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Synthesis of Schiff bases of 2-amino benzo[d]thiazole from higher hetero aldehydes and ketones using Mo-Al₂O₃ composite-based organocatalyst

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A new metal supported organo catalyst has been successfully employed to carry out the high yield Schiff's base reaction at ambient temperature. This methodology has the benefits of straightforward conditions, excellent yields, no work-up, environmental benign process and reusable solid catalyst. The catalyst speeds up the Schiff base reaction with less time and several Schiff bases have been synthesized and characterized with IR, ¹H and ¹³C NMR techniques.

Keywords: E-Imines, Mo-Al₂O₃composite, Environmentally benign condensation, organocatalyst

Catalysts in heterogeneous-based organic transformations typically have exhibited good activity, high surface area, stability, and recyclability. Furthermore, mesoporous materials modified with various metal ions (M_x^+) and metal oxides (M_xO_y) have been used as solid acidic catalysts for various industrially significant organic transformations¹ like Mannich reactions^{1d}, Schiff base reaction^{1e}, Prins reaction^{1f}, and ester condensation reaction^{1g}.

The chemistry of Schiff base compounds has proven to be a fascinating area of research in previous years. Compounds with imine or azomethine (-C=N-) functional groups in their structure are known as Schiff bases. The extensible imine group is a part of the molecule that is active and results in diverse biologically active molecules. Schiff bases demonstrate beneficial biological processes such as anti-tubercular, anti-cancer, anti-bacterial, antipain-relieving, and inflammatory, anti-glycation properties. Additionally, Schiff's bases are utilized as corrosion inhibitors, dyes, pigments, intermediates in organic synthesis, stabilizers for polymers, and catalysts.

The method of synthesizing imines first described by Schiff involved the condensation of carbonyl compounds and primary amines followed by azeotropic distillation to remove the generated water. Molecular sieves² are used to remove the formed water in the reaction. Recently, in-situ dehydrating agents are developed like trimethyl orthoformate³, tetraethyl orthosilicate⁴, PPTS⁵, P₂O₅/Al₂O₃⁶, B(OCH₂CF₃)₃⁷, $Al_2O_3^8$ and SiO_2^{9} . In the recent literature, there were Schiff base reactions with reagents like MgSO₄ & $CuSO_4^{10}$, $Er(OTf)_3^{11}$, $Yb(OTf)_3^{12}$, Piperidine¹³, $Ti(OR)_4^{14}$, K-10¹⁵, Magnesium perchlorate¹⁶, CaO¹⁷, ionic liquids¹⁸, Fly-ash:H₂SO₄¹⁹, perchloric acid²⁰, SiO_2 -H₃PO₄²¹, tungsten oxide modified AlTUD-1 mesoporous acid²², solid acidic FeCl₃/Bentonite²³, $SOCl_2/Ether^{24}$, ethanol²⁵, silica supported²⁶ and using some mechanical grinding²⁷ and water suspension methods²⁸, although most of these reports are ineffective for more potent aromatic and hetero aldehydes.

The reported methods have numerous drawbacks, such as high reaction temperatures, prolonged reaction durations, expensive chemicals, moisturesensitive catalysts, and special equipment requirements. Additionally, the reported reaction is applicable to strong nucleophilic amines and higher carbonyl compounds. Therefore, it is in our interest to report on a Schiff's base reaction involving a bulky heterocyclic base, higher aromatic and heterocyclic aldehydes, and certain ketones.

Experimental Details

Materials and methods

Each reaction was conducted at room temperature under magnetic stirring conditions. The chemicals are purchased from Sigma-Aldrich, Alfa Aesar, and E.Merck. Silica gel 60F₂₅₄ TLC plates are used for analytical thin-layer chromatography. UV light is used to perform the resolution of the established chromatogram (254 nm). Using the Mettler FP51 instrument, the melting points of each Schiff base have been determined in open glass capillaries. Infrared spectra (KBr, 4000-400 cm⁻¹) were captured on an OMNIC Fourier transform spectrophotometer. Bruker AV 400 & 500 NMR spectrometers operating at 500 MHz & 100 MHz were used to record ¹H, ¹³C NMR spectra in CDCl₃ solvent, using TMS as the internal standard.

