

One-Pot Synthesis of 1-Amidoalkyl-2-naphthols Catalyzed by Nano-SnCl₄.SiO₂

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Article history:

Received 2/5/2012

Accepted 13/7/2012

Published online 1/9/2012

Keywords:

Amidoalkyl naphthols

Nano-SnCl₄.SiO₂

Multi- component reaction

2-Naphthol

Abstract

Nano-SnCl₄.SiO₂ as a reusable and efficient catalyst was prepared from reaction of SnCl₄ with nano silica gel at room temperature. The structure of nano-SnCl₄.SiO₂ evaluated by ATR, and XRD. The SEM and TEM photograph of this catalyst improve the size of its particles in nanometer. 1-amidoalkyl-2-naphthols were prepared *via* multi-component reaction of 2-naphthol, aldehydes, and amides in the presence of nano-SnCl₄.SiO₂ under solvent free condition at 90 °C. Short reaction times, high yields, scale up and easy work-up are the advantages of this protocol.

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1. Introduction

Multi component reaction of 2-naphthol, aldehydes and amides in the presence of an acidic catalyst produce 1-amidoalkyl 2-naphthols as the important synthetic building blocks. 1-amidoalkyl 2-naphthols are precursors for the synthesis of 1-aminomethyl-2-naphthol derivatives which exhibit important cardiovascular, bradycardiac [1], and hypertensive [2] activity. Previously, some acidic catalysts such as [bmim]HSO₄ [3], cyanuric chloride [4], sulphamic acid [5], silica-supported molybdotophosphoric acid [6], H₃PW₁₂O₄₀ [7], H₄SiW₁₂O₄₀ [8], I₂ [9], [TEBSA][HSO₄] [10], KHSO₄ [11], P₂O₅ [12], silica sulfuric acid [13], zirconyl (IV) chloride [14], HClO₄-SiO₂ [15] and thiamine hydrochloride [16] have been utilized for synthesis of 1-amidoalkyl-2-naphthols. However, some of the catalysts used suffer drawbacks such as long reaction time, toxicity, cost and unavailability of the catalyst. Therefore, there is still need for a green catalyst which can overcome one or more drawbacks and also an environmentally benign procedure to synthesize of amidoalkyl naphthols.

Tin tetrachloride as a powerful lewis acid is a liquid which is highly volatile, corrosive, and difficult to handle. It is hydrolyzed to produce HCl in the presence of moisture. Silica-supported SnCl₄ [17, 18] is a mild solid Lewis acid that promotes acidic catalyzed organic reactions. This catalyst does not need special precautions for preparation, handling, or storage. It can be stored at an ambient temperature for months without losing its catalytic activity. This catalyst was previously applied for the one pot synthesis of β -acetamidoketones [19], acylals [20], and silylation of hydroxyl group [21].

Nano silica supported tin chloride (nano-SnCl₄.SiO₂) as an efficient acidic catalyst was prepared *via* a reaction of nano-SiO₂ with SnCl₄.

The dimensions of nanoparticles were observed with SEM. The particle sizes of the commercial silica gel and synthesized nano-SnCl₄.SiO₂ were about 24 nm and 42 nm (figure 1) respectively. Because the nano-SnCl₄.SiO₂ produces HCl in water, we have determined the acidic capacity of resin by titration. We have found that 1 g of catalyst produced 4.95 mmol of H⁺.

