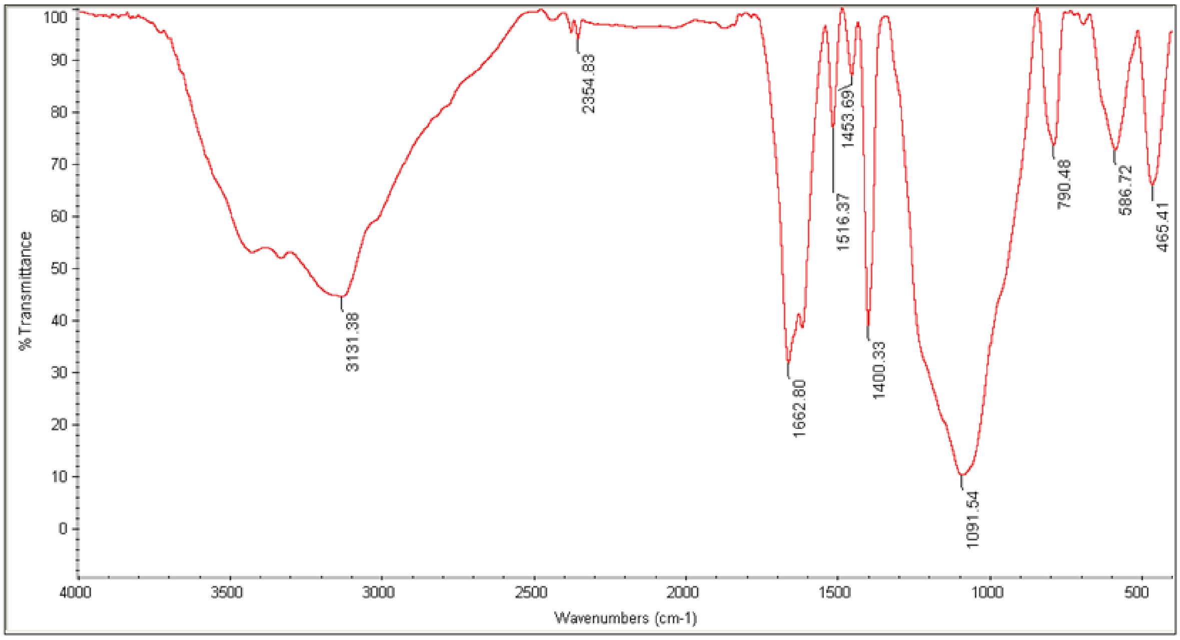


Figure 4: The FT-IR spectrum of STCDFS

Table 1: The FT-IR information of STCDFS

Peak (cm^{-1})	Ascribed bond
465	Si–O (rocking)
587	Fe–O
790	Si–O–Si (symmetric stretching)
1092	Si–O–Si (asymmetric stretching)
1400	C–N^+ (stretching)
1454	C–H of methyl group (bending)
1516 and ~1617	Aromatic C=C (stretching)
~3019	C–H of phenyl moiety (stretching)
~2523-3770	O–H of $H_2PO_4^-$ and adsorbed H_2O on STCDFS surface

Figure 5 demonstrates the XRD pattern of SCBFH. The broad peak at 2θ ≈ 19.4-32.3º corroborated presence of the amorphous form of silica in the nanomaterial structure. Existing a cubic spinel structure of nano-magnetite in the SCBFH structure was confirmed by appearing the sharp peaks at 2θ ≈ 30.1, 35.4, 43.2, 53.3, 57.1, and 62.7º in the XRD pattern. Magnetic behavior of STCDFS was investigated by VSM analysis (Figure 6). Saturation magnetization (Ms) of the nanomaterial was found to be ~3.99 emu.g^{-1}. Ms of the used nano-magnetite as precursor to fabricate STCDFS was 56.7 emu.g^{-1} [14]. Coating silica layer on the nano-magnetite, and grafting *N*¹-(Si-pr)-(*N*¹,*N*¹,*N*⁴,*N*⁴-tetramethylbenzene-1,4-diaminium) moiety on the silica surface caused decrement of Ms in STCDFS compared with the nano-magnetite.

