

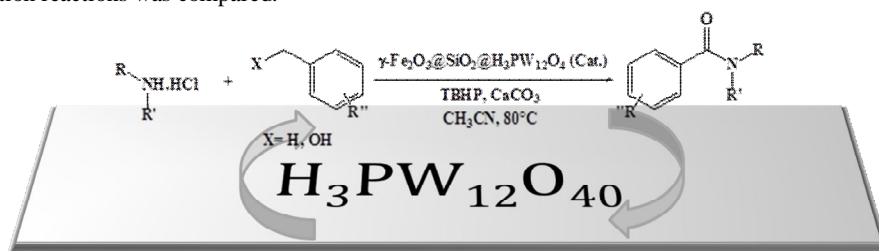
Tungstophosphoric Acid Supported on Silica-encapsulated γ -Fe₂O₃ Nanoparticles Catalyzed Oxidative Amidation

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We have used tungstophosphoric acid to catalyze oxidative amidation reaction from benzyl alcohols and methylarenes with hydrochloride salts of amines. To achieve this purpose, modified magnetic nanoparticles (γ -Fe₂O₃@SiO₂@H₃PW₁₂O₄₀) were applied as catalyst and TBHP as external oxidant. After optimizing, different derivatives of benzamides were synthesized in good yields. Also, the result of two oxidative amidation reactions was compared.



Keywords: Oxidative amidation, Amid, Magnetic nanoparticles, Catalytic reaction

INTRODUCTION

Amide bond formation is an interesting reaction because of its importance in synthesis of polymers, natural products, pharmaceuticals and fine chemicals. Also, its widespread presence in biological compounds such as proteins and peptides, is brilliant. Amide synthesis can be occurred in both catalytic and no catalytic conditions. Classical methods, with no catalyst, are incorporated when carboxylic acid be activated by some reagents such as phosgene or thionylchloride which is limited with different problems including environmental harm, reagent toxicity, side reactions and so on. Additionally, in these methods, an extra oxidation step must be occurred (the conversion of aldehydes or alcohols to carboxylic acid), which is another problem. Because of the mentioned restrictions, one pot oxidative amidation systems by various metal catalysts such as Fe [1,

2], Cu [3,4,5], Pd [6], Zn [7,8], Ru [9], Au [10], La [11], Mn [12] and metal free [13] are developed. Recently, some advanced procedures have been reported, such as: Amidation of amines and carboxylic acids [14], transamidation of primary amides [15], alkynes [3,16], oxidative amidation of alcohols with amines (ammonia, primary and secondary amines) [2,7,9,10,17], aldehydes with amines (ammonia, primary and secondary amines) [5, 18], and alkynes with amines and azides [19]. In most of these methods, homogenous catalysts are provided.

Heteropolyacids are efficient, practical and green acids. They reveal catalytic role as an oxidative reagent in different vital reactions. Diverse oxidation systems have been reported to prove the cooperation of heteropolyacids and various external oxidant. Bamoharramet *et al.* [20] used [NaP₅W₃₀O₁₁₀]¹⁴⁻ system for oxidation of aromatic aldehydes. Ishi *et al.* applied hydrogen peroxide together with heteropolyacids for epoxidation of olefins and allylic alcohols, ketonization of alcohols and diols and oxidation of

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1,2-diols and olefins [21]. Titania-supported heteropolyacid has been used by Popa *et al.* for ethanol oxidation. Several species of heteropolyacids have been used by Tundo *et al.* to oxidize the alcohols and sulfides with hydrogen peroxide [22]. Guldi and Wasserscheid have reported spectroscopic and electrochemical characterization of heteropolyacids for selective biomass oxidation to formic acid [23].

