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Synthesis of Pyrano [2,3,d] Pyrimidines under green chemistry

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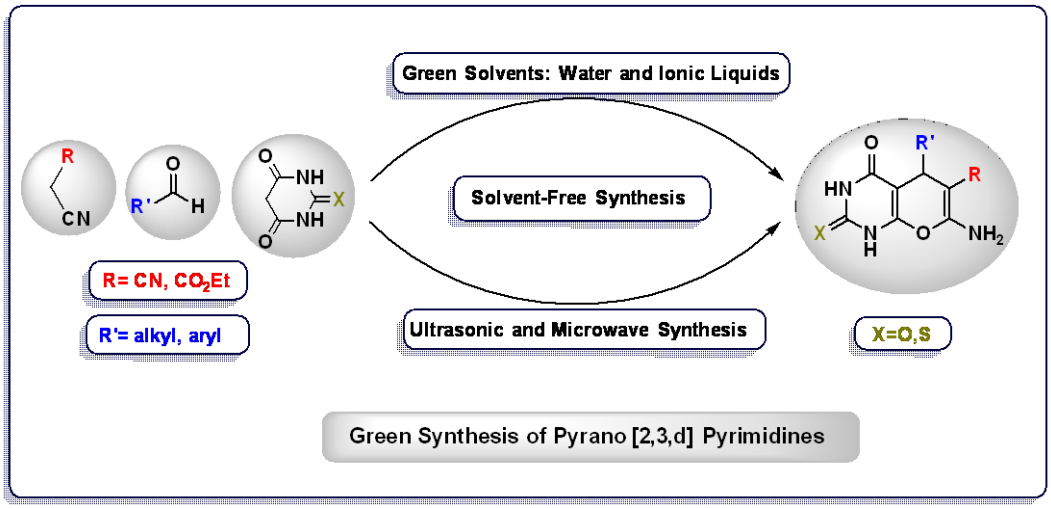
Abstract

Pyrano [2,3,d] pyrimidines derivatives are very important and valuable compounds in the fields of medicine, biological and pharmaceutical. In this context, we study effects and benefits of various green synthetic strategies in the synthesis of pyrano [2,3,d] pyrimidines derivatives. Green chemistry is design, advancement and use of chemical products and systems that minimize or remove the utilization and formation of destructive and toxic materials. It is a universal method that is feasible to all areas of science chemistry. All synthetic processes involve the use of various substances. Unfortunately majority of the chemical reagents and substances are applied in industry and chemistry laboratories are hazardous and toxic compounds which led to environmental damage, through pollution, threats to human health and resource evacuation, we need to develop and utilize more environmentally friendly protocols. Therefore, all prevalent and old approaches clearly give detrimental impacts to the mankind and all living beings. Green strategies open up multitudinous possibilities for performing rapid organic reactions and functional group transformations more effectively. It is quite obvious from the growing number of emerging publications in this area that the possibility to utilize green chemistry technology allows reaction conditions to be accessed that are very important and valuable for organic synthesis. In this review, we study effects and benefits of various green synthetic strategies in the synthesis of pyrano[2,3,d]pyrimidines derivatives. Due to unique benefits of green reactions, new green protocols are being discovered for eco-friendly synthesis of large number of organic, pharmaceutical and natural compounds. As a result, green synthetic reactions are observing a fresh spring.

Graphical abstract



1. Introduction

Heterocyclic chemistry is one of the most important and valuable branches in chemistry science. Heterocyclic compounds are very vital and effective in synthesis of biological, pharmaceutical compounds [1]. Also, these compounds are backbone of most of the pharmaceutical active materials and antibiotics [2]. Multicomponent reactions (MCRs) have attracted remarkable attentions as a powerful means to synthesize medicinal and natural

compounds [3-4].A multicomponent reaction (MCR) is commonly introduced as a reaction where three or more substrates react with together to form a special product in particular a natural product [5-6].The MCR strategy presents an efficient and straightforward path to produce complexity and diversity in a single operation [7-8]. A large number of multicomponent reactions demonstrate benefits in atomic economy, environmental friendliness, simplified steps, optimal and useful use of resources[9-11].

