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STUDY OF RELATIVE EFFICIENCY OF MAGNETICALLY ACTIVE BRONSTED ACID WITH LEWIS ACID CATALYST FOR QUINOXALINE SYNTHESIS UNDER IDENTICAL ENVIRONMENT

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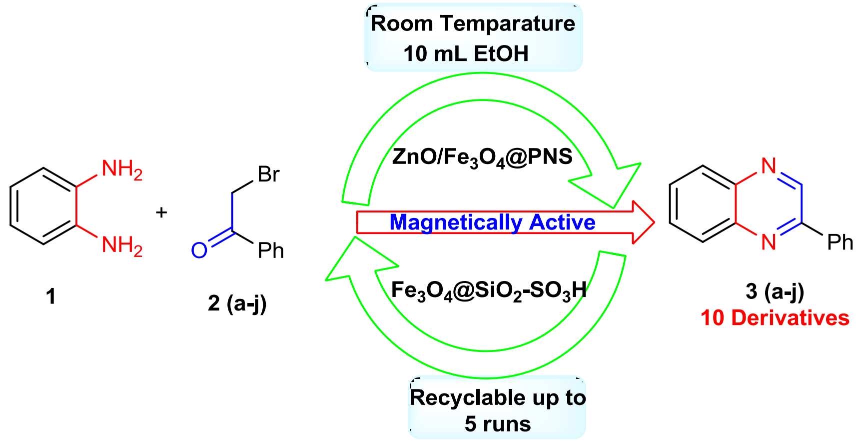
ABSTRACT

A comparative study of catalytic efficiency of magnetically active Bronsted acid ($Fe_3O_4@SiO_2-SO_3H$) and Lewis acid ($ZnO/Fe_3O_4@PNS$) has been accomplished for synthesis of quinoxaline. Quinoxalines were synthesized via cyclo-condensation of *o*-phenylene diamine with phenacyl bromide at room temperature in presence of 10 wt.% catalyst. Both the catalyst showed analogous results under identical condition. The magnetically active, solid supported catalyst with Bronsted acid cites or Lewis acid cites offered near about same efficiency for present transformation. The methodology boasts a hassle-free catalyst preparation and separation process, operates under energy-conserving conditions, yields a commendable (96-86%) output of the targeted moieties, and exhibits catalyst recyclability. These appealing features position it as a superior and eco-friendly alternative to traditional methods for synthesizing quinoxalines.

KEYWORDS: Diamine; Phenacyl bromide; Quinoxaline; $ZnO/Fe_3O_4@PNS$; $Fe_3O_4@SiO_2-SO_3H$; Room temperature; magnetically active.

GRAPHICAL ABSTRACT



1. INTRODUCTION

Quinoxalines are a versatile class of nitrogen containing heterocyclic compounds and they constitute useful intermediates in organic synthesis and medicinal chemistry.[1] It is widely recognized that a broad spectrum of synthetic compounds, natural products, agrochemicals, and medicinal medications. Considering the varied array of heterocyclic units, quinoxalines stand out as a pivotal scaffold found in a multitude of biologically and pharmaceutically significant compounds.[2,3,4] Quinoxaline derivativeshavebroad spectrum of therapeutical activities similar to antiviral,[5] antibacterial and antifungal,[6] antioxidant and antiproliferative,[7] antineoplastic,[8] antitumor,[9-11] antitubercular agent,[12,13] antimicrobial,[14] and anticancer.[15,16] These hetero moietieshave beenalso used in electroluminescent devices[17] and organic semiconductors.[18]

Owing to such diverse applications, synthesis of quinoxaline has grabbedattention of researchers. Several synthetic routes for quinoxalines have been established. Primarily, quinoxalines were preparedthroughcyclo-condensation of 1,2-diaminobenzene with several organic moieties such as 1,2-diketones,[2, 19] α-haloketone,[20] α-ketoesters or α-ketoacids,[21] α-hydroxy ketone,[22] vicinal diols[23], dimethyl and diethyl acetylene dicarboxylate,[24] epoxides,[25] etc.