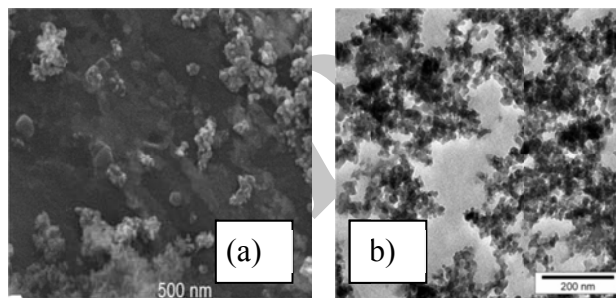


Fig. 1. a) SEM and b) TEM photograph of nano-SnCl₄.SiO₂

For the determination of the loading amount of Sn on 1 g of 50% SnCl₄.SiO₂, we have added hot water in resin and filtered the obtained Sn(OH)₄ as float solid. The calculated loading amount of Sn in catalyst is 63.6 mg g⁻¹. For the identification of the structure of nano-SnCl₄.SiO₂, we have studied ATR spectra of nano-SiO₂, nano-SnCl₄.SiO₂ and SnCl₄ (Figure 2). In all of the spectra, very broad OH stretching bands are observed with a strong intermolecular hydrogen bonding. In ATR spectra of nano-SnCl₄.SiO₂ and SiO₂, the absorption bands for Si-OH and Si-O-Si appear in ~700 cm⁻¹ and ~1100 cm⁻¹ respectively. In ATR spectrum of nano-SnCl₄.SiO₂, the O-Sn-Cl, Si-OH and Si-O-Si absorption bands are observed in 900, 700 and 1100 cm⁻¹ respectively.

The X-ray diffraction (XRD) patterns of nano-SiO₂ and nano-SnCl₄.SiO₂ are shown in figure 3. Nano-SiO₂ XRD pattern has a strong peak in 2 θ value of 21.8024° with FWHM equal to 0.1771. But nano-SiO₂ XRD pattern has strong peaks in 2 θ

values of 14.1121° and 21.7195° with FWHMs equal to 0.4723 and 1.4170 respectively.

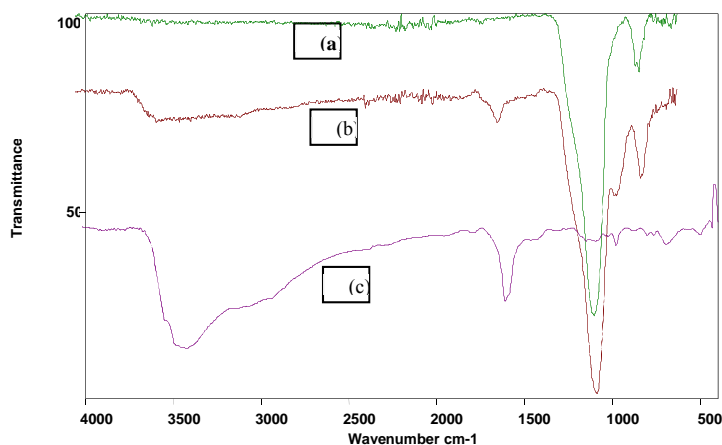


Fig. 2. ATR spectrum of: (a) nano-SiO₂, (b) nano-SnCl₄.SiO₂, and (c) SnCl₄

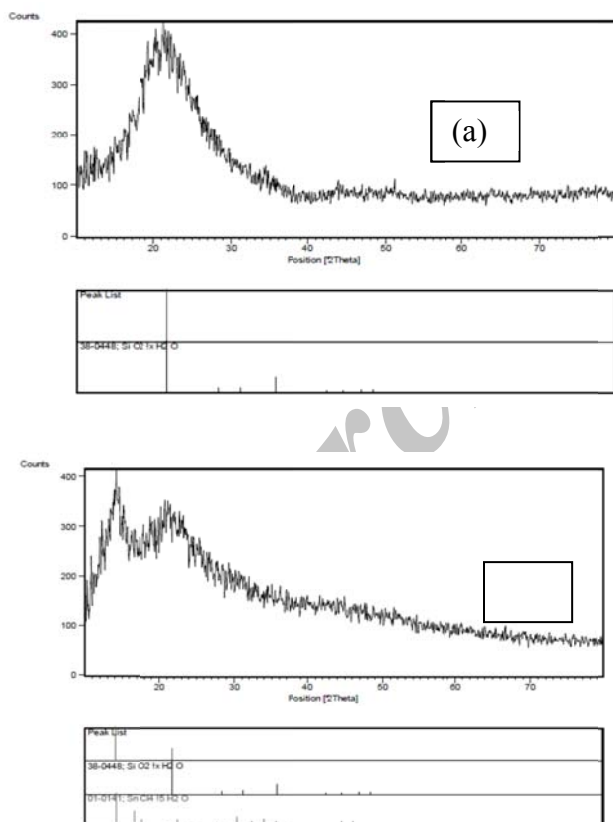


Fig. 3. X-ray diffraction (XRD) pattern of a) nano-SiO₂ and b) nano-SnCl₄.SiO₂.

