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REVIEW

Various Synthetic Pathways for the Synthesis of 3,4-Disubstituted Isoxazole by One Pot Multicomponent Reaction

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Abstract:

Isoxazole is a five membered heterocyclic compound which is an azole with an oxygen atom next to nitrogen. The substituted isoxazole derivatives show various biological activities like antimicrobial, antiviral, anti-inflammatory, antioxidant, anticancer etc. Isoxazoles are found in some natural products and also in number of drugs. Especially 3– methyl–4-arylmethylene substituted isooxazole is used as a main constituent in synthetic organic chemistry. Various approaches have been made for diverse synthesis of 3,4-distituted isoxazoles via multicomponent reaction using different catalyst. Each catalyst has its own effect on the synthetic rate, yield and reaction time of reaction. In this mini review we have reported the various catalytic effect on the single pot multicomponent synthesis of 3,4-disubstituted isoxazolones.

Keywords: catalysis; 3,4-disubstituted isoxazolones; multicomponent reaction; single-pot; three component reaction

1. Introduction

Multi component reactions (MCRs) [1] are single pot operation in which more than two reactants react in a single vessel to form a product which consist almost all the element of reactant, with no by product. Due to this, multi component reactions are proved to be an ideal and eco-friendly reaction system. The product obtained in one pot synthesis involves much fewer steps. High efficiency, eco-friendly, easy work-up, minimum waste, atom economy, time-saving, energy saving, waste reduction etc are some of the remarkable features of these reactions and known to exhibit great significance in preparation of biologically & pharmaceutically active compounds as well as in the synthesis of natural product [2, 3]. Hence it is a strong synthetic methodology recommended by many researchers and scientist for the synthesis of various valuable heterocyclic products of medicinal importance in one pot. Heterocycles are the cyclic compounds consist atoms of at least two different elements as member of its ring. While cyclic compounds having same elements is known as homocyclic compounds.

The heterocycles containing nitrogen atom with an oxygen atom is consider as an important class of compounds in medicinal chemistry. One such heterocyclic compound containing oxygen and nitrogen as heteroatom is isoxazole. The heterocyclic compounds containing isoxazole ring plays an important role in pharmaceutical industry and organic synthesis. Isoxazole is five membered heterocyclic compounds containing oxygen and nitrogen as hetero atoms with oxygen atom next to nitrogen.

The isoxazole derivative constitute a superficial class of heterocycles having many medicinal and biological activities such as cox-2-inhibitor [4], antimicrobial [5], analgesic [6], antioxidant [7], antitumour [8], antifungal [9], anti-inflammatory [10], anticonvulsant [11], antiviral [12], antimycobacterial [13], antagonists [14], immunosuppressive [15], HDAC inhibitor [16], anti-HIV [17], antibiotics [18, 19], antiandrogents

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[20, 21], anticancer [22]. The isoxazole is a core constituent of number of drugs like cox-2-inhibitor, nitric oxide donor furaxan. Isoxazole ring (Fig.1) is found in many antibiotics such as cloxacillin, dicloxacillian and flucloxacillin.

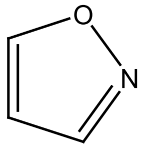


Figure 1. Isoxazole.

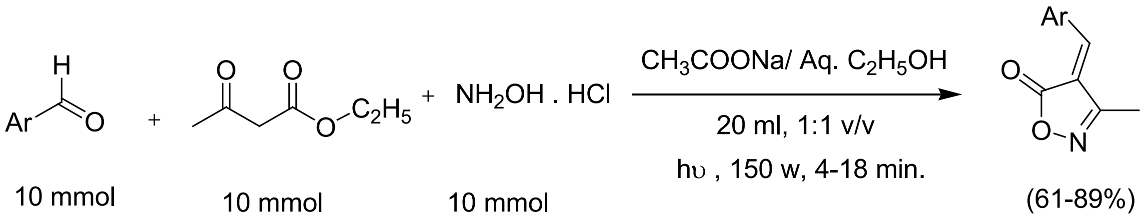
The nitrogen hetero atom shows electron withdrawing effect and the oxygen atom shows electron donating effect. As neutral molecules isoxazoles, undergo electrophillic substitution reaction readily at the position 4 than benzene. The isoxazole have capability to undergo reactions such as protonation, quaterization, complexation, oxidation, reduction, carbionic condensations, thermolysis, photolysis, transformation into other heterocyclic ring systems and reaction with electrophiles, nucleophiles and grignard reagents. Due to the importance of isoxazole-5(4H)-one, many researchers have been interested in the development of highly efficient synthetic methodology.

Looking at the importance of 3,4-disubstituted isoxazoles, recently organic chemists have paid much attention at the generation of new methodologies for the synthesis of such compound. In this review, we have tried to cover the various methodologies adopted synthesizing isoxazolones via one pot multi-component reactions. The details have been shared below.

