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H-β -Zeolite Catalyzed Transamidation of Carboxamides, Phthalimide, Formamides and Thioamides with Amines under neat Conditions

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Efficient transamidation of unactivated carboxamides, phthalimides, formamides and thioamides with amines under solvent-free conditions using H-β-zeolite as a green and recyclable heterogeneous catalyst is described. Easy work up, high purity of the products, recyclability and environmentally-friendly nature of the catalyst are the attractive features of the present methodology. This is the first report for the transamidation of thioamides under heterogeneous conditions.

Amides are important functional groups used in many organic transformations, also they are basic functional moieties in both chemistry and biology.[1] Amides are versatile synthetic intermediates for the construction of pharmacologically important molecules containing nitrogen and oxygen heterocycles (Fig. 1).[2] Generally, amides are prepared by the reaction of carboxylic acid derivatives with amines.[3] These methods suffer from common limitations such as stoichiometric amounts of coupling reagents, poor atom efficiency and formation of the large amount of by-products.[4] To overcome these problems, novel amide bond forming reactions have been developed in recent years.[5]

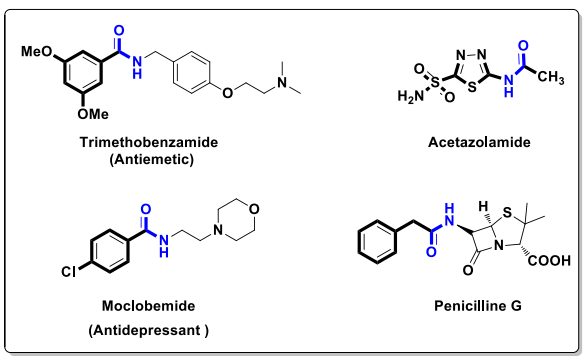


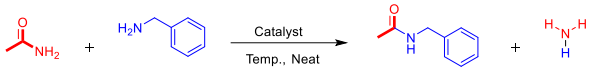
Figure 1. Drugs containing amide moiety.

Transamidation is also a convenient and straightforward method to obtain the desired amides, in conventional transamidation reactions it requires harsh conditions, longer reaction time and high temperatures (> 250º C) for breaking the amide bond because of the poor electrophilic character of the amide carbonyl group.[6] An enzyme mediated transformation was also reported, however it requires very longer reaction time up to six days.[7] To overcome these disadvantages, efforts were made by several research groups using metal-catalysed homogeneous[8] and heterogeneous[9] transamidation reactions. In addition, very recently metal-free catalysed transamination reactions were also reported, such as boric acid, hypervalentiodine, hydroxylamine hydrogen chloride, borate esters, benzoic acid, ionic liquids, 1,4-dioxane, benzotriazole, imidazole, N,N-dialkylformamide dimethyl acetals.[10] These methods also suffers from certain drawbacks, such as the separation and purification of the target products from the reaction mixture, selectivity and limited substrate scope.

Recently, we have developed an efficient organocatalyzed transamidation reactions under homogeneous as well as heterogeneous conditions.[11] In continuation of our interest and the advantages of these heterogeneous catalysis,[12,13] herein we report a novel transamidation of carboxamides and thioamides with amines using H-β-zeolite as a heterogeneous catalyst. This catalyst has an attractive features such as high reactivity, solvent-free, environmentally friendly nature of the catalyst, easy separation and recyclability.

We started our studies on the transamidation of acetamide **1a** with benzyl amine **2a** as model substrates (Table 1). First, the reaction was performed without any catalyst and solvent, the complete lack of reactivity was observed even at 100°C (Table 1, entry 1). Employing 5 wt. % of H-β-zeolite as a catalyst under solvent-free condition, the reaction resulted 88% of the desired transamidation product **3a** (Table 1, entry 2). Further to increase the yield of the transamidation product, we raised the temperature to 130ºC along with 10wt. % of catalyst loading; yield of product was increased to 93% (Table 1, entry 3). However, with increase of catalyst to 20 wt.% under these conditions we isolated the corresponding transamidation product in 97% yield (Table 1, entry 4). Further no improvements were observed when the reaction carried out in other solvents like DMA, DMF, DMSO, Xylene, Mesitylene and Toluene (Table 1, entries 5-10). When lowering

