



Original Article

Silica Supported 1-(2-(Sulfoxy)ethyl)pyridin-1-ium Chloride (SiO₂/[SEP]Cl) as an Efficient and Solid Acid Catalyst for the Synthesis of Quinoxaline Derivatives

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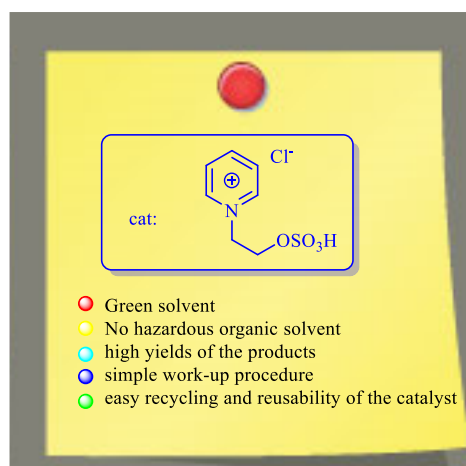
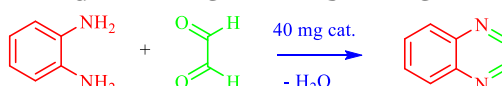
SiO₂/[SEP]Cl

One-pot reaction

ABSTRACT

Silica supported 1-(2-(sulfoxy)ethyl)pyridin-1-ium chloride has been explored as highly efficient catalyst for reactions under mild and green conditions for the synthesis of quinoxaline derivatives. The green and mild methods offer appealing attributes such as use of a recyclable, use of EtOH as green solvent and 60 °C condition, short reaction times, a simple workup and high yield of the products.

GRAPHICAL ABSTRACT



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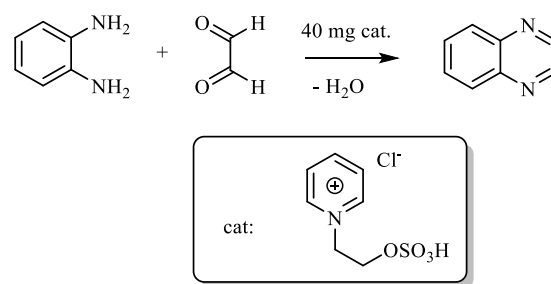
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Introduction

Among the heterocyclic compounds, quinoxaline and their derivatives are the important compounds due to their biological and pharmacological properties which perform antifungal [1], insecticide [2], antibacterial [3], anticancer, antimalarial, anti-HIV [4] and antibiotics [5] activities. In addition, the quinoxaline derivatives are utilized in industry, in dyes, fluorescent dyes, materials having electroluminescence properties and in the synthesis of organic semiconductors [6]. The methods reported in the literature for the synthesis of quinoxaline utilize various catalytic systems such as ultrasonic [7], $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ (II) [8], heteropoly acids [9], Si/MCM-41 [10], phosphosulfonic acid [11], boron sulfonic acid [12], citric acid [13], bismuth (III) triflate [14], ammonium chloride [15], and Zn(L-proline) [16]. In recent years, ionic liquid has received considerable interest due to their unique physical and chemical properties such as: designable chemical properties, high electrical conductivity, dual solvent-catalytic role [17], non-volatile, heat resistance, non-flammable and the ability to dissolve many organic and mineral and organic-metal compounds [18], energy storage and conversion of materials and devices [19]. Nowadays, there is a great attraction for many chemists in the synthesis of such compounds. The low vapor pressure of these compounds increases their potential as an alternative to organic volatile solvents without environmental problems and corrosion. Their fire-retardant nature minimizes the risk of flammable chemical processes [20]. Ionic liquids are a suitable medium for dissolving gases such as CO_2 , H_2 and O_2 in reactions that take place under ultra-critical conditions. Ionic liquids are easily extracted and recyclable [21].

As a part of our ongoing efforts in developing green catalysts, and their applications in catalytic organic reactions, and synthesis of heterocyclic compounds, and in this research, we would like to report the preparation of silica supported 1-(2-(sulfoxy)ethyl)pyridin-1-ium chloride

($\text{SiO}_2/[\text{SEP}]\text{Cl}$) and its application as highly efficient and green catalyst for synthesis of quinoxaline derivatives (Scheme 1).