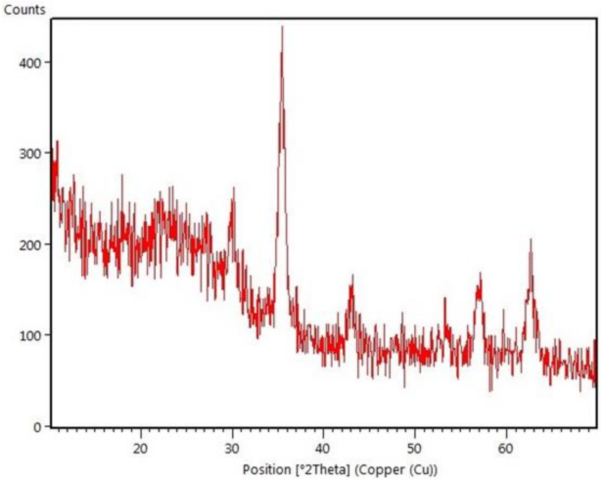


Figure 5: The FT-IR spectrum of STCDFS

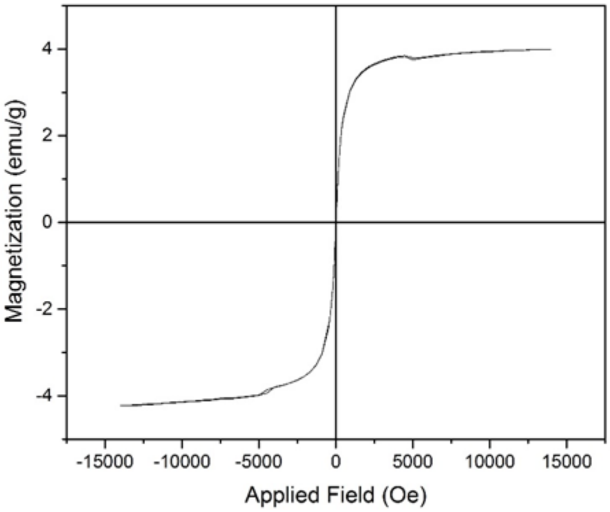
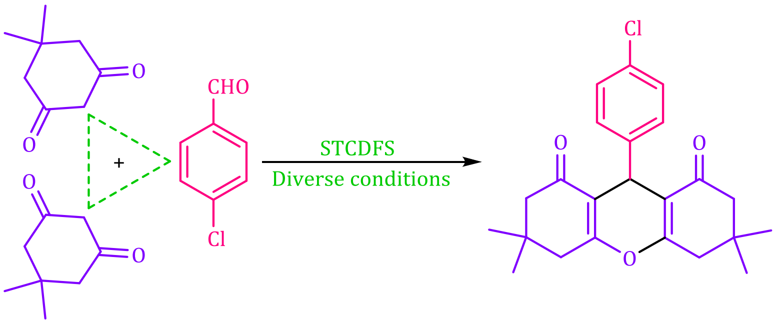


Figure 6: The VSM diagram of STCDFS

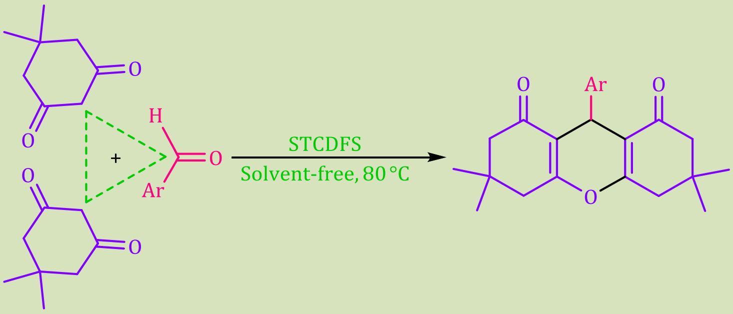
Catalytic Utility of STCDFS

Catalytic utility of STCDFS was investigated for the fabrication of 1,8-dioxo-octahydroxanthenes. In this regard, the fabrication of xanthene **h** from 4-chlorobenzaldehyde (0.25 mmol) and dimedone (0.5 mmol) in solvent-free conditions was designated as a model reaction (Scheme 2), and effect of two main factors (catalyst dosage and temperature) was perused on that (this study was done using 0.020-0.037 g of STCDFS at 70-85 °C). Completion of the reaction was occurred without formation of any byproduct using 0.035 g of STCDFS at 80 °C; in this test, isolated yield of xanthene **h** was 98%, and the reaction time was 5 min. No improvement in the results was observed when STCDFS dosage was increased up to 0.037 g or the temperature up to 85 °C. The domain and productivity of STCDFS for the fabrication of 1, 8-dioxo-octahydroxanthenes were perused via utility of several substituted benzaldehydes in the reaction. Table 2 presents the gained results. High yields of the xanthenes were acquired in short times for benzaldehydes possessing electron-donating, halogen or electron-attracting groups on their *para*, *meta*, or *ortho* positions. High catalytic productivity and widespread domain of STCDFS to fabricate 1,8-dioxo-octahydroxanthenes were confirmed by the results.