Preparation of Mo-Al₂O₃ composite

A conical flask was filled with around 20 mL of ethanol, to which 5 mmol (0.98 g) NH_4MoO_3 was added, and the mixture was then sonicated to create the colloidal suspension. 20 mL of ethanol containing basic alumina (Al₂O₃) (5 mmol, 0.51 g) was added to another conical flask and sonicated to get a colloidal suspension. The two solutions were mixed dropwise using a separating funnel and agitated with a magnetic stirrer for 24 h to create a homogeneous mixture. Once the Mo-Al₂O₃ composites had been made, the mixture was sonicated to make a fine powder. The mixture was stirred for 4 h at room temperature. The solution is filtered with a Buchner funnel using Whatman filter paper at room temperature. The obtained solid is dried at 110°C for 5 h in an oven and grind with a pestle and mortar, afforded the Mo-Al₂O₃ composites as a fine powder. This catalyst was calcined at 400°C for 2 h using a muffle furnace.

General synthesis procedure for the Schiff base compound

5 mL of methanol as a solvent was taken in a round bottom flask. Then 2-amino benzo[d]thiazole (0.46 g, 5 mmol), aromatic aldehyde (5 mmol) was added and stirred for 2 min then to the flask, (0.5 mmol, 0.23 g) Mo-Al₂O₃ composite (5 mol%) (5% with respect to the weight of aromatic aldehyde) and *p*-toluene sulphonic acid (7.5 mmol, 1.291 g) were added into the reaction mixture. The reaction was maintained with constant stirring at room temperature (30°C) for 4 h. At the end of the reaction, 20 mL of ethyl acetate was added to the reaction, and the catalyst was recovered from the reaction mass by filtration. The ethyl acetate layer was collected and recrystallized with hexane/ethyl acetate to get the pure product. In this condensation, the utilized reactants, reaction time and yields are presented in Table 1. The product was characterized to IR, H¹ and ¹³C NMR spectra. Preparation and characterization of the catalyst and NMR and IR spectra of the synthesized product are given in Supplementary Information. The complete characterization data of all synthesized Schiff bases are summarized below.

(E)-N-(benzo[d]thiazol-2-yl)-1-(6-methoxy-

naphthalen-1-yl) methanimine (3a): Yield, 91%; yellow colour solid; m.p. 206-209°C; ¹H NMR (500 MHz, CDCl₃) δ ppm: 9.05 (s, 1H), 8.13 (s, 1H), 7.78-7.68 (m, 4H), 7.36-7.30 (d, 2H), 7.19-7.03 (m, 3H), 3.85 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 169.39, 160.26, 138.25, 134.25, 132.61, 130.97, 129.11, 127.70, 125.86, 123.48, 121.84, 120.00, 115.29, 106.41, 55.61. IR (CHCl₃) υ 3307, 2929, 2846, 2780, 2353, 2162, 1975, 1849, 1694, 1618, 1466, 1325, 1266, 1162, 1095, 1010, 900, 810, 748, 672 cm^{-1.}

(*E*)-1-(anthracen-9-yl)-N-(benzo[d]thiazol-2-yl) methanimine (3b): Yield, 89%; dark red colour solid; m.p. 214-216°C; ¹H NMR (500 MHz, CDCl₃) δ ppm: 8.88-8.86 (s, 1H), 8.51-8.47 (s, 1H), 7.93-7.91 (m, 3H), 7.60-7.58 (d, 2H), 7.47-7.45 (d, 2H), 7.36-7.25 (d, 2H), 7.19-7.17 (d, 2H), 7.15-7.05 (d, 2H), 7.04-7.01 (s, 1H). ¹³C NMR (100 MHz, CDCl₃) δ 168.57, 145.13, 140.91, 135.17, 134.02, 132.33, 131.15, 129.42, 127.43, 127.08, 125.62, 123.42, 121.38, 116.55. IR (CHCl₃) υ 3323, 2901, 2842, 2806, 1975, 1623, 1548, 1451, 1325, 1252, 1154, 1049, 908, 767, 720 cm⁻¹.