Among these approaches, condensation of 1,2-diaminobenzene with phenacyl bromide is the most attractive and frequently used root. This synthetic approachof quinoxaline has included numbers of catalysts like $HClO_4$-SiO_2,[26] silica-supported dodecatungstophosphoric acid,[27] TMSCl,[28] β-cyclodextrin,[29] silica-supported phosphomolybdic acid,[30] miceller SDS,[31]

T_3PDMSO or T_3P,[32] N-Bromosuccinimide[33] and ionic liquid[34] etc. Addition to these, catalyst-free approach[35] has been reported earlier.

On the other hand, among these methods severalcatalytic process were comes with one or more limitationsresemble torough reaction parameters, inadequate yields, required toxic and expensive catalysts or components, use of polar solvent leading to tedious work-up processes. To overawed such shortcomings, researchers diverted towards eco-friendly reactions, use of greener solvents or applying efficient and easy available reactants and catalysts, process under mild circumstances. Accordingly, the invention of resourceful and eco-friendly approach for the production of quinoxalinesis quiet exceedingly preferred. Recently, solid-supported reagents, magnetically active catalyst, metal oxide nano-catalysts and eco-friendly approaches such as room temperature protocol, use of aqueous medium have been developed by many researchers and exploited them in plenty of organic transformations.[36-40] They have significant contribution in organic synthesis owing to their notable benefits such as high surface area, outstanding efficiency, stability, recyclability, nontoxic and effortlessness preparation and safe handling.

Owing to this, recently we have reported Fe_3O_4@SiO_2-SO_3H and polyphosphoric acid functionalized silica (SiO_2@PPA) for quinoxalines synthesis.[2, 41] We have extended our work in the direction of design the efficient protocols for the production of organic moieties using magnetically active and heterogeneous catalyst.[42-45] Herein, we have demonstrated catalytic evaluation of magnetically active silica sulfuric acid (Fe_3O_4@SiO_2-SO_3H) and pea nut shell supported ZnO nano-catalyst (ZnO/Fe_3O_4@PNS) and compared efficiency for the quinoxaline production. The catalytic efficiency for quinoxalines synthesis from diamine and phenacyl bromide under room temperature. Previously, we have prepared Fe_3O_4@SiO_2-SO_3H nano-material and estimated catalytic efficiency for quinoxalines,[2] in present report we have carried out comparative study between magnetically active Bronsted acid with Lewis acid and obtained results were systematically evacuated. As per my best knowledge, described synthesis and comparative study has not reported earlier and this will give new direction to the researchers for catalyst selection.

2. Experimental Section

2.1. Reagents and chemicals

Peanut shell (PNS), zinc acetate, ferrous chloride, ferric chloride, silica powder (70-230 mesh) and H_2SO_4 were procured from Sigma Aldrich. All reactants and solvents ($CHCl_3$, ethyl acetate, ethanol, double distilled water) required for quinoxalines were sourced from a commercial chemical supplier and employed without recrystallization and distillation.

2.2. Preparation of Fe_3O_4@SiO_2-SO_3H Nanoparticles

We have prepared catalyst followed by same procedure reported in our previous work.[2] We have used prepared catalyst for quinoxaline production.

2.3. Preparation of Fe_3O_4@PNS.

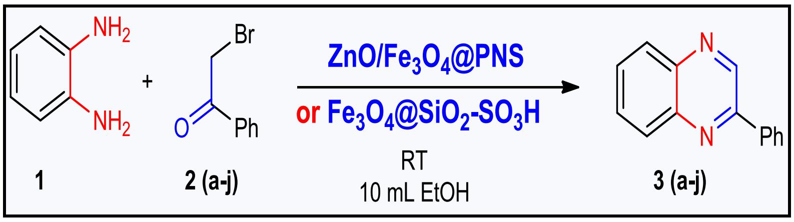
5 gm of peanut shell (PNS) is added in 250 mL beaker with $FeCl_3$ $6H_2O$ (6.66 gm) and $FeCl_2$ $4H_2O$ (3.66gm) in 30 mL distilled water. Obtained mixture was heated at 85 ^oC for 1 h then add 1N NaOH (20 mL) gradually with nonstop stirring for another 1 h. Resulted solid was detached and washed with water, ethanol and acetone (10 mL x 2) then dried it at 70 ^oC in oven for 1 h.