Table 1. Optimisation studies on reaction of acetamide with benzylamine[a]



1a 2a 3a

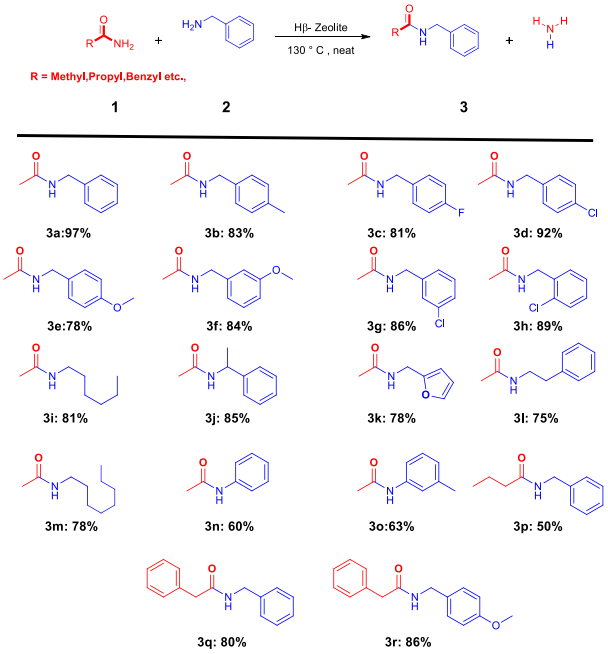
S.no.	Catalyst (wt.%)	Solvent	Temperature (°C)	Yield (%)[b]
1	-	-	100	nr
2	Hβ Zeolite (5)	-	130	88
3	Hβ Zeolite (10)	-	130	93
4	**Hβ Zeolite (20)**	**-**	**130**	**97**
5	Hβ Zeolite (20)	DMA	130	35
6	Hβ Zeolite (20)	DMF	130	63
7	Hβ Zeolite (20)	DMSO	130	32
8	Hβ Zeolite (20)	Xylene	130	72
9	Hβ Zeolite (20)	Mesitylene	130	81
10	Hβ Zeolite (20)	Toluene	130	89
11	Hβ Zeolite (20)	-	120	84
12	Hβ Zeolite (20)	-	100	57
13	Naβ Zeolite (20)	-	130	79
14	Amberlyst-15 (20)	-	130	72
15	HZSM-5 (20)	-	130	52
16	-	-	130	18
17	-	-	150	26

[a] Conditions: 4 mmol of **1a**, 4 mmol of **2a**, catalyst, and solvent (2 mL were used, 24 hrs. [b]Isolated yields after column chromatography.

the reaction temperature from 120 ºC to 100 ºC, under these conditions, the conversion also declined (Table 1, entries 11 and 12). Under similar reaction conditions we are also checked the other solid acid catalysts like Na-β-zeolite, amberlyst-15 and HZSM-5 and observed moderate yields (Table 1, entries 13-15). In addition, we performed the reactions at 130 ºC and 150 ºC without catalyst, but low yield was observed (Table 1, entries 16 and 17). Based on the above optimized reaction conditions, we explored the substrates scope; a variety of benzylamines smoothly coupled with acetamide and gave the corresponding transamidation products. The transamidation of acetamide with a variety of benzylamines like electron donating as well as moderately withdrawing groups on the phenyl ring resulted the transamidation products (**3a-h**) in good to excellent yields (78-97%). The long chain amines like hexylamine, α-methyl benzyl amine, furfurylamine and phenyl ethylamine also gave good yields of 67-81% (**3i-3l**). The present methodology not only applicable for benzylic amines but also works for octyl amine as well as anilines (**3m-3o**). These conditions also well tolerated to the other amides such as extending aliphatic amides and benzamides giving good yields of corresponding transamidation products (**3p -3r**). With the above optimization conditions, we extended the transamidation of different amides like aromatic, hetero aromatic, phthalimides and formamides with various amines (Table 3). Under these optimised conditions substituted benzyl amines as well as benzamide (electron donating and withdrawing) groups easily converted to the corresponding transamidation products (**4a-4d**) in moderate to good yields.

