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A Greener, one pot synthesis of 1, 3-diphenyl-3-(phenylamino) propan-1-one derivatives

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ABSTRACT

The utilisation of melamine trisulfonic acid (MTSA) as a heterogeneous solid acid catalyst has been found to exhibit exceptional efficiency in facilitating a wide range of organic transformations. Numerous scientists have dedicated significant endeavours towards harnessing MTSA as an environmentally friendly catalyst in the synthesis of N- and O-containing heterocyclic compounds. Additionally, MTSA has been employed for the protection of alcohol, phenols, amines, aldehydes, and ketones, as well as for the acetylation of alcohols and phenols, and the N-formylation of amines. A highly effective catalytic technique has been established for the synthesis of a range of β-amino carbonyl chemical derivatives. This protocol involves a one-pot three-component Mannich reaction, utilising MTSA as the catalyst and ethyl alcohol as the solvent. The aforementioned methodology exhibits the capacity to provide the needed goods at a satisfactory rate of 90-96%, while also demonstrating a commendable efficiency in terms of reaction time. One notable benefit of employing this heterogeneous catalyst is its ability to be retrieved and utilised multiple times without experiencing a decline in its effectiveness. This review aims to examine the preparation and applications of MTSA (Methyltrioxorhenium) in the context of organic synthesis.

Key words: *Melamine trisulfonic acid (MTSA), Heterogeneous, Recyclable, Green synthesis, Solid acid catalyst, Heterocyclic compounds.*

Introduction

In recent decades, multi-component reactions (MCRs) have demonstrated significant efficacy and efficiency as bond-forming methodologies in the fields of organic, combinatorial, and medicinal chemistry, particularly within the framework of green chemistry. The multi component reactions (MCRs) exhibit a high degree of flexibility and possess atom economy, as they progress through a series of reaction equilibria to ultimately produce the desired end product. In addition to several other reaction parameters, the characteristics of the catalyst are of considerable importance in determining the outcomes of yield, selectivity, and overall applicability in a given chemical process (Karimi *et al*, 2010; Kumar *et al.*, 2018; Ampolu *et al.*, 2019; Rai *et al.*, 2010; Anastas *et al.*, 2000; Ampolu *et al.*, 2023, and Anastas *et al.*, 2002). Therefore, the pursuit of a cost-effective, gentle, reusable, and versatile catalyst for multi component reactions (MCRs) continues to be a subject of considerable interest. Catalysis has played a crucial role in mitigating pollution resulting from chemical processes inside our environment. The utilisation of catalysts in organic reactions enhances efficiency and selectivity, leading to the reduction of significant quantities of by-products and waste chemicals. This principle constitutes a basic cornerstone of the field of green chemistry. Homogeneous acid catalysts, including HF, HCl, CH_3COOH, HBr, CF_3COOH, and H_2SO_4, are extensively employed in several significant industrial processes. However, these catalysts possess certain drawbacks in terms of their handling, corrosive nature, complicated work-up procedures, and generation of toxic waste (Stolle *et al*., 2015; James *et al.*, 2012; Ampolu *et al.*, 2023; Hernandez *et al.*, 2015; Sunitha *et al.*, 2017; Ampolu *et al.*, 2017; Ampolu *et al.*, 2019). Therefore, in recent years, there has been a significant focus on the exploration of chemical processes or techniques that are environmentally friendly. Consequently, the investigation into heterogeneous catalysts for the purpose of fine chemical synthesis has emerged as a prominent field of study. The possible benefits associated with these materials in comparison to homogeneous systems include improved ease of handling, reduced issues related to reactor and plant corrosion, and enhanced ecologically friendly waste disposal methods.

