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

JAYAVEERSINH MAHIDA,RAVI B. PATEL

Shree P. M. Patel institute of P.G. Studies & Research in science, Anand, Gujarat, India

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.

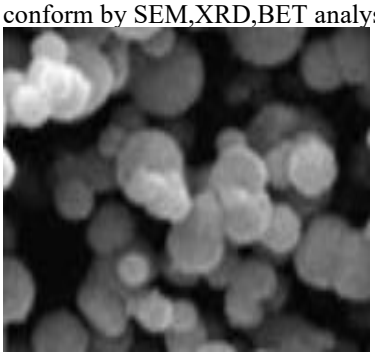
Keywords: *Dimidone, Solvent free, green catalyst. high reusability.*

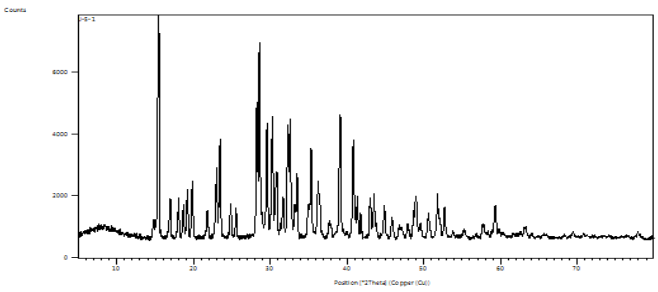
1. INTRODUCTION

This catalyst are design for the homogeneous catalyst for the differ path for the new heterogeneous catalyst. Aluminums 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 all, they sodium aluminum silicates for the abrogation of dyes from environment, and Ahemad nakhaei et all they are introduce the heterogeneous catalyst for the 1,4 dihydropyridine Hantzch 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 $Al(OH)_3$: 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 rice 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 90^0C 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 300^0C 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 (^{0}C)	Time	Yield(%)
1	$Al(OH)_3$(0.1)	Solvent free	110^0C	15-30	64-75(TW)
2	$Al(OH)_3$(0.2)	Solvent free	110^0C	15-30	67-77(TW)
3	$Al(OH)_3$(0.3)	Solvent free	110^0C	15-30	71-79(TW)
4	$Al(OH)_3$(0.4)	Solvent free	110^0C	15-30	73-82(TW)
5	$Al(OH)_3$(0.5)	Solvent free	110^0C	15-30	73-84(TW)
6	Sio-$Al(OH)_3$(0.1)	Solvent free	110^0C	15-30	71-74(TW)
7	Sio-$Al(OH)_3$(0.2)	Solvent free	110^0C	15-30	72-81(TW)
8	Sio-$Al(OH)_3$(0.3)	Solvent free	110^0C	15-30	75-82(TW)
9	Sio-$Al(OH)_3$(0.4)	Solvent free	110^0C	15-30	79-89(TW)
10	Sio-$Al(OH)_3$(0.5)	Solvent free	110^0C	15-30	91-96(TW)
11	$Y(otf)_3$(5%)	H_2O	100^0C	360-420	87(1)
12	HCl	H_2O	100^0C	360-420	49(1)
13	TFA	H_2O	100^0C	360-420	55(1)
14	HoAC	H_2O	100^0C	360-420	42(1)
15	$SbCl_3$-SiO_2	Solvent free	120^0C	30	91(2)
16	$Zn(OAc)_2$	H_2O			120
17	Any Organic acid	H_2O			120
18	HPA1,HPA2	Solvent free			110
19	$CeCl_3.7H_2O$	IL			110
20	TBA	Solvent free			100
21	CBSA	Solvent free			100
22	$B(C_6F_5)_3$	Solvent free			R
23	SBSSA	EtOH			65
24	Amberlyst-15	CH3CN			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:110^0C,NF-Not formed, NR-Not reported

*PC	Aldehyde	Yield (TW)	Yield (Rp)
JBR#01	C_6H_5	96	90(27)
JBR#02	2-NO_2 C_6H_5	NF	NR
JBR#03	3-NO_2 C_6H_5	96	94(31)
JBR#04	4-NO_2 C_6H_5	99	96(31)
JBR#05	2-OH C_6H_5	96	74(32)
JBR#06	4-OH C_6H_5	92	88(31)
JBR#07	2-Cl C_6H_5	94	93(31)
JBR#08	4-Cl C_6H_5	97	94(31)
JBR#09	3,4 Br-$MeOC_6H_3$	93	89(32)
JBR#10	3-Br C_6H_5	NF	NR
JBR#11	4-Br C_6H_5	97	95(31)

Conclusion: **Conclusion:** our team are selected the good yield contained derivatives for the study of catalytic potential for the reaction via green way so we got the highest amount of yield at the 0.5% at the 110^0C and got the 91-96% of yield the past researchers are formed the 80-96%(3,4,5,6,8,10,11,12,21,24,25,29,32,31), but the got the Hugh time for 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 recrysattlion does not required they are the complete conversion of the reaction but the spectroscopic conformation we are the performed the recrysallation on via hot 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.

Analytical data:

JBR#01

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

IR:1226(-C-Nstr),1487,1529(CH_2-CH_2),1635(-C=O,Carbonylgroup),2960(CHstr),3054(CH Str),3755(NH str)

NMR:69.02(1to5ppm,12.43=12H,allnon 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

^{1}HNMR: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)

^{13}CNMR: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

^{1}HNMR: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)

^{13}CNMR: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

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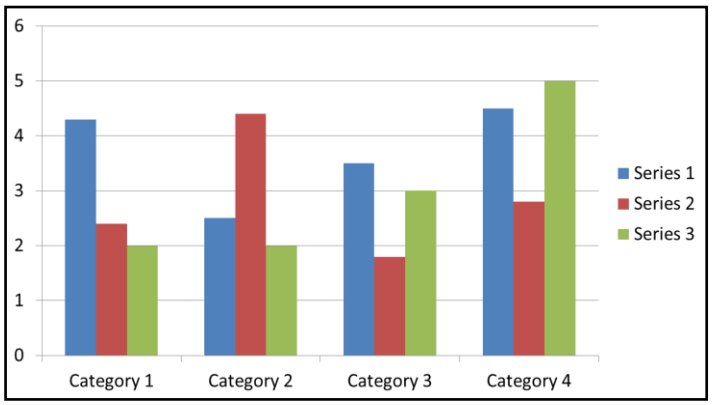

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For Example:

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

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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

[2] *H. S. Nalwa, Editor, "Magnetic Nanostructures", American Scientific Publishers, Los Angeles,* ***(2003)****.*

11.3. Chapter in a Book

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11.4. Conference Proceedings

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11.5. Patent

[5] *C. E. Larsen, R. Trip and C. R. Johnson, "Methods for procedures related to the electrophysiology of the heart", U.S. Patent 5,529,067,* ***(1995)*** *June 25.*