EXPERIMENTAL

Catalyst Synthesis

Procedure for catalyst synthesis has been reported previously [21]. Briefly, 5 mmol $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ and 2.5 mmol $\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$ salts were dissolved in 100 ml deionized water under vigorous stirring (800 rpm), then NH_4OH solution (25%, w/w, 30 ml) was added to the above mixture at room temperature until the pH was raised to 11. The addition of NH_4OH solution followed to maintain pH of the reaction between 11 and 12 at which a black suspension was formed. The resulting black dispersion was continuously stirred for 1 h at room temperature and then refluxed for 1 h. Coating a layer of silica on the surface of the $\gamma\text{-Fe}_2\text{O}_3$ nanoparticles was achieved by adding ethanol (40 ml) to purified nanoparticles then heated for 1 h at 40 °C. Subsequently, tetraethylorthosilicate (TEOS, 10 ml) was charged to the reaction vessel, and the mixture was continuously stirred for 24 h. The silica-coated nanoparticles were collected by a magnet, followed by washing five times with EtOH, diethylether and drying at 100 °C in vacuum for 12 h. Prepared nanoparticles at this stage are heated at 300 °C in furnace for 3 h to convert $\text{Fe}_3\text{O}_4 @ \text{SiO}_2$ to sustainable $\gamma\text{-Fe}_2\text{O}_3 @ \text{SiO}_2$ nanoparticles. Tungstophosphoric acid (1 g) was added to 1 g former suspension of $\gamma\text{-Fe}_2\text{O}_3 @ \text{SiO}_2$ nanoparticles in water and mixed in room temperature for 24 h. Residue was collected by a magnet, followed by washing 3-4 times with distilled water and then dried for 24 h at 80 °C in oven. The desired catalyst was formed.

General Procedure

Oxidative amidation of benzyl alcohol. Hydrochloric salts of amines (1.5 mmol, 1 equiv.) and Calciumcarbonate (2.5 equiv.) were dissolved in acetonitril (4 ml) and $\gamma\text{-Fe}_2\text{O}_3 @ \text{SiO}_2\text{-H}_3\text{PW}_{12}\text{O}_{40}$ (20 mg) was added, then benzyl alcohol (0.75 mmol, 1.5 equiv.) and TBHP (5 equiv., 70

wt% in H_2O) were added. The mixture was stirred at 80 °C for 6 h. After finishing reaction, Hydrogenchloride (1 N, 5 ml) and EtOAc (5 ml) were added, then magnetic nanoparticles were separated from reaction medium by an external magnet. The mixture was extracted with EtOAc and the organic phase was washed with saturated aqueous solution of NaHCO_3 (10 ml). The solvent was dried over anhydrous Na_2SO_4 , then removed under vacuum and the crude product was obtained. Mixture of products were purified by column chromatography to obtain our desired product.

Oxidative amidation of toluene (methylarens). As the same, Hydrochloric salts of amines (1.5 mmol, 1 equiv.) and calciumcarbonate (2.5 equiv.) were dissolved in acetonitril (4 ml) and $\gamma\text{-Fe}_2\text{O}_3 @ \text{SiO}_2\text{-H}_3\text{PW}_{12}\text{O}_{40}$ (20 mg) was added, then Toluene, instead of benzylalcohol, (0.5 ml) and TBHP (8 equiv., 70 wt% in H_2O) were added and the mixture was stirred at 80 °C under Ar gas for 10 h. After finishing the reaction, our desirable product was gained by the same purifying manner.

RESULT AND DISCUSSION

Previously, we reported tungstophosphoric acid supported on silica encapsulated $\gamma\text{-Fe}_2\text{O}_3$ ($\gamma\text{-Fe}_2\text{O}_3 @ \text{SiO}_2\text{-H}_3\text{PW}_{12}\text{O}_{40}$) as a magnetic, recoverable catalyst in synthesis of formamidines [24] and nitrones [25]. Also, we reported different procedures of oxidative amidation of alcohols [26]. In this regard, to achieve new applicable, more eco-friendly method for oxidative amidation reactions we considered $\gamma\text{-Fe}_2\text{O}_3 @ \text{SiO}_2 @ \text{H}_3\text{PW}_{12}\text{O}_{40}$ as superparamagnetic heterogeneous catalyst to accelerate oxidative amidation of benzyl alcohol (Fig. 1) and methyl arenes (Fig. 2) with amine hydrochloride salt.

Various primary, secondary and tertiary amides were synthesized by this method. The synthesized *N*-benzyl benzamide (compound 1d) is chosen as a model reaction to optimize the oxidative amidation conditions (Table 1).

The results are summarized in Table 1. The first reaction is carried out under the following conditions: benzyl alcohol (1 mmol), amine hydrochloride salt (1.5 mmol), no catalyst, TBHP as oxidant (8 equiv.), CaCO_3 as base (2.5 equiv.), and CH_3CN (4 ml) at 80 °C. The corresponding product is obtained lower than 10% yield (Table 1, entry 1). Addition

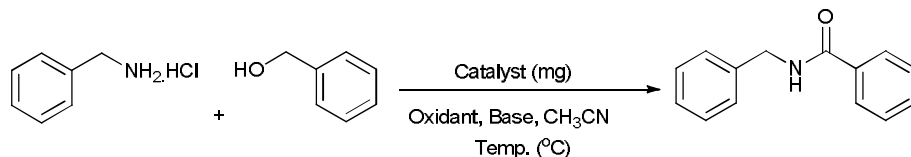


Fig. 2. Schematic of oxidative amidation reaction from benzyl alcohol.