Scheme 2: The model reaction

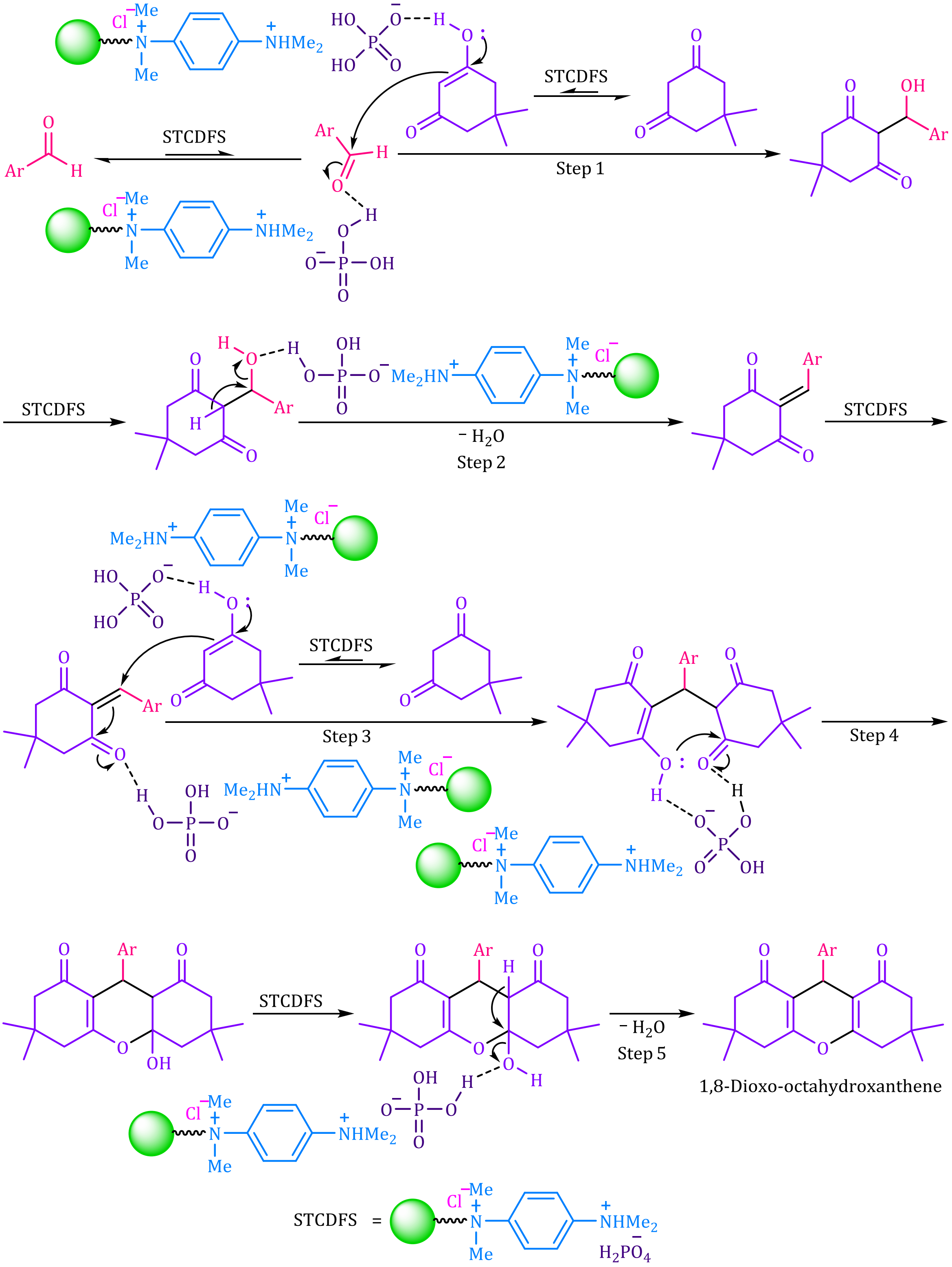
Table 2: The fabrication of 1,8-dioxo-octahydroxanthenes using STCDFS