In addition, aliphatic amines could be transformed into desired transamidation products in good yields (**4e-4f**). Notably, isonicitinamide and thiophene carboxamides as examples of

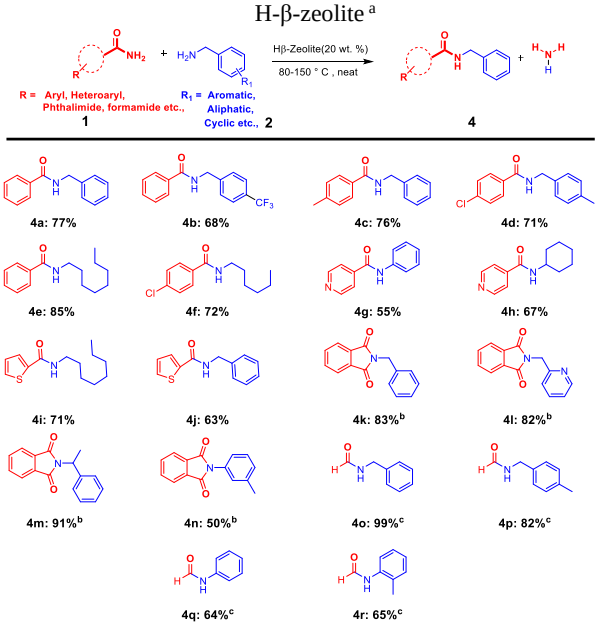
Table 2. Scope for transamidation of different amides amines [a]



[a]Reaction conditions: 4 mmol of **1**, 4 mmol of **2**, catalyst 20.wt.% at 130 °C for 24 hours, isolated yield.

heterocyclic amides were successfully converted into transamidation products in good yields (**4g-4j**). Similarly, the corresponding transamidation products were also obtained in good to excellent yields with phthalimides and formamides as the amide sources with different amines (**4k-4r**).

Table 3. Broad substrate scope of the transamidation catalysed by H-β-zeolite [a]



[a]Reaction conditions: 4 mmol of **1**, 4 mmol of **2**, catalyst 20.wt. % at 150 °C for 30 hrs, isolated yield. [b]Reaction carried out at 150 °C for 36 hrs. [c]Reaction carried out at 80 °C for 24 hrs.

After successful demonstration of transamidation of carboxamides with H-β-zeolite as heterogeneous catalyst, we

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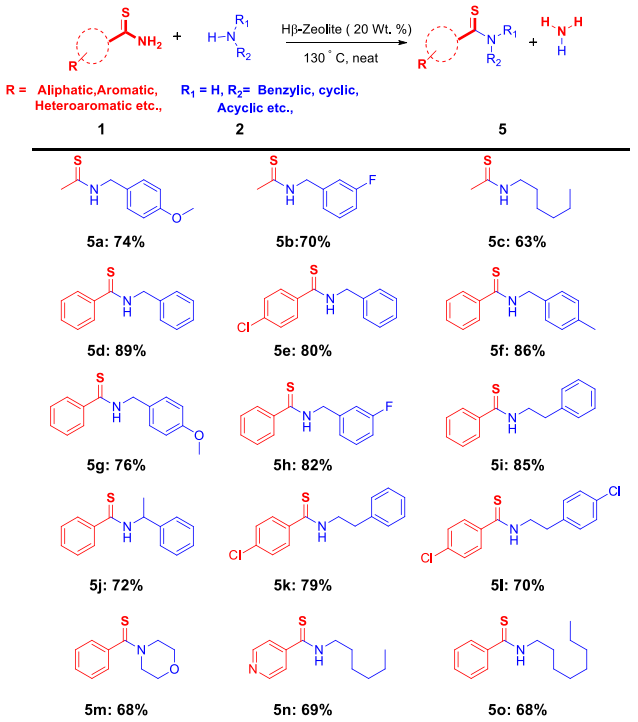
turned our attention towards the transamidation of thioamides, as thioamides are also core units in heterocyclic chemistry, material science and they are useful intermediates in many organic transformations.[14a-f] Compared to transamidation of carboxamides, very few reports are available for transamidation of thioamides under homogeneous conditions, but to the best of our knowledge, no reports exist on heterogeneous conditions for transamidation of thioamides.