The preparation of fused heterocycles has occupied an eminent place in heterocyclic chemistry. One of the most important this compounds is Pyrano[2,3-d]pyrimidine Derivatives. The pyrano[2,3-d]pyrimidine is an unsaturated six membered heterocycle thatis constructed by fusion of pyran and pyrimidine rings together and consisting of one oxygen atom at position number 8 and two nitrogen atoms at position number 1 and 3 respectively [12].During recent years, much attention has been focused on synthesis ofPyrano [2,3,d] pyrimidines and their derivative due to their numerous application in pharmacological fields. These compounds possess wide range of pharmacological activities such as antitumour [13], ardiotonic[14], antimalarial [15], antibronchitic [16], exhibit antihypertensive activity [17], analgesic [18-19], antiviral evaluation [20], antimicrobial and antifungal activities (figure1) [21-22].

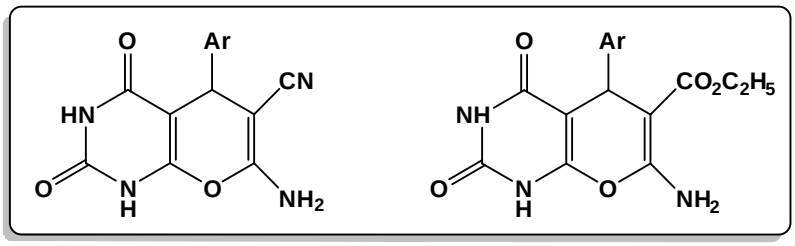


Figure1. Examples of Pyrano [2,3,d] Pyrimidines derivatives with biological and pharmacological activities.

The pyrano[2,3-d]pyrimidines are building blocks used to evaluate their antimicrobial activities and various derived natural products are also used as a drug for insomnia treatment [23].

The synthesis of organic compounds via green, mild a simple method is important goal of organic chemistry. Therefore, every day chemists in particular organic researchers are looking for finding novel and convenient systems to carry out chemical reactions. Since pyrano [2,3,d] pyrimidines are very effective and valuable compounds in the medicine and pharmaceutical fields, therefore finding modern and milder systems for synthesis of these compounds is one of the most important priorities among organic chemistry researches. During recent years, various reagents and catalyst such as triethylamine, potassium carbonate, pyridine, phosphorus pentoxide (P_2O_5), Phosphorus pentasulfide (P_2S_5), piperidine and Zn[(L)proline]$_2$ have been used for the synthesis of pyrano[2,3,d]pyrimidine derivatives [24-30]. Despite great importance of the above protocols in organic synthesis, unfortunately, most of them have major drawback such as unfavorable yields, formation of side product, long reaction time, high temperatures, harsh reaction conditions, use of strong bases, expensive and toxic or metallic catalysts as well as environmentally unfriendly due to use of hazardous organic solvents as reaction medium. The processes are not only of interesting and importance from an environmental point of view but in most of cases also present considerable synthetic aspects in terms of yield, efficiency, practicality and simplicity of reaction procedures. Therefore, there is a genuine requirement to develop a convenient, versatile and environmentally benign system for the synthesis of pyrano[2,3,d]pyrimidine derivatives. In this regard, green chemistry presented a series of valuable, efficient and environmentally friendly strategies to synthesize these compounds. Now, in this paper, we wish to focus on reported green strategies for the synthesis of pyrano[2,3,d]pyrimidine derivatives and also we will explore results and advantages of these strategies.

2. Green chemistry

The use of destructive and toxic chemical materials in chemistry laboratories and the chemical industry have led to increase the concerns in case of the health and safety of workers and environmental pollution. On the other hand, the chemical industry is very vital and important for the world economy because chemical reagents and

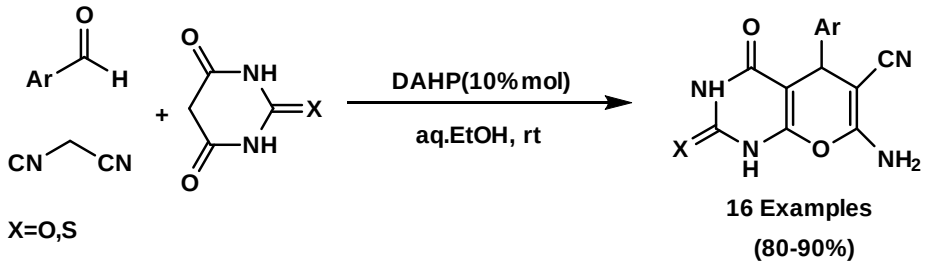
materials are used in large scale for the production of food supplements, cosmetic materials, pharmaceutical and natural products.
In the past decades Green Chemistry has become a research field of great interest due to replace greener reagents and solvents instead of hazardous and toxic materials [31].
Green chemistry is introduced as "design, advancement and use of chemical products and systems that minimize or remove the utilization and formation of destructive and toxic materials" [32].
Now, green chemistry is a new and valuable strategy of looking at organic synthesis and the design of drug molecules, offering considerable environmental and economic benefits over traditional synthetic processes. Green Chemistry is presenting series of new and valuable strategies that can overcome the exacting limitations in traditional synthetic techniques [33].
The accomplishment of reactions in the presence of green mediums (use of recyclable ionic liquids, greener and safer alternative solvents) or under solvent-free conditions, microwave and ultrasonic technologies are some of types of green synthetic strategies.
Now, we wish to classify the various reported protocols based on using green synthetic strategies.