A simple mechanistic approach for the synthesis of isoxazolones includes two steps reaction [23]. Initially, hydroxylamine hydrochloride reacts with ethylacetoacetate to offer oxime which further undergoes ring closure to give substituted isooxazole. Later, the knoevenagel condensation between substituted aldehydes and isooxazole takes place which results into the final product 3,4-disubstituted isooxazolones. Some other methods like solid state grinding and solid state heating [24] have been also reported, however these methods have some limitations like low yield, use of large amount of catalyst, time consuming, complex reaction condition (such as high temperature). That's why green methodology based procedures are still in high demand

2. Various synthetic pathways for the preparation of isoxazolone derivatives

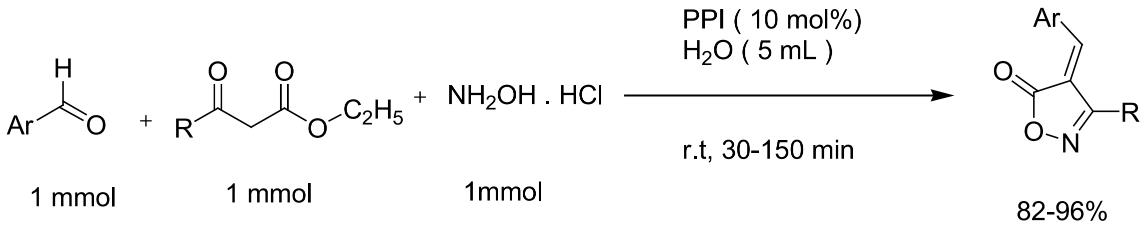
In 2013 Saikh et al. have demonstrated the synthesis of isoxazolones through multi-component reaction of hydroxyl amine with substituted aldehydes and ethylacetoacetate by using sodium acetate as catalyst in aqueous ethanol irradiated with 150W tungsten lamp [25]. Due to inexpensive, readily availability and nontoxic nature of sodium acetate, it is widely used as weak base catalyst for knoevenagel reaction and aldol condensation (Scheme 1).



Ar = Ph, 2,4–diOMePh, 4-OHPh, 3–OMe-4-OHPh, 4–OMePh, 3,4-diOMePh

Scheme 1.

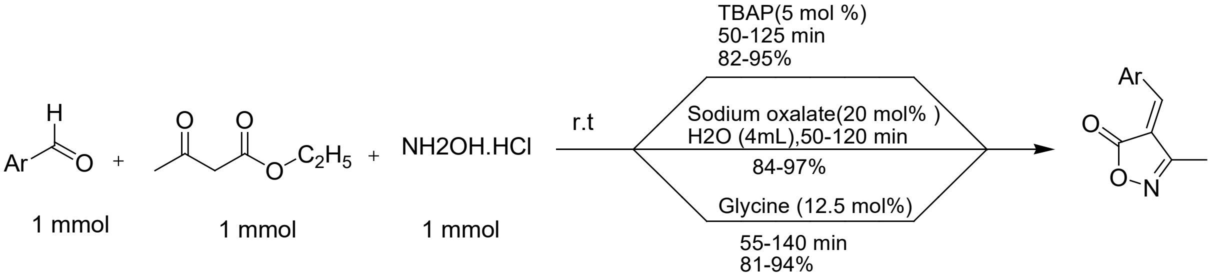
Later, in 2017 Kiyani and his co-workers have been reported synthesis of substituted isooxazolones by using an ecofriendly and efficient poatassium phthalimide (PPI) for the single pot reaction of $NH_2OH.HCl$ with ethylacetoacetate and substituted aldehydes in H_2O under mild reaction conditions gave isoxazole derivative in good yield [26]. Other solvents like ethanol, 1,4-dioxane, cyclohexane, hexane and acetone were also studied but they were unable to give good results as compared to water. PPI played great role as it is inexpensive, commercially available, green, mild and efficient basic catalyst (Scheme 2).



Ar = 2-Furyl, Ph, 3-OHPh, 3-coumarinyl, 4-MePh, 4-OHPh, 3-OMe-4-OHPh, 4-OHPh, 2-Thienyl, 4-OH-3-NO_2Ph, 4-N(Me)$_2$Ph, 2-OHPh; R = Me, CH_2Cl, Ph

Scheme 2.

