Original Article: $Bi(OTf)_3$ as a Highly Potent Catalyst for the Synthesis of Mannich Bases under Milder Conditions

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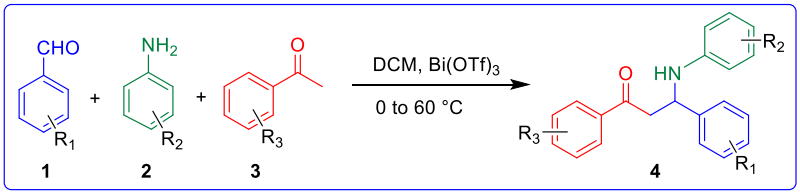
ABSTRACT

A simple, eco-friendly friendly and efficient procedure for the synthesis of Mannich Baseshas been developed via multi-component and one-pot reactions of various aldehydes with aniline, and acetophenone and catalytic amount of $Bi(OTf)_3$ reagent in a DCM solvent.The $Bi(OTf)_3$ acts as a highly potent catalyst (0.5 to 1 mol%) for a for the synthesis of Mannich base (1,3-diphenyl-3-(phenylamino)propan-1-one). This protocol is also compatible with a variety of hetero aldehyde carbonyl compounds in excellent yields. Thus, this practical method is developed as a notable medium for these derivatives *via* a multicomponent reaction.



Introduction

The Multi-component reaction has been projected as one of the most classical Mannich reactions and the reaction was utilized for the synthesis of *β*-amino carbonyl scaffolds (Mannich bases) by a one-pot reaction of aldehyde, amine; the reaction was discovered in 1917 [1]. As per literature reports, the Mannich bases are flexible organic chemicals used for many reactions as important intermediates [2–6] and broadly applicable for the synthesis of alkaloids [6]; this

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scaffold is also used in medicinal/pharmaceutical chemistry [7,8].

For the preparation of the mannich product, the researcher used numerous reagents, since for the last decade, Brønsted acid-based catalysts like acidic ionic liquids [9], $HClO_4$-SiO_2 [10], polymer-supported sulfonic acid [11], camphor sulfonic acid [12], acidic surfactants [13], $H_3PW_{12}O_{40}$ [14], and HCl [15], have been broadly applied for the production Mannich products, which provides an easy connection to Mannich bases. However, this protocol has limitations, requiring a large quantity of catalyst to perform the reaction, yielding less and having a longer reaction time. The reagents such as Lewis acids have been used for the preparation of Mannich bases includes $BiCl_3$[16], $NbCl_5$[17] $ZrOCl_2{\cdot}8H_2O$ [18], $Zn(OTf)_2$ [19], $Yb(OPf)_3$ [20], $CeCl_3{\cdot}7H_2O$/CAN [21,22], and $Ga(OTf)_3$ [23]. These reagents are applied either in a solution-phase and solvent-free conditions. The reaction was also performed in the organometallic complexes of Sb(III) [24], Ti(IV) [25], Zr(IV) [26, 27], Bi(III) [28], along with other Lewis acids, such as sulfonium [29] iodonium salts [30], which also deserve a very effective catalyst for this transformation.

Numerous acidic solvents were used for the Mannich base synthesis. Guoying Zhao *et al.* have prepared brønsted acidic ionic liquids including ($[Bmim]^+[HSO_4]^-$), ($[Bmim]^+[H_2PO_4]^-$), ($[Hmim]^+Tsa^-$) and ($[Hmim]^+Tfa^-$)for the Mannich base synthesis [31].

Kun Li et al. developed a novel method for the Mannich base synthesis; the novel lipase was used for the direct synthesis of Mannich base in water solvent [32]. Chandra Mukhopadhyay *et al*. have reported that the Boric acid and glycerol catalysed efficient synthesis of Mannich base by the one-pot, three-component reaction with aldehydes, aromatic amines, and cyclic ketones in water at room temperature to produce the desired Mannich base in moderate to good yields [33].