Additional strong peak in 2θ value of 14.1121° show that Sn was bond to SiO₂ and formed proposed Si-O-Sn moiety. According to above data, we have suggested the following structure for nano-SnCl₄.SiO₂ (Fig. 4).

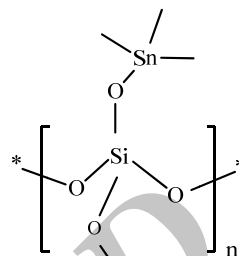


Figure 4: Suggested structure for nano-SnCl₄.SiO₂.

2. Experimental

2.1. Materials and Characterization

Products were characterized by FT-IR (ATR), ¹H-NMR and by comparison of their physical properties with those reported in the literature. FT-IR (ATR) spectra were run on a Bruker, Equinox 55 spectrometer. ¹H-NMR spectra were obtained using a Bruker Avans 400 MHz spectrometer (DRX). Melting points were determined by a Buchi melting point B-540 B.V.CHI apparatus. The SEM of nano particles determined with VEGA/TESCAN scanning electron microscope and TEM photograph was prepared by Leo 912AB OMEGA microscope. The X-ray diffraction (XRD) patterns of materials were recorded by employing a Philips Xpert MPD diffract meter equipped with a Cu K α anode ($\lambda = 1.54 \text{ \AA}$) in the 2θ range from 10 to 80° .

2.2. General procedure for the synthesis of 1-amidoalkyl-2-naphthols

A mixture of 2-naphthol (1mmol), aldehyde (1 mmol), amide (1.2 mmol) and 35% nano-SnCl₄.SiO₂ (0.015 g) was heated at 90°C . The

progress of the reaction was monitored by TLC. After completion of the reaction, the mixture was washed with chloroform and filtered to recover the catalyst. The filtrate was evaporated, and the crude product was re-crystallized from ethanol and chloroform (80:20) to obtain the pure 1-amidoalkyl 2-naphthols derivatives in 79–97% yields

3. Results and discussion

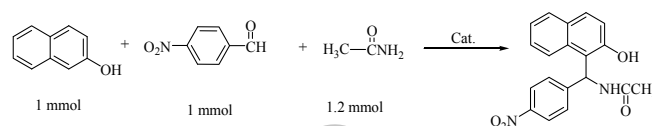
In continuation of our investigations on the application of solid acids in organic synthesis [19-26], we have investigated the synthesis of 1-amidoalkyl 2-naphthols in the presence of nano-SnCl₄.SiO₂ as an acidic catalyst. Initially to find the best reaction conditions, the reaction of 2-naphthol (1 mmol), 4-nitrobenzaldehyde (1 mmol) and acetamide (1.2 mmol) was performed under various conditions and different quantities of 35% SnCl₄.SiO₂ (table 1). According to the obtained data, the best condition is the reaction at 90 °C in solvent free condition and 0.015 g of 35% nano-SnCl₄.SiO₂ (entry 12, table 1). These conditions are comparable with other applied conditions. According to the best condition, we have synthesized 1-amidoalkyl-2-naphthols via condensation of 2-naphthol with various amides and aldehydes (scheme 1, table 2).

With aldehydes containing electron withdrawing group such as 4-nitrobenzaldehyde, the yield of product is higher in shorter time (entries 6-9, table 2). On the contrary, the aldehydes with electron releasing groups act with lower yields in longer times (entry 5, table 2).

Aliphatic aldehydes were also examined, but the yields of them were low as compared to those of products from aromatic aldehydes. The reactions of urea or thiourea with acetamide and 2-naphthol were examined, but no corresponding products were produced. Also, aniline was utilized and N-

[(2-hydroxynaphthalen-1-yl)-*p*-nitrophenyl-methyl]aniline as a green solid with high yield (90%) was produced (Scheme 2).