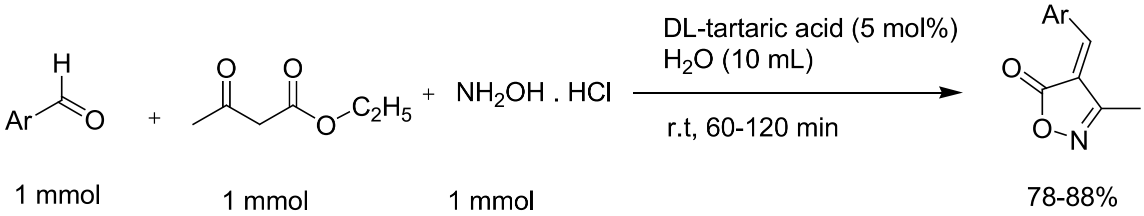
In 2014 Kiyani and assosciates have developed the synthesis of 3,4-disubstitutedisoxazoles by using tetrabutyl ammonium per chlorate (TBAP)/ glycine/ sodium oxalate as catalyst at room temperature for the one pot multicomponent reaction of ethylacetoacetate, numerous substituted aldehydes and hydroxylamine hydrochloride in aqueous medium [27]. Various other solvents for example ethanol, 1,4-dioxane, dichloromethane, hexane and acetone were also examined by the authors, but the good yield is obtained in water only. In comparison to glycine and sodium oxalate TBAP gives product within short reaction time (Scheme 3).



Ar = 4-MePh, 3-OHPh, 4-OHPh, 3-OMe-4-OHPh, 4-N(Me)$_2$Ph, 3-Thienyl, 2-OHPh, Ph, 4-OMePh, 4-NHCOMePh, 2-OH-4-N(Et)$_2$Ph, 2-Thienyl; R = Me, Ph, CH_2Cl

Scheme 3.

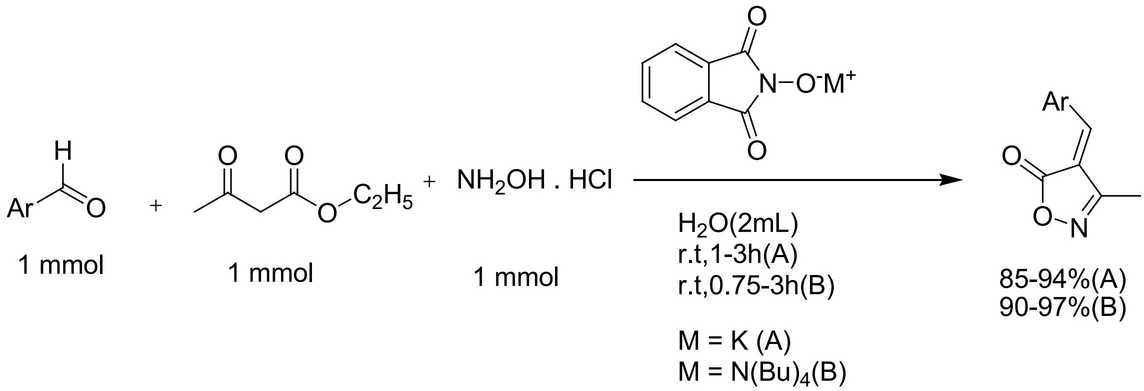
In 2015 Khandebharad et al. have reported the synthesis of isoxazolone derivatives through a multi-component transformation of ethylacetoacetate, various aromatic aldehyde and hydroxylamine hydrochloride by using tartaric acid as a biodegradable organocatalyst in water as solvent at room temperature [28]. The author also studied reaction conditions by using other catalyst such as sodium citrate, sodium benzoate and dl-tartaric acid. While good product yield and less reaction time were observed in case of dl-tartaric acid (Scheme 4).



Ar = Ph, 4-OMePh, 4-MePh, 4N(He)$_2$Ph, 2-Thienyl, 4-ClPh, 2-OHPh, 4-OHPh, 2-Fuyl

Scheme 4.

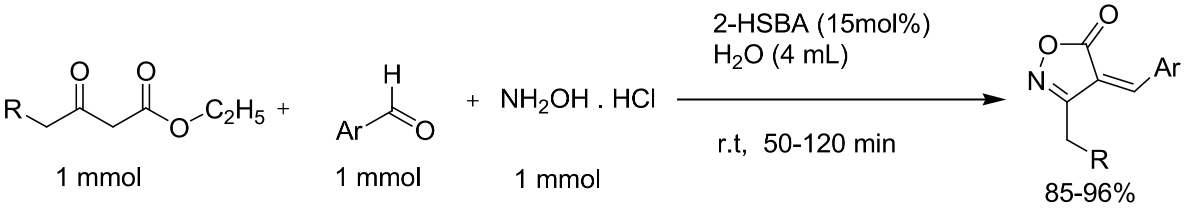
In 2016 Dekamin and associates demonstrated the synthesis of isoxazolone derivative through single-pot reaction of substituted aldehydes, ethylacetoacetate and $NH_2OH.HCl$ by using tetrabutylammonium or potassium salts of phthalimide-N-oxyl as catalyst at low temperature in aqueous medium [29]. Other solvents like DMSO, water, cyclohexane and ethanol were also examined by author while best result was acquired in water (Scheme 5).