The Mannich reaction is a significant process in the synthesis of diverse chemical compounds. The use of Mannich products is prevalent in the synthesis of peptides, lactams, amino alcohols, and as precursors for the synthesis of amino acids. In this context, we have successfully synthesised β-amino carbonyl compounds by a three-component one-pot synthesis known as the Mannich reaction. This reaction was conducted using MTSA as a highly efficient and reusable heterogeneous catalyst. The traditional catalysts commonly employed in the classical Mannich reaction, which involves the reactivity of aldehydes, amines, and ketones, mostly consist of mineral acids and organic acids. The synthesis of â-amino carbonyl compounds via the Mannich reaction has been extensively studied in the literature (Santhi *et al.*, 2021; Komali *et al*., 2022). Various catalysts have been investigated for this purpose, including MgO/ZrO_2 (Deepak *et al*., 2010), 2,4-dibromosuccinic acid (Mir Rasul *et al*., 2014) Dipyridine copper chloride (Venu *et al*., 2008), $NbCl_5$, $Re(PFO)_3$, 2,4,6 trichloro [1,3,5] triazine (TCT) (Chang *et al*., 2010), Bismuth(III) triflate (Arda *et al*., 2012), $Al(NO_3)_3.9H_2O$ (Wang *et al*., 2010), and Yttrium (III) chloride, These catalysts have been shown to effectively promote the Mannich reaction.

Nevertheless, these methodologies are associated with several limitations, including a prolonged response time (Venu *et al*., 2008), a substantial catalyst loading (Deepak M.N. *et al*., 2010), reduced yields (Mir Rasul *et al*., 2014), potential toxicity (Wang *et al*., 2010), and challenges in isolating the final product (Arda *et al*., 2012). Therefore, as an extension of our research aimed at the development of environmentally acceptable methods for heterocyclic synthesis, we have undertaken an effort to synthesise â-amino carbonyl compounds by the Mannich reaction involving aromatic aldehyde, aniline, and acetophenone in ethyl alcohol as the solvent. The melamine trisulfonic acid (MTSA) catalyst, known for its inclusion of SO_3H groups, is highly appealing due to its versatility in facilitating organic processes. This catalyst not only enhances convenience but also offers economic and environmentally friendly benefits. Metal-organic frameworks (MOFs) have been extensively investigated as very efficient catalysts for the synthesis of N- and O-containing heterocyclic compounds. Additionally, they have been employed for the protection of alcohol, phenols, amines, aldehydes, and ketones, as well as for the acetylation of alcohols and phenols, and the N-formylation of amines. Furthermore, the catalyst exhibits the ability to be retrieved and employed

repeatedly, demonstrating sustained activity and efficiency throughout multiple cycles. The purpose of this study is to provide a concise overview of current advancements in research pertaining to the diverse organic transformations facilitated by MTSA, which serves as a heterogeneous and recyclable catalyst.

Experimental

Materials and Methods

All compounds employed in this study are of Analytical-grade and used without further purification. All chemicals were obtained from Sigma Aldrich, and doubly distilled water was used as needed during the experiment. All of the reactions were performed under standard laboratory conditions at a temperature of around 25 °C.