Table 1. Synthesis of *N*-[(2-hydroxynaphthalen-1-yl)-*p*-nitrophenyl-methyl]acetamide under various conditions



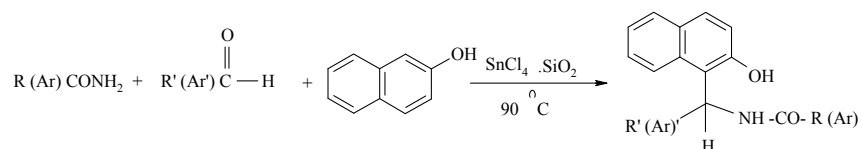
Ent.	Catalyst (g)	Condition Solvent/T °C	Time (h)/ yield ^a (%) ^{ref.}
1	20% SnCl ₄ .SiO ₂ (0.05)	70 °C	0.3/42
2	30% SnCl ₄ .SiO ₂ (0.05)	70 °C	0.3/65
3	50% SnCl ₄ .SiO ₂ (0.05)	70 °C	0.3/75
4	50% SnCl ₄ .SiO ₂ (0.05)	90 °C	0.3/92
5	50% SnCl ₄ .SiO ₂ (0.05)	EtOAc/Reflux	12/85
6	50% SnCl ₄ .SiO ₂ (0.05)	CHCl ₃ /Reflux	3/70
7	50% SnCl ₄ .SiO ₂ (0.05)	H ₂ O	24/0
8	50% SnCl ₄ .SiO ₂ (0.05)	EtOH	12/65
9	20% nano SnCl ₄ .SiO ₂ (0.01)	90 °C	0.2/63
10	20% nano SnCl ₄ .SiO ₂ (0.015)	90 °C	0.2/76
11	35% nano SnCl ₄ .SiO ₂ (0.01)	90 °C	0.2/73
12	35% nano SnCl ₄ .SiO ₂ (0.015)	90 °C	0.2/94
13	35% nano SnCl ₄ .SiO ₂ (0.015), 2 nd run	90 °C	0.2/84
14	35% nano SnCl ₄ .SiO ₂ (0.015), 3 rd run	90 °C	0.2/72
15	HClO ₄ /SiO ₂	S.F. /110	0.5/95 ¹⁵
16	HClO ₄ /SiO ₂	MW	0.2/91 ¹⁵
17	Silica chloride	MW	0.3/94 ²⁹
18	P ₂ O ₅	S.F./60	0.1/96 ¹²
19	[TEBSA][HSO ₄]	S.F./12	0.15/88 ¹⁰
20	KHSO ₄	S.F./10	0.5/96 ¹¹
21	TCT	S.F./10	0.7/93 ⁴
22	H ₃ PW ₁₂ O ₄₀	Et ₄ NCl/100	1.4/95 ⁷
23	MeSO ₃ H	S.F./85	0.4/ 92 ³⁰
24	H ₄ SiW ₁₂ O ₄₀	S.F./110	0.25/82 ⁸
25	H ₃ PMo ₁₂ O ₄₀ .xH ₂ O/SiO ₂	S.F./120	0.25/90 ⁶

^a Isolated yield.

This product was formed by reaction of 2-naphthol with imine (1) (scheme 3). The reaction among 4-nitrobenzaldehyde, phenol and benzamide produce N-[(2-hydroxybenzene-1-yl)-*p*-nitrophenyl

methyl]benzamide with medium yield (60 %)

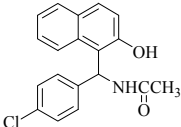
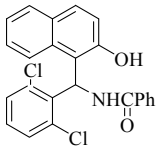
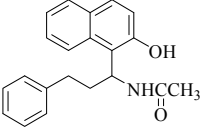
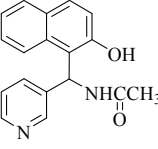
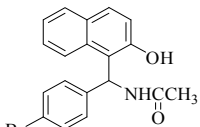
(Scheme 4).



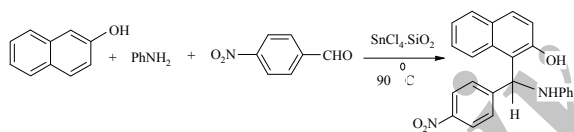
Scheme 1

Table 2. Synthesis of 1-amidoalkyl-2-naphthols in the presence of 35% nano-SnCl₄.SiO₂ in solvent free condition at 90 °C^a.