(*E*)-N-(benzo[d]thiazol-2-yl)-1-(pyren-1-yl)methanimine (3c): Yield, 81%; light yellow colour solid; m.p. 221-223°C; ¹H NMR (500 MHz, CDCl₃) δ ppm: 9.24 (s, 1H), 8.29-8.27 (s, 1H), 8.19-8.17 (m, 4H), 8.14-8.07 (d, 2H), 8.00-7.90 (s, 1H), 7.90-7.86 (s, 1H), 7.41-7.09 (m, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 168.94, 143.02, 136.68, 135.48, 131.65, 131.06, 127.08, 126.88, 124.36, 123.69, 122.77, 121.63, 115.93. IR (CHCl₃) v 3050, 2916, 2870, 2330, 2158, 2085, 2001, 1928, 1823, 1767, 1595, 1525, 1443, 1356, 1330, 1235, 1158, 900, 810, 748, 700 cm⁻¹.

(*E*)-N-(benzo[d]thiazol-2-yl)-1-(furan-2-yl)methanimine (3d): Yield, 99%; black colour solid; m.p. 163-165°C; ¹H NMR (500 MHz, CDCl₃) δ ppm: 8.95 (s, 1H), 7.81 (s, 1H), 7.56-7.50 (d, 2H), 7.46-7.26 (d, 2H), 7.14-7.02 (d, 2H). ¹³C NMR (100 MHz, CDCl₃) δ 167.70, 149.13, 141.20, 140.76, 129.76, 129.08, 126.17, 122.42, 121.05, 117.77. IR (CHCl₃) υ 3299, 3050, 2916, 2846, 2749, 2612, 2338, 2158, 2018, 1713, 1607, 1561, 1517, 1443, 1314, 1266, 1201, 1114, 1036, 928, 884, 740, 697 cm⁻¹.

(*E*)-N-(benzo[d]thiazol-2-yl)-1-(thiophen-2-yl) methanimine (3e): Yield, 99%; yellow colour solid; m.p. 172-173°C; ¹H NMR (500 MHz, CDCl₃) δ ppm: 9.12 (s, 1H), 7.91-7.76 (m, 3H), 7.75-7.50 (s, 1H),



(Contd.)



7.26-7.15 (d, 2H), 7.07-7.00 (s, 1H). ¹³C NMR (100 MHz, CDCl₃) δ 167.39, 158.57, 150.06, 136.68, 135.33, 130.28, 128.95, 128.49, 126.13, 122.37, 120.96, 118.22. IR (CHCl₃) υ 3319, 3273, 2964, 2932, 2859, 2167, 1947, 1759, 1635, 1545, 1455, 1407, 1314, 1247, 1131, 1049, 1005, 920, 892, 754, 723, 677 cm⁻¹.

(*E*)-N-(benzo[d]thiazol-2-yl)-1-(2-chloro-8-methylquinolin-4-yl) methanimine (3f): Yield, 99%; dark red colour solid; m.p. 190-192°C; ¹H NMR (500 MHz, CDCl₃) δ ppm: 8.32 (s, 1H), 8.22-7.39 (m, 6H), 7.31-7.08 (d, 1H), 5.69-5.59 (s, 1H), 2.72-2.62 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 168.30, 148.28, 146.75, 137.50, 136.44, 130.92, 129.09, 128.85, 127.09, 126.99, 126.93, 126.80, 125.98, 125.91, 125.57, 123.59, 121.55, 116.44, 100.49, 53.82. IR (CHCl₃) υ 3323, 3163, 3053, 2996, 2963, 2918, 2372, 2082, 1801, 1626, 1595, 1531, 1446, 1395, 1325, 1252, 1174, 1084, 979, 935, 885, 812, 750, 682 cm⁻¹.