2.4. Preparation of ZnO/Fe_3O_4@PNS.

1 gm of product obtained in first step and zinc acetate (10 mmole) was stirred in 20 mL distilled water at room temperature still to formation of precipitation. The p^H of reaction vessels was kept constant at 10 with addition of NH_4OH. Resulted solid was filtered and washed with distilled water for several times and then dried for 24 h at 70 ^oC.

2.5. The general procedure for quinoxalines synthesis

In a 100 mL round bottom flask, diamine (1mmol) and phenacyl bromide (1 mmol) were combined in 10 mL EtOH in presence of 10 wt.% ZnO/Fe_3O_4@PNS **or** Fe_3O_4@SiO_2-SO_3H, was magnetically stirred at room temperature. Reaction progress was monitored by TLC, and upon completion, the vessel was extracted with ethyl acetate. The catalyst was separated through simply using external strong magnet, catalyst washed thrice with 10 mL of ethyl acetate each time, and the combined washings were added to the ethyl acetate extract. Evaporation under vacuum on a rotary evaporator yielded the crude solid product, which was purified into a pure quinoxaline derivative through recrystallization and column chromatography.



***Scheme 1: Synthesis of quinoxalines at room temperature using magnetically active nano-catalyst*.**

2.6. Spectral analysis of some reprehensive compounds[26-35]

2-Phenyl quinoxaline (3a): IR (KBr, cm^{-1}): 718, 810, 960, 1395, 1530, 1630, 1705, 1922, 2830, 2921; ^1H NMR (300 MHz, $CDCl_3$, δ ppm): 7.50 (s, 3H), 7.68 (m, 2H), 8.17 (m, 4H), 9.38 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$, δ ppm): 125.8, 129.0, 129.5, 129.9, 130.0, 130.2, 130.7, 137.1, 141.1, 142.9, 143.6; MS (m/z) = 207 [M+1]+.

2-(4-Methylphenyl)quinoxaline (3b):IR (KBr, cm^{-1}): 706, 814, 948, 1417, 1533, 1668, 1722, 1930, 2846, 2916; ^1H NMR ($CDCl_3$, δ ppm): 2.53 (s, 3H), 7.34–7.39 (d, 2H), 8.04–8.10 (d, 4H), 9.35 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$, δ ppm): 20.1, 126.1, 128.4, 128.9, 129.6, 129.9, 130.5, 139.3, 142.1, 144.0.

2-(4-Methoxyphenyl) quinoxaline (3c):IR (KBr, cm^{-1}): 744, 834, 944, 1169, 1418, 1449, 1476, 1528, 1593, 2931; ^1H NMR ($CDCl_3$, δ ppm): 3.84 (s, 3H), 6.95–7.04 (d, 2H), 7.58–7.68 (m, 2H), 8.00–8.07 (m, 3H), 8.20–8.26 (d,2H), 9.21 (s, 1H); ^{13}C NMR (75 MHz,$CDCl_3$, δ ppm): 27.1, 53.7, 113.2, 127.6, 128.2, 128.9, 129.3, 130.7, 142.2, 160.3; MS (ESI)+: m/z = 237 $[M+1]^+$.

2-(4-Bromophenyl)-7-methylquinoxaline (3i): IR (KBr, cm^{-1}): 704, 818, 942, 1417, 1532, 1669, 1721, 1929, 2847, 2931; ^1H NMR ($CDCl_3$, δ ppm): 2.54 (s, 3H), 6.74 (d, 2H), 7.66–7.69 (m, 2H), 8.00–8.03 (m, 2H), 8.22–8.29 (m, 2H), 9.24 (s, 1H); ^{13}C NMR ($CDCl_3$, δ ppm): 19.8, 127.4, 128.9, 129.3, 129.6, 130.2, 131.9, 139.4, 140.6, 144.1; MS (m/z) = 298 $[M+1]^+$.

2-(Biphenyl-4-yl) quinoxaline (3j): IR (KBr, cm^{-1}): 714, 832, 906, 944, 1116, 1294, 1409, 1524, 1669, 1717, 2931; ^1H NMR ($CDCl_3$, δ ppm): 7.31–7.34 (d, 1H), 7.38–7.46 (t, 2H), 7.58–7.64 (d, 2H), 7.74–7.83 (m, 4H), 8.06–8.13 (t, 2H), 8.24–8.30 (d, 2H), 9.29 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$, δ ppm): 123.0, 126.5, 127.9, 128.6, 129.1, 129.7, 132.4, 133.6, 140.5, 141.7, 144.2.