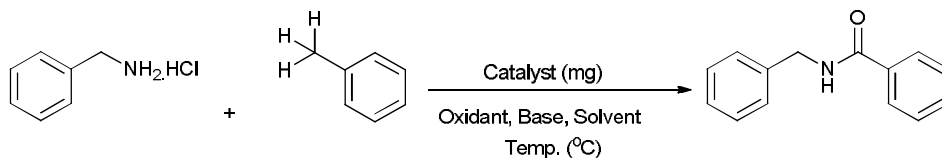


Fig. 2. Schematic of oxidative amidation reaction from benzyl alcohol.

Table 1. The Results of Screening the Optimal Conditions^a

Entry	Catalyst	(mg)	Oxidant	Base	Temp. (°C)	Yield (%) ^b
1	-	10	TBHP	CaCO ₃	80	<10
2	N.P.	10	TBHP	CaCO ₃	80	<10
3	N.P.@HPW	10	-	CaCO ₃	80	N.D.
4	N.P.@HPW	10	TBHP	CaCO ₃	25	35
5	N.P.@HPW	10	TBHP	CaCO ₃	60	69
6	N.P.@HPW	10	TBHP	CaCO ₃	80	87
7	N.P.@HPW	10	TBHP	Na ₂ CO ₃	80	<10
8	N.P.@HPW	10	TBHP	K ₂ CO ₃	80	51
9	N.P.@HPW	10	TBHP	NaOH	80	29
10	N.P.@HPW	10	TBHP	Et ₃ N	80	35
11	N.P.@HPW	10	H ₂ O ₂	CaCO ₃	80	50
12	N.P.@HPW	10	mCPBA	CaCO ₃	80	25
13	N.P.@HPW	10	UHP	CaCO ₃	80	25
14	N.P.@HPW	5	TBHP	CaCO ₃	80	40
15	N.P.@HPW	20	TBHP	CaCO ₃	80	91
16	N.P.@HPW	25	TBHP	CaCO ₃	80	91

^aAll reactions were performed with benzyl alcohol (1 mmol), benzyl amine hydrochloride salt (1.5 mmol), CH₃CN (4 ml), oxidant (5 equiv.), and Base (2.5 equiv.) under Ar for 6 h.

^bIsolated yield.

Table 2. The Results of Screening the Optimal Conditions^a

Entry	Catalyst	(mg)	Oxidant	Solvent	Base	Temp. (°C)	Yield (%)
1	N.P.@HPW	10	TBHP	Toluene	CaCO ₃	100	45
2	N.P.@HPW	10	TBHP	Solv. Sys. ^c	CaCO ₃	80	45
3	N.P.@HPW	10	TBHP	Solv. Sys. ^d	CaCO ₃	80	40
4	N.P.@HPW	10	TBHP	Solv. Sys. ^e	CaCO ₃	80	35
5	N.P.@HPW	10	TBHP	Solv. Sys. ^f	CaCO ₃	80	67
6	N.P.@HPW	10	TBHP	Solv. Sys. ^f	CaCO ₃	25	36
7	N.P.@HPW	10	TBHP	Solv. Sys. ^f	CaCO ₃	60	54
8	N.P.@HPW	10	TBHP	Solv. Sys. ^f	CaCO ₃	80	59
9	N.P.@HPW	10	TBHP	Solv. Sys. ^f	Na ₂ CO ₃	80	<10
10	N.P.@HPW	10	TBHP	Solv. Sys. ^f	K ₂ CO ₃	80	33
11	N.P.@HPW	10	TBHP	Solv. Sys. ^f	NaOH	80	37
12	N.P.@HPW	10	TBHP	Solv. Sys. ^f	Et ₃ N	80	29
13	N.P.@HPW	10	H ₂ O ₂	Solv. Sys. ^f	CaCO ₃	80	45
14	N.P.@HPW	10	mCPBA	Solv. Sys. ^f	CaCO ₃	80	25
15	N.P.@HPW	10	UHP	Solv. Sys. ^f	CaCO ₃	80	55
16	N.P.@HPW	5	TBHP	Solv. Sys. ^f	CaCO ₃	80	32
17	N.P.@HPW	15	TBHP	Solv. Sys. ^f	CaCO ₃	80	74
18	N.P.@HPW	20	TBHP	Solv. Sys. ^f	CaCO ₃	80	80
19	N.P.@HPW	25	TBHP	Solv. Sys. ^f	CaCO ₃	80	80
20	N.P.@HPW	-	TBHP	Solv. Sys. ^f	CaCO ₃	80	<10
21	N.P.@HPW	20	-	Solv. Sys. ^f	CaCO ₃	80	N.D.
22	N.P.	20	TBHP	Solv. Sys. ^f	CaCO ₃	80	<10

