

FULL PAPER

Applied Organometallic Chemistry

An efficient, multicomponent synthesis of aminoalkylnaphthols via Betti reaction using ZSM-5 as a recoverable and reusable catalyst

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1 | INTRODUCTION

Multicomponent reactions (MCRs) are an important class of synthetic protocols in organic and medicinal chemistry with green chemistry criteria such as high degree of atom economy, waste prevention, no usage of hazardous compounds, energy efficiency, and applications in combinatorial chemistry.^[1] These reactions are single step one-pot transformations proceeding through more than two subsequent reactions in which the product of the first is a substrate for the second.^[2,3] Also, these reactions constitute an especially attractive synthetic strategy as they provide easy and rapid access to large libraries of organic compounds with diverse substitution patterns. Besides, MCRs are rather environmentally benign and atom economic, as they require short reaction time and avoid costly purification processes as well as protection– deprotection steps.^[4] During the past few decades, many one-pot MCRs have been reported for the construction of new carbon–carbon, carbon–heteroatom, and hetero atom–hetero atom bonds.^[5–7] One of the important reactions in the field of MCRs is the synthesis of Betti bases, which is found to be much crucial due to its unique and wide spread applications. The first ever work on these derivatives was reported in the beginning of the 20th century by distinguished Italian chemist Mario Betti via a modified Mannich reaction named the Betti reaction.^[8]

The design and application of environmentally friendly catalysts to reduce the number of toxic wastes are critical for improving the chemical synthetic protocols. A simple, mild, efficient, and eco-friendly method was developed for the synthesis of a series of Betti bases, 1-(α -aminoalkyl)naphthols, via a one-pot, multi-component reaction from aldehydes, β -naphthol and secondary amines in the presence of H-ZSM-5 as a catalyst at room temperature. The key advantages of the new protocol are environmentally friendly as it offers some interesting promising reaction prerequisites such as mild condition, safe, minimal waste, low cost, short reaction time and atom efficiency, easy workup, high to excellent yields, and possessing excellent functional group tolerance to synthesize structurally diverse derivatives. The catalyst is readily recovered by simple decantation and recycled several times with no significant loss in the catalytic activity.

K E Y W O R D S

aminoalkylnaphthols, Betti reaction, catalyst, X-ray diffraction, ZSM-5

2-naphthols (Betti bases, Figure 1) from β -naphthol, aldehyde, and ammonia or urea in an ethanolic solution of potassium hydroxide in two steps for a long time (9–36 h).^[9,10]

Recently, the importance of Betti bases is well established in pharmaceutical chemistry because of their bioactivities, including antibacterial,^[11,12] antifungal,^[13] anti-inflammatory,^[14] anticancer,^[15] antitubercular,^[16,17] antitumor,^[18,19] fungicidal,^[20,21] hypotensive,^[22] and bradycardiac activities (Figure 2).^[23] Also, the literature reveals that the Betti bases are valuable ligands for transition metals in asymmetric synthesis,^[24-26] chiral auxiliaries for the synthesis of α -amino phosphonic acids,^[27,28] chiral shift reagents for carboxylic acids,^[29] and intermediates in the synthesis of heterocycles.^[22,30,31] Owing to the wide variety of applications of the Betti bases as building blocks, it is paramount to build up synthetic strategies around this nucleus. Therefore, the vast range of biological effects associated with this scaffold has resulted in the Betti bases ring system being considered as a privileged structure.

