NANO $\text{SbCl}_5 \cdot \text{SiO}_2$: AN EFFICIENT CATALYST FOR THE SYNTHESIS OF QUINOXALINE DERIVATIVES AT ROOM TEMPERATURE UNDER SOLVENTLESS CONDITION

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ABSTRACT

Nano $\text{SbCl}_5 \cdot \text{SiO}_2$ as an eco-friendly and efficient nanocatalyst was applied for quinoxaline derivatives preparation with improved yield. In this protocol, α -diketones and 1,2-diamines were condensed in the presence of nanocatalyst at room temperature under solventless conditions. The method gave good yields of quinoxaline derivatives in short reaction times in comparison with earlier methods. Using nontoxic and inexpensive materials, simple work-up, short reaction times and high yields of the products are the advantages of this method.

Keywords: nano $\text{SbCl}_5 \cdot \text{SiO}_2$, nano-catalyst, quinoxaline derivatives, solventless, green condensation reactions

INTRODUCTION

The quinoxaline moiety is an important structural motif in the preparation of substances with pronounced biological activities¹. The quinoxaline core is presented in many drugs with antitumor²⁻³, anticancer⁴, antiamebic⁵, anticonvulsant⁶, antimalarial⁷, antioxidant⁸ and anti-protozoal activity⁹. Some fluorescent dyes¹⁰, electroluminescent materials¹¹, chemically controllable switches¹², and organic semiconductors also contain the quinoxaline moiety¹³. Because of important applications of quinoxaline compounds in both medicinal and industrial fields, a number of protocols have been developed for the synthesis of quinoxaline derivatives. Improved methods have been reported by using different catalysts such as gallium(III) triflate¹⁴, montmorillonite K10¹⁵, sulfamic acid¹⁶, cupric sulfate pentahydrate¹⁷, Zn (l-proline)¹⁸, I_2 ^{19,20}, Ni nanoparticles²¹, cellulose sulfuric acid (CSA)²², silica sulfuric acid (SSA)²³, sulfated titania²⁴, Zn^{2+} -montmorillonite K10 clay²⁵, L-proline²⁶, $\text{HClO}_4 \cdot \text{SiO}_2$ ²⁷, Ruthenium catalyst²⁸, acidic ionic liquids²⁹, β -cyclodextrin³⁰, methanol/acetic acid³¹ and the absence of any catalyst in water^{32,33} have been used for the preparation of quinoxalines.

However, most of the reported methodologies still have certain limitations such as expensive and air sensitive catalysts, toxicity and high boiling point of solvents and restrictions for large scale applications. Critical product isolation procedures, large amounts of catalysts and highly toxic wastes in scaling up for industrial applications leading to environmental issues are other limitations. Thus, the development of a simple and efficient method under solventless conditions for constructing these heterocycles has been advocated.

The use of inorganic solid acids as heterogeneous catalyst have received considerable importance in organic synthesis³⁴. Because of easy handling, enhanced reaction rates, high selectivity and easy separation of the products that such processes permitting the recycling and reuse of catalyst with operational and economical advantages. Antimony pentachloride (SbCl_5), a thin, colored and fuming liquid, is used in industry and organic synthesis³⁵. Since it fumes in air and reacts with moisture to form HCl, its handling and usability in the liquid form is laborious. Thus, the supported form is indeed preferable. It has been claimed that the supported SbCl_5 is a solid superacid. SbCl_5 is used extensively in organic synthesis as a Lewis acid for enhancing a variety of organic reactions such as Friedel-Crafts alkylation³⁶, electrophilic additions to alkenes and 1,3- dienes³⁷ and aromatization of enamine³⁸.

In continuation of our investigation on application of solid acids in organic synthesis³⁹⁻⁴⁴, we have used nano $\text{SbCl}_5 \cdot \text{SiO}_2$ for the synthesis of quinoxalines by condensation of benzil derivatives (1) with 1,2-phenylenediamine derivatives (2) in good to excellent yields (Scheme 1). The effect of solvent, molar ratio of reactants, amount of catalyst, temperature and time of reaction were investigated.

