

One pot MCR for the 9-phenyl-1,2,3,4,5,6,7,8,9,10-decahydroacridine derivatives for good yield via Silica supported green catalyst and solvent free condition

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Abstract: Organic synthesis is the very useful for the modification of the drug discovery and its applicable for the modify for the change the route of drug discovery as well as pharmacology study, the current situation of the environment is very harsh and environmentally friendly sources are costly then the tradition method of synthesis, so our worked is focus on designing the environment as well as economically viable route of synthesis with the help of some novel silica supported metal catalyst. in this type catalyst design from collected solid harmful environment metal waste of aluminum foil. our team is collected aluminum waste from food sellers and then wash with water aluminum converted to aluminum hydroxide. they use for the formulation of gel formation of medicine. and its not harmful like aluminum metal, as per survey aluminum is the second highest metal waste on the earth.

Keywords: Dimidone, Solvent free, green catalyst, high reusability.

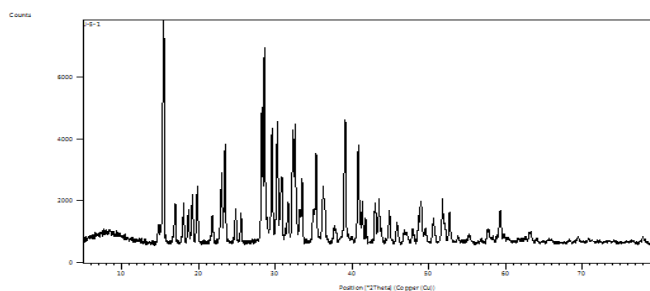
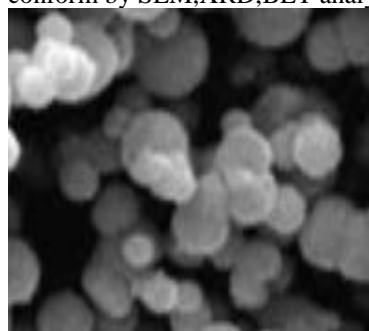
1. INTRODUCTION

This catalyst are design for the homogeneous catalyst for the differ path for the new heterogeneous catalyst. Aluminum hydroxide is the base its docking on the porous silica. for the increase the activity of the catalyst with lowest amount of the catalyst. the nano particle, Green catalyst and increase the reusability or activity of catalyst is the immerging field for the new catalyst design and change the reaction route for the deployment in the synthesis field, so our team is focused on this thing, our goal is the docking molecule on silica, green catalyst and its reusability is almost double then other nano particle or docking material they used as catalyst in previous work. design the catalyst motivated from Nasser H et al, they sodium aluminum silicates for the abrogation of dyes from environment, and Ahemad nakhaei et al

they are introduce the heterogeneous catalyst for the 1,4 dihydropyridine Hantzsch reaction for modification study, Aluminum, ferrous, Titanium like metals nano particle or its compositions are used as a catalyst in reactions, they required 30 to 460 min for the ending of reaction. and they reusability is approximately 5 cycles they are gave good yield after the 5 cycles they continue reduced its yield. they are gave not effective yield in case silica supported results but our team are introduce successful silica supported aluminum hydroxide have good activity and reusability for this type reactions.

Material and method:

Silica supported $\text{Al}(\text{OH})_3$: First collect the waste aluminum foil from food court then wash with water and remove all stacked material of food, then cuts in small pieces after cutting the dissolve in waste HCl they HCl is by product of pharmaceutical company, the addition of aluminum pieces is slowly in HCl at the time of the addition the evaluation of hydrogen gas and rise the temperature and then the cool it, after the cool the addition of Sodium hydroxide pallet and set Ph=2.0 to 7.0 and above and white precipitation is formed, its filter by vacuum filter and dry in hot air oven for 90°C for 2 hour after the drying the make the slurry of Silica and the addition of aluminum hydroxide in slurry and starring for 2 hours after the remove the solvent from slurry the dry and kept in muffle furnace for 1 hour 300°C after this the fine powder is ready they used as a green catalyst, it's conform by SEM, XRD, BET analysis.



Experimental section: 2.5 mmole aldehyde ,5 mmole cyclohexane-1,3-dione with 1.5 mmole Ammonium acetate mix well then addition of catalyst in variety of amount with different temp e and check the efficiency of catalyst .monitor the reaction on TLC plate after the completion of reaction the all reaction mass dissolve in suitable solvent like MeOH,EtOH at the time of purification process all product are dissolve in solvent then filter it and collect the catalyst and wash with EtoH and use for the next cycle.