Scheme 1: Synthesis of quinoxaline derivatives under the catalysis of $\text{SiO}_2/[\text{SEP}]\text{Cl}$

Material and methods

Chemicals and natural flake graphite powder (325 mesh, 99.95%) were purchased from the Aldrich Chemical Company. ^1H NMR and ^{13}C NMR spectra were measured for samples in CDCl_3 or DSMO-d_6 using a 400 MHz Bruker AVANCE instrument at 400 and 100 MHz, respectively, using Me_4Si as internal standard. The progress of the reactions was monitored by TLC on Silica Gel PolyGram SIL G/UV 254 plates. Melting points were measured in open capillary tubes using an electro thermal 9100 apparatus. Fourier transform infrared (FT-IR) spectra were recorded from KBr pellets using a Perkin Elmer Spectrum 65 FT-IR model instrument.

Preparation of $\text{SiO}_2/[\text{SEP}]\text{Cl}$

Silica-supported 1-(2-(sulfoxy)ethyl)1-pyridine-1-ium-chloride was prepared according to the literature procedure [22, 23]. A mixture of 2-chloro ethanol (0.40 g, 5 mmol) in CH_2Cl_2 (40 mL) was added drop-wise to a pyridine (0.36 g, 5 mmol) in a round bottomed flask at 0°C , and the resulting mixture was stirred for 24 h at $100\text{--}120^\circ\text{C}$. Then, a solution of chlorosulfonic acid (0.58 g, 5 mmol) in dry CH_2Cl_2 (40 mL) was added drop-wise to a mixture of reaction at 0°C over a period of 10 min. The CH_2Cl_2 layer was then decanted and the resulting residue was triturated with CH_2Cl_2 (3×10 mL) before being dried under vacuum at 90°C to give $\text{SiO}_2/[\text{SEP}]\text{Cl}$ as a viscous brown oil.

Spectral data of $\text{SiO}_2/[\text{SEP}]\text{Cl}$ are as follows. Viscous brown oil; IR (KBr): OH ($3200\text{--}3600\text{ cm}^{-1}$), C=N (1658 cm^{-1}), C=C (1426 cm^{-1}), S=O (1232 cm^{-1}), and S-O (614 cm^{-1}). ^1H NMR (400 MHz, $\text{DMSO-}d_6$): δ 3.13–3.16 (t, $J=5.6$ Hz, 2H), 3.93–3.970 (t, $J=8$ Hz, 2H), 8.10–8.17 (t, $J=12$ Hz, 2H), 8.61 (s, 1H), 8.92–8.99 (t, $J=6.4$ Hz, 2H), 9.00–9.42 (d, 1H). ^{13}C NMR (100 MHz, $\text{DMSO-}d_6$): 60.7, 61.8, 128.2, 142.2, 146.0.

General procedure for synthesis of quinoxaline derivatives under catalysis of $\text{SiO}_2/[\text{SEP}]\text{Cl}$

To a mixture of orthophenylene diamine (1.0 mmol), and 1,2-dicarbonyl compounds (1.0 mmol) in ethanol (5 mL), was added the catalyst $\text{SiO}_2/[\text{SEP}]\text{Cl}$ (40 mg), and the mixture was stirred at 60°C for an appropriate time (Table 2). The progress of the reaction was monitored by TLC. After completion of the reaction, the catalyst was isolated by filtration. The remaining supernatant liquid was evaporated to leave the crude product which was purified by recrystallization from the absolute EtOH. All the synthesized products were known compounds, and were characterized by their physical properties and spectral (FT-IR, ^1H NMR, ^{13}C NMR) analysis, and compared with the reported corresponding data.

2,3-Diphenylquinoxaline

white solid m.p $125\text{--}127$ [lit. $128\text{--}129$]²², FT-IR (KBr): 1556 cm^{-1} ; ^1H -NMR (FT-300 MHz, CDCl_3/TMS): δ 7.33977(bs, 6H, Ar-H) 7.54183(bs, 4H, Ar-H) 7.74584(bs, 2H, Ar-H) 8.20007(bs, 2H, Ar-H) ; ^{13}C NMR (300 MHz, CDCl_3): 128.290, 128.896, 129.121, 129.913, 130.066, 138.921, 141.115, 153.384; MS: $m/z = 282$ (M+).