Therefore, the synthesis of this core has received increasing attention to synthetic organic chemists and biologists. Recently, several methodologies and green procedures for the synthesis of Betti reactions have also been successfully developed using catalysts such as Brønsted and Lewis acids such as SnCl₄.5H₂O,^[32] ionic liquids,^[33] nano-sulfated zirconia,^[34] ZrO (OTf)₂,^[35] polymer-supported sulfonic acid,^[36] Bi (NO₃)₃.5H₂O,^[37] [HMIM]C (CN)₃,^[38] nanocrystalline MgO,^[39] supported copper triflate,^[40] ZnCl₂,^[41] GO-MnO₂-Au ternary nanocomposite,^[42] and sulfated polyborate.^[43] Although these methods/reactions have their advantages, there are demerits such as the use of expensive low selectivity catalysts, harsh reaction conditions, expensive reagents, moisture sensitivity, toxic solvent/reagents, the use of excess or stoichiometric amounts of reagents, non-recoverability of the catalysts, and/or recoverable with tedious separation procedures, involving lots of toxic waste generation besides a long reaction time and low yield of the desired product. To overcome these problems, a general efficient methodology is needed utilizing zeolite socony mobil-5 (ZSM-5) as an inexpensive and benign catalyst, which



consists appropriate amount of silica and alumina. It belongs to the pentasil family of zeolites with smaller crystal size and higher surface area showing improved catalytic activity and selectivity.^[44] Its chemical formula, Na_nAl_nSi_{96-n}O₁₉₂·6H₂O, shows the unit cell composition of the zeolite. The existence of aluminum and Si/Al ratio of ZSM-5 catalyst causes to possess acidic property.^[45] This property makes the catalyst to function as catalyst support, adsorbent, and so forth. Also, it possesses many advantages such as excellent solubility in different solvents, high reactivity, easy handling, low cost, and ecofriendly nature. Because of these features, ZSM-5 has been used as a catalyst in organic transformations.^[46] Therefore, considering the wide range biological applications of Betti bases and the unique properties of ZSM-5 as an ideal catalyst. As a part of our program aiming at developing an efficient and environmentally friendly catalvtic system for common organic transformations,^[47-50] herein we to report an efficient, simple, and modified Mannich-type synthesis of Betti bases or 1-(α -aminoalkyl)-2-naphthols from 2-naphthol, aromatic aldehydes and different aliphatic amines (cyclic) using H-ZSM-5 zeolite as catalyst (10 mol%) at room temperature in quantitative yield (Scheme 1).

2 | RESULTS AND DISCUSSION

Initially, a model reaction was conducted comprising three components, 2- naphthol (1), benzaldehyde (2a), and pyrrolidine (3a) to form 4a to optimize the reaction conditions with varying solvent and best ideal catalytic system loading as these are the important parameters, which affect the yield of the desired product to a great extent (Table 1). For optimization of the reaction, we have screened various catalysts and solvents (Table 1, Entries 1-8) along with H-ZSM-5 zeolite as a catalyst. To find out the best, eco-friendly, and efficient catalyst for this reaction, we performed the same reaction with various catalysts such as SiO₂, TiO₂-SiO₂, BF₃SiO₂, PTA, TiO₂, ZrO₂, TiO₂-ZrO₂, and H-ZSM-5 (Table 1). We observed that the best results were obtained using catalyst H-ZSM-5 zeolite in terms of product yield and reaction time (Table 1, Entries 13 and 14). To study the real efficacy of the catalyst for Betti base synthesis, a blank reaction was carried out in absence of catalyst at room temperature (r.t.) without keeping in inert condition. We found that low yield of Betti base was obtained without catalyst even after keeping the reaction for 12 h at r.t. (Table 1, Entry 15). Further, we studied the effect of various organic solvents for the synthesis of Betti base to increase the product yield. Various conventional organic solvents, such as MeOH, EtOH, THF, and DCM afforded

FIGURE 2 Biologically active Betti bases

H-ZSM-5

1.3-2h DCM, rt \mathbf{R}^2

OH

(4a-t)



H

3

SCHEME 1 Synthesis of Betti bases using ZSM-5 zeolite catalyst

moderate to good yields at r.t. (Table 1, 39%-92%, Entries 8-15). Among all these entries, best results were obtained using zeolite catalyst at r.t. in benzene and DCM solvents to the corresponding products in 89% and 91% yield within 2 h (Table 1, Entries 13 and 14), respectively. It is worth mentioning that the reaction works efficiently at ambient conditions so there is no need to carry out the trial in an inert atmosphere. After finding the ideal catalyst and solvent system, we have investigated the effect of different amounts of catalyst loading for this transformation. To find out the suitable catalyst amount loading, a model reaction was performed using 5 and 20 mol% of H-ZSM-5 zeolite as a catalyst at r.t. in DCM (Table 1, Entries 16 and 17). We found that the best results with 10 mol% of catalyst loading resulted in an excellent yield of 92% in minimum duration of time. The use of a higher amount of catalysts (20 mol%) neither improves the yield nor reaction time. So 10 mol% of the catalyst was found to be the best condition to carry out the reaction.