(*E*)-N-(benzo[d]thiazol-2-yl)-1-(5-methoxy-3Hindol-3-yl) methanimine (3g): Yield, 92%; black colour solid; m.p. 187-189°C; ¹H NMR (500 MHz, CDCl₃) δ ppm: 8.97 (s, 1H), 7.83-7.70 (m, 4H), 7.43-7.29 (m, 4H), 3.82 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 168.48, 156.22, 144.00, 140.89, 131.76, 129.32, 126.88, 125.96, 123.51, 121.48, 116.38, 114.37, 113.08, 55.69. IR (CHCl₃) v 3315, 3155, 3104, 2910, 2834, 2698, 2507, 2150, 1959, 1857, 1631, 1579, 1541, 1474, 1412, 1368, 1302, 1240, 1201, 1139, 1075, 942, 838, 794, 717, 681 cm⁻¹.

(*E*)-N-(benzo[d]thiazol-2-yl)-1-(4-chlorophenyl) ethan-1-imine (3h): Yield, 99%; white colour solid; m.p. 108°C; ¹H NMR (500 MHz, CDCl₃) δ ppm: 8.26-8.12 (d, 2H), 8.10-7.86 (t, 3H), 7.50-7.44 (d, 1H), 7.33-7.18 (d, 2H), 2.67 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 168.57, 143.52, 141.33, 131.83, 130.35, 129.41, 126.94, 125.74, 123.53, 121.64, 116.23, 29.43. IR (CHCl₃) υ 3312, 3108, 2910, 2834, 1764, 1694, 1607, 1517, 1474, 1322, 1229, 1139, 1083, 1005, 962, 861, 736, 704 cm⁻¹.

(*E*)-N-(benzo[d]thiazol-2-yl) propan-2-imine (3i): Yield, 99%; white colour solid; m.p. 98-99°C; ¹H NMR (500 MHz, CDCl₃) δ ppm: 7.47-7.30 (d, 2H), 7.29-7.12 (s, 1H), 7.11-7.03 (s, 1H), 1.25 (s, 6H). ¹³C NMR (100 MHz, CDCl₃) δ 167.05, 148.40, 128.88, 126.30, 123.06, 120.87, 117.60, 29.31. IR (CHCl₃) υ 3397, 3265, 3065, 2901, 2865, 2710, 2366, 2158, 1913, 1717, 1615, 1548, 1446, 1361, 1294, 1185, 1119, 1032, 1013, 931, 888, 736, 677 cm⁻¹.

(*E*)-N-(benzo[d]thiazol-2-yl) cyclohexanimine (3j): Yield, 99%; black colour solid; m.p. 112-113°C; ¹H NMR (500 MHz, CDCl₃) δ ppm: 7.45-7.42 (d, 2H), 7.23-7.08 (s, 1H), 7.78-7.68 (m, 4H), 7.05-7.00 (s, 1H), 2.59-2.24 (m, 3H), 1.86-1.69 (d, 2H), 1.26 (s, 6H). ¹³C NMR (100 MHz, CDCl₃) δ 167.46, 149.41, 128.86, 126.32, 122.13, 121.14, 117.60, 41.78, 29.96, 27.05. IR (CHCl₃) υ 3369, 3191, 2995, 2924, 2859, 2252, 2049, 1736, 1623, 1536, 1240, 1100, 1049, 915, 857, 731, 689 cm⁻¹.

(*E*)-N-(benzo[d]thiazol-2-yl)-1,1-diphenylmethanimine (3k): Yield, 99%; red colour solid; m.p. 119- 120° C; ¹H NMR (500 MHz, CDCl₃) δ ppm: 7.78-7.60 (m, 4H), 7.57-7.46 (d, 2H), 7.45-7.30 (m, 6H), 7.36-7.30 (d, 2H), 7.27-7.24 (s, 1H), 7.13-7.05 (s, 1H). ¹³C NMR (100 MHz, CDCl₃) δ 167.41, 149.42, 137.22, 132.06, 129.86, 127.88, 125.68, 122.14, 120.87, 117.96. IR (CHCl₃) υ 3299, 3202, 3073, 2924, 2859, 2162, 2045, 1990, 1720, 1618, 1522, 1432, 1381, 1314, 1266, 1181, 1126, 1049, 951, 903, 810, 767, 697 cm⁻¹.