^aAll reactions were performed with benzylamine hydrochloride salt (1 mmol), CH₃CN (2.5 ml), oxidant (8 equiv.), toluene (0.5 ml), and base (2.5 equiv.) under Ar at 80 °C for 10 h. ^bIsolated yield. ^cTHF/ Toluene (5/1). ^dEthyl acetate/Toluene (5/1). ^eDioxane /Toluene (5/1). ^fAcetonitrile/Toluene (5/1).

of γ -Fe₂O₃@SiO₂ (Nano particle, summarized in Table 1. as N.P.) has no significant effect on the reaction yield (Table 1, entry 2) indicating that the presence of a catalyst is necessary for this reaction.

The influence of CaCO₃ is better than other bases such as K₂CO₃, Na₂CO₃, NaOH and Et₃N (Table 1, entries 6-10). In the next step, temperature role is investigated, which shows increasing to 80 °C has crucial effect on the yield of product (Table 1, entries 4-6). Next, the effect of oxidant on the reaction yield is investigated. Therefore, the reaction has been carried out in the presence of several oxidizing reagents, such as: *m*-CPBA, H₂O₂, and UHP, which the result is the same as oxidant-free conditions. But TBHP is different; no product is observed in the absence of any oxidant (Table 1, entries 11-14 and 3). This shows that the presence and type of oxidant are important for this reaction. Using 5 mg of the catalyst has undesirable effect. The optimum amount of catalyst is 20 mg, and the yield is not significantly enhanced by increasing this amount to 25 mg (Table 1, entries 14-16).

For oxidative amidation of methyl arens, similarly, the synthesized *N*-benzyl benzamide (compound 1d) is chosen as the model reaction to optimize the conditions, because the results can be compared (Table 2). The first reaction is carried out under the following conditions: benzylamine hydrochloride salt (1.5 mmol), γ -Fe₂O₃@SiO₂-H₃PW₁₂O₄₀ as catalyst (10 mg), TBHP as oxidant (8 equiv.), CaCO₃ as base (2 equiv.), and toluene (0.5 ml) at 100 °C. The corresponding product is obtained in 45% yield (Table 2, entry 1). *N*-benzyl benzamide is synthesized in lower than 10% yield in the absence of any catalyst (Table 2, entry 21 and 23). This shows necessity of catalyst presence for this reaction is the same as operation for benzyl alcohol. To improve the yield of product, other conditions were optimized. The result of base effect is the same as benzyl alcohol reaction, the influence of CaCO₃ was better than other bases such as K₂CO₃, Na₂CO₃, KOH and Et₃N (Table 2, entries 8-12). In temperature optimizing, 80 °C is the best for operating reaction. Increasing temperature from 25 °C to 60 °C and finally 80 °C is important because of solving amine hydrochloride salts. In this condition, base can be effective for neutering acidity of salt. Next, the effect of different solvent systems is checked. To compare THF/toluene, ethyl acetate/toluene, dioxane/toluene, acetonitrile/

toluene, as binary solvents (solvent system), toluene with two roles simultaneously, and acetonitrile/toluene is found the best solvent system (Table 2, entry 2-5). Raw toluene, because of poor solubility of amine salts, even in high temperatures, and catalyst agglomeration, even with sonication, are not an applicable medium. Thus, solvent systems, including mixture of two solvents, have been used. Reactions were carried out by several types of oxidizing reagents, such as: *m*-CPBA, H₂O₂, and UHP, and in oxidant-free conditions, too. TBHP is chosen as an effective oxidant. In the absence of any oxidant, yield of reaction is troublesome and no product is detected (Table 2, entries 22).