1

With the optimized conditions in hand (Table 1, Entry 14), the scope of the reaction was investigated considering the nature of the various cyclic amines (five- and six-membered), functionalized aromatic aldehydes, and 2-naphthol, allowing access to a library of substituted amino arylnaphthols (Table 2, Entries 1–20). A wide range of aromatic aldehydes with substituents at the *ortho*, *meta*, or *para*-positions were treated with pyrrolidine and 2-naphthol under the optimized condition to obtain a good yield of the product (Table 2, Entries 1–5). It was revealed that all the reactions were

completed efficiently within 2 h, and the expected products were obtained in good to excellent yields (76%-92%). Variety of substituted benzaldehyde were also successfully treated with different aliphatic cyclic amines such as piperidine, morpholine, and 2-naphthol giving rise to corresponding products within 1-2 h in 76%-92% yields. Several electron-releasing or electron-withdrawing substituents at the ortho, meta, and para-positions of aromatic aldehydes showed no significant effect on product yield and reaction time. However, aromatic aldehydes bearing groups like -NO₂, -OMe, and -Cl also displayed better reactivity and gives convincible yields. Similarly, we have also tried to attempt the reaction with proline and hydroxyl proline (Table 2, Entries 19 and 20) under optimized conditions, but no corresponding Betti base was obtained. Also, the model reaction was scalable up to 10 g with mechanical overhead stirring resulted in the formation of the desired product. On the other hand, the applicability of this protocol on aliphatic/heteroaromatic aldehydes with amines (aliphatic/cyclic) is under progress. Further, the application of this protocol for the synthesis of natural products and different drugs is targeted to be dealt considering the optimized conditions. The spectral data and melting point of the synthesized compounds are in agreement with literature data (supporting information). Additionally, a single X-ray crystallographic study was conducted and the molecular structure of compounds 4a (CCDC 2063270) and 4b (CCDC 2063272) was unambiguously established (Figure 3).



Entry	Catalyst	Amount of catalyst (mol% or g)	Solvents	Time (h) ^a	Yield(%) ^b
1	Phosphotungestic acid (PTA)	0.250	Toluene	Overnight	49
2	TiO ₂	0.250	Toluene	12	51
3	ZrO ₂	0.250	Toluene	12	54
4	TiO ₂ -ZrO ₂	0.250	Toluene	12	59
5	SiO ₂	10 mol%	Toluene	12	61
6	TiO ₂ -SiO ₂	10 mol%	Toluene	12	57
7	BF ₃ -SiO ₂	10 mol%	Toluene	12	41
8	H-ZSM-5	0.250	Toluene	5	73
9	H-ZSM-5	10 mol%	МеОН	8	46
10	H-ZSM-5	10 mol%	EtOH	9	54
11	H-ZSM-5	10 mol %	THF	11	39
12	H-ZSM-5	10 mol%	CH ₃ CN	6	71
13	H-ZSM-5	10 mol%	Benzene	2	89
14	H-ZSM-5	10 mol%	DCM	2	92
15	H-ZSM-5		_	12	41
16	H-ZSM-5	5 mol%	DCM		73
17	H-ZSM-5	20 mol%	DCM		91

Note: Reaction conditions: Benzaldehyde/pyrrolidine/2-naphthol = 1.2:1.0:1.0, with H–ZSM–5 zeolite catalyst at room temperature. At these reaction conditions (in bold), the product gives good yields.

^aReaction is monitored by TLC. ^bIsolated yield.

