An Efficient Silica Supported Fluoroboric Acid Catalysed Synthesis of Quinoxaline Derivatives

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Abstract

Synthesis of some new quinoxaline derivatives (3a-p) from the reaction between substituted 1,2 diketone and substituted 1,2 diamino arene by using silica supported fluoroboric acid as an heterogeneous catalyst and PEG as a green solvent. Reaction route reported herein carries the attractive features like clean, mild, efficient acidic condition with short reaction time with quantitative yield of the product. Thus, this method flashes the combo of simple and efficient synthesis with easy isolation and purification of the desire product.

Keywords: Quinoxaline, 1,2 diketone, 1,2 diamine, HBF₄-SiO₂, PEG

Introduction

Quinoxalines are incredibly valuable intermediate for the improvement of biologically and pharmacologically interested molecule. Bezimidazole derivatives showed broad spectrum in the field of medicinal chemistry including antibacterial,¹ antiviral,² antifungal,³ antihyperglycemic,⁴ and anticancer.⁵ In addition quinoxaline derivatives have been evaluated as photoinitiators,⁶ electroluminescent,⁷ dyes⁸ and in luminescent studies.⁹ The well-known interest in the molecule containing benzimidazole scaffold has provoked widespread studies for their synthesis.

Due to the huge collection of applications, different synthetic roots have been developed for the synthesis of quinoxaline derivatives. It includes the reaction of 1,2-diamines and α-keto-oximes,¹⁰ the coupling of α-diazo ketones with aryl 1,2-diamines,¹¹ oxidative coupling of ene-1,2-diamines and epoxides,¹² reaction of aromatic 1,2-diamines with α-haloketones,¹³ reaction of diethyl bromomalonate with aryl-1,2-diamines,¹⁴ and reductive cyclization of 2-nitroanilines with 1,2-dicarboxyl,¹⁵ intramolecular cyclization of dialdimines,¹⁶ and oxidative cyclization of o-phenylenediamines with α-hydroxyketones.¹⁷

Number of oxidative reagents are used for the synthesis of quinoxaline like DDQ,¹⁸ Oxone,¹⁹ benzfuroxan,²⁰ MnO₂,²¹ benzoquinone,²² NaHSO₃,²³ Pb(OAc)₄,²⁴ tetracyanoethylene,²⁵ have been engaged. Although, day by day several synthetic route have been developed for the synthesis of quinoxalineby using variety of catalyst such as CuSO₄·5H₂O,²⁶ oxalic acid,²⁷ CAN,²⁸ KHSO₄,²⁹ sulfamic acid,³⁰ Yb(OTf)₃,³¹ Zn₂+–K10-clay,³² DABCO,³³ o-iodoxybenzoic acid,³⁴ HClO₄–SiO₂,³⁵ polyaniline-sulfate salt,³⁶ Zr(DS)₄³⁷. Along with this literature observation also flash on the solvent system used for the synthesis of quinoxalines.³⁸-⁴⁰ On the basis of above observed literature, there is a need to create a new mode for the synthesis of quinoxaline derivatives. Answer to this demand herein we report a new method.
for the synthesis of quinoxaline derivatives by employing HBF₄-SiO₂ as a heterogeneous catalyst (Scheme 1).

Scheme 1: Synthesis of 2,3-diphenylquinoxaline derivatives (3a-p).

Experimental

Materials and Methods

All the chemicals were used laboratory grade and purified previously to use. Melting points of all synthesized compounds were recorded in open capillary tubes and are uncorrected. IR spectra were recorded in KBr pellets on FTIR Shimadzu spectrophotometer, and ¹H NMR spectra in were scanned on an AVANCE 300 MHz spectrometer using DMSO and TMS as an internal standard. The MS were recorded on an El-Shimadzu-GC-MS spectrometer. Elemental analysis was carried out on a Carlo Ebra 106 Perkin-Elmer model 240 analyzer.

Preparation of Tetrafluoroboric Acid Adsorbed on Silica Gel (HBF₄-SiO₂)

The heterogeneous catalyst HBF₄-SiO₂ was prepared by the report reported method.⁴¹ A mixture of silica gel (26.7 g, 300–400 mesh) and 40% aq. HBF₄ (3.3 g, 8.25 mL, 15 mmol) in diethyl ether (75 ml) was stirred for 3 hrs. Concentrate the mixture and dried the residue under vacuum at 100 °C for 72 hrs to afford HBF₄-SiO₂ (0.5 mmol HBF₄/g) as a free-flowing power.