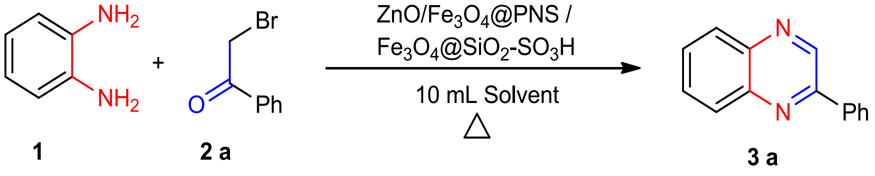
3. RESULT AND DISCUSSION

We have planned to evaluate catalytic efficiency of magnetically active silica sulfuric acid and magnetically active ZnO supported PNS. Our research started with preparation of catalyst, it was synthesized followed by procedure reported in literature. After the successful synthesis of catalyst, we havecompared the catalytic efficiency of both the catalystfor the quinoxalines production. In previous report, we have outlined the optimized parameter for quinoxaline synthesis in presence of Bronsted acid (Fe_3O_4@SiO_2-SO_3H)catalyst from diamine and diketones. The appropriate solvent, effect of catalyst loading and reaction temperature was well evaluated for quinoxaline synthesis. To compared efficiency, both catalysts evaluated under identical atmosphere.

Before the use of optimized conditions, that was verified for ZnO/Fe_3O_4@PNS and Fe_3O_4@SiO_2-$SO_3H$with the model reaction as ortho-phenyldiamine (1 mmol) stirred with phenacyl bromide (1 mmol) and catalyst in 10 mL solvent. We have evaluated all these parameters and observedoutcomes are displayed in table 1. Primarily, we have checked the effect of solventon the model reaction conducted in presence of 10 wt.% catalyst at room temperature. We have demonstrated reaction in varying solvents like ethanol, methanol, DMSO, DCM and water. The obtained results indicated that ethanol has been appropriate solvent for the planned reaction. It reported85 % to 87% yield of quinoxaline within 2 h (Table 1, entry 1) stirring at room temperature, while other solvents like DMSO, DCM, methanol and water (Table 1, entries 1-5) were failed to compete the result of ethanol under prescribed reaction parameter. Furthermore, to study the role of temperature we have carried out same reaction at varying temperature from room temperature to 40 ^{o}C, 60 ^{o}C and 80 ^{0}C. Entries 6 to 8 showed that increasing the temperature up to 40 ^{o}C and 60 ^{o}C was suitable for current transformation while negative impact on outcome of reaction were observed on rising the temperature at 80 ^{0}C. It may due to the decomposition of reactants or evaporation of solvent at higher temperature. By means of energy economy, we preferred to carry out reaction at room temperature rather than at 40 ^{o}C or 60 ^{o}C.

Meanwhile, the amount of catalyst is also an important phase to outline optimized conditions and achieve most appropriate scheme for quinoxaline synthesis from diamine and phenacyl bromide at room temperature. The proposed reaction was accompanied with loading of altering amount of ZnO/Fe_3O_4@PNS **or** Fe_3O_4@SiO_2-SO_3H catalyst. Results expressed that the lower amounts like 7.5 wt. % and 5 wt. % (Table 1, entries 11 and 12) of catalyst was insufficient to produce remarkable yield of target molecule while higher amount as 12.5 wt. % of catalysthas not displayed enhancement in outcome of studied transformation (Table 1, entries 9). Similarly, further addition in catalytic amount (15 wt. %) lead to negative impact that produced only 76-75 % yield (Table 1, entry 10). After the investigation of these factors, we finalizedas 10 wt. % was appropriate amount of catalystto produce outstanding yieldof quinoxaline, via 2 h stirring of reactants in 10 mL ethanolat room temperature.

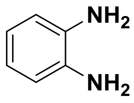
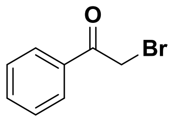
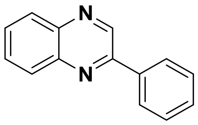
Table 1: Optimization of reaction parameters for the synthesis of quinoxaline using ZnO/Fe_3O_4@PNS and Fe_3O_4@SiO_2-SO_3H.