Product	Ar	Time (min)	Yield (%)[a]	M.p. (°C) [lit.]
a	C_6H_5	5	97[b]	201-203 (200-202) [48]
b	$4\text{-}CH_3C_6H_4$	5	97[b]	217-219 (218-220) [47]
c	$4\text{-}(PhCH_2O)C_6H_4$	10	92	155-157 (153-155) [48]
d	$2,5\text{-}(CH_3O)_2C_6H_3$	10	95	170-172 (172-174) [53]
e	$3,4\text{-}(CH_3O)_2C_6H_3$	7	93	177-179 (174-176) [47]
f	$4\text{-}CH_3OC_6H_4$	5	97[b]	245-247 (243-245) [48]
g	$3\text{-}O_2NC_6H_4$	5	98[b]	168-170 (171-173) [50]
h	$2\text{-}ClC_6H_4$	7	98[b]	222-224 (223-225) [48]
i	$2,4\text{-}Cl_2C_6H_3$	7	98[b]	254-256 (257-259) [51]
j	$4\text{-}ClC_6H_4$	5	98[b]	231-233 (229-230) [50]

[a] *Isolated yield*[b] *The reaction nearly completed*

Scheme 3 represents the reaction mechanism, which was proposed on basis of the literature [48, 49]. $H_2PO_4^-$ of STCDFS catalyzes the fabrication of 1,8-dioxo-octahydroxanthenes thru activation of the electrophiles (by its acidic hydrogen) and the nucleophiles (by its negative oxygen as a weak base) in stages 1, 3, and 4, facilitating elimination of H_2O in stages 2 and 5, and accelerating tautomerization of dimedone in stages 1 and 3.



Scheme 3: The mechanism

STCDFS were compared with some catalysts in terms of medium, temperature, time, and yield of the reaction. For this study, the fabrication of xanthene **a** was chosen. The data, which are tabulated in Table 3, confirmed superiority of STCDFS in comparison with the catalysts in two or more of the factors. Lastly, STCDFS reusability was investigated. The fresh STCDFS catalyzed the fabrication of xanthene **j** in 98% in 5 min. In first recycling, the product was constructed in 95% in 7 min; in second recycling, the yield decreased to 87%, and the reaction time rose up to 10 min. Hence, STCDFS was reusable for one times without remarkable loss of its performance.

Table 3: Comparing STCDFS with some catalysts to fabricate xanthene **a**

Catalyst	Conditions	Time (min)	Yield (%)	Ref.
STCDFS	Solvent-free, 80 ºC	5	97	-
Salicylic acid	Solvent-free, 70 ºC	10	90	[47]
Ph_3CCl	Solvent-free, 110 ºC	50	95	[48]
Nano-CeO_2	H_2O, reflux	60	95	[49]
W/Cu@g-C_3N_4^{a}	Solvent-free, 80 ºC	60	92	[50]
$ZnFe_2O_4$@Fe_3O_4	EtOH, reflux	30	91	[51]
Bis(PEG)phthalate-Cu(II)	H_2O, r.t.	30	97	[52]
[Hbim]BF_4	MeOH, r.t., ultrasonic	45	85	[53]
Nano-CeO_2/Al_2O_3	EtOH, reflux	60	95	[54]
Fe_3O_4 nanoparticles	Solvent-free, 100 ºC	30	89	[55]

^{a} WCl_6/$CuCl_2$ supported on graphitic carbon nitrid

Conclusion

Briefly, we have developed a novel magnetic nanocatalyst, namely *N*^{1}-(Si-pr)-(*N*^{1},*N*^{1},*N*^{4},*N*^{4}-tetramethylbenzene-1,4-diaminium) chloride dihydrogen phosphate grafted on Fe_3O_4@SiO_2. It could efficaciously catalyze the fabrication of 1,8-dioxo-octahydroxanthene from aryl aldehydes and dimedone by its $H_2PO_4^-$ group; the acidic hydrogens of $H_2PO_4^-$ could activate the electrophiles, and the negative oxygen (as a weak base) could activate the nucleophiles. High performance, excellent yields, quick fabrication of the products, utility of solvent-free technique, and eco-friendly conditions are some privileges of our protocol.

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Disclosure Statement

No potential conflict of interest was reported by the authors.

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Authors' Contributions

A. Zare has defined the project idea, and supervised it; he has also interpreted the analyses, and written the article and revised it. F. Mostaghar has done the experimental works of the project.

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