General procedure for the preparation of 2,3-diphenylquinoxaline derivatives (3a-p)

Equimolar mixture of 1,2 diketone 1 (0.1 mmol), 1,2 diamino arene (0.1 mmol) and HBF₄-SiO₂ (0.20 g, 0.1 mmol) in DMSO (10 ml) was stirred at 60-70 °C for an appropriate time (monitored by TLC). After completion of reaction, reaction mixture cooled at room temperature, poured in cold water and washed with CHCl₃. The obtained crude product was recrystallize by aq. Acetic Acid. Finally melting point of the final product was compared those of the authentic sample and found to be ideal. Further, by spectral characterization structural determination of the newly synthesized compounds was done.

| Entry | 1,2-diamine | 1,2-Diketone | Product | Time (min) | Yield (%) |
|-------|-------------|--------------|---------|------------|-----------|
| 3a    |             |              |         | 12         | 92        |

Table 1: The scope of various substituents for the synthesis of quinoxaline derivatives.
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|   | Structure 1 | Structure 2 |  |  
|---|---|---|---|---|
| 3b | ![Structure 3b](image1) | ![Structure 3b](image2) | 20 | 85 |
| 3c | ![Structure 3c](image3) | ![Structure 3c](image4) | 15 | 90 |
| 3d | ![Structure 3d](image5) | ![Structure 3d](image6) | 22 | 87 |
| 3e | ![Structure 3e](image7) | ![Structure 3e](image8) | 20 | 88 |
| 3f | ![Structure 3f](image9) | ![Structure 3f](image10) | 24 | 82 |
| 3g | ![Structure 3g](image11) | ![Structure 3g](image12) | 17 | 89 |
| 3h | ![Structure 3h](image13) | ![Structure 3h](image14) | 22 | 85 |
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Spectral data of newly synthesized compounds

2,3-diphenylquinoxaline (3a)

IR (KBr): $\nu$ 3138, 1686, 1535, 1481, 1372, 1298 cm$^{-1}$; $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.80 (d, 2H, $J = 8$ Hz), 7.67 (d, 2H, $J = 8$ Hz), 7.62-7.39 (m, 10H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 156.84, 141.39, 137.52, 129.64, 128.93, 127.25, 126.37, 125.88; MS: m/z 282 (M$^+$); Anal. calcd. for C$_{20}$H$_{14}$N$_2$: C, 85.08 (85.31); H, 5.00 (4.83); N, 9.92 (10.11).

2,3-bis(4-bromophenyl)quinoxaline (3b)

IR (KBr): $\nu$ 3093, 1665, 1556, 1542, 1440, 1385, 1291 cm$^{-1}$; $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.95-7.80 (m, 8H), 7.74-7.60 (m, 4H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 156.37, 142.61, 136.17, 132.40, 131.75, 130.89, 130.26, 129.37, 128.64, 120.53; MS: m/z 439 (M$^+$); Anal. calcd. for C$_{20}$H$_{12}$Br$_2$N$_2$: C, 54.58 (54.29); H, 2.75 (2.46); Br, 36.31 (36.72); N, 6.36 (6.81).

2,3-di-p-tolylquinoxaline (3c)

IR (KBr): $\nu$ 3084, 2970, 1666, 1572, 1519, 1450, 1337, 1284 cm$^{-1}$; $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.93-7.67 (m, 4H), 7.52 (d, 2H, $J = 10$ Hz), 7.48 (d, 2H, $J = 10$ Hz), 7.30 (d, 2H, $J = 8$ Hz), 2.11 (s, 6H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 154.34, 142.67, 135.97, 131.38, 130.22, 128.42, 126.84, 125.23, 124.06, 122.54; MS: m/z 310 (M$^+$); Anal. calcd. for C$_{22}$H$_{18}$N$_2$: C, 85.13 (85.6); H, 5.85 (5.24); N, 9.03 (9.78).

dibenzo[a,c]phenazine (3d)

IR (KBr): $\nu$ 3148, 1659, 1582, 1555, 1460, 1334, 1310 cm$^{-1}$; $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 8.92 (d, 2H, $J = 8$ Hz), 8.24 (d, 2H, $J = 10$ Hz), 7.88-7.48 (m, 8H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 143.52, 131.63, 130.71, 130.64, 129.27, 128.13, 126.37, 124.08, 123.40, 122.10; MS: m/z 280 (M$^+$); Anal. calcd. for C$_{20}$H$_{12}$N$_2$: C, 85.13 (85.6); H, 5.85 (5.24); N, 9.03 (9.78).