Ar = Ph, 4-NO_2Ph, 4-CNPh, 2-OHPh, 4-MePh, 2,4-$(OMe)_2$Ph, 4-ClPh, 3-NO_2Ph, 4-OMePh, 3-OHPh, 4-OHPh, 4-OH-3-OMePh, 2-Furyl, 4-$N(Me)_2$Ph, 2-Thienyl, (E)-2-Phenylethenyl

Scheme 5.

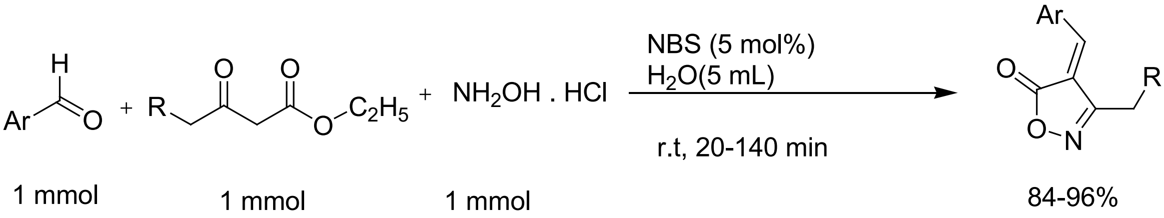
In 2015, Kiyani et al., described the multi-component synthesis of disubstituted isoxazolone derivatives by using 2-Hydroxy-5-sulfobenzoic acid (2-HSBA) as an organocatalyst at room temperature by the reaction of ethylacetoacetate, various substituted aldehydes and hydroxylamine hydrochloride in aqueous medium [30]. Various solvents like DMSO, ethanol, dioxane, dichloromethane, hexane and acetone were also examined but best results were found in water (Scheme 6).



Ar = 4-ClC_6H_5, 4-$NO_2C_6H_5$, 4-$OCH_3C_6H_5$, 3-OHC_6H_5, 4-OHC_6H_5, 4-OH-3-$OMeC_6H_4$, 4-$NHCOMeC_6H_5$, 2-Thienyl, 4-CNC_6H_5, 2-OHC_6H_5, Ph, 4-$CH_3C_6H_5$, 2,4-$(OMe)_2C_6H_4$, 3-Thienyl, 2-OH-4$N(Et)_2C_6H_4$, CH=CH C_6H_5; R = Cl, H

Scheme 6.

In 2015, Kiyani have developed another protocol for the preparation of disubstituted isoxazolones via one pot reaction of ethylacetoacetate with $NH_2OH.HCl$ and various substituted aldehydes by using catalytic amount of N-bromosuccinimide (NBS) in aqueous medium at room temperature [31]. A wide range of solvents like cyclohexane, ethanol, 1,4-dioxane, hexane and acetone were also demonstrated in this reaction by the authors (Scheme 7).

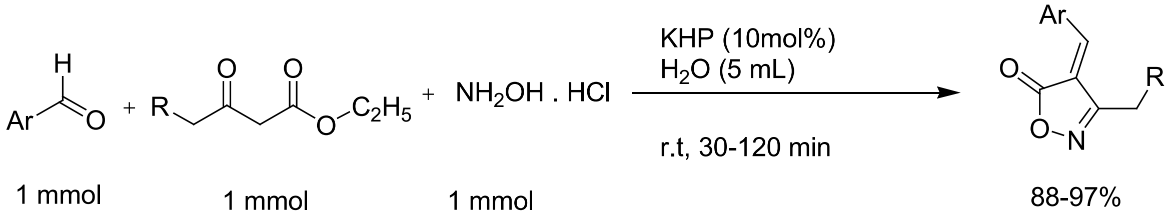


Ar = Ph, 4-NO_2Ph, 4-CNPh, 2-OHPh, 4-MePh, 4-OH-3-OMePh, 4-$N(Me)_2$Ph, 2-Thienyl, 2-Furyl, 3-

Coumarinyl, 3-Thienyl, 4-ClPh, 3-NO$_2$Ph, 4-OMePh, 3-OHPh, 4-OHPh, 4-OH-3-NO$_2$Ph; R = H, Cl

Scheme 7.

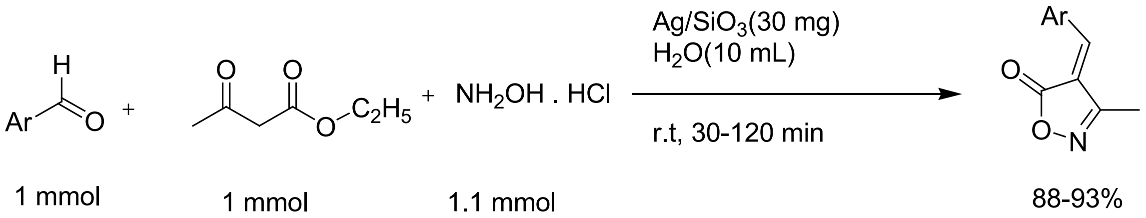
In 2015 Kiyani and associate have reported the preparation of disubstituted isoxazolones in the presence of Potassium Hydrogen Phthalate (KHP) as green and inexpensive catalyst by the reaction of ethylacetoacetate, various aromatic aldehydes and NH$_2$OH.HCl at low temperature in aqueous medium [32] (Scheme 8).