To obtain the solid-state bonding and special connectivity information of these amino arylnaphthols was analyzed by single-crystal X-ray diffraction technique. Molecular structure of compounds 4a and 4b is shown in Figure 3. The details of single-crystal data and structure refinement details of both the compounds are given in Tables S1 and S2. The X-ray diffraction analysis reveals that both the compounds are structurally intact, and the naphthol ring is almost planar concerning the aromatic benzene. The fivemembered pyrrolidine ring of the compounds 4a and 4b is stabilized by a strong intramolecular O—N bonding with a bond distance of 2.591 and 2.606 Å, respectively. Selected bond length, bond angle, and torsional angle of the compounds 4a and 4b are tabulated in Tables S3 and S4.

2.1 | Catalyst recyclability studies

From an economic and industrial perspective, successive recovery and reusability of the catalysts are the foremost important aspects. Therefore, the reusability of the catalyst in the model reaction under optimized reaction conditions was evaluated. After completion of the reaction, as indicated by TLC, the catalyst was removed and recovered by filtration under suction from the reaction mixture. Further, it was attempted to investigate the reusability of the H-ZSM-5 zeolite catalyst more than three times. After the first run of the reaction, a 92% yield was obtained and then the catalyst was recovered by filtration and dried in the oven overnight at 120°C. The recovered catalyst was dried again under vacuum for 12 h and used

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 TABLE 2
 Synthesis of varied aminoalkyl naphthol derivatives via Scheme 1

Entry	Aldehyde	Amine	Product	Product No.	Time (hr)	% Yield ^a
1	CHO	HN	N OH	4a	2	92
2	CI	HN N	ClN CLN CLOH	4b	1.3	89
3	CHO OCH ₃	HN N	OCH3 N OH	4c	2	83
4	H ₃ CO CHO		H ₃ CO	4d	1.5	81
5	H ₃ C	HN N	H ₃ C	4e	2	85
6	CHO		N OH	4f	2	86
7	Br	H O	Br, N, O OH	4g	1.5	81

(Continues)

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TABLE 2 (Continued)

Entry	Aldehyde	Amine	Product	Product No.	Time (hr)	% Yield ^a
8	CHO Br		Br O N OH	4h	2	79
9	H ³ CO CHO		H ₃ CO	4i	2	83
10	CI	$\begin{pmatrix} H \\ 0 \end{pmatrix}$	Cl N OH	4j	1.3	79
11	N CHO			4 k	2	76
12	HOCHO		HO N OH	41	2	85
13	O ₂ N CHO	(N)	O ₂ N N OH	4m	2	83
14	CI CHO		CI N OH	4n	2	81

TABLE 2 (Continued)

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Entry	Aldehyde	Amine	Product	Product No.	Time (hr)	% Yield ^a
15	CHO	$\begin{pmatrix} H \\ N \\ 0 \end{pmatrix}$	Cl N N OH	40	2	79
16	CHO	N H	N OH	4p	1.3	87
17	H ₃ CO CHO	N H	H ₃ CO	4q	1.3	83
18	CHO Br	N H	Br N OH	4r	1.3	81
19	CHO	C OH	No reaction	4s	12	
20	СНО	HO'' HO'' OH	No reaction	4t	12	

Note: Reaction conditions: Benzaldehydes/alicyclic amine/2-naphthol = 1.2:1.0:1.0, with H–ZSM–5 (10 mol%) catalyst at room temperature. ^aIsolated yield.

up to three more runs for the reaction. It was revealed that after recovering and reuse of the catalyst displays a minimal decrease in yields without loss of catalytic activity. The product **4a** was obtained in 92%, 87%, 82%, and 73% yields after three runs (Table 3, Entries 1–4), thus proving the catalyst recyclability.