6-bromo-2,3-diphenylquinoxaline (3e)

IR (KBr): $\nu$ 3070, 1664, 1590, 1520, 1450, 1392, 1348 cm$^{-1}$; $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 8.32 (s, 1H), 7.92 (d, 1H, $J = 7.0$ Hz), 7.56 (d, 1H, $J = 7.0$ Hz), 7.50-7.36 (m, 4H), 7.30 (d, 2H, $J = 8.0$ Hz); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 143.52, 131.63, 130.71, 130.64, 129.27, 128.13, 126.37, 124.08, 123.40, 122.10; MS: m/z 280 (M$^+$); Anal. calcd. for C$_{20}$H$_{13}$BrN$_2$: C, 66.50 (66.27); H, 3.63 (3.42); Br, 22.12 (22.74); N, 7.75 (7.31).

6-bromo-2,3-bis(4-bromophenyl)quinoxaline (3f)

IR (KBr): $\nu$ 3132, 1678, 1548, 1563, 1434, 1390, 1286 cm$^{-1}$; $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.89-7.42 (m, 8H), 8.23 (s, 1H), 7.72 (d, 1H, $J = 7.0$ Hz), 7.42 (s, 1H, $J = 7$ Hz); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 159.76, 158.34, 143.95, 139.42, 135.28, 133.62, 132.58, 131.86, 130.83, 129.71, 122.39, 119.62; MS: m/z
517 (M⁺); Anal. calcd. for C₂₉H₁₁Br₃N₂: C, 46.28 (46.63); H, 2.14 (2.57); Br, 46.18 (46.35); N, 5.40 (5.11).

6-bromo-2,3-di-p-tolylquinoxaline (3g)

IR (KBr): v 3143, 2968, 1688, 1546, 1572, 1462, 1350, 1310 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 8.42 (s, 1H), 7.84 (d, 1H, J = 7 Hz), 7.56 (s, 1H, J = 7 Hz), 7.84-7.36 (m, 8H), 2.29 (s, 6H); ¹³C NMR (100 MHz, CDCl₃): δ 156.68, 156.48, 143.34, 140.96, 136.58, 134.33, 132.26, 131.72, 130.57, 120.64, 118.55, 20.82; MS: m/z 388 (M⁺); Anal. calcd. For C₂₂H₁₇BrN₂: C, 67.88 (67.25); H, 4.40 (4.73); Br, 20.53 (20.17); N, 7.20 (7.63).

11-bromodibenzo[a,c]phenazine (3h)

IR (KBr): v 3137, 1642, 1568, 1538, 1472, 1364, 1288 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 8.90 (d, 2H, J = 10 Hz), 8.84 (d, 2H, J = 10 Hz), 8.76 (d, 2H, J = 12 Hz), 8.23 (s, 1H), 7.88 (d, 2H, J = 12 Hz), 7.85 (d, 1H, J = 8 Hz), 7.56 (d, 1H, J = 8 Hz); ¹³C NMR (100 MHz, CDCl₃): δ 142.67, 140.71, 133.64, 132.25, 131.63, 130.83, 129.38, 128.76, 128.07, 127.46, 122.15, 118.68; MS: m/z 358 (M⁺); Anal. calcd. For C₂₉H₁₁BrN₂: C, 66.87 (66.42); H, 3.09 (3.34); Br, 22.24 (22.73); N, 7.80 (7.28).

6-nitro-2,3-diphenylquinoxaline (3i)

IR (KBr): v 3061, 1660, 1594, 1518, 1448, 1398, 1341 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 8.63 (d, 1H, J = 8 Hz), 8.59 (d, 1H, J = 8 Hz), 7.95 (d, 2H, J = 10 Hz), 7.82 (d, 2H, J = 10), 7.68 (s, 1H), 7.43-7.30 (m, 6H); ¹³C NMR (100 MHz, CDCl₃): δ 160.32, 157.66, 144.85, 138.27, 130.83, 130.08, 129.62, 128.96, 128.26, 127.54, 124.84, 122.30; MS: m/z 327 (M⁺); Anal. calcd. For C₂₀H₁₃O₂N₃: C, 73.38 (73.56); H, 4.00 (4.26); N, 12.84 (12.64); O, 9.78 (9.32).