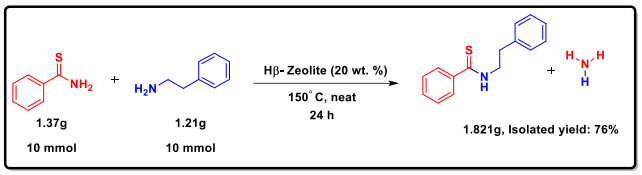
We report herewith transamidation of thioamides with both aliphatic and aromatic amines under heterogeneous conditions (Table 4). The present method also works very well for various thioamides and amines. Substituents bearing electron donating and withdrawing groups on the phenyl ethyl amine and phenyl propyl amines underwent to this procedure and generated the corresponding products in good yields (**5a**-**5e**). Furthermore, α-methyl benzyl amine and cyclohexyl amines were also gave moderate to good yields (**5f** & **5g**). The reaction proceeds well for aromatic thioamides with functionalized amines (Me, F, and OMe) to give corresponding transaminated products in moderate to good yields (**5h**-**5k**). In addition to thioamides, aliphatic and 2-phenyl ethyl amines could also be obtained in good yields (**5l** and **5m**). The hetero aromatic amide was also selectively gave the good yield of corresponding product (**5n**).

Table 4. Scope of transamidation of thioamides[a]



[a]Reaction conditions 4 mmol of **1**, 4 mmol of **2**, Catalyst 20. Wt. % at 130 °C for 30 hours, isolated yield.

To validate the feasibility of the present transformation for industrial applications, we performed the reaction at gram scale process under the optimised conditions, for 24hrs. The corresponding thioamidation product was isolated in 76% yield (Scheme 1). Under the similar reaction conditions we checked the reaction in the absence of catalyst only 37% yield of transthioamidation product was obtained.



Scheme 1. Gram scale transthioamidation

To check the recyclability of H-β-zeolite catalyst for the transamidation reactions under the optimized conditions, after the completion of the reaction the catalyst was recovered by simple filtration, washed with dichloromethane and oven dried at 50º C for 20 min and reused. The recyclability was conducted with model substrates acetamide with benzyl amine, and transamidation product was obtained up to four cycles with no significant change of the conversion and yield (Fig. 2). This study indicates that the present catalytic system works well for transamidation of carboxamides and thioamides with a wide range of amines under solvent-free conditions. SEM images of fresh and reused catalyst was confirmed the same morphology (figure 3). Further, the catalyst (fresh and reused) has been characterized by using XRD and FT-IR[15] (see supporting information). The XRD analysis of used catalyst matched well with fresh catalyst.

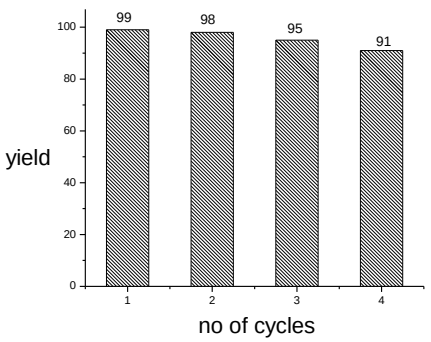


Figure 2. Recyclable study of Hβ-Zeolite catalysed transamidation of acetamide with benzyl amine. Yields were determined by GC.

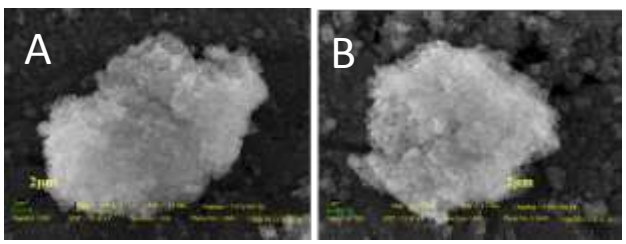
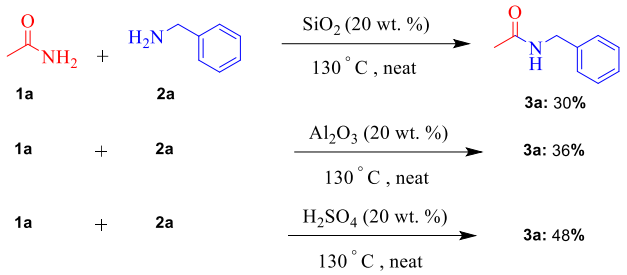


Figure 3. SEM images of H-β-Zeolite (A) Fresh and (B) reused.