6-Nitro-2,3-diphenylquinoxaline

red solid m.p $185\text{--}187$ [lit. $185\text{--}187$]²⁴, FT-IR (KBr): 1656 cm^{-1} (stretching C=N); ^1H -NMR (FT-300 MHz, CDCl_3/TMS): δ 7.38(bs, 6H, Ar-H) 7.56(bs, 4H, Ar-H) 8.28(bs, 1H, Ar-H) 8.45(bs, 1H, Ar-H) 9.02(bs, 1H, Ar-H); ^{13}C NMR (300 MHz, CDCl_3):; MS: 123.269, 125.512, 128.450, 129.667, 129.854, 129.953, 130.666, 137.950, 139.870,

143.390, 147.801, 155.621, 156.176; MS: $m/z = 327$ (M+).

Dibenzo[a,c]phenazine

yellow solid m.p $224\text{--}226$ [lit. $223\text{--}225$]²¹, FT-IR (KBr): 1604 cm^{-1} (stretching C=N); ^1H -NMR (FT-300 MHz, CDCl_3/TMS): δ 7.71(s, 4H, Ar-H) 7.85(s, 2H, Ar-H) 8.35(s, 2H, Ar-H) 8.43(s, 2H, Ar-H) 9.33(s, 2H, Ar-H); ^{13}C NMR (300 MHz, CDCl_3): 122.875, 126.448, 128.023, 128.925, 129.356, 130.205, 130.661, 132.019, 141.323, 141.876; MS: $m/z = 280$ (M+).

Acenaphtho[1,2-b]quinoxaline

yellow solid m.p $241\text{--}242$ [lit. $238\text{--}240$], FT-IR (KBr): 1614 cm^{-1} (stretching C=N); ^1H -NMR (FT-300 MHz, CDCl_3/TMS): δ 7.74–7.81(m, 4H, Ar-H) 8.05(d, $J=8.16$, 2H, Ar-H) 8.20–8.23(m, 2H, Ar-H) 8.4(d, $J=6.87$, 2H, Ar-H); ^{13}C NMR (300 MHz, CDCl_3): 122.328, 128.679, 129.285, 129.442, 129.694, 129.900, 131.248, 136.479, 140.707, 153.595; MS: $m/z = 254$ (M+).

Result and Dissection

Characterization of Catalyst $\text{SiO}_2/[\text{SEP}]\text{Cl}$

The silica supported 1-(2-sulfooxy)ethyl)1-pyridine-1-ium-chlorid was synthesized according to the literature procedure [23]. As depicted in Scheme 1-a, first step was prepared by the reaction between pyridine and 2-chloroethano that led to ionic liquid 2B. Next, 1-(2-sulfooxy)ethyl)1-pyridine-1-ium-chloride was synthesized using reaction of chlorosulfonic acid that, dropwise and slowly added to the ionic liquid B for 45–60 min at 0°C , which afforded 3C (Figure 1).

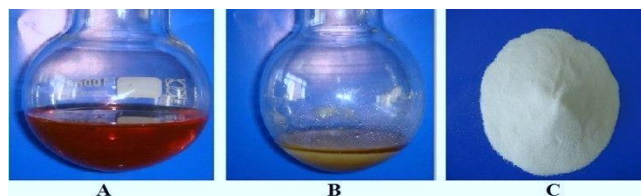


Figure 1: reaction of (a) 2-chloroethanol and pyridine, (b) 1-(2-sulfooxy)ethyl)1H-pyridine-1-ium-chloride, (c) silica-supported 1-(2-sulfooxy)ethyl)1-pyridine-1-ium-chloride

The structure of the prepared catalyst was established by performing different analytical methods such as Fourier transform infrared (FT-IR) spectroscopy, and ^1H and ^{13}C NMR spectra as described below.

Figure 2 illustrates the Fourier transform (FT-IR) spectra of the pyridine (A), 1-(2-sulfoxy)ethyl)1*H*-pyridine-1-ium-chloride (B), and silica-supported 1-(2-sulfoxy)ethyl)1-pyridine-1-ium-chloride (C). The O-H stretching vibration due to the carboxylic group is observed in the range of 3200-3600 cm^{-1} for B and C. The broad peaks at 1658 and 1426 cm^{-1} shown in the spectrum are assigned to the stretching vibrations of C=N and C=C groups, respectively. Also, the characteristic peaks at 1232 and 614 cm^{-1} in the spectra of C can be attributed to the stretch vibrations of the S=O and S-O groups, respectively, which has appeared in the Figures 2A, 2B and 2C.