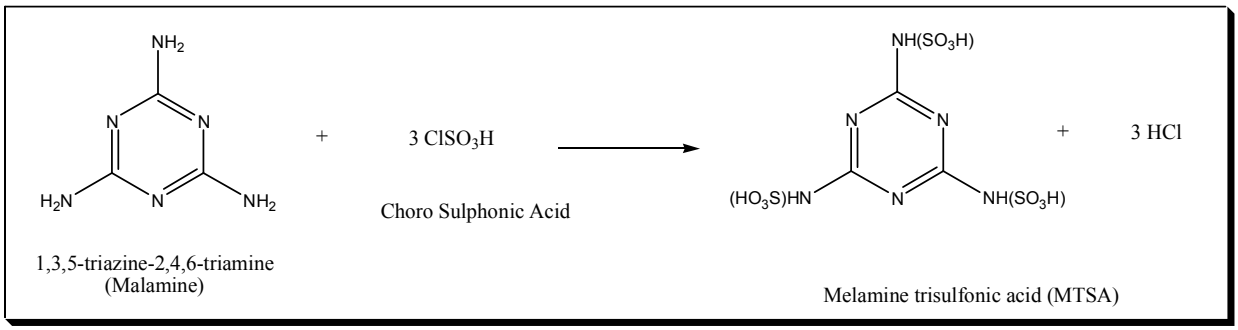
Characterization

Fourier Transmission Infra-Red (FTIR) spectroscopy, specifically with the Perkin Elmer- Spectrum RX-IFTIR instrument, was utilised for the purpose of identifying the functional groups that are present in the synthesised product. The X-ray diffraction patterns were obtained by utilising a Bruker X-Ray Diffractometer, employing graphite filtered CuK radiation with a wavelength of 1.54 Angstroms. The X-ray source was operated at 40 kilovolts, and the scanning rate was set at 3 minutes per degree (2θ) within the range of 20 to 80 degrees. The morphology and size of the particles were assessed using a 200 KeV Transmission Electron Microscope (TECNAI 200 Kv TEM- Fei, Electron Optics). The elemental composition of particles was evaluated by the utilisation of scanning electron microscopy with energy-dispersive X-ray spectroscopy (SEM-EDAX) utilising the JEOL JSM 5600 instrument and the INCA Oxford EDS model.

This work employed Fourier Transmission Infra-Red (FTIR) spectroscopy, specifically employing the Perkin Elmer- Spectrum RX-IFTIR equipment, as a means to identify the functional groups contained in the synthesised product. The X-ray diffraction patterns were acquired using a Bruker X-Ray Diffractometer. The diffraction studies were performed utilising CuK radiation that underwent filtration via graphite, resulting in a wavelength (λ) of 1.54 angstroms. The X-ray source was operated at a voltage of 40 kV, while the scanning rate was configured to be 3° per minute. The scanning range extended from an angle of 2θ = 20 degrees to 80 degrees. The morphology and size of the particles were evaluated with a 200 KeV Transmission Electron Microscope (TECNAI 200 Kv TEM- Fei, Electron Optics). The elemental composition of particles was determined using scanning electron microscopy with energy-dispersive X-ray spectroscopy (SEM-EDAX) on the JEOL JSM 5600 apparatus equipped with the INCA Oxford EDS Model.

General procedure for the synthesis of melamine trisulfonic acid (MTSA)

A total of 10 ml (150.4 m.mol) of chlorosulfonic acid was introduced into a 250 ml suction flask that is equipped with a gas inlet tube for the purpose of passing HCl gas over a solution that absorbs it, specifically water. A total of 6.32 grammes (equivalent to 50.14 m.mol) of Melamine were incrementally introduced over duration of 30 minutes, maintaining the ambient temperature. Hydrochloric acid gas was promptly released from the reaction vessel. Following the addition of melamine, the mixture underwent thorough agitation for a duration of 30 minutes. Meanwhile, the remaining hydrochloric acid (HCl) was removed using suction. The mixture underwent trituration with n-hexane (20 ml) followed by filtration. The solid residue underwent a washing



Scheme 1. Synthesis of Melamine trisulfonic acid (MTSA)

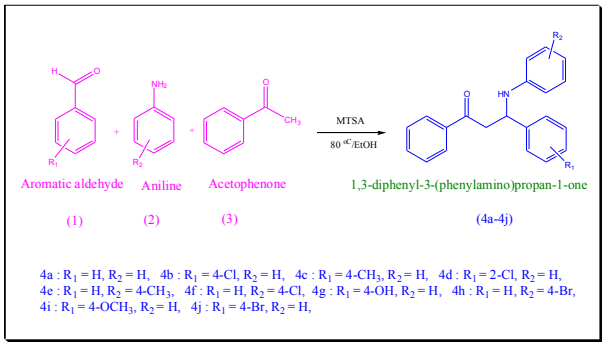
procedure using n-hexane (20 ml) and subsequent drying under desiccator. A quantity of 15.2 gr, representing 85% purity, of Melamine trisulfonic acid was acquired in the form of a solid substance with a white coloration (*Shirini et al.*, 2010).