The greener synthesis of the Mannich base is a challenging task for the chemist since for the last decade there is a large number of green methods are reported for the Mannich reaction. Mahmood Kamali *et al.*reported the solvent-free greener method for the synthesis of Mannich Bases from 4-Hydroxy-pyridine-2-one [34]. Also, Mahboob Ghadami *et al.* reported the sodium dodecyl sulfate micellar mediated Mannich reaction with the ultrasound irradiation of various aldehydes, aromatic amines, and acetophenone derivatives. However, this protocol usually requires higher loading of catalyst. Therefore, the development of highly potent, less-costly, and non-toxic methods for the Mannich reaction is still highly desired. These studies revealed that the compounds with a *β*-amino carbonyl compound core have various biomedical properties, including antidiabetic, antimicrobial, antioxidant, antidyslipidemic, and anticancer activities [35-36]. However, the majority of these reported protocols have limitations such as the use of harsh conditions, loading of catalyst, toxic reagents, longer reaction time and low yields. Therefore, there is a need to develop a novel protocol for the synthesis of *β*-amino carbonyl compounds under mild conditions. However, we are always interested in the development of novel method for the synthesis of bioactive compounds [37-40]. This work aimed to develop a novel greener methodology for the synthesis of *β*-amino carbonyl compounds.

Experimental

All the apparatus and chemicals were used as per standard laboratory guideline. The M.P. of novel compound was done with melting point apparatus thermal IA9100 (Bibby Scientific

Limited, Staffordshire, UK). The FTIR of the compound was recorded over the Bruker FTIR instruments. The ^{1}HNMR, and ^{13}CNMR were recorded over the Bruker -300MHz, Bruker-400 MHz instruments.

General procedure for the synthesis of Mannich base

The mixture of solution contain benzaldehyde (1mmol), acetophenone (1mmol), and amine (1mmol) in a $Bi(OTf)_3$ (1mL) and dry DCM (5mL) and mixture was vigorously stirred at room temperature for 12-14 h. Once the reaction was completed, the product was isolated by the evaporation of solvent and purified by simple column chromatography to afford the desire pure Mannich base in very good yields.

1,3-diphenyl-3-(phenylamino)propan-1-one (4A)

White solid, M.P. 170-171 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.95–7.87 (m, 2H), 7.64–7.58 (m, 1H), 7.54–7.46 (m, 4H), 7.40–7.36 (m, 2H), 7.32–7.26 (m, 1H), 7.18–7.15 (m, 2H), 6.74 (ddd, *J*=7.6, 2.2, 1.5 Hz, 1H), 6.62 (dt, *J*=8.6, 1.6 Hz, 2H), 5.06 (dd, *J*=7.6, 5.6 Hz, 1H), 4.60 (br s, 1H), 3.60 (dd, *J*=16.2, 5.4 Hz, 1H), 3.48 (dd, *J*=16.3, 7.6 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 198.38, 147.10, 143.20, 136.80, 133.60, 129.15, 128.50, 128.80, 128.45, 127.40, 126.34, 17.81, 113.34, 54.70, 46.32.

3-(4-chlorophenylamino)-1,3-diphenylpropan-1-one (4B)

White solid, M.P. 170-171, °C; IR (KBr, ν cm^{-1}): 3365.44, 1660.14, 14980.60, 570.20, 507.23, ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.80–7.70 (m, 2H), 7.60 (ddd, *J*=6.8, 4.2, 1.4 Hz, 1H), 7.55–7.48 (m, 2H), 7.48–7.34 (m, 2H), 7.38–7.44 (m, 2H), 7.08–7.01 (m, 1H), 6.42–6.77 (m, 1H), 4.90 (dd, *J*=7.8, 5.0 Hz, 1H), 4.54 (s, 1H), 3.42 (dd, *J*=8.2, 2.6 Hz, 1H), 3.63 (dd, *J*=8.4, 6.7 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 197.13, 146.54, 143.63, 143.52, 134.56, 129.96, 129.94, 129.78, 129.23, 128.54, 127.36, 125.49, 116.98, 55.93, 46.45.