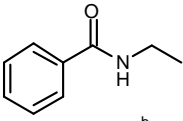
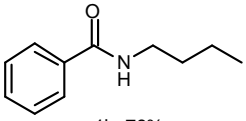
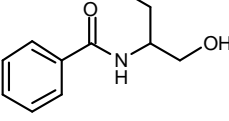
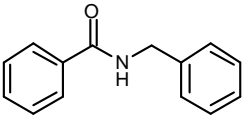
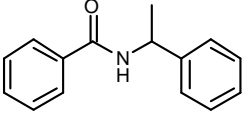
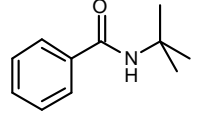
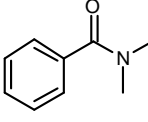
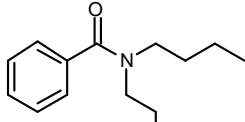
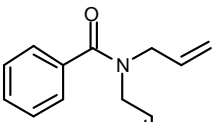
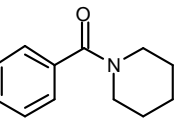
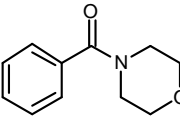
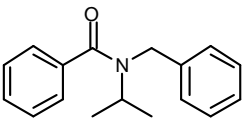
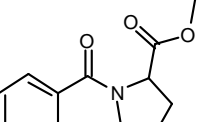
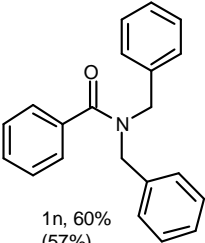
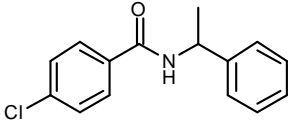
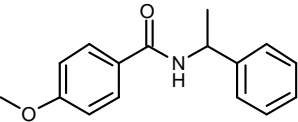
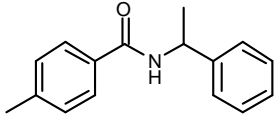
Optimum conditions as follows: for the first reaction, benzyl alcohol (1 mmol), benzylamine hydrochloride salt (1.5 mmol), γ -Fe₂O₃@SiO₂-H₃PW₁₂O₄₀ as catalyst (10 mol%), TBHP as oxidant (5 equiv.), and CaCO₃ as base (2.5 equiv.) at 80 °C. For the second reaction, amines hydrochloride salt (1 mmol), CH₃CN (2.5 ml), oxidant (8 equiv.), toluene (0.5 ml), and base (2.5 equiv.) under Ar at 80 °C for 10 h. Series of primary, secondary and tertiary amides are synthesized by reacting various amine hydrochloride salts (aliphatic and aromatic) with toluene (Table 2). As can be seen in Table 2, aliphatic amines like propylamine, butyl amine, and 2-amino-1-butanol and *tert*-butyl amine transmittes to relevant amides in good yields (respectively 50 (48), 78 (73), 66 (58) and 9 (80)%). Notably, no oxidation occurs in alcoholic function in 1c. Benzyl amine and methylbenzylamine coupled with benzyl alcohol (and toluene) in 91(80)% (1d) lead to 93 (90)% (1e) yield. Various secondary amines (cyclic and aliphatic) are also under the reaction conditions and corresponding tertiary amides are synthesized in moderate to good yields (1g-n).

Decreasing the isolated yields for some cases, because of steric factors, is a predictable outcome. All these products were characterized by recording melting points (in some cases), IR, ¹H NMR, and ¹³C NMR spectra.

CONCLUSIONS

We have developed an efficient and direct catalytic oxidative synthesis of primary, secondary and tertiary amides from alcohols and methylarens catalyzed by

Table 3. Preparation of Various Benzamides in the Presence of $\gamma\text{-Fe}_2\text{O}_3@SiO_2\text{-H}_3PW_{12}O_{40}$ ^a

 1a, 50% ^b (48%) ^c	 1b, 78% (73%)	 1c, 66% (58%)	 1d, 91% (88%, 83%, 79%) ^d (80%)
 1e, 93% (90%)	 1f, 61% (55%)	 1g, 70% (65%)	 1h, 56% (52%)
 1i, 68% (65%)	 1j, 74% (73%)	 1k, 82% (82%)	 1l, 52% (50%)
	 1m, 75% (74%)	 1n, 60% (57%)	
 1o, 78% (71%)	 1p, 50% (49%)	 1q, 79% (70%)	

^aReaction conditions: benzyl alcohol (1 mmol), benzyl amine hydrochloride salt (1.5 mmol), CH_3CN (4 ml), oxidant (5 equiv.), and Base (2.5 equiv.) under Ar at 80 °C for 6 h. ^bYield of benzyl alcohol reaction is presented in parentheses. ^cYield of toluene reaction is presented in parentheses. ^dThe reaction was accomplished with recycled catalyst.

heteropolyacids supported on $\gamma\text{-Fe}_2\text{O}_3@SiO_2$. This process provides a practical synthesis for different amide derivatives. More studies about other catalytic methods, in this reaction class, are ongoing.

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