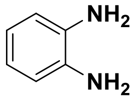
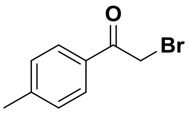
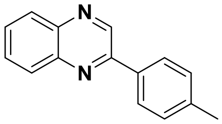
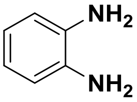
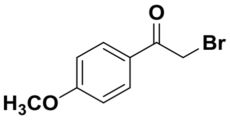
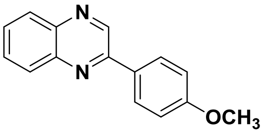
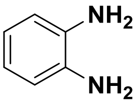
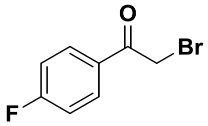
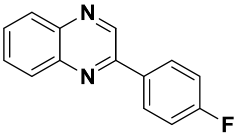
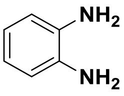
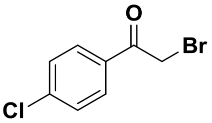
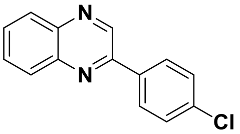
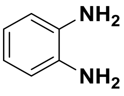
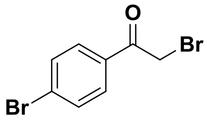
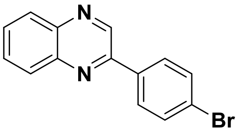
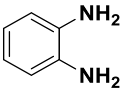
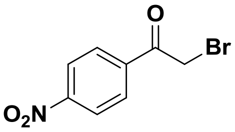
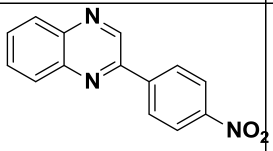
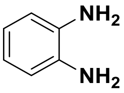
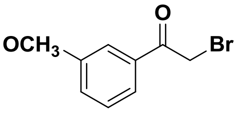
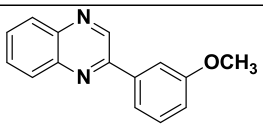
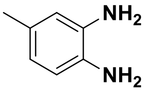
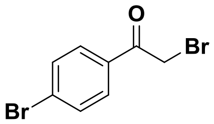
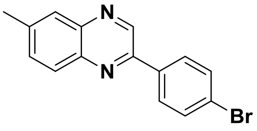
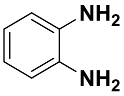
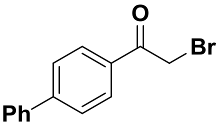
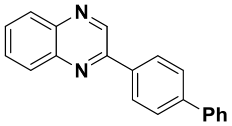


Entry	Catalyst amount	Solvent (10 mL)	Temperature (°C)	Reaction Time$^{(b)}$ (h)	Yield$^{(c)}$ (%)	
					ZnO/Fe_3O_4@PNS	Fe_3O_4@SiO_2-SO_3H
1	10 wt.%	Ethanol	RT	2	85	87
2	10 wt.%	DMSO	RT	5	78	80
3	10 wt.%	DCM	RT	5	72	70
4	10 wt.%	Methanol	RT	4	80	80
5	10 wt.%	Water	RT	10	45	57
6	10 wt.%	Ethanol	40	2	88	88
7	10 wt.%	Ethanol	60	2	85	86
8	10 wt.%	Ethanol	80	2	75	76
9	12.5 wt.%	Ethanol	RT	2	85	88
10	15 wt.%	Ethanol	RT	2	76	75
11	7.5 wt.%	Ethanol	RT	4	62	66
12	5 wt.%	Ethanol	RT	6	48	55

[a]Reaction conditions: Diamine (1 mmol), phenacyl bromide (1 mmol), solvent 10 mL.
[b]monitored by TLC; [c]Isolated yield.

Table 2: Synthesis of series of quinoxaline and comparative yield for used catalysts.