2,3-bis(4-bromophenyl)-6-nitroquinoxaline (3j)

IR (KBr): v 3048, 1643, 1551, 1536, 1412, 1416, 1309 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 8.79 (d, 1H, J = 8 Hz), 8.67 (d, 1H, J = 8 Hz), 8.29-7.63 (m, 8H), 7.86 (s, 1H); ¹³C NMR (100 MHz, CDCl₃): δ 157.21, 155.73, 140.15, 136.36, 130.12, 129.78, 129.32, 128.48, 128.05, 127.28, 123.51, 121.73; MS: m/z 485 (M⁺); Anal. calcd. For C₂₀H₁₁Br₂O₂N₃: C, 49.52 (49.23); H, 2.29 (2.61); Br, 32.94 (32.72); N, 8.66 (8.34); O, 6.60 (6.16).

6-nitro-2,3-di-p-tolylquinoxaline (3k)

IR (KBr): v 3048, 2983, 1656, 1576, 1536, 1433, 1382, 1269 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 8.66 (d, 1H, J = 8 Hz), 8.56 (d, 1H, J = 8 Hz), 7.98 (d, 2H, J = 10 Hz), 7.82 (d, 2H, J = 10 Hz), 7.52 (s, 1H), 2.34 (s, 6H); ¹³C NMR (100 MHz, CDCl₃): δ 160.86, 158.38, 141.56, 140.29, 137.26, 133.67, 130.74, 129.46, 128.59, 127.13, 121.73, 24.10; MS: m/z 355 (M⁺); Anal. calcd. For C₂₀H₁₃O₂N₃: C, 74.35 (74.66); H, 4.82 (4.53); N, 11.82 (11.42); O, 9.00 (9.37).

11-nitrodibenzo[a,c]phenazine (3l)

IR (KBr): v 3078, 1642, 1581, 1517, 1462, 1370, 1288 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 8.72 (d, 1H, J = 8 Hz), 8.65 (d, 1H, J = 8 Hz), 7.99 (d, 4H, J = 10 Hz), 7.83 (d, 4H, J = 10 Hz), 7.66 (s, 1H); ¹³C NMR (100 MHz, CDCl₃): δ 148.39, 142.74, 140.80, 131.25, 130.86, 130.15, 129.06, 128.86, 127.47, 126.68, 121.73, 120.96; MS: m/z 325 (M⁺); Anal. calcd. For C₂₀H₁₁O₂N₃: C, 73.84 (73.63); H, 3.41 (3.82); N, 12.92 (12.45); O, 9.84 (9.37d).
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2,3-diphenylpyrido[2,3-b]pyrazine (3m)
IR (KBr): ν 3037, 1676, 1590, 1478, 1386, 1265 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 8.84 (d, 1H, J = 10 Hz), 7.81 (d, 1H, J = 10 Hz), 7.59-7.38 (m, 10H), 7.33 (d, 1H, J = 10 Hz); ¹³C NMR (100 MHz, CDCl₃): δ 156.22, 154.73, 150.48, 148.84, 140.62, 138.56, 132.16, 130.74, 129.34, 128.08, 127.47, 126.19; MS: m/z 283 (M⁺); Anal. calcd. For C₁₉H₁₃N₃: C, 80.54 (80.37); H, 4.62 (4.57); N, 14.83 (14.38).

2,3-bis(4-bromophenyl)pyrido[2,3-b]pyrazine (3n)
IR (KBr): ν 3072, 1659, 1562, 1550, 1448, 1364, 1280 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 8.67 (d, 1H, J = 10 Hz), 8.54 (d, 1H, J = 10 Hz), 7.86 (d, 2H, J = 8 Hz), 7.68 (d, 2H, J = 8 Hz), 7.48 (m, 4H), 7.36 (s, 1H); ¹³C NMR (100 MHz, CDCl₃): δ 158.87, 156.98, 152.67, 150.52, 146.74, 136.14, 132.82, 131.43, 130.74, 128.08, 126.27, 124.33; MS: m/z 440 (M⁺); Anal. calcd. For C₁₉H₁₁Br₂N₃: C, 51.73 (51.29); H, 2.51 (2.72); Br, 36.23 (36.18).

