



**EVALUATION OF CATALYTIC ACTIVITY OF NEWLY PREPARED
CuFe₂O₄@SiO₂@C₃-Imid-C₄SO₃-PW MAGNETIC NANOPARTICLES IN THE
SYNTHESIS OF 1,8-DIOXO-OCTAHYDROXANTHENES UNDER SOLVENT-FREE
CONDITIONS**

Nasrinsadat Hosseininasab¹, Abolghasem Davoodnia^{1,*}, Faramarz Rostami-Charati², Amir Khojastehnezhad³, Niloofar Tavakoli-Hoseini³

¹ *Department of Chemistry, Mashhad Branch, Islamic Azad University, Mashhad, Iran*

² *Department of Chemistry, Faculty of Science, Gonbad Kavous University, Gonbad, Iran*

³ *Young Researchers and Elite Club, Mashhad Branch, Islamic Azad University, Mashhad, Iran*

Abstract: Catalytic activity of newly prepared CuFe₂O₄@SiO₂@C₃-Imid-C₄SO₃-PW magnetic nanoparticles is studied in the synthesis of 1,8-dioxo-octahydroxanthenes by the reaction of dimedone with aromatic aldehydes. The reactions occur under solvent-free conditions and the process is operative with various aromatic aldehydes, giving the corresponding products in high yields over short reaction times. In addition, the magnetic nanocatalyst could be easily recovered from the reaction mixture by a magnet and reused several times.

Keywords: CuFe₂O₄@SiO₂@C₃-Imid-C₄SO₃-PW, Magnetic nanoparticles, 1,8-Dioxo-octahydroxanthenes, Solvent-free conditions

Introduction

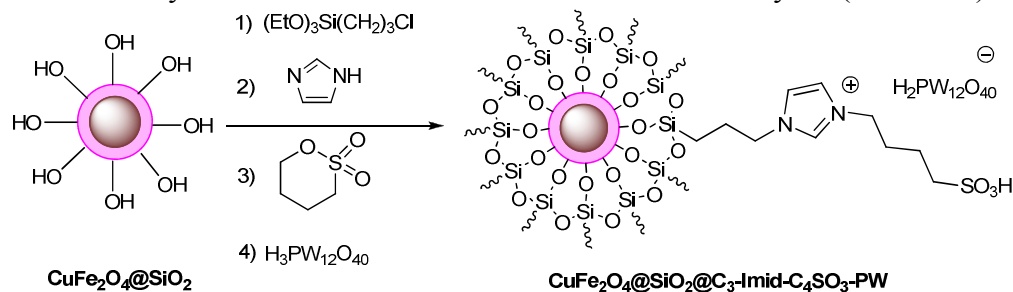
Compounds derived from xanthenes are of importance as they have various biological properties such as antimicrobial^{i,ii}, anti-inflammatoryⁱⁱⁱ, analgesicⁱⁱⁱ, antiviral^{iv}, antifungal^v, and antioxidant^{vi} activities. They are also used in laser technologies^{vii}, and found in fluorescent materials for visualization of biomolecules^{viii}, and in dyes^{ix}. In particular, xanthenediones constitute a structural unit in a number of natural products^{x,xii} and have been used as versatile synthons because of the inherent reactivity of the inbuilt pyran ring^{xii}. These compounds also exhibit diverse and interesting biological activities such as anticancer^{xiii,xiv}, anti-tubercular^{xv}, anti-inflammatory^{xv}, antioxidant^{xvi}, and antibacterial^{xvii} properties. Xanthenediones are generally

*Corresponding author. Tel.: +98-51-38435000; Fax: +98-51-38429520; E-mail: adavoodnia@mshdiau.ac.ir; adavoodnia@yahoo.com