3-(4-bromophenyl)-1-phenyl-3-(phenylamino)propan-1-one (4D)

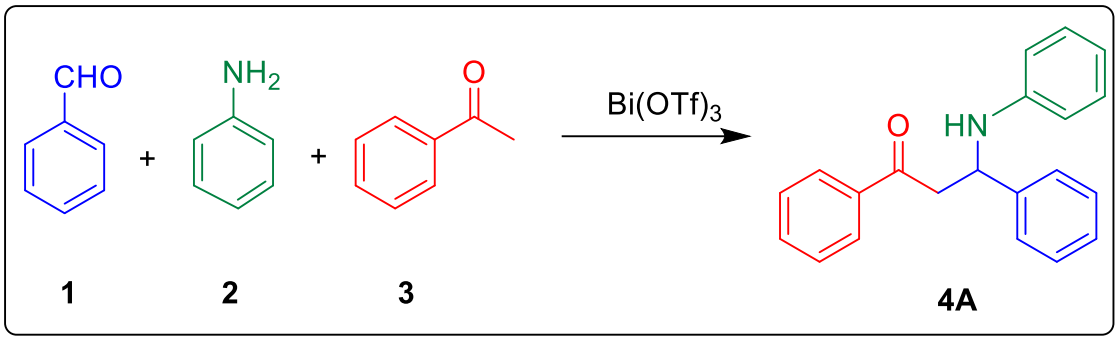
Colorless solid; 83% yield; mp 131-133 °C; FT-IR cm^{-1}, 3294, 3029, 161, 1610, 1504, 1450, 1230, 1012, ; ^{1}H NMR (300 MHz, $CDCl_3$): d 3.41 (d, *J*=6.4 Hz, 2H), 4.95 (t, *J*=6.1 Hz, 1H), 6.62 (d, *J*=7.4 Hz, 2H), 6.75 (t, *J*=7.8 Hz, 1H), 7.16 (t, *J*=7.8 Hz, 2H), 7.31–7.60 (m, 7H), 7.89(d, *J*=7.8Hz, 2H); ^{13}C NMR (50 MHz, $CDCl_3$): d 41.4, 54.3, 113.8, 120.2, 121.5, 128.21, 128.4, 128.8, 130.1, 131.7, 133.4, 136.2, 141.2, 145.6, 197.2.

Results and Discussion

Presently, there is an increasing demand on the introduction of economical and environmentally benign processes based on the principles of green chemistry. Herein, we have used a catalytic amount of $Bi(OTf)_3$ for the preparation of Mannich base. The initial reaction on the ($Bi(OTf)_3$ catalysed (0.5 mol%) three-component Mannich reaction of benzaldehyde, acetophenone, and aniline in a dry DCM solvent. The reaction mixture was stirred at room to afford the corresponding *β*-amino ketone **4A** in a 55% yield. Accordingly, we investigated the effect of different mol percentages of $Bi(OTf)_3$ (**Table 1**). When the reaction proceeded with 0.5mol% of $Bi(OTf)_3$ at room temperature for 12 h, giving compound 4A with 60% yield, the reaction was performed with increasing in temperature to 60 °C; the formation of product was observed with increase in yields by **77% (Table 1, entry** 4) and decrease in reaction time. The results we have obtained above highlight the importance of the temperature for the reaction. Then, the reaction was performed in the 1.0 mol% of $Bi(OTf)_3$ with 60 °C, obtaining desired products

with 87% yield (**Table 1, entry 5**). Further increase of the temperature failed to improve the yield of the reaction; compound **4A** was formed in 85% yield when the reaction was performed at 60 °C, with 1.5 mol% of $Bi(OTf)_3$.