Scheme 1. Condensation of benzil with 1,2-phenylenediamine in the presence of nano $\text{SbCl}_5 \cdot \text{SiO}_2$ at room temperature under solventless conditions

EXPERIMENTAL

Material

Chemicals were purchased from the Merck Chemical Company in high purity. The IR spectra were recorded on FT-IR Magna 550 Nicolet using with KBr plates. ¹H-NMR spectra were obtained using a Bruker Avance 400 MHz DRX spectrometer. Melting points were determined without correction using a Büchi melting point B-540 Büchi apparatus. SEM and TEM analysis were carried out on VEGA/TESCAN scanning electron microscope and Leo 912AB OMEGA transmission electron microscopy, respectively. The X-ray diffraction (XRD) patterns of materials were prepared by employing a Philips Xpert MPD diffractometer equipped with a Cu K α anode ($\lambda = 1.54 \text{ \AA}$) in the 2θ range from 5 to 80°.

Preparation of catalyst

The catalyst was prepared by stirring a mixture of SbCl_5 (0.7 ml) and nano silica gel (20 nm, 1 g) in 5 ml chloroform for 1 h at room temperature. The slurry was filtered and washed with chloroform (2×5 ml). The obtained solid (62% w/w nano $\text{SbCl}_5 \cdot \text{SiO}_2$) was dried at ambient temperature for 2 h and then stored in a dry container⁴⁵.

General procedure for the synthesis of quinoxalines under solventless conditions

A mixture of 1,2-phenylenediamine derivative (1 mmol), 1,2-diketones (1 mmol) and nano $\text{SbCl}_5 \cdot \text{SiO}_2$ (62%w/w) (0.15 g) was stirred at room temperature under solventless condition. The reaction was monitored by TLC (*n*-hexane:ethyl acetate, 7:3). After completion of the reaction, the mixture was washed with chloroform (2×5 mL) and filtered to recover the catalyst. The solvent was evaporated and the crude product was recrystallized from ethanol (5 mL) to afford pure quinoxaline derivatives.

Spectroscopic data

2,3-diphenylquinoxaline (Table 1, 3a)

M.p. = 123–124 °C (123–125 °C [46]). ¹H NMR (CDCl_3 , TMS, 400 MHz): δ 7.3 (m, 6H); 7.52 (d, 4H, $J = 7.35 \text{ Hz}$); 7.76 (dd, 2H, $J = 3.3 \text{ Hz}$, $J = 3.45 \text{ Hz}$); 8.19 (dd, 2H, $J = 3.3 \text{ Hz}$, $J = 3.45 \text{ Hz}$) ppm; ¹³C NMR (CDCl_3 , TMS, 100 MHz): 128.27, 128.8, 129.26, 129.88, 129.93, 139.18, 141.29, 153.5 ppm.

6-methyl-2,3-diphenylquinoxaline (Table 1, 3b)

M.p. = 119–121 °C (118–120 °C [47]). ¹H NMR (CDCl_3 , TMS, 400 MHz):

δ 2.5 (s, 3H); 7.25 (m, 6H); 7.55 (m, 4H); 7.59 (d, J = 8.75 Hz, 1H); 7.98 (s, 1H); 8.02 (d, J = 8.5 Hz, 1H), ppm; ^{13}C NMR (CDCl_3 , TMS, 100 MHz): 21.94, 127.15, 128.12, 128.22, 128.51, 129.35, 131.28, 138.27, 138.65, 139.25, 140.22, 152.40, 153.25 ppm.