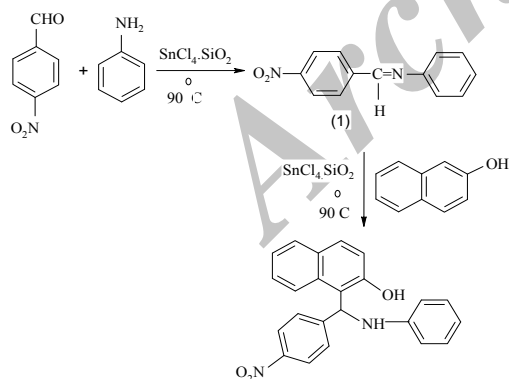
Ent.	R' (Ar')	R (Ar)	Product	Time (min)/ Yield (%) ^a	M.P. °C	
					Found	reported ^{ref}
1	2-MeO-C ₆ H ₄	CH ₃		40/81	240-241	241-242 ¹²
2	C ₆ H ₅	Ph		25/90	229-231	230-232 ⁴
3	3-NO ₂ -C ₆ H ₄	Ph		13/94	241-242	242-243 ¹²
4	4-MeC ₆ H ₄	Ph		30/83	209-210	209-211 ¹⁰
5	2-HOC ₆ H ₄	CH ₃		50/80	213-215	-
6	4-O ₂ NC ₆ H ₄	CH ₃		12/97	236-238	237-238 ¹²
7	4-O ₂ NC ₆ H ₄	Ph		15/95	226-228	228-229 ¹²
8	4-O ₂ NC ₆ H ₄	CH ₃ O		20/92	206-208	205-207 ³¹
9	4-O ₂ NC ₆ H ₄	H ₂ C=CH		18/93	231-232	-

10	4-ClC ₆ H ₄	CH ₃		20/93	230-231	229-231 ⁴
11	2,6-Cl ₂ C ₆ H ₃	Ph		14/92	214-215	-
12	C ₆ H ₅ CH ₂ CH ₂	CH ₃		65/79	191-192	-
13	3-pyridyl	CH ₃		20/93	192-193	-
14	4-BrC ₆ H ₄	CH ₃		20/96	231-233	230-232 ⁴

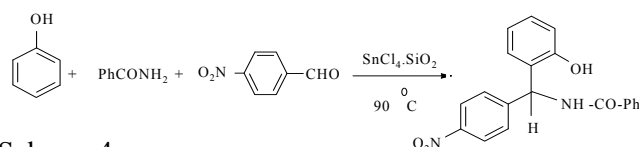
2-naphthol (1mmol), aldehyde (1 mmol), amide (1.2 mmol) and SnCl₄.SiO₂ (0.015 g) were used.