General procedure for the synthesis of 1,3-diphenyl-3-(phenylamino)propan-1-one derivatives

A solution containing 10 mmol of an aromatic aldehyde, 10 mmol of aniline, and 10 mmol of an aromatic ketone in 10 ml of ethanol was prepared. To this solution, 500 mg of MTSA, which had been pre-activated in a microwave oven for 2 minutes, was added. The resulting mixture was then subjected to reflux at a temperature of 80 °C for duration of 3 hours. The progress of the reaction was assessed using thin-layer chromatography, employing a solvent mixture of n-hexane and ethyl acetate in a ratio of 3:1. The obtained results are presented in Table 1. Following the conclusion of the reaction, the catalyst was subsequently isolated and subjected to a drying process in preparation for its subsequent use (Huiting *et al.*, 2023, Heydari *et al.*, 2022). The findings suggest that the catalyst demonstrates a sustained level of activity after being utilised on five separate occasions, with no discernible decline in performance. The reaction mixture underwent filtration, resulting in the formation of the matching pure chemical through re-crystallization using ethanol. The pure substances were subjected to analysis using FT-IR, 1H-NMR, and MASS spectroscopy techniques in order to determine their characteristics.

Plausible mechanism for the formation of 1, 3-diphenyl-3-(phenylamino) propan-1-one derivatives

The feasible reaction mechanism of this one-pot three component reaction can be elucidated by referring to previous research reported in the literature (Lenka *et al.*, 2016). The reaction mechanism is depicted in Scheme 2. The initial step involves the synthesis of imine 6, which is produced through the reaction between an aromatic aldehyde and an amine in the presence of a catalyst. The subsequent reaction involves the catalytic synthesis of enol 7 from acetophenone. The intermediate imine 6 was produced in the initial reaction, followed by the subsequent reaction of the in situ created enol 7 to provide the appropriate derivatives 4, specifically 1,3-diphenyl-3-(phenylamino) propan-1-one.

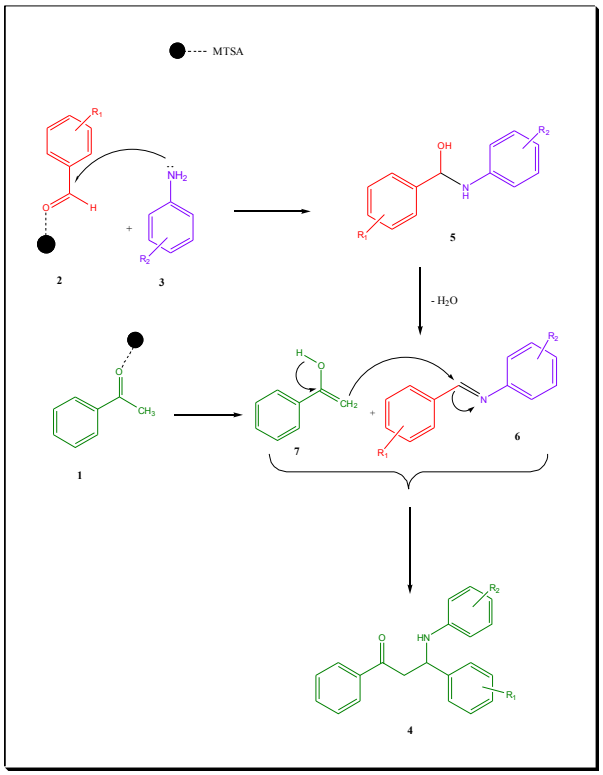


Scheme 2. Synthesis of 1,3-diphenyl-3-(phenylamino) propan-1-one derivatives

Spectral data for the synthesized 1,3-diphenyl-3-(phenylamino)propan-1-one derivatives

