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ALUM AN EFFICIENT CATALYST FOR SYNTHESIS OF 4-ARYL-3-METHYLISOXAZOLE-5(4H)-ONE DERIVATIVES

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Abstract:

Isoxazole and its derivatives were synthesized by one pot cyclocondensation reaction of aldehyde, hydroxylamine hydrochloride and ethyl acetoacetate with alum with small amount of SiO_2.

Key Words: Aldehyde, Ethyl Acetoacetate, Hydroxylamine Hydrochloride, Alum & SiO_2

1. Introduction:

Multi -component reaction are Introduced by strecker in 1850. Multicomponent reaction gives some importance characteristics like high yield, mild reaction conditions, ecofriendly nature and reduces the time period of reaction mechanism. Other than this MCR gives three components reaction gives best result in short time with good yield. In one pot three components system easy constructional with organic substance hence it avoid the complicated process. Oxygen and nitrogen containing heterocyclic compound having beneficial class in medicinal chemistry due to their diversified properties and use. isoxazole structure is also play an importance for heterocyclic which having favorable properties for pharmaceutical area such as antifungal, antitumor, antioxidant, antimicrobial, antiviral, insecticidal and antituberculsis [10, 11] etc. The alum is a convenient reagent uses for synthetic transformation, it is soluble in organic solvent. The alum show high reactivity with low toxicity gives good yield. The present protocol helps to development of simple and efficient method for the synthesis of heterocyclic isoxazole.

2. Experimental:

2.1 Experimental Section: All chemical were purchased from Merck, sdfcl were commercially available and were used as received without further purification .all reaction conducted at room temperature melting points were measured by open capillary method incorrectly. IR data collected on (range 4000-400). NMR Data recorded in DMSO –d6 as solvent by Bruker Avance Neo 500 MHz spectrometer.

2.2 General Procedure for the Synthesis: The appropriate reaction mixture ethyl acetoacetate (2mmol), hydroxylamine hydrochloride (2mmol), aromatic aldehyde (2mmol) and 10 % alum with small amount of (0.100 gm) SiO_2 in 12 ml ethanol was reflux up to 3-4 hours as per required product formation gradually check during reaction (monitored by TLC analysis). The precipitated was wash with 5% water and recrystallized using 95 % ethanol to afford the product. All products are known compound and identified by physical data with reported in this literature.

Reaction:

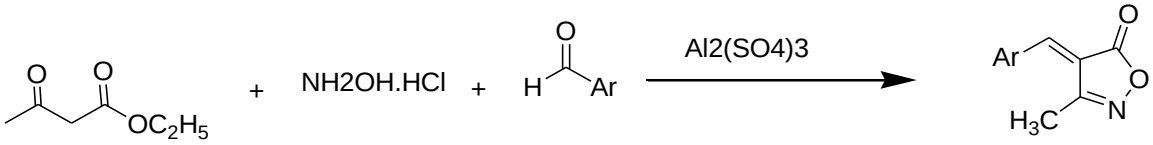

Figure 1: One pot three component condensations of ethyl acetoacetate, hydroxylamine hydrochloride and aldehyde with alum gives isoxazole

Table 1: Synthesis isoxazole from various aldehyde

Compound	Aldehydes	Time	Yield	Found mp
A1	4-$MeOC_6H_5CHO$	3	80	170-175
A2	2-OH C_6H_5CHO	4	65	190-195
A3	4-OH -3$CH_3OC_6H_5CHO$	3	75	142-144
A4	4-Cl-C_6H_5CHO	4	Trace	-------
A5	C_6H_5CHO	3	80	142-143
A6	3 $NO_3C_6H_5CHO$	4	-----	-------

Spectral Data for Selected Product:

Characterization of Compounds:

- ENTRY A3 (4 hydroxy-3 methoxybenzylidene)-3- methylisoxazole -5(4H)-one
  Yellow white crystal mp-142
  1H-NMR (500MHz-DMSO d6)- δ 8.52 , δ 7.91, δ 7.91-7.76 , δ 6.97 , δ 3.86, δ 2.52
  13 C –NMR (125MHz-DMSO d6) – δ 168.86 , δ 162.00 , δ 153 , δ 147 , δ 124 , δ 113.57 , δ 155.41 , δ 11.12
- ENTRY A5- -(4 benzylidene)-3- methylisoxazole -5(4H)-one
  Yellow crystal: mp 142-144
  1H-NMR (500MHz-DMSO d6)- δ 8.42 , δ 7.94, δ 7.56-7.61 , δ 7.56 , δ 2.29
  13 C –NMR (125MHz-DMSO d6) – δ 167.64 , δ 162.00 , δ 151.41 , δ 132.28 , δ 130.99 , δ 128.51 , δ 118.69 , δ 11.11

3. Results and Discussion:

Alum as an efficient catalyst having white colour pawder safe in uses, ecofriendly, nontoxic, non flammable, clean, inexpensive soluble in ethanol. When alum with small amount of SiO_2 react with ethyl acetoacetate, aldehyde with hydroxylamine hydrochloride gives arylmethylene isoxazol-5-(4H)-one derivatives via the one pot three component process. There was traces amount of product formation observed in the present of solvent. The use of ethanol gives best result with good yields. Reaction carried out in two parts, in first stage, ethyl acetoacetate reacts with hydroxylamine hydrochloride to gives ethyl 3-(hydroxyimino) butanoate. In second stage aldehyde react with above product gives Knoevenagel reactions gives 3 –methyl -4- arylmethylene isoxazole-5(4H) one.

Table 2: Percentage of Catalyst

Enter	Catalyst %	Time	Yield
1	5	3	30
2	10	3	80
3	15	3	80
4	20	3	80

4. Conclusion:

In conclusion, aromatic aldehyde were react with eaa and hydroxylamine hydrochloride in presence of sodium hypophosphite 10% catalyst in water all result are show in table. The aromatic aldehydes are with electron donating group gives the product with good in short time. ortho substitute group required more time and low yield for 2- hydroxyl benzaldehyde, while electron withdrawing group are not gives product. The one pot three compound systems gives 3 methyl 4arylmethalen isoxazole in ethanol at 80 ^{0}C temperature.

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6. Conflict of Interest:

The author have declared that no conflict of interest exists.

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