6-nitro-2,3-diphenylquinoxaline (Table 1, 3c)

M.p. = 186–188 °C (185–187 °C [47]). ^1H NMR (CDCl_3 , TMS, 400 MHz): δ 7.34 (m, 6H); 7.55 (d, J = 6.75 Hz, 4H); 8.32 (d, J = 9.23 Hz, 1H); 8.49 (d, J = 7 Hz, 1H); 9.05 (s, 1H), ppm; ^{13}C NMR (CDCl_3 , TMS, 100 MHz): 125.45, 128.32, 129.28, 129.58, 129.74, 129.83, 130.45, 138.36, 138.57, 139.97, 143.52, 147.74 ppm.

2,3-bis(4-methoxyphenyl)quinoxaline (Table 1, 3d)

M.p. = 146–148 °C (145–147 °C [46]). ^1H NMR (CDCl_3 , TMS, 400 MHz): δ 3.81 (s, 6H); 6.86 (d, J = 8.25 Hz, 4H); 7.48 (d, J = 8.25 Hz, 4H); 7.53 (m, 2H); 8.1 (m, 2H), ppm; ^{13}C NMR (CDCl_3 , TMS, 100 MHz): 55.12, 111.57, 127.47, 128.53, 130.15, 131.45, 140.78, 153.23, 159.58 ppm.

2,3-bis(4-methoxyphenyl)-6-methylquinoxaline (Table 1, 3e)

M.p. = 124–126 °C (125–127 °C [47]). ^1H NMR (CDCl_3 , TMS, 400 MHz): δ 2.65 (s, 3H); 3.72 (s, 6H); 6.56 (d, J = 8.75 Hz, 4H); 7.42 (d, J = 8.5 Hz, 4H); 7.55 (d, J = 8.5 Hz, 1H); 7.85 (s, 1H); 8.1 (d, J = 8.5 Hz, 1H) ppm; ^{13}C NMR (CDCl_3 , TMS, 100 MHz): 21.25, 55.47, 112.25, 127.48, 128.27, 130.78, 131.43, 131.56, 139.72, 140.08, 141.12, 152.19, 152.75, 160.12 ppm.

2,3-bis(4-methoxyphenyl)-6-nitroquinoxaline (Table 1, 3f)

M.p. = 191–193 °C (192–194 °C [47]). ^1H NMR (CDCl_3 , TMS, 400 MHz): δ 3.81 (s, 6H); 6.72 (d, J = 8 Hz, 4H); 7.53–7.58 (m, 4H); 8.15 (d, J = 9.25 Hz, 1H); 8.35 (d, J = 9.25 Hz, 1H); 8.95 (s, 1H) ppm; ^{13}C NMR (CDCl_3 , TMS, 100 MHz): 55.25, 113.15, 122.74, 124.49, 130.25, 130.41, 131.74, 131.82, 144.46, 155.74, 155.89, 160.90 ppm.

2,3-dimethylquinoxaline (Table 1, 3g)

M.p. = 105–106 °C (105–107 °C [48]). ^1H NMR (CDCl_3 , TMS, 400 MHz): δ 2.59 (s, 6H); 7.52–7.57 (m, 2H); 7.85–7.89 (m, 2H), ppm; ^{13}C NMR (CDCl_3 , TMS, 100 MHz): 23.08, 128.21, 128.48, 128.69, 140.95 ppm.

2,3,6-trimethylquinoxaline (Table 1, 3h)

M.p. = 112–114 °C (113–115 °C [48]). ^1H NMR (CDCl_3 , TMS, 400 MHz): δ 2.45 (s, 3H); 2.65 (s, 6H); 7.41 (d, J = 8.5, 1H); 7.68 (s, 1H); 7.81 (d, J = 8.25, 1H), ppm; ^{13}C NMR (CDCl_3 , TMS, 100 MHz): 21.5, 23.58, 127.14, 127.45, 131.48, 139.17, 139.48, 141.56, 152.43 ppm.