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

IR (KBr): 3444, 3315 (2^0-amine, NH-Str), 1664, 1606 cm^{-1} (Carbonyl, C=O Str) 1541,1450 (C=C, aromatic Str), ^1H NMR (400 mHz, $CDCl_3$): δ 4.01-4.04 (m, 1H, NH), 4.98-5.04 (m, 2H, CH_2), 6.68-6.80 (m, 2H, Ar-H), 7.04-7.14 (m, 4H, Ar-H), 7.21-7.30 (m, 2H, Ar-H), 7.37-7.54 (m, 3H, Ar-H), 7.87-7.91 (m, 2H, Ar-H), ESI-MS m/z(%): 302 ($[M+H]^+$ 100).



Scheme 3. Plausible mechanism for the formation of 1,3-diphenyl-3-(phenylamino)propan-1-one derivatives.

3-(4-Chlorophenyl)-1-phenyl-3-(phenylamino) propan-1-one(4b)

IR (KBr): 3433, 3308(2^0-amine, NH Str), 1664, 1606 (Carbonyl, C=O Str), 1528, 1440 (C=C, aromatic Str), 724, 704 cm^{-1} (-Cl Str).^{1}H NMR (400 MHz, $CDCl_3$): δ 3.54 (m, 1H, CH_2), 3.57 (m, 1H, CH_2), 4.04 (m, 1H, NH), 6.62-6.80 (m, 2H, Ar-H), 7.09-7.30 (m, 2H, Ar-H), 7.38-7.48 (m, 2H, Ar-H), 7.54-7.61 (m, 2H, Ar-H), 7.86-7.90 (m, 2H, Ar-H), ESI-MS m/z(%): 336 ($[M+H]^+$ 100).

1-phenyl-3-(phenylamino)-3-(P-tolyl) propan-1-one(4c)

IR(KBr) : 3389, 3317(2^0-amine,NH Str), 1668,1603 (Carbonyl,C=O Str), 1512, 1498(C=C,aromatic Str), 286cm-1(CH_3). ^{1}H NMR (400 mHz, $CDCl_3$): δ 2.32(s,3H,-CH_3), 4.07 (m,1H,NH), 6.63-6.72(m, 2H, Ar-H), 6.88-6.92(m,2H, Ar-H), 7.07-7.15(m, 2H, Ar-H), 7.27-7.48(m,4H,Ar-H), 7.86-7.91(m, 2H, Ar-H), ESI-MS m/z(%) : 316 ($[M+H]^+$ 100).

3-(2-Chlorophenyl)-1-phenyl-3-(phenylamino) propan-1-one(4d)

IR (KBr) : 3394, 3314(2^0-amine,NH Str), 1669,1602 (Carbonyl,C=O Str), 1590,1566 (C=C,aromatic Str), 775, 686cm-1(-Cl Str). ^{1}H NMR (400 mHz, $CDCl_3$): δ 3.14(m, 1H,CH_2), 3.19(m, 1H,CH_2),4.01 (m,1H,NH), 4.49(m,1H,CH) 6.58-6.73(m,2H, Ar-H), 7.06-7.25(m,2H,Ar-H), 7.30-7.34(m,2H,Ar-H), 7.40-7.56(m,2H, Ar-H), 7.86-7.89(m,2H,Ar-H), ESI-MS m/z(%) : 336 ($[M+H]^+$ 100).

1,3-diphenyl-3-(p-tolylamino) propan-1-one(4e)

IR (KBr) : 3389, 3323(2^0-amine,NH Str), 1673, 1666(Carbonyl, C=O Str), 1511,1493, (C=C, aromatic Str), 283cm-1(-CH_3 Str). ^{1}H NMR (400 mHz, $CDCl_3$): δ 2.32(s,3H,-CH_3),4.07 (m,1H,NH), 6.63-6.72(m,2H, Ar-H), 6.88-6.92(m,2H,Ar-H), 7.07-7.28(m,2H,Ar-H), 7.32-7.44(m,4H,Ar-H), 7.48-7.91(m,2H,Ar-H). ESI-MS m/z(%) : 316 ($[M+H]^+$ 100).