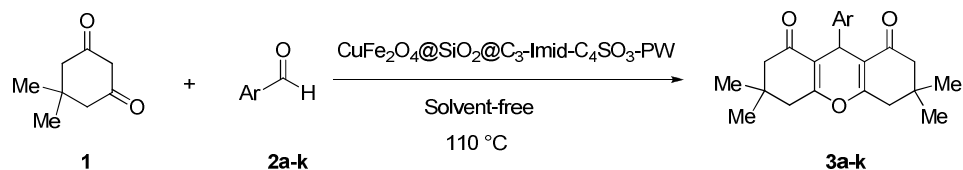
synthesized by condensation of aromatic aldehydes with 1,3-cyclohexanedione or 5,5-dimethyl-1,3-cyclohexanedione (dimedone) using various promoting agents such as triethylbenzylammonium chloride^{xviii}, trimethylsilylchloride^{xix}, ZnO nanoparticles^{xx}, H₃PW₁₂O₄₀/MCM-41^{xxi}, SmCl₃^{xxii}, hydrotrope^{xxiii}, L-proline^{xxiv}, alumina supported acidic ionic liquid^{xxv}, ceric ammonium nitrate supported HY-zeolite^{xxvi}, Keplerate-type giant nanoporous isopolyoxomolybdate^{xxvii}, β -cyclodextrin grafted with butyl sulfonic acid^{xxviii}, [BPY]HSO₄^{xxix}, and sulfonic acid functionalized ionic liquids^{xxx}. Many of these methodologies, however, suffer from disadvantages such as unsatisfactory yields, expensive catalysts, long reaction times, toxic organic solvents, and harsh reaction conditions. Therefore, development of new efficient recyclable catalysts for the synthesis of 1,8-dioxo-octahydroxanthenes is still in great demand.

Although, acidic ionic liquids (ILs) with low toxicity, high thermal stability, and extremely low vapor pressure, are suitable substitute catalysts for conventional acids^{xxxi-xxxiv}, the danger of catalyst leaching and the difficulties of product isolation from ILs are still big challenges to overcome. The immobilization of ILs on solid supports can solve these problems and satisfy the requirements from chemical industry of the ease of separation and recovery of catalyst from reaction mixture^{xxxv}. In this regard, there are several reports for immobilizing ILs using magnetic nanoparticles (MNPs) supports^{xxxvi-xxxviii}. The main advantage of MNPs supported catalysts is that they can be isolated efficiently from the reaction medium through a simple magnetic decantation thereby eliminating the requirement of catalyst filtration and centrifugation. A number of applications in biomolecular sensors^{xxxix}, drug and gene delivery^{xl}, and magnetic resonance imaging (MRI)^{xli} have been also reported for MNPs.

In this vein, and in continuation of the previous works, firstly by Luo and co-workers^{xlii}, for the immobilization of phosphotungstic acid (H₃PW₁₂O₄₀, denoted as PW in the present paper), with a Keggin structure, on MNPs^{xliii,xliv}, recently, a novel acidic IL immobilized on CuFe₂O₄@SiO₂ MNPs, denoted as CuFe₂O₄@SiO₂@C₃-Imid-C₄SO₃-PW, was synthesized (Scheme 1) and fully characterized in our group and successfully applied as highly efficient catalyst in the synthesis of 1*H*-pyrazolo[1,2-*b*]phthalazine-5,10-diones^{xlv}. These findings encouraged us to explore other applications of these new MNPs in the synthesis of organic compounds. Therefore, in line with our interest in heterocycles^{xlvi-xlx} and in the development of convenient methods using reusable catalysts^{xlxi-xlxvii}, in the present study we report the results of our investigation into the application of CuFe₂O₄@SiO₂@C₃-Imid-C₄SO₃-PW as catalyst in the synthesis of 1,8-dioxo-octahydroxanthenes by the reaction of dimedone with aromatic aldehydes (Scheme 2).



Scheme 1. Preparation of CuFe₂O₄@SiO₂@C₃-Imid-C₄SO₃-PW MNPs



Scheme 2. Synthesis of 1,8-dioxo-octahydroxanthenes catalyzed by CuFe₂O₄@SiO₂@C₃-Imid-C₄SO₃-PW MNPs

Experimental

All chemicals were purchased from Merck and Aldrich and used without purification. Ultrasonication was performed by Soltec sonicator (Italy, 2200ETH S3) at a frequency of 40 kHz and a nominal power of 260 W. Melting points were measured on a Stuart SMP3 melting point apparatus. The ¹H NMR spectra were measured on a Bruker 300 and 400 spectrometers using tetramethyl silane (TMS) as internal standard. IR spectra were recorded on a Tensor 27 Bruker spectrophotometer in KBr disks.

Preparation of CuFe₂O₄@SiO₂@C₃-Imid-C₄SO₃-PW MNPs.