Result and discussion: this synthesis is focused on the green as well as novel silica supported catalyst for the economically viable synthesis route for the deployment of new thing in catalysis. the good yield ,high reusability, so, our team are waste aluminum foil converted to efficient and capable green catalyst for the env-eco catalyst. they are gave the same efficiency other catalyst like SbCl₃,HPA1,HPA2,LGPCO, the check appropriate time, temperature and amount of the catalyst for this reaction, we conclude that the solvent free condition and the various solvent are use for the reaction at variety of temperature for the optimization of reactions condition for the good yield, and after the all solvent .

Table:01 comparative study with previously reported catalyst

Sr	Amount of catalyst	Solvent	Temp (°C)	Time	Yield(%)	Yield (Rp)
1	Al(OH) ₃ (0.1)	Solvent free	110 ⁰ C	15-30	64-75 ^(TW)	90 ⁽²⁷⁾
2	Al(OH) ₃ (0.2)	Solvent free	110 ⁰ C	15-30	67-77 ^(TW)	NR
3	Al(OH) ₃ (0.3)	Solvent free	110 ⁰ C	15-30	71-79 ^(TW)	94 ⁽³¹⁾
4	Al(OH) ₃ (0.4)	Solvent free	110 ⁰ C	15-30	73-82 ^(TW)	96 ⁽³¹⁾
5	Al(OH) ₃ (0.5)	Solvent free	110 ⁰ C	15-30	73-84 ^(TW)	93 ⁽³¹⁾
6	Sio-Al(OH) ₃ (0.1)	Solvent free	110 ⁰ C	15-30	71-74 ^(TW)	94 ⁽³¹⁾
7	Sio-Al(OH) ₃ (0.2)	Solvent free	110 ⁰ C	15-30	72-81 ^(TW)	89 ⁽³²⁾
8	Sio-Al(OH) ₃ (0.3)	Solvent free	110 ⁰ C	15-30	75-82 ^(TW)	NR
9	Sio-Al(OH) ₃ (0.4)	Solvent free	110 ⁰ C	15-30	79-89 ^(TW)	95 ⁽³¹⁾
10	Sio-Al(OH) ₃ (0.5)	Solvent free	110 ⁰ C	15-30	91-96 ^(TW)	91-96 ^(TW)
11	Y(otf) ₃ (5%)	H ₂ O	100 ⁰ C	360-420	360-420	87 ⁽³⁾
12	HCl	H ₂ O	100 ⁰ C	360-420	360-420	49 ⁽⁴⁾
13	TFA	H ₂ O	100 ⁰ C	360-420	360-420	55 ⁽⁵⁾
14	HoAC	H ₂ O	100 ⁰ C	360-420	360-420	42 ⁽¹⁾
15	SbCl ₃ -SiO ₂	Solvent free	120 ⁰ C	30	91 ⁽¹⁾	91 ⁽¹⁾

16	Zn(OAc) ₂	H ₂ O	120
17	Any Organic acid	H ₂ O	120
18	HPA1,HPA2	Solvent free	110
19	CeCl ₃ .7H ₂ O	IL	110
20	TBA	Solvent free	100
21	CBSA	Solvent free	100
22	B(C ₆ F ₅) ₃	Solvent free	R
23	SBSSA	EtOH	65
24	Amberlyst-15	CH ₃ CN	Ref
25	LGPCO	Solvent free	Ref
26	GSMO	Solvent free	Ref
27	ZnCl ₂ -SiO ₂	Solvent free	Ref
28	FeCl ₃ -SiO ₂	Solvent free	Ref

Table:02

time:15-30 min, Amount of catalyst:0.5 mmole ,temp:110⁰C,NF-Not formed, NR-Not reported

*PC	Aldehyde	Yield (TW)	Yield (Rp)
JBR#01	C ₆ H ₅	96	90 ⁽²⁷⁾
JBR#02	2-NO ₂ C ₆ H ₅	NF	NR
JBR#03	3-NO ₂ C ₆ H ₅	96	94 ⁽³¹⁾
JBR#04	4-NO ₂ C ₆ H ₅	99	96 ⁽³¹⁾
JBR#05	2-Cl C ₆ H ₅	96	74 ⁽³²⁾
JBR#06	4-Cl C ₆ H ₅	92	88 ⁽³¹⁾
JBR#07	4-Cl C ₆ H ₅	94	93 ⁽³¹⁾
JBR#08	4-Cl C ₆ H ₅	97	94 ⁽³¹⁾
JBR#09	3,4-Br- MeOC ₆ H ₃	93	89 ⁽³²⁾
JBR#10	3-Br C ₆ H ₅	97	95 ⁽³¹⁾
JBR#11	4-Br C ₆ H ₅	97	95 ⁽³¹⁾