3-((4-chlorophenyl) amino)-1,3-diphenylpropane-1-one(4f)

IR(KBr) : 3616(2^0-amine,NH Str), 1615(Carbonyl, C=O Str), 1541, 1466(C=C, aromatic Str), 780,727cm-1(-Cl Str). ^{1}H NMR (400 mHz, $CDCl_3$): δ 3.18(m, 1H,CH_2), 3.22(m, 1H,CH_2), 4.03 (m,1H,NH),4.35-4.45(m,1H,CH), 6.58-6.73(m,2H, Ar-H), 7.05-7.13(m,2H,Ar-H), 7.18-7.28(m,2H,Ar-H), 7.31-7.45(m,2H,Ar-H), 7.87-7.94(m,2H,Ar-H). ESI-MS m/z(%) : 336 ($[M+H]^+$ 100).

3-(4-hydroxyphenyl)-1-phenyl-3-(phenylamino) propan-1-one(4g)

IR(KBr): 3375 (2^0-amine, NH Str), 3171(-OH Str), 1615 (Carbonyl,C=O Str), 1470,1412cm-1(C=C, aromatic Str). ^{1}H NMR (400 mHz, $CDCl_3$): δ 3.15-3.22 (m, 2H,CH_2), 4.06 (m,1H,NH), 4.41-4.48(m,1H,CH), 5.32(m,1H,-OH), 6.71-6.81 (m,2H, Ar-H), 7.02-7.15 (m,2H,Ar-H), 7.25-7.41 (m,2H,Ar-H), 7.44-7.50(m,2H,Ar-H), 7.54-7.93(m,2H,Ar-H). ESI-MS m/z(%) : 318 ($[M+H]^+$ 100).

3-((4-bromophenyl) amino)-1,3-diphenylpropan-1-one(4h)

IR(KBr) : 3443(2^0-amine,NH Str), 1659, 1603(Carbonyl, C=O Str), 1538, 1488(C=C,aromatic Str), 589(-Br Str)

^{1}H NMR (400 mHz, $CDCl_3$): δ 3.13(m, 1H,CH_2), 3.18(m, 1H,CH_2), 4.07 (m,1H,NH), 4.45-4.49(m,1H,CH), 6.58-6.63(m,2H, Ar-H), 7.18-7.28(m,2H,Ar-H), 7.31-7.57(m,4H,Ar-H), 7.85-7.89(m,2H, Ar-H). ESI-MS m/z(%) :381 ($[M+H]^+$ 100).

Table 1. Synthesis of 1,3-diphenyl-3-(phenylamino)propan-1-one derivatives.

S. No.	Compound	R_1	R_2	Time (Hrs)	Yield (%)
1	1,3-diphenyl-3-(phenylamino)propan-1-one(4a)	H	H	3	96
2	3-(4-Chlorophenyl)-1-phenyl-3-(phenylamino)propan-1-one(4b)	4-Cl	H	3.25	95
3	1-phenyl-3-(phenylamino)-3-(P-tolyl)propan-1-one(4c)	4-CH_3	H	3	96
4	3-(2-Chlorophenyl)-1-phenyl-3-(phenylamino)propan-1-one(4d)	2-Cl	H	3.25	95
5	1,3-diphenyl-3-(p-tolylamino)propan-1-one.(4e)	H	4-CH_3	3.25	95
6	3-((4-chlorophenyl)amino)-1,3-diphenylpropane-1-one(4f)	H	4-Cl	3.5	94
7	3-(4-hydroxyphenyl)-1-phenyl-3-(phenylamino)propan-1-one(4g)	4-OH	H	4	94
8	3-((4-bromophenyl)amino)-1,3-diphenylpropan-1-one(4h)	H	4-Br	3.5	95
9	3-(4-methoxyphenyl)-1-phenyl-3-(phenylamino)propan-1-one(4i)	4-OCH_3	H	4	94
10	3-(4-bromophenyl)-1-phenyl-3-(phenylamino)propan-1-one (4j)	4-Br	H	3.5	95