2.2 | Exploration of the possible mechanism

The mechanism of this multi-component reaction involves the mechanism similar to that of Michael addition/Knoevenagel condensation cascade process.^[51] A tentative mechanism for the H-ZSM-5 zeolite-catalyzed



TABLE 3 The recycle study of H-ZSM-5 zeolite for Betti base synthesis

Entry	Recycle study	%Yield (isolated)
1	I run	92
2	II run	87
3	III run	82
4	IV run	73



SCHEME 2 Proposed mechanism for the synthesis Betti base using H-ZSM-5 catalyst

Betti base synthesis is proposed in Scheme 2. First, H-ZSM-5 coordinates to benzaldehyde and generates the ZSM-5-aldehyde intermediate **(I)**, which reacts with aniline to generate the imine intermediate **(II)** with the removal of water and catalyst. In the next step, the imine intermediate is activated when H-ZSM-5 binds to the nitrogen **(III)**, which promotes the following nucleophilic addition of β -naphthol to give the desired product **(IV)** and regenerates H-ZSM-5. From literature, it is found that the formation of imine intermediate is likely the rate-determining step. H-ZSM-5 is an acidic catalyst that provides efficient acidic sites to form a coordinate bond between oxygen atom (or nitrogen atom) which can activate the formation of imine intermediate, followed by facilitating the nucleophilic addition.^[52]

3 | CONCLUSIONS

In summary, we have developed a new efficient method for the synthesis of a series of Betti base via Mannich approach using H-ZSM-5 zeolite as a heterogeneous catalyst in a one-pot reaction, by reacting readily available different amines, substituted aromatic aldehydes, and 2-naphthol at r.t. Reaction condition was optimized by studying the effect of catalyst, the effect of solvent, and varying reaction temperatures for the synthesis of Betti bases. The noteworthy advantages of this protocol are (a) high yields with short reaction time; (b) a wide range of substrate scope using aromatic aldehydes and cyclic amines (wide substrate compatibility); (c) great activity, easy separation using cheap catalyst in comparison with many other catalysts used for the synthesis of Betti bases; (d) easy work-up procedure (clean reaction profile) and cost efficiency; and (e) reactions at r.t. Altogether, the present method is a useful and attractive strategy for the preparation of different Betti base $(1-(\alpha-aminoalky))$ -2-naphthol) derivatives simply by varying different substrates.

4 | EXPERIMENT

4.1 | Material and methods

Analytical grade reagents were purchased from Sigma-Aldrich or Fluka or Acros were used as supplied. The

FIGURE 3 Molecular structure of compounds **4a** and **4b**

melting points of the products were determined by open capillaries and are uncorrected. The ¹H and ¹³C nuclear magnetic resonance (NMR) spectra were recorded on Bruker AVANCE III spectrometers at 300 and 400 MHz, respectively. Chemical shifts (δ) are reported in ppm from the standard internal reference tetramethylsilane (TMS). The following abbreviations are used: s = singlet, d = doublet, t = triplet, and m = multiplet. The homogeneity of the compounds was described by TLC on aluminum silica gel 60 F₂₅₄ (Merck) detected by ultraviolet (UV) light (254 nm) and iodine vapors. The catalysts were characterized by various analytical techniques.

4.2 | General procedure for the synthesis of the Betti base 4(a-t)

A mixture of 2-naphthol (1.0 eq.), cyclic amine (1.0 eq.), and benzaldehyde (1.2 eq.) was dissolved in DCM and stirred at r.t. in the presence of 10 mol% of catalyst within appropriate time as shown in Table 2. After completion of reaction, as indicated by TLC, the catalyst was removed and recovered by filtration from the reaction mixture and the filtrate solution was concentrated using rotary evaporator to get crude product and then purified through the silica gel column chromatography to obtain corresponding Betti base in excellent yield as indicated in Table 2.

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AUTHOR CONTRIBUTIONS

Rangappa S. Keri: Funding acquisition; methodology; project administration; supervision. Mahadeo Patil: Conceptualization; methodology. Srinivasa Budagumpi: Data curation. Sasidhar B. S.: Formal analysis.

CONFLICT OF INTEREST

There is are conflicts to declare.

DATA AVAILABILITY STATEMENT

The data that support the findings of this study are available in the supporting information of this article.

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SUPPORTING INFORMATION

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