A mixture of CuFe₂O₄@SiO₂ (1.0 g), dispersed ultrasonically, and (3-chloropropyl)triethoxysilane (1.0 ml) in dry toluene (6 ml) was stirred at room temperature for 20 min and then refluxed for 24 h. The reaction mixture was cooled to room temperature and the solid was isolated by a magnet and repeatedly washed with toluene and dried under vacuum at 80 °C for 7 h. The resulting MNPs were ultrasonically dispersed in dry toluene (5 ml) for 20 min at 60 °C and then imidazole (8 mmol) was added and the mixture was heated under reflux for 14 h. After cooling to room temperature, the new MNPs were collected and repeatedly washed with toluene and diethyl ether and dried under vacuum at 70 °C for 3 h to form CuFe₂O₄@SiO₂@C₃-Imid MNPs. After sonication of the later MNPs, in dry toluene (5 ml) for 15 min at 60 °C, 1,4-butane sultone (5 mmol) was added dropwise during 20 min and the mixture was refluxed for 6 h. After cooling to room temperature, the solid was collected using a permanent magnet and repeatedly washed with dry toluene and dried under vacuum at 70 °C for 2 h to form CuFe₂O₄@SiO₂@C₃-Imid-C₄SO₃ MNPs. These MNPs (1.0 g) were then ultrasonically dispersed in dry THF (5 ml) for 15 min at 60 °C and PW (1.5 mmol) was added and sonication continued for another 1 h. The resulting MNPs were isolated by magnetic decantation and washed with dry THF and dried under vacuum at 60 °C for 12 h to form CuFe₂O₄@SiO₂@C₃-Imid-C₄SO₃-PW MNPs^{xlv}.

General procedure for the synthesis of 1,8-dioxo-octahydroxanthenes 3a-k catalyzed by CuFe₂O₄@SiO₂@C₃-Imid-C₄SO₃-PW MNPs

To a mixture of dimeredone **1** (2 mmol), and an aldehyde **2a-k** (1 mmol), in a round bottom flask, CuFe₂O₄@SiO₂@C₃-Imid-C₄SO₃-PW (0.04 g) was added. The mixture was heated in the oil bath at 110 °C for 5-10 min. After completion of the reaction, monitored by TLC, the reaction mixture was cooled to room temperature and hot ethanol was added. The catalyst was recycled by magnetic decantation and washed with dry THF and dried under vacuum at 60 °C for 2 h. The combined filtrates were concentrated and allowed to stand at room temperature until

precipitation occurred. The precipitate was recrystallized from ethanol to give compounds **3a-k** in high yields.

3,3,6,6-Tetramethyl-9-phenyl-1,8-dioxo-octahydroxanthene (3a): ^1H NMR (300 MHz, DMSO- d_6 , δ , ppm): 0.88 (s, 6H, 2CH $_3$), 1.02 (s, 6H, 2CH $_3$), 2.16 (AB $_q$, $\Delta\nu = 56.7$ Hz, $J_{AB} = 16.2$ Hz, 4H, 2CH $_2$), 2.40-2.60 (m, 4H, 2CH $_2$ overlapped with solvent), 4.51 (s, 1H, CH), 7.05-7.25 (m, 5H, arom-H); FT-IR (KBr disk, ν , cm^{-1}): 3065, 2958, 1662, 1625, 1492, 1468, 1361, 1199, 1167, 1140, 1002, 892, 743, 700.

9-(4-Bromophenyl)-3,3,6,6-tetramethyl-1,8-dioxo-octahydroxanthene (3c): ^1H NMR (400 MHz, CDCl $_3$, δ , ppm): 1.00 (s, 6H, 2CH $_3$), 1.11 (s, 6H, 2CH $_3$), 2.17 (d, $J = 16.4$ Hz, 2H, CH $_2$), 2.25 (d, $J = 16.4$ Hz, 2H, CH $_2$), 2.47 (s, 4H, 2CH $_2$), 4.70 (s, 1H, CH), 7.18 (d, $J = 8.0$ Hz, 2H, arom-H), 7.34 (d, $J = 8.0$ Hz, 2H, arom-H); FT-IR (KBr disk, ν , cm^{-1}): 3035, 2951, 2876, 1661, 1624, 1486, 1469, 1408, 1360, 1198, 1166, 1139, 1070, 1008, 842.