En.	1,2 diamine	α-bromoketone	Product	Time (hr)	Yield (%)		Melting Point	
					ZnO/ Fe_3O_4 @PNS	Fe_3O_4@ SiO_2- SO_3H	Obs.	Rep. [26-35]
3a				2	85	87	76-78	75-78

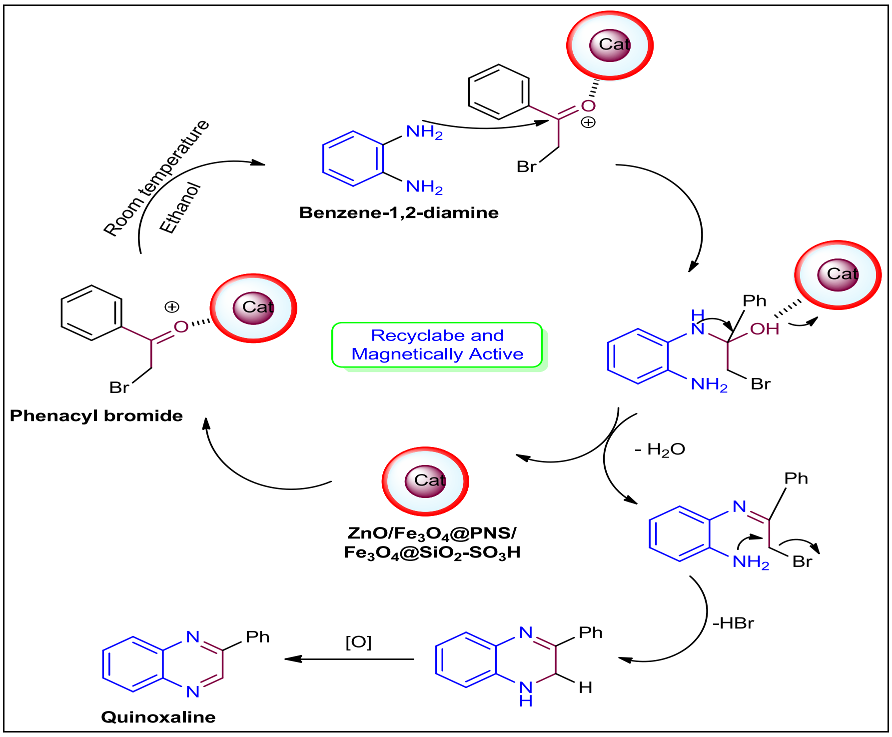
3b				2.5	82	83	86-90	84-90
3c				2.5	80	82	94-97	94-98
3d				1	89	91	113-116	112-118
3e				1	87	90	129-130	128-130
3f				1.5	86	88	128-130	128-131
3g				1.5	90	90	190-191	190-191
3h				2.5	86	88	88-90	87-88
3i				2	83	87	127-129	126-129
3j				2.5	78	83	75-76	73-76

[a]Reaction conditions: Diamine (1 mmol), phenacyl bromide (1 mmol), ethanol 10 mL, Catalyst (10 wt.%). [b]monitored by TLC; [c]Isolated yield.

Having the finest conditions for the model reaction, we examined the scope using different substrates and synthesised ten derivatives of quinoxaline, the results are shown in table 2. The substituted phenacyl bromidewere reacted with ortho phenyldiamine at room temperature, same reactions were carried out in presence of Bronsted acid (Fe_3O_4@SiO_2-SO_3H) and Lewis acid (ZnO/Fe_3O_4@PNS).We found that the procedure has been applied to quinoxalines, which offered uniformly very good to excellent yield of target molecule. A variety of structural types and functional groups were well-tolerated. Electron donating and electron withdrawing moieties were accelerated to the reaction and conserved throughout the reaction. All synthesized compounds are reported. We hope designed protocol may be quite fit to large production of quinoxaline derivatives. The obtained results exhibited that both strategies were quiet fit for quinoxaline production, the type of acid doesn't altered the conspicuous results. Magnetically active supported nano-catalyst, either Bronsted acid or Lewis acid were offered eco-friendly and efficient approach for quinoxalines production. The structural conformation of prepared quinoxalines was done through spectral analysis (IR, NMR, Mass) and analytical data thatover and done viacomparedwith literature data.[26-35]

3.1. Probable mechanism for the quinoxaline synthesis

The catalytic action can be illustrated through mechanism. Herein, we dispatched the probable mechanism for quinoxaline production attending with Bronsted acid ($Fe_3O_4@SiO_2$-SO_3H) or Lewis acid ($ZnO/Fe_3O_4@PNS$) catalyst in scheme 2. The reaction was expected to follow the acid-catalyzed condensation reaction and corresponding to mechanism reported in literature.[46] From this, we clearly concluded that, reported catalyst has played vial role in activation of carbonyl groups of bromoketone, which fascinated attack of diamine, again catalyst supported to dehydration reaction and dehalogenation simultaneously resulting in the formation of cyclic product. The final product was obtained via air oxidation.