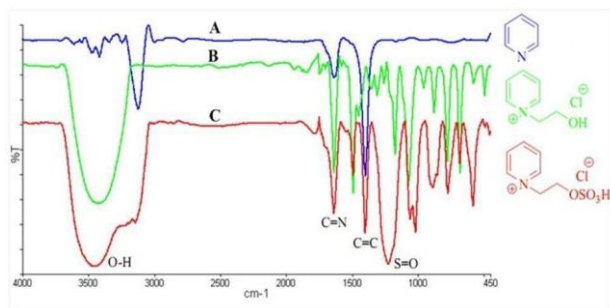


Figure 2: Comparative FT-IR spectra for (a) pyridine, (b) 1-(2-sulfoxy)ethyl)1*H*-pyridine-1-ium-chloride, (c) silica-supported 1-(2-sulfoxy)ethyl)1-pyridine-1-ium-chloride

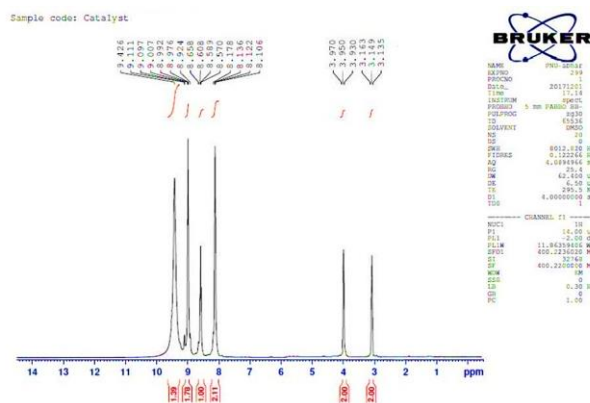


Figure 3: ^1H NMR spectrum of silica-supported 1-(2-sulfoxy)ethyl)1-pyridine-1-ium-chloride

The ^1H and ^{13}C NMR spectra of catalyst are presented in Figure 3 and 4. In the region of 8.61 ppm, a unique peak related to the acidic hydrogen (SO_3H). The ^{13}C NMR spectrum of the silica-supported 1-(2-sulfoxy)ethyl)1-pyridine-1-ium-chloride exhibited five signals in agreement with the proposed structure.

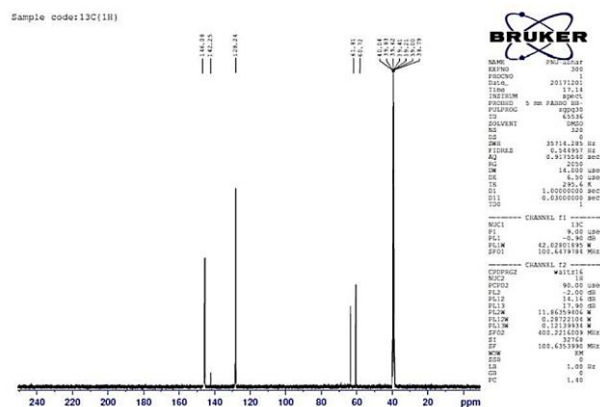


Figure 4: ^{13}C NMR spectrum of silica-supported 1-(2-sulfoxy)ethyl)1-pyridine-1-ium-chloride

Catalytic activity of the silica-supported 1-(2-sulfoxy)ethyl)1-pyridine-1-ium-chloride for the synthesis of quinoxaline derivatives

To assess the catalytic potential of the newly prepared silica-supported 1-(2-sulfoxy)ethyl)1-pyridine-1-ium-chloride) as heterogeneous catalyst in organic transformations, we decided to examine its activity in one-pot synthesis of quinoxaline derivatives from the reaction of aromatic *o*-phenyl diamine and 1,2-dicarbonyl. Preliminarily, we carried out the reaction of the synthesis quinoxaline, we chose the one-pot condensation reaction between *o*-phenyl diamine and benzil as model reaction.