9-(4-Chlorophenyl)-3,3,6,6-tetramethyl-1,8-dioxo-octahydroxanthene (3e): ^1H NMR (300 MHz, CDCl $_3$, δ , ppm): 1.13 (s, 6H, 2CH $_3$), 1.24 (s, 6H, 2CH $_3$), 2.30-2.55 (m, 8H, 4CH $_2$), 5.50 (s, 1H, CH), 7.02-7.30 (m, 4H, arom-H); FT-IR (KBr disk, ν , cm^{-1}): 3030, 2964, 2952, 1661, 1625, 1590, 1489, 1469, 1413, 1361, 1334, 1198, 1166, 1140, 1111, 1089, 1013, 1003, 981, 844.

9-(3-Nitrophenyl)-3,3,6,6-tetramethyl-1,8-dioxo-octahydroxanthene (3h): ^1H NMR (300 MHz, DMSO- d_6 , δ , ppm): 0.88 (s, 6H, 2CH $_3$), 1.02 (s, 6H, 2CH $_3$), 2.18 (AB $_q$, $\Delta\nu = 57.6$ Hz, $J_{AB} = 16.2$ Hz, 4H, 2CH $_2$), 2.55-2.60 (m, 4H, 2CH $_2$), 4.62 (s, 1H, CH), 7.50-7.70 (m, 2H, arom-H), 7.95-8.04 (m, 2H, arom-H); FT-IR (KBr disk, ν , cm^{-1}): 3059, 2957, 1655, 1621, 1529, 1475, 1352, 1238, 1200, 1166, 1141, 1002, 928, 815, 796.

9-(4-Hydroxyphenyl)-3,3,6,6-tetramethyl-1,8-dioxo-octahydroxanthene (3k): ^1H NMR (300 MHz, DMSO- d_6 , δ , ppm): 0.88 (s, 6H, 2CH $_3$), 1.01 (s, 6H, 2CH $_3$), 2.14 (AB $_q$, $\Delta\nu = 54.9$ Hz, $J_{AB} = 16.1$ Hz, 4H, 2CH $_2$), 2.42-2.58 (m, 4H, 2CH $_2$ overlapped with solvent), 4.40 (s, 1H, CH), 6.56 (d, $J = 8.3$ Hz, 2H, arom-H), 6.92 (d, $J = 8.3$ Hz, 2H, arom-H), 9.14 (s, 1H, OH); FT-IR (KBr disk, ν , cm^{-1}): 3412, 3023, 2963, 1663, 1614, 1595, 1514, 1450, 1425, 1361, 1245, 1200, 1166, 1108, 1003, 931, 839.

Results and discussion

In our initial study, the catalytic efficiency of CuFe $_2$ O $_4$ @SiO $_2$ @C $_3$ -Imid-C $_4$ SO $_3$ -PW was optimized in the synthesis of 9-(4-chlorophenyl)-3,3,6,6-tetramethyl-1,8-dioxo-octahydroxanthene **3e** via the reaction of dimedone **1** (2 mmol), with 4-chlorobenzaldehyde **2e** (1 mmol) in various reaction conditions (Table 1). Because of advantages of solvent-free conditions in chemical reactions including formation of cleaner products, simplification of work-ups, and environmental compatibility, we decided to investigate the efficiency of CuFe $_2$ O $_4$ @SiO $_2$ @C $_3$ -Imid-C $_4$ SO $_3$ -PW in the model reaction under solvent-free conditions in different catalyst amounts and temperatures. A blank, no catalyst, reaction that proceeded under solvent-free conditions at high temperature (entry 1) did not lead to a significant yield of **3e**. We were pleased to see that the reaction was efficiently catalyzed by CuFe $_2$ O $_4$ @SiO $_2$ @C $_3$ -Imid-C $_4$ SO $_3$ -PW under solvent-free conditions at elevated temperature leading to a high yield of product **3e**. The best result was conducted at 110 °C in the presence of 0.04 g of CuFe $_2$ O $_4$ @SiO $_2$ @C $_3$ -Imid-C $_4$ SO $_3$ -PW (entry 10). The higher amount of the catalyst or temperature had no significant effect on the yield and reaction time. Subsequently, our investigation showed that the compound **3e** can be obtained in good yield in different solvents including H $_2$ O, EtOH, MeOH, MeCN, and CHCl $_3$.

However, the reaction times are shorter under solvent-free conditions. Therefore, solvent-free conditions using 0.04 g of the catalyst at 110 °C was selected in all subsequent reactions.