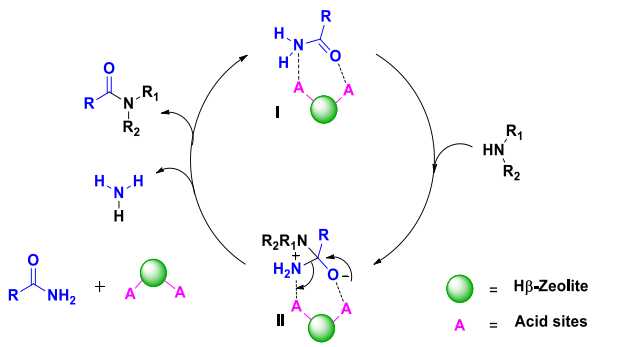
Further, we performed some additional experiments, to establish the mechanism and to know acidic properties of the catalyst

(Scheme 2). Under the optimised conditions, the reactions were performed with silica, alumina and H_2SO_4 independently, low yield of the desired product was obtained compared with H-β-zeolite. Based on these observations, we concluded, that both Bronsted acid and Lewis acid cites of H-β-zeolite may play a crucial role for this transamidation.



Scheme 2. Controlled experiments

Generally, H-β-zeolite has been used as an acid catalyst in organic transformations. This catalyst is reported to have Bronsted acid cites in the external surface and Lewis acid sites predominantly at the internal surface. Based on our studies and literature reports we proposed plausible reaction mechanism (Scheme **3**).[16] Initially, starting amide bond gets activated by co-ordinating with acid sites (both Lewis & Bronsted) of the zeolite to generate the intermediate **I**. Nucleophilic addition of amine to electrophilic centre of the amide carbon of the intermediate **I** leads to the formation of another intermediate **II**. Finally, removal of ammonia from intermediate **II** yield the desired transamidation product.



Scheme 3. Plausible reaction mechanism

In conclusion, we have developed a green protocol for transamidation and thioamidation of amides using H-β-zeolite as heterogeneous recyclable catalyst. The present protocol is compatible with a wide range of amides, thioamides including phthalimide and formamide and obtained the corresponding transaminated products in good to excellent yields under solvent-free conditions.

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Information (ESI) available: [details of any supplementary information available should be included here]. See DOI: 10.1039/c000000x.

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Graphical Abstract

H-β -Zeolite Catalyzed Transamidation of Carboxamides, Phthalimide, Formamides and Thioamides with Amines under neat Conditions

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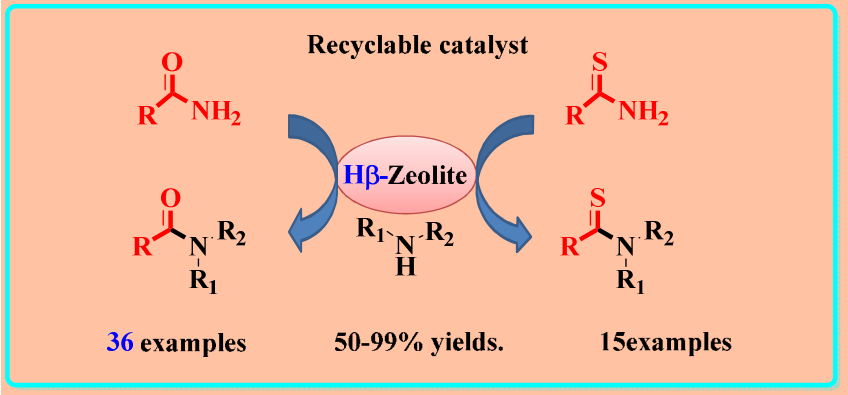

Efficient transamidation of unactivated carboxamides, phthalimides, formamides and thioamides with amines under solvent-free conditions using H-β-zeolite as a green and recyclable heterogeneous catalyst is described.