Ar = Ph, 3-NO$_2$Ph, 4-OMePh, 3-OHPh, 4-0HPh, 4-OH-3-NO$_2$Ph, 4-N(Et)$_2$Ph, 2- Furyl, 2-Thienyl, 3-Thienyl, 3-Coumarinyl, 4-ClPh, 4-NO$_2$Ph, 4-CNPh, 2-OHPh, 4-MePh, 4-OH-3-OMePh, 4-N(Me)$_2$Ph; R = H, Cl

Scheme 8.

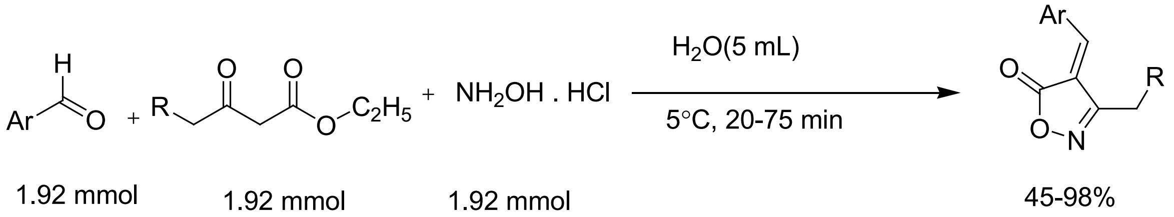
In 2016 Maddila et al. have demonstrated a green methodology for the preparation of disubstituted isoxazolones by using Ag/SiO2 in water at low temperature through the reaction of aryl substituted aldehydes with ethylacetoacetate and hydroxylamine hydrochloride [33]. Various other catalyst such as NaOH, K$_2$CO$_3$, Na$_2$S, pyridine, BF$_4$, SiO$_2$, Al$_2$O$_3$, Fe$_2$O$_3$, CeO$_2$, AgNO$_3$, Ag/SiO$_2$, HClO$_4$/SiO$_2$ and FeCl$_3$/SiO$_2$. A wide range of solvents as DMF, acetonitrile, ethanol, dioxane, methanol, hexane and water were also examined while the best results were obtained in water (Scheme 9).



Ar = C$_6$H$_5$, 4-OHC$_6$H$_5$, 4-EtC$_6$H$_5$, 2,4 diMeC$_6$H$_4$, 2,4,6-(OMe)$_3$C$_6$H$_3$, 2,5-(OMe)$_2$C$_6$H$_4$, 2,3-(OCH$_3$)$_2$C$_6$H$_4$, 3-OHC$_6$H$_5$, 3,4 diOHC$_6$H$_4$, 2,5-diOHC$_6$H$_4$, 4-N(Me)$_2$C$_6$H$_5$, 4-OMeC$_6$H$_5$

Scheme 9.

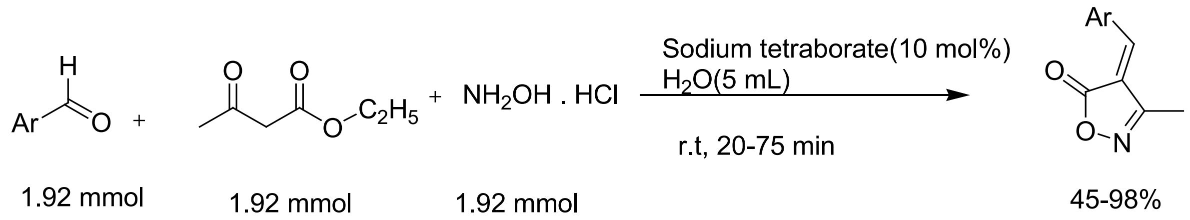
In 2015 Chavan et al. have described another efficient preparation of disubstituted isoxazolone derivatives through multi-component reaction of numerous aryl substituted aldehydes, ethylacetoacetate and hydroxyl amine hydrochloride in water at 5 °C [34] (Scheme 10).



Ar = Ph, 4-NHCOMePh, 4-OmePh, 3-OMe-4-OHPh, 2-OMePh, 3-Indole, 4-OEtPh, CH=CHPh, 3,4-OCH$_2$OPh, 4-FPH, 2-OHPh, 3-OHPh, 3-FPH, 4-OHPh, 4-MePh, 4-N(Me)$_2$Ph; R$_1$ = H, Cl; R$_2$ = Me, Et

Scheme 10.