With successfully optimized conditions in hand, and in order to evaluate the scope of this catalytic transformation, a range of *ortho*-, *meta*- and *para*-substituted aromatic aldehydes undergo reaction with dimedone under optimized conditions to afford 1,8-dioxo-octahydroxanthene derivatives (Table 2). As shown in Table 2, all electron-rich as well as electron-poor aromatic aldehydes reacted successfully and gave the products in high yields within short reaction time. However, it was found that the aromatic aldehydes with electron-withdrawing groups reacted faster than those with electron-donating groups as would be expected. These results clearly indicate that the CuFe₂O₄@SiO₂@C₃-Imid-C₄SO₃-PW acts as highly active catalyst in this methodology.

Table 1. Optimization of reaction parameters for synthesis of compound **3e** catalyzed by CuFe₂O₄@SiO₂@C₃-Imid-C₄SO₃-PW^a

Entry	Catalyst (g)	Solvent	T (°C)	Time (min)	Isolated Yield (%)
1	-----	-----	110	100	-----
2	0.01	-----	70	65	70
3	0.01	-----	90	40	84
4	0.01	-----	110	25	92
5	0.02	-----	90	30	90
6	0.02	-----	110	15	93
7	0.03	-----	90	20	90
8	0.03	-----	110	12	94
9	0.04	-----	90	15	92
10	0.04	-----	110	7	96
11	0.04	-----	120	7	95
12	0.06	-----	110	10	93
13	0.06	-----	120	10	93
14	0.04	H ₂ O	Reflux	25	85
15	0.04	EtOH	Reflux	30	82
16	0.04	MeOH	Reflux	45	80
17	0.04	MeCN	Reflux	30	77
18	0.04	CHCl ₃	Reflux	50	61

^aReaction conditions: dimedone **1** (2 mmol), and 4-chlorobenzaldehyde **2e** (1 mmol).

Table 2. Synthesis of 1,8-dioxo-octahydroxanthenes **3a-k** catalyzed by CuFe₂O₄@SiO₂@C₃-Imid-C₄SO₃-PW MNPs^a

Entry	Ar	Product	Time (min)	Isolated (%)	Yield	m.p. (°C)	
						Found	Reported
1	C ₆ H ₅	3a	8	89		204-206	201-203 [xix]
2	3-BrC ₆ H ₄	3b	7	90		190-192	188-190 [xxx]
3	4-BrC ₆ H ₄	3c	7	91		232-234	232-233 [xx]
4	2-ClC ₆ H ₄	3d	8	92		225-228	225-226 [xx]

5	4-ClC ₆ H ₄	3e	7	96	228-230	230-232 [xix]
6	4-MeC ₆ H ₄	3f	8	91	219-221	221-223 [xxvii]
7	4-MeOC ₆ H ₄	3g	10	89	246-248	247-249 [xxvii]
8	3-O ₂ NC ₆ H ₄	3h	6	94	174-176	171-173 [xxii]
9	4-O ₂ NC ₆ H ₄	3i	5	96	228-230	225-226 [xxii]
10	3-HOC ₆ H ₄	3j	10	88	223-225	224-226 [xxvii]
11	4-HOC ₆ H ₄	3k	10	90	248-250	244-246 [xxii]

^aReaction conditions: dimedone **1** (2 mmol), aldehyde **2a-k** (1 mmol), CuFe₂O₄@SiO₂@C₃-Imid-C₄SO₃-PW (0.04 g), 110 °C, solvent-free.

The recovery and catalytic activity of recycled CuFe₂O₄@SiO₂@C₃-Imid-C₄SO₃-PW MNPs was explored in the synthesis of compound **3e**. After the completion of the first reaction, the reaction mixture was cooled to room temperature and hot ethanol was added. The catalyst was retrieved from the reaction mixture using an external magnet and washed with dry THF and dried under vacuum at 60 °C for 2 h before being reused in the same reaction. The catalyst could be used at least four times with little loss of activity (96, 94, 92, and 89% yields for first to fourth use, respectively).

Conclusion

In conclusion, we showed that newly synthesized CuFe₂O₄@SiO₂@C₃-Imid-C₄SO₃-PW MNPs efficiently catalyze the synthesis of 1,8-dioxo-octahydroxanthenes in the reaction of dimedone with aromatic aldehydes under solvent-free reactions. Electron-poor aldehydes reacted slightly faster than electron-rich ones. In addition, the catalyst can be easily recovered by a magnet and reused in the next runs. Further applications of this new catalyst for other reaction systems are currently under investigation.

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