Table 1. Optimization of Mannish base

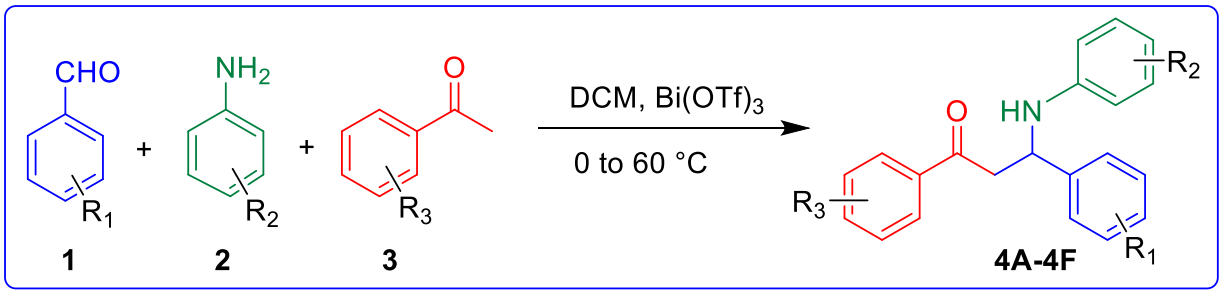


Sr/No	HFIP Catalyst in Mol%	Solvent	Temp	Time	Yields
1	NO	DCM	R.T	12	-
2	0.1	DCM	RT	12	20
3	0.5	DCM	RT	12	60
4	0.5	DCM	60 °C	8	77
5	1.0	DCM	60°C	6	87
6	1.5	DCM	60°C	6	85
7	1.5	DCM	60 °C	6	85

Note; at higher temperature and more equivalence of reagent the side product will form more.

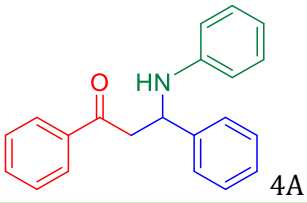
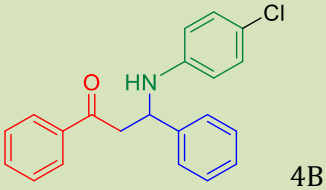
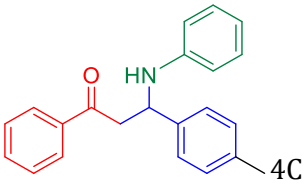
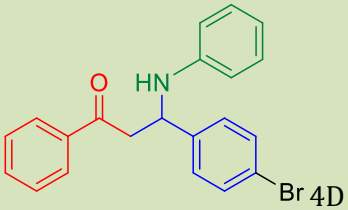
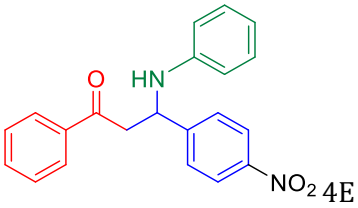
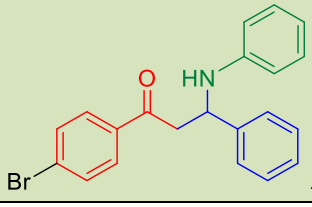
Further, we performed the reaction with $Sc(OTf)_3$, $Yb(OTf)_3$ and AgOTf in DCM solvent, and the result was good for the AgOTf similar to the $Bi(OTf)_3$ catalyst.

With the optimized reaction condition in hand, then, we examined the scope multicomponent Mannich reaction with various substrates with optimized reaction condition. As shown in **Scheme 1, Table 2**, all the substrates proceed with smooth reaction with the optimized condition to afford *β*-amino ketones in a moderate to very good yields. However, the time reaction for the completion of the reaction in case of the acetophenone with substituted aromatic aldehydes and substituted aromatic amines was longer compared with that of normal benzaldehyde and aniline.