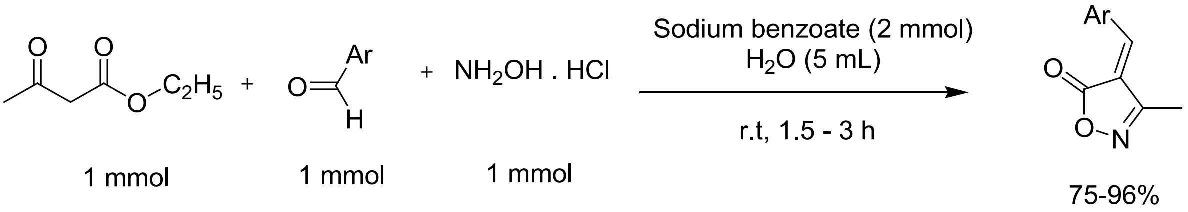
In 2013 Kiyani and co-workers have reported the preparation of 3,4-disubstituted isoxazolones via a one pot reaction various substituted aldehydes, $NH_2OH.HCl$ and ethylacetoacetate by employing $NaBH_4$ at room temperature in aqueous medium [35]. Different solvents like dioxane, acetone, hexane and ethanol were also demonstrated (Scheme 11).



Ar = Ph, 4-OMePh, 2,4-diClPh, 4-CH_3Ph, 4-NO_2Ph, 4-ClPh, 2-Furyl, 2-Thienyl, 3-NO_2Ph, 4-OH-3-NO_2Ph, 3-Chromenyl, 4-OHPh, 4-OH-3-OMePh, 3-OHPh, 2-OHPh, 4-N(Me)$_2$Ph

Scheme 11.

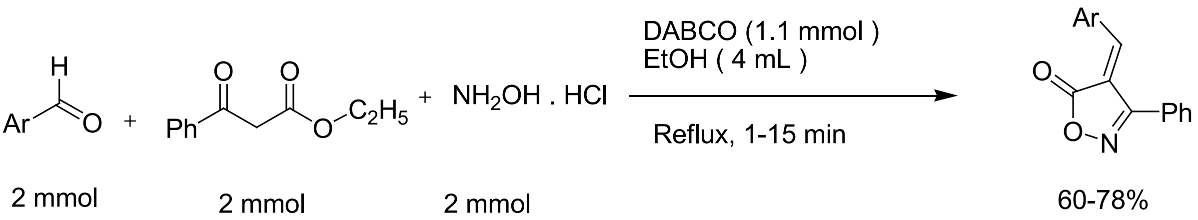
In 2011 Liu and associates demonstrated an efficient and green methodology by the reaction of ethylacetoacetate with substituted aldehyde and $NH_2OH.HCl$ by thw use of sodium benzoate as catalyst in aqueous medium at low temperature [36]. Various other solvents for example, ethanol, dioxane, cyclohexane and acetone were also examined however the best results were obtained in water. Sodium benzoate was utilized as an efficient, novel and green catalyst for the knoevenagel condensation of aldhydes with active methylene compounds such as ethyl acetoacetate and malonitrile to offered substitute olefins (Scheme 12).



Ar = Ph, 2-Furyl, CH=CHPh, 4-OMePh, 3-OMe-4-OHPh, 2-OHPh, 4-OHPh

Scheme 12.

In 2012 Mirzazade and co-workers demonstrated an eco-friendly protocol for synthesizing the disubstituted isoxazolones through the reaction of hydroxylamine hydrochloride, ethylacetoacetate and highly substituted aldehydes catalyzed by 1,4 diazobicycle[2,2,2] octane (DABCO) in refluxing ethanol [37] (Scheme 13).

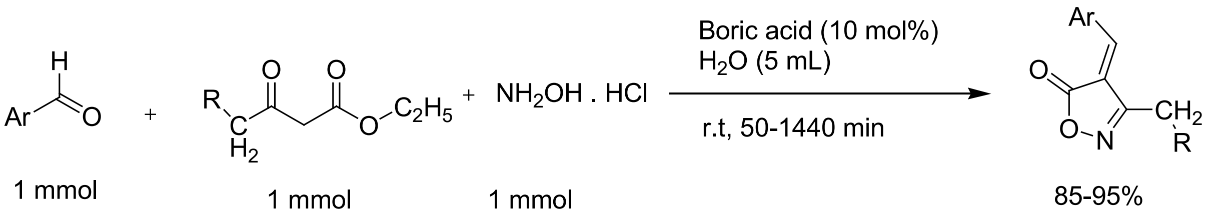


Ar = Ph, 4-ClPh, 4-BrPh, 2,4-OHPh, 4-MePh, 4-OHPh, CH=CHPh, 2-Naphthyl, 2-OCH_3Ph, 4-OMePh, 4-NMe_2Ph, 2,4-OCH_3Ph

Scheme 13.