2,3-dimethyl-6-nitroquinoxaline (Table 1, 3i)

M.p. = 130–131 °C (130–132 °C [48]). ^1H NMR (CDCl_3 , TMS, 400 MHz): δ 2.75 (s, 6H); 8.01 (d, J = 9 Hz, 1H); 8.34 (d, J = 9 Hz, 1H); 8.78 (s, 1H), ppm; ^{13}C NMR (CDCl_3 , TMS, 100 MHz): 23.27, 122.24, 124.79, 129.86, 139.83, 143.63, 147.04, 156.27, 157.21 ppm.

2,3-dihydro-5,6-diphenylpyrazine (Table 1, 3j)

M.p. = 154–155 °C (154–156 °C [49]). ^1H NMR (CDCl_3 , TMS, 400 MHz):

δ 3.72 (s, 4H); 7.24–7.35 (6H, m), δ 7.41 (4H, dd, J = 7.9 and 1.8 Hz) ppm; ^{13}C NMR (CDCl_3 , TMS, 100 MHz): 43.27, 128.24, 129.72, 131.86, 139.43, 164.81 ppm.

2,3-Di-p-tolylquinoxaline (Table 1, 3k)

M.p. = 146–147 °C (147–148 °C [19]). ^1H NMR (CDCl_3 , TMS, 400 MHz): δ 2.33 (s, 6H); 7.21 (d, J = 7.9, 4H); 7.48 (d, J = 8.0, 4H); 7.66 (m, 2H); 8.19 (m, 2H) ppm; ^{13}C NMR (CDCl_3 , TMS, 100 MHz): 21.27, 128.24, 128.75, 129.66, 129.83, 135.63, 138.44, 140.83, 153.71 ppm.

RESULTS AND DISCUSSION

XRD, SEM and TEM analysis of catalyst

The structure of the prepared catalyst was identified by powder XRD. X-ray patterns of the nano $\text{SbCl}_5\cdot\text{SiO}_2$ were recorded. The XRD of nano SiO_2 showed no considerable peaks of crystallinity (Figure 1a). However, the XRD of nano $\text{SbCl}_5\cdot\text{SiO}_2$ (Figure 1b) showed small amount of crystallinity with the characteristic peaks. To obtain a visual image of the nano $\text{SbCl}_5\cdot\text{SiO}_2$, scanning electron microscopy (SEM) and transmission electron microscopy (TEM) were carried out. By SEM and TEM images, some information about the morphology of the catalyst nano particles was obtained as presented in (Figure 2a-d). As can be seen from the figure, the sample shows a nano crystalline structure.

Synthesis of quinoxalines in the presence of nano $\text{SbCl}_5\cdot\text{SiO}_2$ under solventless conditions

To demonstrate the generality and scope of this method, we examined the reaction of 1,2-diamine with a number of differently substituted 1,2-diketone under solventless conditions. The results of this study are presented in table 1.

Quinoxaline derivatives were synthesized over nano $\text{SbCl}_5\cdot\text{SiO}_2$ using various 1,2-diketones and 1,2-diamines at room temperature; the results are presented in table 1. As shown in table 1, all reactions have proceeded very efficiently at room temperature and no undesirable side-reactions were observed, although the yields were highly dependent on the substrate used. The effects of electron deficiency and of the nature of substituents on the aromatic ring of diamine and diketone on the yield of quinoxalines were small. For instance, electron-donating substituents present in diamine part, increased the yields of products, whereas the effect is reverse with the electron-withdrawing substituents (Table 1). On the other hand, electron-donating substituents with aromatic 1,2-diketone decreased the product yields. However, the variations in the yields were very little. The results summarized in table 1 clearly indicate the scope of the reaction with respect to the synthesis of quinoxaline derivatives on nano $\text{SbCl}_5\cdot\text{SiO}_2$ supported catalyst. The proposed mechanism for synthesis of quinoxaline may be visualized to occur *via* reactions as depicted in scheme 2.