Scheme 2



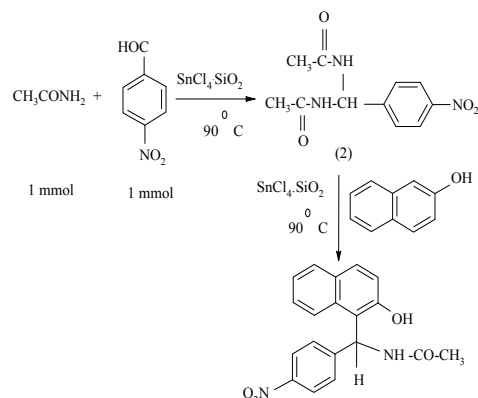
Scheme 3



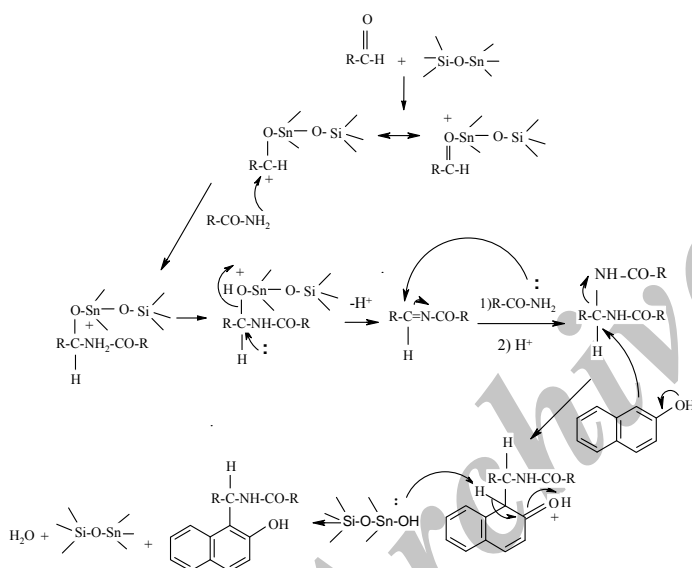
Scheme 4

Previously, two type of mechanism were suggested for 1-amidoalkyl-2-naphthols formation. In the first one, the ortho-quinonemethides (O-QMs) [3-9] and in the second one an N-acylimine [16] was introduced as intermediate. For investigation about mechanism of 1-amidoalkyl-2-naphthols formation, when we have reacted 1 mmol of 4-nitrobenzaldehyde with 1 mmol of 2- naphthol, only 14-(4-nitrophenyl)-14H-dibenzo[*a,j*]xanthene was formed without *o*-QMs intermediate. When we have reacted 1 mmol of acetamide with 1 mmol of 4-nitrobenzaldehyde in the presence of SnCl₄.SiO₂ in 90 °C, the product N, N'-alkylidenebis amide (2) was formed, isolated and identified. By reaction of (2) with 2- naphthol in the presence of SnCl₄.SiO₂ in 90 °C, the final product N-[(2-hydroxynaphthalen-1-yl)-*p*-nitro phenyl-methyl]acetamide was formed (scheme 5). According to our data, we have suggested a reasonable mechanism for the

formation of 1-amidoalkyl-2-naphthols in scheme 6.



Scheme 5



Scheme 6

4. Conclusion

In conclusion, a highly efficient synthesis of amidoalkynaphthols by the multi-component condensation of aromatic aldehydes, β -naphthol and urethane or amides catalyzed by nano-SnCl₄.SiO₂ is reported. This method offers significant advantages such as high conversions, easy handling, cleaner reaction profile and shorter reaction times which makes it a useful and

attractive process for the rapid synthesis of substituted amidoalkynaphthols.

Selected spectroscopic data

N-[(2-hydroxynaphthalen-1-yl)-*o*-hydroxy phenyl-methyl]acetamide (table 2, entry 5)

FT-IR (ATR, neat)=3387, 3300-3000, 1624, 1601, 1450, 1537, 750.¹HNMR (400 MHz, DMSO-d₆): 1.85 (s, 3H), 6.67 (brs, 2H), 6.97 (brs, 1H), 7.2 (m, 5H), 7.66 (brs, 1H), 7.72 (d, *J*=4.8 Hz, 1H), 8.11 (brs, 1H), 8.34 (brs, 1H), 9.38 (brs, 1H), 9.81 (brs, 1H) ppm.

N-[(2-hydroxynaphthalen-1-yl)-*p*-nitrophenyl-methyl]acrylamide (table 2, entry 9).

FT-IR (ATR, neat)= 3411, 3000-3200, 1650, 1624, 1602, 1439, 1514, 1506, 1340, 822, 747.

¹HNMR (400 MHz, DMSO-d₆): 5.6 (d, *J*=9.6 Hz, 1H), 6.1 (d, *J*=16 Hz, 1H), 6.6 (dd, *J*=16 and 9.6 Hz, 1H), 7.2 (m, 3 H), 7.37 (m, 3 H), 7.82 (m, 2 H), 8.1 (d, *J*=8.4 Hz, 2H), 8.85 (brs, 1H), 10.16 (s, 1H) ppm.

N-[(2-hydroxynaphthalen-1-yl)-2,6-dichlorophenyl methyl]benzamide (table 2, entry 11)

FT-IR (ATR, neat)=3419, 3300-3000, 1628, 1578, 1486, 1515, 743.¹HNMR (400 MHz, DMSO-d₆): 7.02 (d, *J*=7.6 Hz, 1H), 7.25 (m, 2H), 7.35 (d, *J*=6.8 Hz, 2H), 7.45 (m, 5H), 7.23 (d, *J*=7.6 Hz, 1H), 7.8 (d, *J*=8 Hz, 1H), 7.84 (d, 1H), 7.94 (brs, 2H), 9.19 (brs, 1H), 9.68 (brs, 1H) ppm.