In 2015 Kiyani and his group have demonstrated another effective single-pot multi-component reaction of various aromatic aldehydes with ethylacetoacetate and $NH_2OH.HCl$ by using boric acid (H_3B0_3) in water

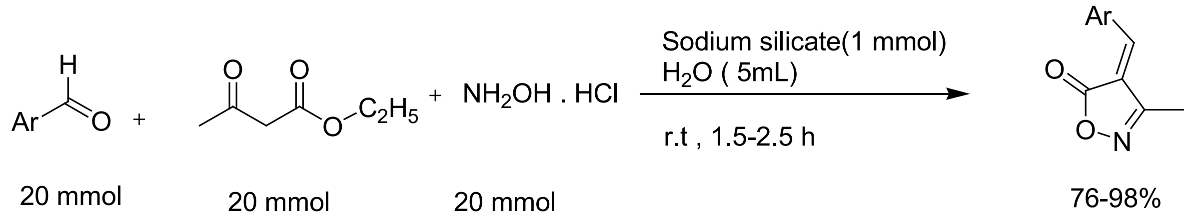
[38]. Various other solvents like ethanol, dioxane, hexane, water and acetone were also demonstrated however best results were obtained in water (Scheme 14).



Ar = Ph, 4-NO_2Ph, 3-OCH_3Ph, 4-ClPh, 3-OHPh, 4-OHPh, 4-CH_3Ph, 4-OCH_3Ph, 4-OHPh, 4-OHPh, 2-OHPh, 3-NO_2-4-OHPh, 3-Penyl, 2,4-diClPh, 2-Thiphenyl, 2-Furyl, 4-N(Me)$_2$Ph, 3-Coumarinyl; R = H, Cl, Ph

Scheme 14.

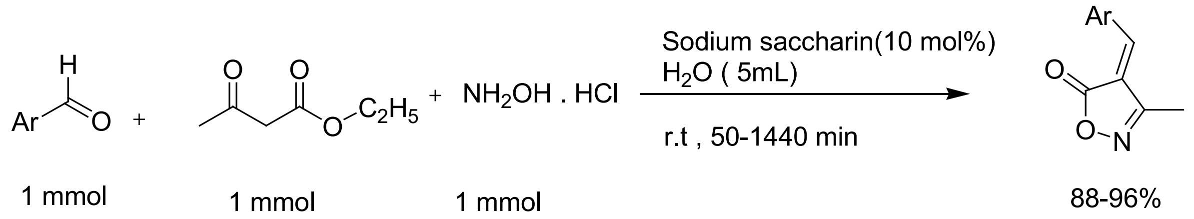
In 2011 Liu and associates have developed the synthesis of isoxazolones through the reaction of hydroxylamine hydrochloride with various aromatic aldehydes and ethyl acetoacetate by using sodium silicate pentahydrate as an efficient catalyst in water at room temperature [39]. Ethanol, dioxane, acetone and cyclohexane were also considered as solvents for this protocol, but the best result was obtained in water only (Scheme 15).



Ar = Ph, 4-OHPh, 2-OHPh, 2-Furyl, CH=CHPh, 4-OMePh, Vanillin

Scheme 15.

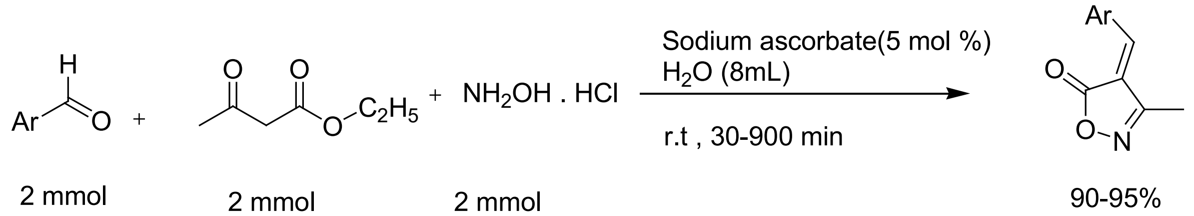
In 2013 Kiyani and co-workers have reported the preparation of 3,4-disubstituted isoxazolone derivatives through the reaction of hydroxylamine hydrochloride with various aromatic aldehydes and ethyl acetoacetate by using sodium saccharin in aqueous medium at low temperature [40]. A wide range of solvents were also tested however best results were obtained in water. Sodium saccharin as a basic green, low-cost, easily available compound was used as catalyst in some organic synthesis (Scheme 16).



Ar = Ph, 2-Pyridyl, 4-OMePh, 3-Coumarinyl, 4-MePh, 2,4-diClPh, 3-OMe-4-OHPh, 4-NO_2Ph, 2-Furyl, 4-N(Me)$_2$Ph, 2-Thiophenyl, 3-Thiophenyl, 4-OHPh, 3-OHPh, 2-OHPh

Scheme 16.