3-(4-methoxyphenyl)-1-phenyl-3-(phenylamino) propan-1-one (4i)

IR(KBr) : 3377(2^0-amine,NH Str), 1664,1602 (Carbonyl, C=O Str),1510, 1510,1 445 (C=C,aromatic Str), 1067, 1027(OCH_3 Str). ^{1}H NMR (400 mHz, $CDCl_3$): δ 3.13-3.18(m, 2H,CH_2), 3.86(s, 3H, -OCH_3),4.07 (m,1H,NH), 4.48-4.51(m,1H,CH), 6.63-6.73(m,2H, Ar-H), 6.88-6.92(m,2H, Ar-H), 7.07-7.15(m,1H,Ar-H), 7.27-7.32(m,1H,Ar-H), 7.44-7.48(m,2H,Ar-H), 7.86-7.91(m,2H,Ar-H), ESI-MS m/z(%) :332 ([M+H]$^+$ 100).

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

IR(KBr) : 3313(2^0-amine,NH Str), 1658, 1608(Carbonyl, C=O Str), 1487, 1446(C=C, aromatic Str), 588(-Br Str)
^{1}H NMR (400 mHz, $CDCl_3$): δ 3.13(m, 1H,CH_2), 3.18(m, 1H,CH_2),4.06 (m,1H,NH), 4.41-4.45(m, 1H, CH), 6.58-6.73(m, 2H, Ar-H), 7.18-7.28(m, 2H, Ar-H), 7.31-7.45(m, 4H, Ar-H), 7.87-7.94(m, 2H, Ar-H), ESI-MS m/z(%) : 381 ([M+H]$^+$ 100).

Results and Discussion

The current reaction was seen under varying quantities of catalyst. The findings indicate that a catalyst dosage of 500mg is sufficient to provide a favourable product yield. Increasing the quantity of catalyst beyond a certain point does not result in a significant change in the yield of the product. Therefore, a quantity of 500mg of catalyst was utilised in order to carry out the reaction. In this investigation, the model reaction (Scheme 1) was conducted using a mixture of benzaldehyde, aniline, and acetophenone in ethanol. The findings are presented in Table 2.

Comparative catalytic activity of MTSA with other catalysts for the synthesis of 1,3-diphenyl-3-(phenylamino)propan-1-one derivatives.

Table 2. Effect of catalyst loading on the formation of 1,3-diphenyl-3-(phenylamino) propan-1-one derivatives.

S. No.	Catalyst loading in (mg)	Time (Hrs)	Yield (%)
1.	200	3	42
2.	300	3	66
3.	400	3	78
4.	500	3	96
5.	600	3	96

Table 3 displays the reaction timings associated with the synthesis of 1,3-diphenyl-3-(phenylamino)propan-1-one derivatives using different catalysts. It has been shown that when utilising different catalysts, particularly under reflux conditions, the reaction durations exhibit a significant increase. The synthesis of 1,3-diphenyl-3-(phenylamino)propan-1-one derivatives under reflux conditions, catalysed by Bismuth nitrate[30], has been documented to exhibit shorter reaction durations. The current methodology presents a cost-effective and readily repeatable approach, hence providing a highly efficient MTSA (Multi-Tasking Synthetic Agent) for achieving desired outcomes. The following table, labelled as Table 3, provides relevant data or information.

Effect of solvent on synthesis of 1,3-diphenyl-3-(phenylamino)propan-1-one derivatives

Table 3. Comparative catalytic activity of MTSA with other catalysts.