To determine the optimal reaction conditions parameters such as the catalyst loading, solvents, and reaction temperature on the reaction were screened. First, the effect of the solvent on the reaction was studied using different solvents such H_2O , EtOH, poly ethylene glycol (PEG) and CH_3CN in the presence of an equal amount (40 mg) of the catalyst at 60 $^\circ\text{C}$. According to the experimental results summarized in Table 1, the best solvent was found to be EtOH in terms of the

reaction yield (entry 4). Next, in order to study the effect of temperature, the reaction was carried out at higher temperatures (room temperature, 40, 60 °C, and reflux point) using EtOH as the solvent of choice and catalyst loading of 40 mg. As shown in Table 1, increasing the temperature resulted no improvement effect neither on the yield nor on the time of the reaction (entry 12). For further studies regarding the effect of catalyst loading, the model reaction

was conducted using various amounts of the catalyst in EtOH at room temperature. From the results summarized in Table 1, it was clear that the catalyst loading of 0.04 gr presented the best conversion. The best results in terms of the reaction rate and yield of the product quinoxaline was obtained when the reaction was carried out in the EtOH as a solvent, 0.04 gr of catalyst and 60 °C.

Table 1: Optimization of reaction conditions for the synthesis of quinoxaline derivatives in the presence of silica-supported 1-(2-sulfoxy)ethyl)1-pyridine-1-ium-chloride

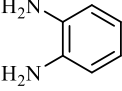
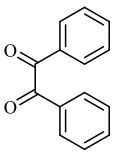
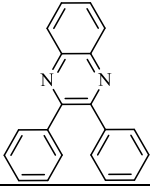
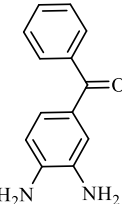
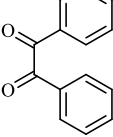
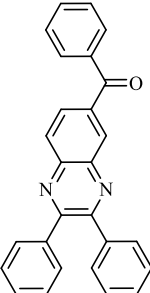
| Entry | Catalyst loading (%) | Solvent | Temperature (°C) | Reaction time (min) | Yield (%) ^a |
|-------|----------------------|------------------------|------------------|---------------------|------------------------|
| 1 | 20 | EtOH | 65 | 20 | 31 ^b |
| 2 | 30 | EtOH | 65 | 20 | 69 ^b |
| 3 | 40 | EtOH | 65 | 20 | 95 |
| 4 | 40 | EtOH | 65 | 15 | 97 |
| 5 | 40 | H ₂ O | 65 | 20 | 53 ^b |
| 6 | 40 | PEG | 65 | 20 | 74 ^b |
| 7 | 40 | CH ₃ CN | 65 | 20 | 60 ^b |
| 8 | 40 | EtOH | r.t. | 20 | 0 |
| 9 | 40 | EtOH/ H ₂ O | 65 | 24 h | 50 ^b |
| 10 | 40 | EtOH | 40 | 20 | Trace |
| 11 | 40 | EtOH | 65 | 20 | 31 ^b |
| 12 | 40 | EtOH | reflux | 15 | 97 |

^a isolated yields. ^b The products are separated using thin layer chromatography