Conclusion: our team are selected the good yield contained derivatives for the study of catalysis potential for the reaction via green way, we got the highest amount of yield at the 0.5% at the 110⁰C and got the 91-96% of yield the past researchers formed the 90-96% (3,4,5,6,360,420,24,25,29,32,31), but the High time 30 the completion of the reactions. so, we are tray to optimization of the waste

after the reaction for the and we go the highest amount of yield in very short time and the more productive thing of this catalyst the recrystallization does not required they are the complete conversion of the reaction but the spectroscopic conformation we are the performed the recrystallization on via hot Analytical data:

JBR#01

9-phenyl-3,4,6,7,9,10-hexahydroacridine-1,8(2H,5H)-dione:

IR:1226(-C-Nstr),1487,1529(CH₂-CH₂),1635(-C=O,Carbonyl group),2960(CHstr),3054(CH Str),3755(NH str)

NMR:69.02(1to5ppm,12.43=12H,all non aromatic ring hydrogen),28.02(6to 8,5.088=5H,aromatic ring hydrogen),3.57(0.64=1H,nitrogen contenting ring hydrogen)

¹³CNMR:28.77,30.66,44.43,52.82,119.35,119.55,119.99,123.62,124.34,129.02,129.10,,137.46,138.45,138.56,147.24,149.26,204.78.

Mass:293.98

JBR#04

9-(4-nitrophenyl)-3,4,6,7,9,10-hexahydroacridine-1,8(2H,5H)-dione:

IR: 1231,1342,1490,1608,1659,2958,3060,3172

¹HNMR:71.85(1to5ppm,13.65=13H,all non aromatic ring hydrogen),28.02(6to 8,5.088=5H,aromatic ring hydrogen),3.57(0.64=1H,nitrogen contenting ring hydrogen)

¹³CNMR:23.20,31.50,33.42,37.90,114.31,124.78,127.25,128.44,131.52,148.78,153.20

MASS:339.25

JBR#08

9-(4-chlorophenyl)-3,4,6,7,9,10-hexahydroacridine-1,8(2H,5H)-dione

¹HNMR:74.70(1to5ppm,13.45=13H,all non aromatic ring hydrogen),20.42(6to 8,3

.62 =4H,aromatic ring hydrogen),6.21(1.11=1H,nitrogen contenting ring hydrogen)

¹³CNMR:23.32,30.44,33.78,36.39,42.70,111.18,115.55,111.78,1129.44,130.15,131.78,142.58,155.47,162.21,200.45

Mass:327.10

ethanol. the variety of derivatives are the formed by the change the substitution of aromatic aldehyde and we got the different derivatives of hexahydroacridine. the conformation of the structure with help of IR by FT-IR Proton NMR, Carbon NMR and Mass spectra.

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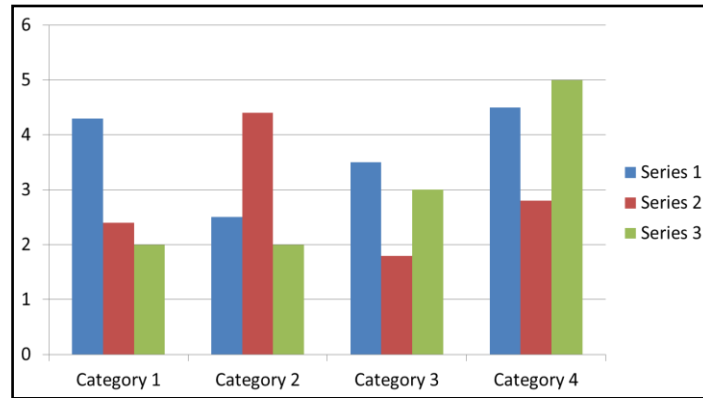


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$$\varphi_{\mu\nu}(z) = \frac{\|k_{\mu\nu}\|^2}{\sigma^2} e^{-\frac{\|k_{\mu\nu}\|^2 \|z\|^2}{\sigma^2}} [e^{ik_{\mu\nu}z} - e^{-\frac{\sigma^2}{2}}] \quad (1)$$

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11.1. Journal Article

- [1] C. D. Scott and R. E. Smalley, “Diagnostic Ultrasound: Principles and Instruments”, *Journal of Nanosci. Nanotechnology.*, vol. 3, no. 2, (2003), pp. 75-80.

11.2. Book

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11.3. Chapter in a Book

- [3] H. V. Jansen, N. R. Tas and J. W. Berenschot, “Encyclopedia of Nanoscience and Nanotechnology”, Edited H. S. Nalwa, American Scientific Publishers, Los Angeles, vol. 5, (2004), pp. 163-275.

11.4. Conference Proceedings

- [4] *J. Kimura and H. Shibasaki, "Recent Advances in Clinical Neurophysiology", Proceedings of the 10th International Congress of EMG and Clinical Neurophysiology, Kyoto, Japan, (1995) October 15-19.*

11.5. Patent

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