N-[(2-hydroxynaphthalen-1-yl)-3-phenylpropyl]acetamide (table 2, entry 12)

FT-IR (ATR, neat)= 3421, 3350-3050, 1636, 1582, 1495, 1516, 740, 703.¹HNMR (400 MHz, DMSO-d₆): 1.99 (s, 3H), 2.3-2.8 (m, 4H), 7.12 (d, *J*=7.6 Hz, 1H), 7.16 (d, *J*=7.2 Hz, 1H), 7.23 (m, 3H), 7.32 (t, *J*=7.6 Hz, 2H), 7.43 (t, *J*=7.6 Hz, 2H), 7.66 (d, *J*=8.8 Hz, 1H), 7.77 (d, *J*=8 Hz, 1H), 8.01 (brs, 1H) ppm, ¹³CNMR (100 MHz, DMSO-d₆): 23.25, 33.03, 36.01, 46.22, 119.11, 120.08,

122.73, 123.02, 126.12, 126.56, 128.68, 128.71, 128.95, 132.75, 142.30, 153.60, 169.09 ppm.

N-[(2-hydroxynaphthalen-1-yl)-3-pyridyl-methyl]acetamide (table 2, entry 13).

FT-IR (ATR, neat)= 3300-3000, 1642, 1577, 1425, 1514, 743.¹HNMR (400 MHz, DMSO-d₆): 1.98 (s, 3H), 7.14 (d, *J*=7.6 Hz, 1H), 7.21 (d, *J*=8.8Hz, 1H), 7.26 (brs, 2H), 7.39 (brs, 1H), 7.51 (d, *J*=6.8 Hz, 1H), 7.81 (m, 3H), 8.36 (brs, 2H), 8.55 (d, *J*=7.2 Hz, 1H), 10.11 (s, 1H) ppm.

N-[(2-hydroxybenzene-1-yl)-*p*-nitrophenyl-methyl]benzamide (scheme 4).

FT-IR (ATR, neat)=3383, 3300-3200, 1649, 1579, 1486, 1548, 1505, 1342, 852, 717.¹HNMR (400 MHz, DMSO-d₆): 7.06 (brs, 1H), 7.49 (brs, 3H), 7.55 (brs, 2H), 7.73 (brs, 2H), 7.92 (brs, 4H), 8.25 (brs, 2H), 9.21 (brs, 2H) ppm.

N, N'-(4-nitrophenylmethylene) diacetamide (compound 2, scheme 5).

FT-IR (ATR, neat) = 3262, 3122, 1667, 1605, 1372, 1564, 1511, 1351, 825.¹HNMR (400 MHz, DMSO-d₆): δ = 1.86 (s, 6H), 6.53 (brs, 1H), 7.55 (d, *J*=8 Hz, 2H), 8.21 (d, *J*=8 Hz, 2H), 8.71 (brs, 2 N-H) ppm.

N-[(2-hydroxynaphthalene-1-yl)-*p*-nitrophenyl-methyl]aniline

FT-IR (ATR, neat)=3400-3000, 1601, 1456, 1341, 1510, 846, 743. ¹HNMR (400 MHz, DMSO-d₆): δ = 6.57 (d, *J*=8 Hz, 2 H), 6.76 (d, *J*=8Hz, 2 H), 7.01 (d, *J*=8Hz, 1H), 7.08 (d, *J*=8.4 Hz, 1H), 7.24 (t, *J*=8 Hz, 1H), 7.3 (d, *J*=8Hz, 1H), 7.38 (t, *J*=8.4 Hz, 1H), 7.56 (d, *J*=8 Hz, 1H), 7.65 (d, *J*=8 Hz, 1H), 7.75 (t, *J*=8 Hz, 2H), 8.1 (t, *J*=8Hz, 1H), 8.15 (d, *J*=8.4 Hz, 2H), 9.74 (s, N-H), 10.3 (s, OH).

Acknowledgment

The Research Council of Yazd University, Central Iron Ore and Strategic of Research Centre

gratefully acknowledged for the financial support for this work.

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