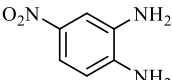
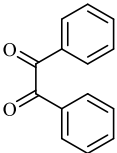
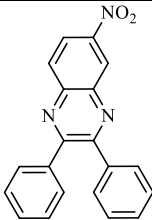
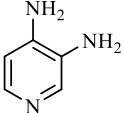
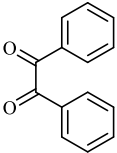
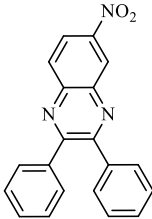
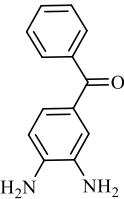
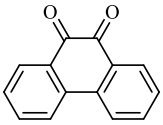
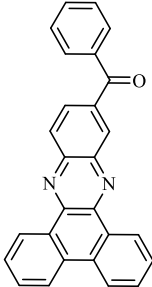
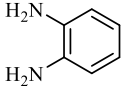
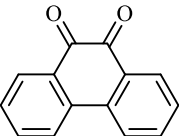
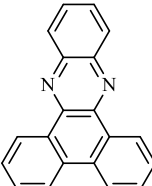
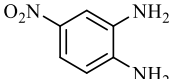
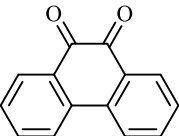
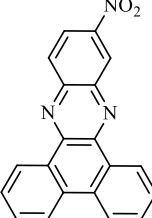
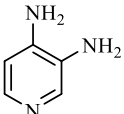
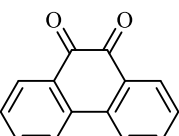
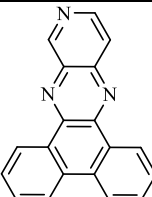
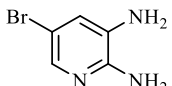
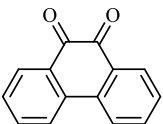
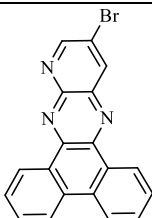
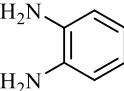
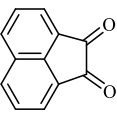
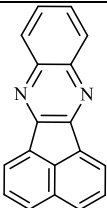
To establish the scope and general applicability of these reactions, a series of variously substituted diamines and 1,2-dicarbonyls were reacted under the aforementioned optimized conditions to furnish the corresponding quinoxaline. The experimental results are summarized in Tables 2.

All the products are known compounds which are characterized by their melting points and spectral (FT-IR, ¹H NMR, ¹³C NMR) analysis and compared with the reported data. The characteristic data for some selected products are presented in the experimental section.

Table 2. Synthesis of quinoxaline derivatives in the presence of catalyst ^a

| Entry | <i>o</i> -diamine | 1,2-dicarbonyl | product | Time (min) | Yield (%) ^b |
|-------|---|---|--|------------|------------------------|
| 1 |  |  |  | 20 | 95 |
| 2 |  |  |  | 95 | 90 |

Continued of table 2:

| | | | | | |
|----|---|---|---|-----|----|
| 3 |  |  |  | 75 | 93 |
| 4 |  |  |  | 600 | 89 |
| 5 |  |  |  | 15 | 87 |
| 6 |  |  |  | 120 | 96 |
| 7 |  |  |  | 20 | 91 |
| 8 |  |  |  | 30 | 90 |
| 9 |  |  |  | 105 | 90 |
| 10 |  |  |  | 480 | 97 |

Continued of table 2:

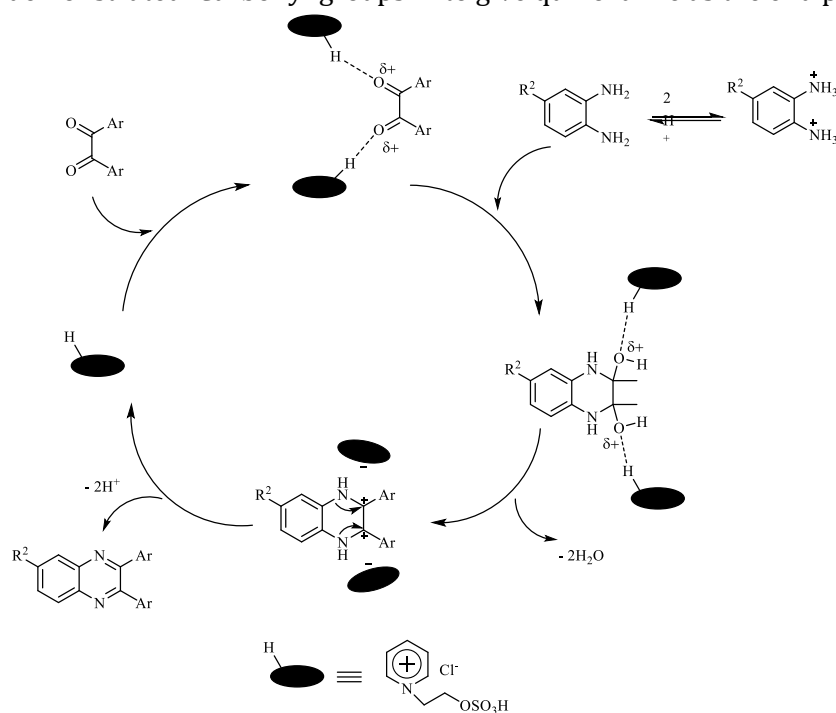
| | | | | | |
|----|--|--|--|-----|----|
| 11 | | | | 160 | 95 |
| 12 | | | | 30 | 95 |
| 13 | | | | 540 | 94 |