Fig. 1. The XRD pattern of (a) nano SiO_2 and (b) nano $\text{SbCl}_5\cdot\text{SiO}_2$

Fig. 2. a) SEM image of nano SiO_2 , b) SEM image of nano $\text{SbCl}_5 \cdot \text{SiO}_2$, c) TEM image of nano SiO_2 , d) TEM image of nano $\text{SbCl}_5 \cdot \text{SiO}_2$

Scheme 2. Proposed mechanism for the synthesis of quinoxaline derivatives

Table 1. Synthesis of quinoxaline derivatives from 1,2-diketones (1mmol) and 1,2-diamines (1mmol) in the presence of nano $\text{SbCl}_5 \cdot \text{SiO}_2$ (0.15 g) at room temperature under solvent-less conditions

Entry	1, 2-diketone	1,2-diamine	Product	Time (min)	Yield ^{Ref.} (%)
1			 3a	8	99 ⁴⁶
2			 3b	10	95 ⁴⁷
3			 3c	15	91 ⁴⁷

4			3d	15	95 ⁴⁶
5			3e	25	95 ⁴⁷
6			3f	20	85 ⁴⁷
7			3g	35	90 ⁴⁸
8			3h	25	91 ⁴⁸
9			3i	40	88 ⁴⁸
10			3j	10	90 ⁴⁹
11			3k	15	92 ¹⁹

Optimization of the reaction conditions

Nano $\text{SbCl}_5 \cdot \text{SiO}_2$ with different loading of SbCl_5 were used and it was observed that as SbCl_5 loading are increased from 10 to 62% w/w, the yield of quinoxalines also are increased. However, on further increasing the SbCl_5 loading to 70 and 100 % w/w the yields are decreased with the formation of side products. Thus 62% w/w of nano $\text{SbCl}_5 \cdot \text{SiO}_2$ was used as the preferred catalyst for further studies (Table 2).

Table 2. Effect of Nano $\text{SbCl}_5 \cdot \text{SiO}_2$ on the synthesis of quinoxaline^a

Number	Catalyst amount(%w/w)	Time (min)	Yield (%) ^b
1	0	24h	0
2	10	40	40
3	30	32	45
4	50	15	80
5	60	9	90
6	62	8	99
7	70	10	72
8	100	10	40

^aReaction conditions: 1,2-diketone (1 mmol), 1,2-phenylenediamine (1mmol) at room temperature under solventless condition.

^bIsolated Yields

To choose the most appropriate medium in this heterocyclization reaction, the condensation of 1,2-phenylenediamine and benzil

was examined at room temperature in the presence of a catalytic amount of nano $\text{SbCl}_5 \cdot \text{SiO}_2$ in various solvents. It seems that CH_2Cl_2 , MeOH, EtOH, CH_3CN , Et_2O , DMF, PhCH_3 , EtOAc and THF have given excellent conversions. CH_3CN was the best among the solvents tested, but, we have find that the reaction have proceeded excellent under solvent-less conditions rather than using solvents (Table 3).

Table 3. Effect of solvents for the synthesis of quinoxaline^a using nano $\text{SbCl}_5 \cdot \text{SiO}_2$

Number	Solvent	Time (min)	Yield (%) ^b
1	CH_2Cl_2	15	95
2	MeOH	20	92
3	EtOH	15	90
4	Et_2O	30	91
5	THF	30	94
6	DMF	15	89
7	CH_3CN	12	96
8	PhCH_3	60	93
9	EtOAc	60	85
10	Solventless	08	99

^aReaction conditions: 1,2-diketone (1 mmol), 1,2-phenylenediamine (1mmol) at room temperature under solvent condition.

^bIsolated Yields

CONCLUSION

In conclusion, we have developed a simple and efficient synthesis of quinoxaline derivatives (11 samples) using nano $\text{SbCl}_5 \cdot \text{SiO}_2$ as catalyst under solventless conditions. Moreover the low cost of the catalyst, solventless condition, low toxicity of the catalyst, short reaction times, simple experimental procedure and high yields of the products are some of advantages of this protocol. We believe that our environmentally benign procedure will find important applications in the synthesis of quinoxalines.

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