2.1. Green Solvents

From scientific laboratories to industrial plants, organic conversions are usually performed in solution, therefore using solvents as the reaction medium. Solvents are an important and inseparable (indispensable)part for the performance of chemical processes [34]. In the chemical industry and chemistry laboratories, solvents belong to the most important group of chemical materials due to the large scales that are used annually [35].Solvents are very vital as aqueous medium for reactions to be accomplished and after the synthesis of a chemical product for extraction, isolation, purification and drying [36]. The majority of solvents are organic chemical materials with destructive and toxic features. In result, despite solvents are very important in world economy, but these reagents (due to its toxic natures properties) are very dangerous for human health and environment [37-38].
The idea of ‘‘green’’ solvents represent the target to minimize the environmental impact resulting from the use of solvents in chemical generation [39-40]. This idea is introduced a series of techniques to carry out organic reactions in greener mediums.
Recently, four important pathways for green mediums have been developed: (i) use of more benign solvents (such as water) that demonstrate more efficient EHS (environmental, health and safety), (ii) use of ionic liquids that demonstrate low vapour pressure, (iii) use of bio-solvents that generated with recyclable resources such as ethanol (generated by fermentation of sugar-containing feeds), (iv)use of solvent-free conditions (the performance of reaction without use of organic solvents) [41-46].

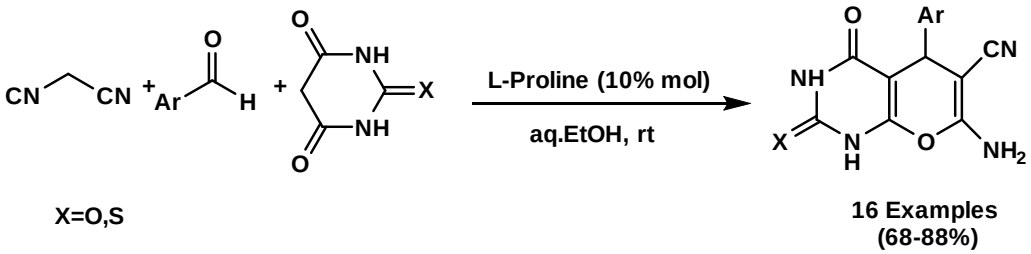
2.2. Organic Synthesis in Water

The accomplishment of reactions in an aqueous medium could be the response to the future of organic synthesis. Water is a valuable universal solvent because it both is very efficient for performance of organic reactions and is very inexpensive as well as environmentally friendly.
Without doubt, alongside being a readily available, inexpensive, benign, nontoxic and nonflammable solvent, water has special structure and physicochemical features which, in many cases, lead to an enhancement of reaction rates and selectivities due to hydrophobic influence, hydrogen bondings, polarity and/or trans-phase interactions [47].
Water is extensively used as a green solvent for the performance of multicomponent reactions.
Organic synthesis “on water” simplifies effective chemical generation in an environmentally safe route [48].
In recent years, Green chemistry that uses water as a solvent to prepare pharmaceutical and natural products has received great attention in organic synthesis [49].For instance, Balalaie and his co-workers exploited diammonium hydrogen phosphate (DAHP) in an aqueous media for the synthesis of pyrano[2,3,d]pyrimidine derivatives (Scheme 1) [50]. The pyrano[2,3,d]pyrimidine derivatives were prepared in about 2 h at room temperature, in aqueous medium (water/ethanol). A general and green protocol with the same reagents has also been developed [51].