(*E*)-(18,5S)-N-(benzo[d]thiazol-2-yl)-8,8-dimethylbicyclo [3.2.1] octan-6-imine (3I): Yield, 90%; light red colour solid; m.p. 221-223°C; ¹H NMR (500 MHz, CDCl₃) δ ppm: 7.97-7.92 (s, 1H), 7.53-7.43 (s, 1H), 7.26-7.13 (d, 2H), 3.11-3.04 (d, 2H), 2.72-2.65 (dd, 1H), 2.46-2.28 (m, 3H), 2.07-2.04 (d, 2H), 1.26 (s, 8H). ¹³C NMR (100 MHz, CDCl₃) δ 169.71, 140.83, 136.57, 133.26, 129.03, 128.55, 128.12, 125.74, 127.43, 125.86, 124.34, 122.22, 114.86, 37.58, 33.53, 31.90, 29.56. IR (CHCl₃) υ 3045, 2986, 2901, 2784, 2648, 2167, 1967, 1810, 1685, 1623, 1579, 1466, 1373, 1280, 1193, 1052, 965, 864, 723, 681 cm⁻¹.

Results and Discussion

Mechanism of the condensation reaction

The condensation followed a well-known acid catalyzed mechanism as shown in Scheme 1. The amine nucleophile attacks of carbonyl carbon of aldehyde or ketone followed by elimination of water give the Schiff bases. The first step was the acidic site of the catalyst bonded with the carbonyl oxygen of the carbonyl group of aldehyde or ketone, and carbocation is formed. This carbocation is then attacked by the amine nucleophile to form an intermediate with a N-atom carrying a positive charge. This positive charge is neutralized by the removal of proton. Further the catalyst is removed and the elimination of water molecule results in the formation of the Schiff base.

Optimization of reaction conditions

In the synthesis, 2-chloro-8-methylquinoline-4carbaldehyde (1f) was reacted with 2-amino benzo[d]thiazole (2) on different temperatures and times using the various catalysts affording N-(benzo[d]thiazol-2-yl)-1-(2-chloro-8-methylquinolin-4-yl) methanimine (3f) as a reference reaction. We experimented with strong acids like HCl, H₂SO₄, weak acids like CH₃COOH, Lewis acids like ZnCl₂, amphoteric salts like Al₂O₃,(NH₄)₂MoO₃ and catalysts like *p*-TSA, *p*-TSA/Al₂O₃ in different conditions in solvents like H₂O, CH₂Cl₂, THF, EtOH, MeOH and



Scheme 1 — Mechanism of synthesis of Schiff's bases byMo-Al₂O₃-PTSA assisted condensation of amine and carbonyl compounds

| Table 2 — Optimization of catalyst | | | | | | | | |
|------------------------------------|---|------------|-------|------|-------|--|--|--|
| Entry | Catalyst | Solvent | Temp | Time | Yield | | | |
| | | | (°C) | (h) | (%) | | | |
| 1 | no catalyst added | Methanol | rt | 24h | nr | | | |
| 2 | HCl | H_2O | rt | 24h | nr | | | |
| 3 | Trimethyl ortho formate | THF | 80°C | 24h | nr | | | |
| 4 | ZnCl ₂ | Toulene | 100°C | 24h | nr | | | |
| 5 | Al_2O_3 | CH_2Cl_2 | rt | 24h | nr | | | |
| 6 | (NH4) ₂ MoO ₃ +Al ₂ O ₃ | CH_2Cl_2 | rt | 24h | nr | | | |
| 7 | p-TSA | CH_2Cl_2 | rt | 24h | <1 | | | |
| 8 | Al ₂ O ₃ /p-TSA | Methanol | rt | 24h | 2 | | | |
| 9 | CH ₃ COOH | Ethanol | 80°C | 24h | 5 | | | |
| 10 | Mo-Al ₂ O ₃ composites in | Methanol | rt | 4h | 99 | | | |
| | p-TSA | | | | | | | |

toluene and the results are shown in Table 2. We found that the reaction 2-chloro-8-methylquinoline-4carbaldehyde (1f) with 2-amino benzo[d]thiazole (2) at room temperature (30° C) in the presence of Mo-Al₂O₃ composites in *p*-TSA in Methanol (Table 2, entry 10) as the good conditions to afforded N-(benzo[d]thiazol-2-yl)-1-(2-chloro-8-methylquinolin-4-yl) methanimine (**3f**) in the high yields (Table 2, entry 10). Without a catalyst, no reaction is seen and the initial ingredients are recovered and unchanged (Table 2, entry 1).