Scheme 2: Possible mechanism for quinoxaline synthesis using **$ZnO/Fe_3O_4@PNS$ or $Fe_3O_4@SiO_2$-SO_3H catalyst.**

3.2. Recyclability test of ZnO/Fe_3O_4@PNS catalyst

To study the recyclability of prepared ZnO/Fe_3O_4@PNS catalyst, we have demonstrated model reaction attending optimized parameters. After the successful conversion, the ZnO/Fe_3O_4@PNS was isolated via using external magnet. Recovered catalyst was washed with water and dried at 70 °C for 5 h in vacuum oven. The dried ZnO/Fe_3O_4@PNS was reused for next run of model reaction, this practice has been continued up to the noticeable defeat in catalytic activity. We found that ZnO/Fe_3O_4@PNS has effectively catalyzed to the current transformation up to fifth cycle as shown in Figure 1. From this exercise, we concluded as the investigated catalyst was capable to produce notable results without any alternation in product and stability of catalyst up to successive five cycles.

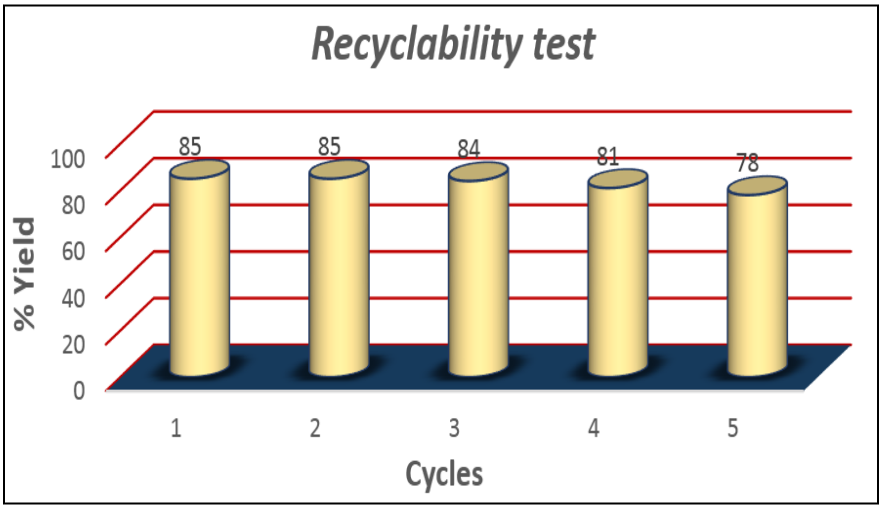


Figure 1: Recyclability of ZnO/Fe_3O_4@PNS catalyst for quinoxaline synthesis.

4. CONCLUSION

In this study, we present an efficient pathway for synthesizing quinoxalines through the cyclo-condensation reaction of diamine and phenacyl bromide derivatives at room temperature. The reaction was successfully catalyzed by 10 wt. % ZnO/Fe_3O_4@PNS as well as Fe_3O_4@SiO_2-SO_3H, demonstrating remarkable yields of quinoxalines under optimized conditions. The prepared catalyst exhibited notable catalytic activity at room temperature, maintaining its efficiency for up to the fifth run. We have evaluated comparative study of effectiveness of both catalysts under identical conditions and parallel results were obtained. The type of catalyst as Bronsted acid (Fe_3O_4@SiO_2-SO_3H) or Lewis acid (ZnO/Fe_3O_4@PNS) cannot much differ the outcomes of present transformation. Additionally, both catalysts offered several advantages, including its heterogeneous nature, straightforward workup process, and operability even at room temperature. Consequently, we propose an effective, high-yield protocol that can be considered as a greener alternative to traditional approaches.

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