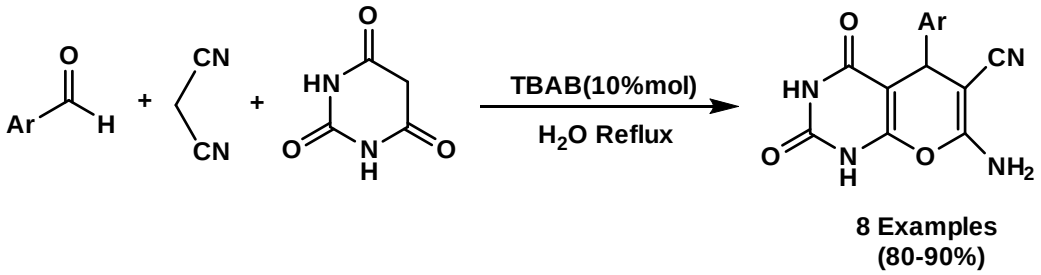
Scheme 1. Synthesis of Pyrano[2,3,d]Pyrimidines derivatives in an aqueous media Catalyzed by DAHP.

L- proline was also successfully applied as a neutral bifunctional catalyst for the condensation of aromatic aldehydes, malononitrile and barbituric-or thiobarbituric acid in aqueous ethanol. (Scheme 2) [52].



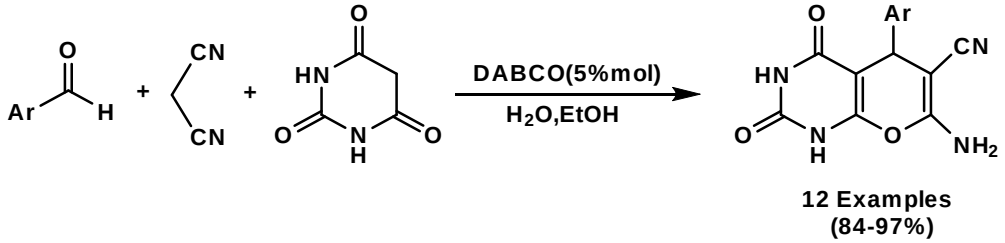
Scheme 2. Synthesis of Pyrano [2,3,d] Pyrimidinesderivativescatalyzed by DAHP.

A practical, valuable and efficient protocol for the preparation of pyrano [2,3,d] pyrimidine derivatives has also described by Mobinikhaledi research group [53]. Tetrabutylammonium bromide (TBAB) in an aqueous medium (water) was demonstrated to be an excellent and green catalytic system for the condensation of aromatic aldehydes, malononitrile and barbituric acid (Scheme 3).



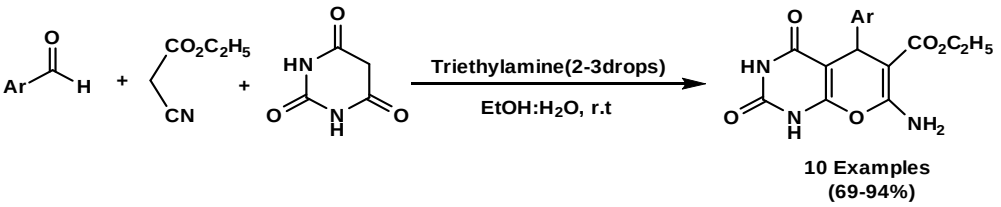
Scheme 3. Synthesis of Pyrano [2,3,d] Pyrimidines derivatives in water Catalyzed by TBAB.

1,4-diazabicyclo[2.2.2]octane (DABCO) in aqueous medium (mixture of water and ethanol) is also an efficient catalyst to synthesize pyrano [2,3,d] pyrimidine derivatives (Scheme 4) [54]. A green and convenient protocol with the same reagents has also been developed [55-56].



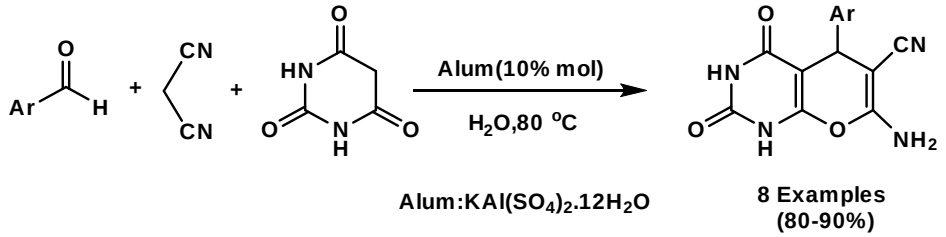
Scheme 4. Synthesis of Pyrano [2,3,d] Pyrimidines derivatives catalyzed by DABCO.