Optimization of the solvent

The yields of the direct Schiff base reaction catalyzed by 5 mol% Mo-Al₂O₃composites in presence of a *p*-TSA catalyst is observed in different solvents like H₂O, DMSO, DMF, CH₂Cl₂, CHCl₃, Toulene, THF, EtOH, MeOH as

| Table 3 — Optimization of solvent | | | | | | | |
|-----------------------------------|-------------------|----------|-----------|--|--|--|--|
| Entry | Solvent | Time (h) | Yield (%) | | | | |
| 1 | no solvent added | 8h | nr | | | | |
| 2 | H_2O | 8h | nr | | | | |
| 3 | DMSO | 8h | nr | | | | |
| 4 | DMF | 8h | nr | | | | |
| 5 | CH_2Cl_2 | 8h | nr | | | | |
| 6 | CHCl ₃ | 8h | nr | | | | |
| 7 | Toulene | 8h | nr | | | | |
| 8 | THF | 8h | nr | | | | |
| 9 | EtOH | 8h | 81 | | | | |
| 10 | MeOH | 4h | 99 | | | | |

Table 4 — Effect of loading of catalyst

| Entry | Catalyst mol (%) | Temperature (T) | Time (h) | Yield ^a (%) |
|-------|------------------|-----------------|----------|------------------------|
| 1 | 1.0 | rt | 24h | 20 |
| 2 | 2.0 | rt | 24h | 32 |
| 3 | 5.0 | rt | 4h | 99 |
| 4 | 8.0 | rt | 24h | 75 |

presented in Table 3. It is observed that the reaction was not adequate for polar aprotic solvents like DMSO, DMF, THF, Toulene, $CHCl_3$, and CH_2Cl_2 . We found that the reaction is effective in polar protic solvents like EtOH, MeOH, and methanol is a suitable solvent for this condensation reaction (Table 3, entry 10).

Loading of the catalyst

The reaction was carried out with various mol% of the catalyst, and the yields are presented in Table 4. For the preparation of Mo-Al₂O₃ composites, we have taken $(NH_4)_2MoO_3$ and Al₂O₃ in 1:2 ratios in



Fig. 1 — Catalyst recycling of Mo-Al₂O₃/p-TSA catalyst for Schiff's base reaction. [Reaction condition: 2-chloro-8-methylquinoline-4-carbaldehyde (5f): 2-amino benzo[d]thiazole (6), 1:1 (5 mmole ratio), Mo-Al₂O₃Composites (0.25 mmol, 0.11 g) and *p*-TSA (7.5 mmol, 1.29 g) at room temperature (30° C)]

methanol. Lower ratios (0.5:1, 0.1:1, etc.) are not successful in conducting this reaction.

Reusability of the catalyst

The reaction of 2-chloro-8-methylquinoline-4carbaldehyde (1f) with 2-amino benzo[d]thiazole (2) was performed in presence of 5% of Mo-Al₂O₃/p-TSA catalyst for 4 h at room temperature. The filtered Mo-Al₂O₃/p-TSA catalyst is dried and reused because of its activity for five consecutive reactions, yielding the product in 60-65% yields. The reusability of the catalyst is shown in Fig. 1.

Conclusion

In conclusion, Schiff base reactions of various higher aromatic and hetero aldehydes (1a-l) with 2-amino benzo[d]thiazole (2) in presence of $Mo-Al_2O_3/p$ -TSA catalyst in methanol are reported. The reported method has the advantages of (a) mild procedure; (b) air-free and solid catalyst; (c) low cost and less workup; (d) the reusability of the catalyst. Moreover, this method is applicable to the synthesis of a wide range of Schiff bases.

Supplementary Information

Supplementary information is available in the website http://nopr.niscpr.res.in/handle/123456789/58776.

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