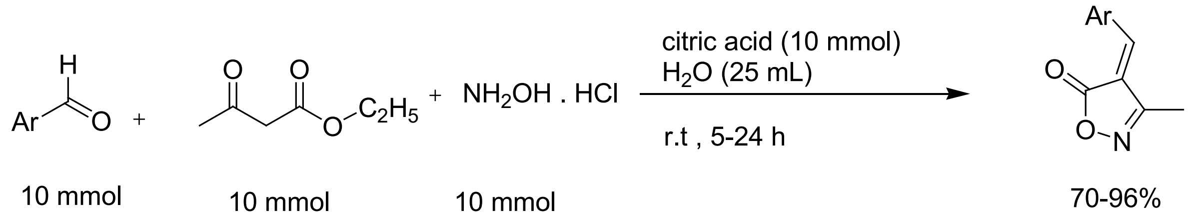
In 2013 Kiyani and associates have described the synthesis of 3,4-disubstituted isoxazolone derivatives through the reaction of various substituted aldehydes with NH_2OH.HCl and ethylacetoacetate by using sodium ascorbate in water at room temperature [41]. THF, acetone, hexane, ethanol, dioxane and cyclohexane were also demonstrated along with water however the best results was obtained only in water in terms of yield and time (Scheme 17).



Ar = C_6H_5, 4-NO_2Ph, 4-ClPh, 4-OHPh, 2-Furyl, 3-OHPh, 2-OHPh, 4- CH_3Ph, 2-Pyridyl, 2-Thienyl, 4-OMePh, 3-Br-4-OH-5-OMePh, 5-OMe-1H-indolyl, 4-OMePh, 4-OH-3-NO_2Ph, 4-OH-3-OMePh, 4-OH-3-NO_2Ph, 3-indolyl

Scheme 17.

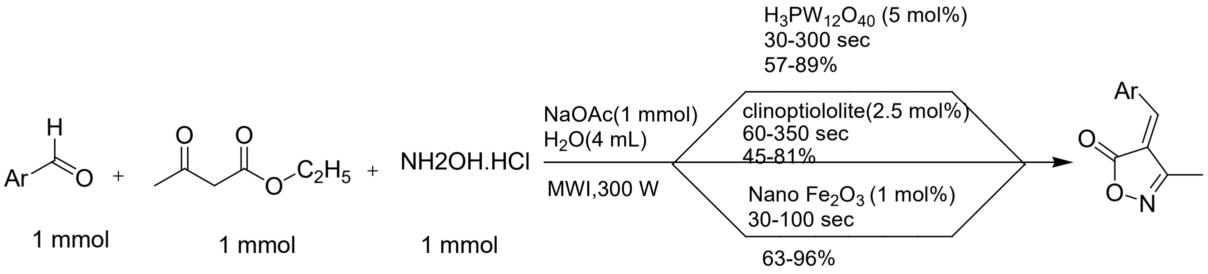
In 2016 Rikani and associate have demonstrated the preparation of 3,4-disubstituted isoxazolones derivatives via reaction of numerous substituted aldehydes with ethylacetoacetate and $NH_2OH.HCl$ by employing natural citric acid in aqueous medium at low temperature [42] (Scheme 18).



Ar = Ph, 4-MePh, -CH=CHPh, 4-N(Me)$_2$Ph, 3-BrPh, 4-OMePh, 2-OMCPh

Scheme 18.

In 2013 Fozooni et al. have demonstrated the single pot synthesis of 3,4-disubstituted isoxazolones derivatives through reaction of ethyl acetoacatate, various aromatic aldehydes and $NH_2OH.HCl$ by employing Fe_2O_3 nanoparticle, $H_3PW_{12}O_{40}$ and clinoptilolite with sodium acetate in water under microwave irradiation [43]. Fe_2O_3 nanoparticle had good performance than other catalyst in terms of yields and reaction time (Scheme 19).



Ar = Ph, *N,N*-(Me)$_2$Ph, 4-PPh, C_4H_3S, 4-OMePh; R = Me, Ph

Scheme 19.

4. Conclusions

In summary, we have tried to focus on the various catalytic approach made for the efficient and green synthesis of disubstituted isoxazolones via multi-component reaction of hydroxylamine hydrochloride, ethyl acetoacetate with various substituted aldehydes. Many new biodegradables and homogenous organocatalysis have been explored for improving selectivity, purity and high yield of 3,4-disubstituted isooxazolones. Particularly, some heterogeneous catalysts have been also used for synthesizing the disubstituted isoxazolones efficiently. Further, 3,4-disubstituted isoxazolones are important organic intermediates for the synthesis of various bio-active molecules.

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