Triethyamine is an efficient base catalyst for the performance of condensation reactions. In this regard, Bahat research group utilized from this reagent in solution of water: ethanol (1:1 ratio) for the condensation of aromatic aldehydes, malononitrile and barbituric acid (Scheme 5) [57,12].

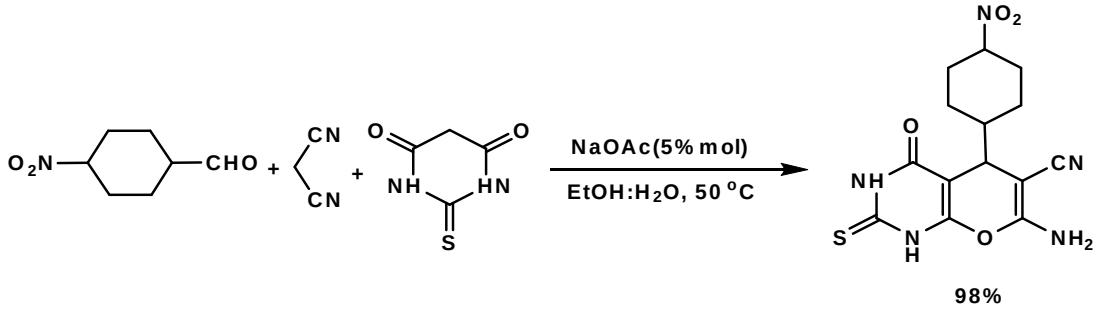


Scheme 5. Synthesis of Pyrano [2,3,d] Pyrimidines derivatives catalyzed by Triethyamine.

Recently, one-pot three-component reaction to synthesize pyrano[2,3-d]pyrimidine derivatives in the presence of water as reaction medium has also been reported [58]. $KAl(SO_4)_2.12H_2O$ (alum) in water was also presented as a valuable and effective system for the eco-friendly and efficient synthesis of Pyrano[2,3-d] pyrimidinone derivetives (Scheme 6) [59]. Green media, lack of toxicity, short reaction times, simple work-up and excellent yields are some valuable aspects of this strategy. Very recently, Khorassani research group surprisingly discovered that synthesis of Pyrano[2,3-d] pyrimidines could also be carried out easily using sodium acetate in an aqueous medium (Scheme 7) [60]. To increase the efficiency of system, mixture of water and ethanol (4:1 ratio) was added as solvent into the reaction medium.



Scheme 6. Synthesis of Pyrano [2,3,d] Pyrimidines derivatives catalyzed by Alum.

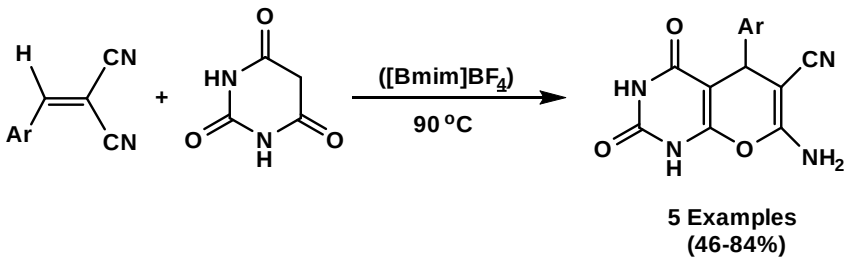


Scheme 7. Synthesis of Pyrano [2,3,d] Pyrimidines derivatives in an aqueous media.

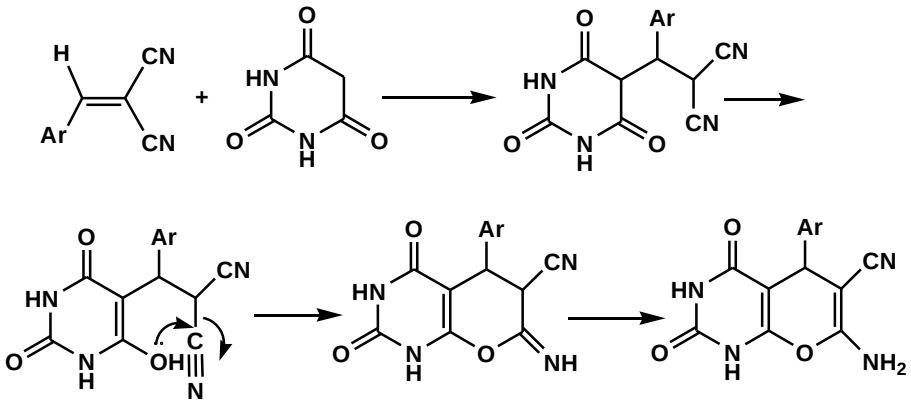
2.3. Organic synthesis using Ionic liquids