2,3-di-p-tolylpyrido[2,3-b]pyrazine (3o)
IR (KBr): ν 3060, 2997, 1683, 1540, 1571, 1466, 1352, 1244 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 8.78 (d, 1H, J = 10 Hz), 8.63 (d, 1H, J = 10 Hz), 7.78 (d, 2H, J = 8 Hz), 7.57 (d, 2H, J = 8 Hz), 7.38 (m, 4H), 7.21 (d, 1H, J = 10 Hz), 2.38 (s, 6H); ¹³C NMR (100 MHz, CDCl₃): δ 154.52, 152.71, 152.67, 145.34, 135.62, 133.08, 132.84, 130.37, 128.19, 127.46, 125.38, 22.44; MS: m/z 311 (M⁺); Anal. calcd. For C₂₁H₁₇N₃: C, 81.00 (81.26); H, 5.50 (5.67); N, 13.49 (13.84).

dibenzo[f,h]pyrido[2,3-b]quinoxaline (3p)
IR (KBr): ν 3127, 1653, 1568, 1582, 1494, 1381, 1268 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 8.87 (d, 2H, J = 8 Hz), 8.71 (d, 1H, J = 10 Hz), 8.41 (d, 2H, J = 8 Hz), 7.88 (d, 2H, J = 8 Hz), 7.80 (d, 2H, J = 8 Hz), 7.43 (d, 1H, J = 10 Hz), 7.20 (d, 1H, J = 10 Hz); ¹³C NMR (100 MHz, CDCl₃): δ 144.75, 142.38, 130.18, 129.62, 128.57, 128.23, 127.22, 126.85, 126.13, 125.78, 124.30, 123.67; MS: m/z 281 (M⁺); Anal. calcd. For C₁₉H₁₁N₃: C, 81.12 (81.67); H, 3.94 (3.48); N, 14.94 (14.52).

Results and Discussion

The route adopted for the synthesis of the title compound was flashed in the scheme 1. By using the method reported elsewhere in the literature the heterogeneous catalyst HBF₄-SiO₂ was prepared and used in the above scheme. The structures of compounds (3a-p) were confirmed by the IR, ¹H NMR and Mass spectral analysis. For the quantitative yield of the final product (3a-p), the model reaction was observed in different criterion. Optimization of the synthetic rout was arranged in order to achieve the Green Chemistry Principles. On that way firstly we try to carry out the same synthesis without catalyst and without solvent. Here we find out that the reaction cannot be proceeding at RT as well as in higher temperature also. But by increasing temperature and introducing the heterogeneous catalyst HBF₄-SiO₂, the % yield of the newly synthesized compounds is increased (Entry 6, Table 1).

Table 2: Optimization of reaction in different reaction condition.

| Entry | Catalyst | Temp (°C) | Time (min)ᵃ | Yield (%)ᵇ |
|-------|----------|-----------|-------------|------------|
| 1     | No Catalyst | RT        | >120        | 0          |
| 2     | No Catalyst | 120       | >120        | 0          |
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Table 2: Influence of the solvent on the reaction.

| Entry | Solvent | Time(min) a | Yield(%) b |
|-------|---------|-------------|------------|
| 1     | Water   | >60         | 12         |
| 2     | EtOH    | >30         | 74         |
| 3     | CH₃CN   | >30         | 58         |
| 4     | DMF     | >30         | 65         |
| 5     | PEG-400 | 12          | 92         |

a Reaction progress monitored by thin-layer chromatography (TLC)
b Yields refer to isolated yield

Conclusion

In summary, by using different substituted 1,2-diketones and 1,2-diamine here we report a new synthetic route for the synthesis of 2,3-diphenyl quinoxaline derivatives. The reported methods incorporate the provisions which are the building blocks of the Green Chemistry, like simple, efficient, eco-friendly method. Among these, the ambient condition is also favorable for the high reaction rate with excellent product yield. With the use of heterogeneous catalyst herein we use PEG-400 as a green solvent which focus the reaction route as a green and safe process and achieve both economic and environmental advantages and also explore in feature for the different organic synthesis.

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