Scheme 1. Synthesis of Mannish base

Table 2. Synthesis of Mannich bases **4A-4F**

	Product	M.P	Yield
1	 4A	170-171 °C	87%
2	 4B	169-172 °C	82%
3	 4C	134-136 °C	82%
4	 4D	131-133 °C	80%
5	 4E	121-124 °C	74%
6	 4F	144-145°C	80%

We have studied the solvent effect on the optimized reaction condition, as shown in Table 3. The reaction proceeded in the benzene, DMF, ether, THF, PEG, DMSO and DCM solvent, The reaction proceeded slowly in the DMF, ether, THF, PEG, DMSO with good to moderate yield, while reaction proceeding in DCM resulted in the higher yield at 60 °C (87%, 5h)(**Table 3**).

It should be noted that no any additional catalyst was employed in this reaction, and equivalent amounts of the starting materials were used. The present method is better than the previous reported method because reaction condition is mild and atom-economic, thus it may have potential applications in organic synthesis. Some of the lewis acid catalyzed methods are shown in Table 4. Most

of the methods show the excellent yields and selectivity.

Table 3. Solvent effect on the HFIP -catalysed Mannich reaction

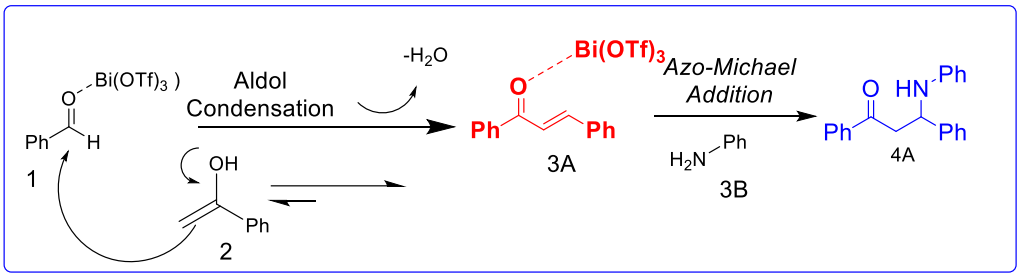
Sr/No	Solvent	Reaction Time (h)	Catalyst	Yield of 4A
1	DMF	7	$Bi(OTf)_3$	55
2	Ether	6	$Bi(OTf)_3$	45
3	THF	6	$Bi(OTf)_3$	62
4	PEG	10	$Bi(OTf)_3$	50
5	DMSO	6	$Bi(OTf)_3$	55
6	**DCM**	**5**	$Bi(OTf)_3$	**85**

Table 4.

Sr/No		Temperature	Yields
1	$Ga(OTf)_3$ (10 mol%), r.t., 0.5 h, ultrasound irradiation	Room Temp	90%[42]
2	5-sulfosalicyclic acid (5 mol%), H_2O, r.t., 3 h	Room Temp	90%[43]
3	Yb(OPf)	Toome Temp	82[44]
4	$Zn(OTf)_2$	Room Temp	75-96[44]
5	$Hf(OTf)_4$		92[46]
6	$Bi(OTf)_3$	Rt to 60 °C	87%

Mechanism

The mechanism of the reaction goes via the aldol condensation followed by the azo-michael addition reaction to produce desired Mannish base (Scheme 2), involving $Bi(OTf)_3$ catalyzed three-component reaction that proceeds via sequential aldol condensation and aza-Michael addition to afford the desired Mannish base (**Scheme 2**). The Mechanism involves the $Bi(OTf)_3$ to strongly activate the carbonyl compounds, hence, the reaction goes via aldol condensation to produce intermediates A, then the compound A reacts with 3 via Azo-michael addition type reaction and gives desire product B.



Scheme 2.Proposed mechanism for Mannish reaction

Conclusion

We have applied $Bi(OTf)_3$ as an efficient catalyst for the Mannich reaction. Under the DCM solvent and 0.5 to 1.0 mol% of $Bi(OTf)_3$ catalyst, the reaction could catalyse with high yielding and diverse in substrate scope. All the synthesized compounds were characterised by using ^{1}HNMR, ^{13}C NMR, FTIR and Mass spectroscopy methods.

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Conflict of Interest

We have no conflicts of interest to disclose.

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