^aReaction conditions: *o*-diamine (1 mmol), 1,2-dicarbonyl (1 mmol), 40 mg catalyst, 5 mL ethanol as solvent and 65 °C. ^b Isolated product

Proposed Catalytic Reaction Mechanisms

In Scheme 2, the plausible reaction mechanism of silica-supported 1-(2-sulfoxy)ethyl)1-pyridinium-chloride is demonstrated. Carbonyl groups

in 1,2-dicarbonyl are activated by catalyst. Then reacts readily with *o*-phenylenediamine. The resultant amino-1,2-diol undergoes dehydration to give quinoxaline as the end product.



Scheme 2: Proposed mechanism for the synthesis of quinoxaline derivatives in the presence of catalyst

Catalyst Recyclability

The recyclability of the catalyst silica-supported 1-(2-sulfooxy)ethyl)1-pyridine-1-ium-chloride was studied for the model reaction under the optimized conditions repeatedly. The change of the catalytic activity in terms of the yield was assessed against the number of cycles of the reaction, as presented in Figure 5. After completion of the reaction in each cycle, the

catalyst was separated from the reaction mixture. The recovered catalyst was then washed with hot ethanol and dried in oven at 75 °C. As seen in Figure 5, the catalyst can be recycled and reused for at least five runs without considerable loss of activity. The slight decrease of catalytic activity could be due to the normal loss of the catalyst during the work-up stage.



Figure 5: Recycling and reuse in quinoxaline synthesis reaction under optimized conditions

As seen in Table 3, the experimental data resulted from the present research are compared to the data reported by other groups in the catalytic synthesis of quinoxaline. In some cases, homogeneous and non-recyclable catalysts or expensive catalysts are used, in some cases the reaction time is long and in some

cases green solvent is not used. However, in this project, the solvent is green, and the catalyst is easily separated and reused at the end of the reaction, this is one of the advantages of this catalyst system.

Table 3: Comparison of catalyst performance with previously reported catalysts in quinoxaline synthesis reaction

| Entry | catalyst | Condition reaction | Time (min) | Yield (%) | [ref.] |
|-------|--|-------------------------|------------|-----------|-----------|
| 1 | sulfated polyborate | solvent-free, 100 °C | 5 | 99 | 24 |
| 2 | TiO ₂ -P25-SO ₄ ²⁻ | EtOH, rt | 5 | 98 | 25 |
| 3 | polyaniline-sulfate salt | 1,2-dichloro ethane, rt | 20 | 95 | 26 |
| 4 | I ₂ | DMSO, rt | 35 | 95 | 27 |
| 5 | Zn[(L)proline] | HOAc, rt | 10 | 95 | 28 |
| 6 | cellulose sulfuric acid | EtOH, rt | 60 | 93 | 29 |
| 7 | montmorillonite K-10 | H ₂ O, rt | 150 | 100 | 30 |
| 8 | CuSO ₄ · 5H ₂ O | H ₂ O, rt | 15 | 96 | 31 |
| 9 | H ₆ P ₂ W ₁₈ O ₆₂ · 24H ₂ O | Acetic acid | 5 | 98 | 32 |
| 10 | silica-supported 1-(2-sulfooxy)ethyl)pyridine-1-ium chloride | EtOH, 65 °C | 20 | 95 | This work |

Conclusion

In this research study, we have successfully prepared a new and recoverable heterogeneous catalyst silica-supported 1-(2-sulfooxy)ethyl)1-pyridine-1-ium-chloride. This prepared catalyst

was explored as an efficient, green, versatile, and heterogeneous catalyst for the synthesis of quinoxaline. The reactions proceeded in EtOH as green solvent to afford the products in excellent yields. The attractive features of the present

research are high yields of the products, use of green solvents, simple work-up procedure, and easy recycling and reusability of the catalyst.

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Conflict of Interest

We have no conflicts of interest to disclose.

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