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Eco-friendly and Effective Catalyzed Synthesis of 1, 3, 4 –Oxadiazole using Silica Supported Poly Phosphoric Acid, Solvent Free and Grinding Method

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Abstract : *The synthetic route were involved condensation, cyclisation, dehydration, Oxidation of aromatic aldehydes and aromatic hydrazide using silica supported poly phosphoric acid by mortor-pistle grinding method under solvent free condition. The catalyst used silica supported poly phosphoric acid has safe, green, easy to prepare and recoverable, gives high yield, reusable catalyst. The structures of the synthesized product ,the derivatives of 1,3,4-Oxadiazole were evaluated by advanced analytic techniques as 13C-NMR, 1H-NMR, IR, LCMS, elementary analysis.*

Key Words: Silica supported poly phosphoric acid, Ultra sound, grinding, aromatic hydrazine, aromatic aldehydes.

I. INTRODUCTION

1, 3, 4-Oxadizoles an important heterocyclic organic compound containing oxygen and Nitrogen have highly active compound 1. It has wide variety of medicinal and biological activity2 including anti-inflammatory, antibacterial, antiviral and broad pharmaceutical activity.1, 3, 4-Oxadiazoles an aromatic heterocyclic compound synthesized by different methods such as ultrasonic techniques3-8 using Ionic liquids9, under microwave irradiations10, Grinding method13.

By considering the reaction mechanism for synthesis of 1, 3, 4-Oxadiazoles in two types first is the dehydrative cyclisation using POCl3, SOCl2, H2SO4 etc. and second one is the oxidative cyclisation using oxidant such as I2-K2CO3, PbO2, FeCl3, KMnO4, CAN, HgO/I2.11 Although some methods have good yield, simple and easy synthetic way, low cost , efficient, recoverable and reusable catalyst12.For further study, synthesis of new class of compound which has medicated property nowadays more important. We promoted synthesis of organic compound with solvent free and easy to handle acid catalyst is PPA/SiO2 in place of simple PPA. Under grinding method, 1, 3, 4-Oxadiazole derivatives synthesised using PPA/SiO2 under solvent free conditions shown in scheme-I.

II. METHODS AND MATERIALS

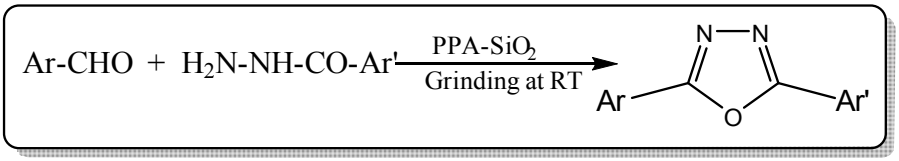
The open capillary method used for knowing the melting point of newly prepared 1,3,4-Oxadiazole derivatives. The progress of reaction monitoring by TLC . The structural data gave from spectral analysis evaluated with known referral structures from researchers.

III. GENERAL PROCEDURE

The mixture of aromatic aldehyde (0.01mole) and acyl hydrazine (0.01mole) under grinding method in mortor for about 10 min in 2-3 min interval of time keep monitoring reaction progress by TLC (n-hexane: EtOH 6:4), after completion of reaction the crude sample were poured into cold water and was extracted with ethyl acetate. The catalyst

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was filtrated as residue. The solvent was removed from crude sample. The sample was washed with water and recrystallized with n-hexane to get the pure sample of 1, 3, 4-Oxadiazole. The structure were confirmed by H1-NMR C13-NMR, IR and mass spectral data and compared with data reported from authentic sample reported.



Scheme – I: Aromatic aldehyde and Benzoylhydrazide under grinding in presence PPA-SiO2. 1, 3, 4-Oxadiazole derivatives.

IV. RESULT AND DISCUSSION

In the study of above scheme under simplest and effective grinding method between aromatic aldehyde and benzoyl hydrazide gave the intermediate as benzoyl hydrazone and the final Product 1,3,4-Oxadiazoles was prepared. The Scope of the catalyst PPA-SiO2 and synthesized 1, 3, 4-Oxadiazoles have vast significance in pharmaceutical areas. The PPA-SiO2 acts as water scavenger as well as an acid catalyst The 1,3,4-Oxadiazole with other catalyst gave 60-80% yield and if the yield product above 90% then it were difficult to remove and recover the catalyst also time consuming required electric instruments13. By using proposed scheme-I, the result as shown inTable-1.

Sr. No	Catalyst	Yield	Time duration	Ref.
1	CsCO3	84 %	30 min	11
2	I2/K2CO3	96%	60 min	11
3	I2/DBU	58 %	110 min	11
4	TiO2	59 %	180 min	13
5	PPA	86 %	30 min	12
6	PPA-SiO2	99 %	30 min	10,12

Table-1: Different catalyst used for 1, 3, 4-Oxadiazole synthesis

Synthesizing 1, 3, 4-Oxadiazole derivatives were found that comparing PPA-SiO2 with other catalyst gives best results. The synthesised heterocyclic compound as 1,3,4-Oxadiazole were identified by melting point reported in references11as shown in table-2.

Sr.No.	Aldehyde	Aromatic hydrazine	Time duration in (min)	Yield (%)	Melting Point in 0c
1	Ph-CHO	Ph-CONHNH2	15	98	139
2	4-CH3-Ph-CHO	Ph-CONHNH2	15	99	126
3	4-OCH3-Ph-CHO	Ph-CONHNH2	20	95	150
4	4-NO2-Ph-CHO	Ph-CONHNH2	16	90	223
5	4-Cl-Ph-CHO	Ph-CONHNH2	15	89	165

Table 2: Synthesised 1,3,4-Oxadiazole derivatives as per scheme – 1 with various aspects.

Ttable -2 gave the suitable explaination about the derivatives , the aromatic aldehyde with electron withdrawing group (EWG) as -NO2 , -X high melting point and nearly same yield as compare to electron donating group (EDG) as –CH3 , -OCH3. The electron donating group has high yield low melting.

The spectra data as H1-NMR, C13 -NMR, IR, Mass spectrum for synthesized oxadiazole were checked from reported references.it has nearly same values. The catalyst PPA supported by Silica absorbs water which eliminated in condensation process to form intermediate substituted benzoylhydrazones. Silica provides space for the reaction .The catalyst used many times with losing its activity even use for several time.

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V. CONCLUSION

Here, the conclusion drawn from the above result and discussion that substituted 1,3,4- oxadiazole synthesized by grinding method in presence of silica supported poly phosphoric acid catalyst at solvent free condition is more effective and efficient process. It has simple route to complete reaction and has easy to handle recoverable, reusable catalyst. The synthesized 1,3,4-oxadiazole has excellent microbial activity. This research on further modification gives new class of heterocyclic compound to study in future.

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