S. No.	Catalyst	Time (hrs)	Temp (°C)	Yield (%)
1.	Dipyridine copper chloride	9	85	95
2.	MgO/ZrO_2	8	80	91
5.	2,3-dibromsuccinic acid	6	R.T.	80
6.	$Al(NO_3)_3$. $9H_2O$	4	R.T.	85
8.	Bismuth nitrate	4	R.T.	89
9.	MTSA	3	80	96

The examination of the reaction medium in the investigations indicated that solvents exerted a significant influence on the reaction. The findings are succinctly presented in Table 4. The results indicated that polar solvents, such as CH_3COOH, CH_3CN, and C_2H_5OH, exhibited significantly superior perfor-

Table 4. Effect of solvent on synthesis of 1,3-diphenyl-3-(phenylamino)propan-1-one derivatives

S. No.	Catalyst	Solvent	Time (hrs)	Yield (%)[a]
1	MTSA	H_2O	48	Trace
2	MTSA	CH_2Cl_2	20	30
3	MTSA	CH_3COOH	12	85,55[b]
4	MTSA	CH_3CN	5	90
5	MTSA	C_2H_5OH	3	96

All reactions were carried out under reflux conditions with 500 mg of catalyst.
[a] Isolation yields [b] Catalyst was reused

mance compared to non-polar solvents. A minimal yield was reported when water was employed as the solvent, perhaps attributable to the agglomeration of the hydrophobic catalyst. Despite the effectiveness of CH_3COOH, the reuse of the catalyst resulted in a low yield. Consequently, ethanol was chosen as the solvent.

Effect of Temperature on synthesis of 1, 3-diphenyl-3-(phenylamino)propan-1-one derivatives.

Table 5 presents the reaction temperature of 80 °C for the synthesis of 1,3-diphenyl-3-(phenylamino) propan-1-one derivatives using the MTSA catalyst. The experimental findings indicated that at temperatures below 80 °C, the yield of the reaction was found to be relatively low, while the duration of the reaction was seen to be comparatively long. The experiment was conducted at a temperature of 80 °C.

Table 5. Effect of temparature on the formation of 1,3-diphenyl-3-(phenylamino) propan-1-one derivatives

S. No.	Catalyst	Temparature (°C)	Time (hrs)	Yield (%)[a]
1	MTSA	R.T.	30	20
2	MTSA	40	20	50
3	MTSA	50	12	75
4	MTSA	70	3	96

All reactions are carried out using 500 mg of catalyst in Ethylalcohol. [a]Isolated yields

Reusability of the catalyst

The catalyst's reusability is a significant advantage that enhances its utility in commercial applications. Therefore, an investigation was conducted on the recovery and reusability of MTSA. In the course of these tests, the reaction mixture was subjected to isolation using ethanol. The catalyst was efficiently recycled by a filtration process following a thorough washing with ethanol and subsequent drying at a temperature of 60 °C. The catalyst was able to be reused for a total of five cycles without any discernible decrease in its catalytic activity. The following table, labelled as Table 6, is presented for reference.

Table 6. Recyclability of the catalyst in the reaction, synthesis of 1,3-diphenyl-3-(phenylamino)propan-1-one derivatives

Run no.	Yield (%)
1	96
2	95
3	94
4	91
5	90

Conclusion

The utilisation of MTSA as a catalyst in the Mannich process, involving aldehydes, amines, and ketones, has demonstrated excellent outcomes. The use of this catalyst has numerous benefits. Firstly, the preparation process for the catalyst is straightforward. Additionally, the catalyst requires a low loading and allows for convenient operation. Moreover, it facilitates high yield without the production of any by-products. Furthermore, the catalyst can be readily recycled and reused, further enhancing its practicality. The primary contribution of this study is in the effective synthesis of MTSA, which demonstrates a harmonious equilibrium between recyclization and activity in the context of the Mannich reaction.

Competing interests: There are no competing interests

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