As mentioned in above, the most of solvents are organic chemicals. As a result of many physical and chemical obstacles, organic solvents cannot often be completely eliminated from the final product during generating even with such drying strategies as increased temperature under decreased pressure or by lyophilization (freeze-drying) [61].Sometimes, small amounts of solvent generally still remain in the terminal product. Furthermore, a final product may also become contaminated by organic solvents in the packaging, warehouse storage [61].

To solve these problems, over the past few decades, reagents such as water, supercritical fluids, fluorous phases, surfaces or interiors of clays, zeolites, silica gels, and alumina have been explored as potential reaction media [62].During recent years, Ionic liquids (ILs) have also been considered as alternatives to organic solvents in chemical and biological processes [62].Recently, ionic liquids (ILs) are attracting increased attention worldwide due to their unique and excellent properties [63-65].Ionic liquids are versatile and efficient solvent for the condensation reactions. In this regard, ionic liquids were mediated for the condensation of arylmethylidenemalononitrile and barbituric acid under neutral conditions (Scheme 8) [66]. In this system, 1-*n*-butyl-3-methylimidazolium tetrafluoroborate ([Bmim]BF_4) used as recyclable ionic liquid in which it also acted both as promoter and reaction medium. Also, authors presented a plausible mechanism for accomplishment of reaction that has been designed in Scheme 9.



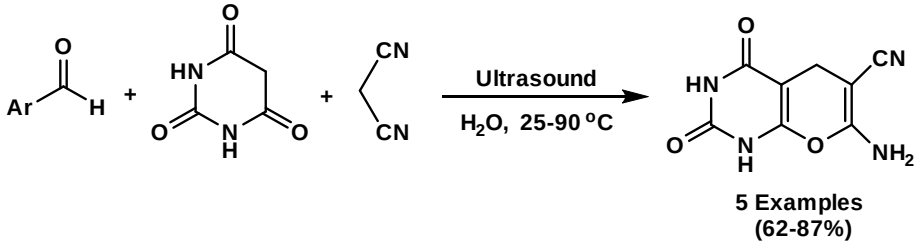
Scheme 8. Synthesis of Pyrano [2,3,d] Pyrimidines derivatives in [Bmim]BF_4 as anionic liquid.



Scheme 9. Plausible mechanism for the synthesis of Pyrano [2,3,d] Pyrimidines in [Bmim]BF_4.

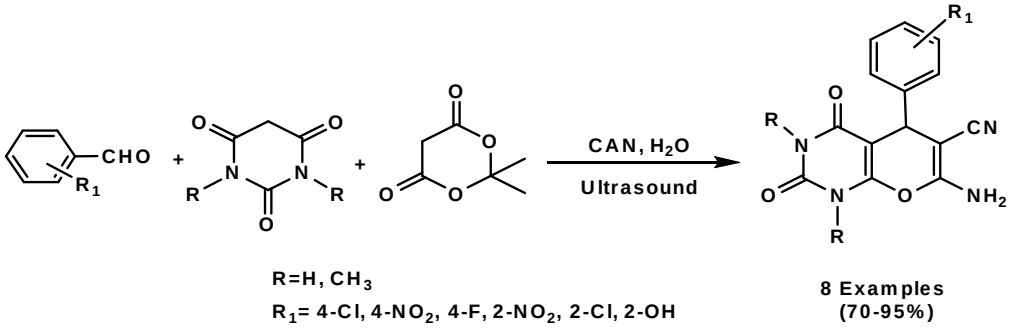
2.4. Ultrasonic-assisted reactions

The use of ultrasound to promote chemical reactions is called sonochemistry. Ultrasound method has surprisingly been used in organic synthesis in the three last decades. Compared with traditional protocols, the technique is more efficient and easily controlled [67]. A wide range of organic reactions can be accomplished in shorter reaction times, higher yields, or milder conditions under ultrasonic irradiation [68].Jin research group developed a clean, mild and good one-pot protocol based on the use of ultrasonic irritation technique in water as solvent (Scheme 10) [69].Efficient condensations were observed with aromatic aldehydes, malononitrile and barbituric acid substrates under mild conditions without use of any metallic and basic catalyst.



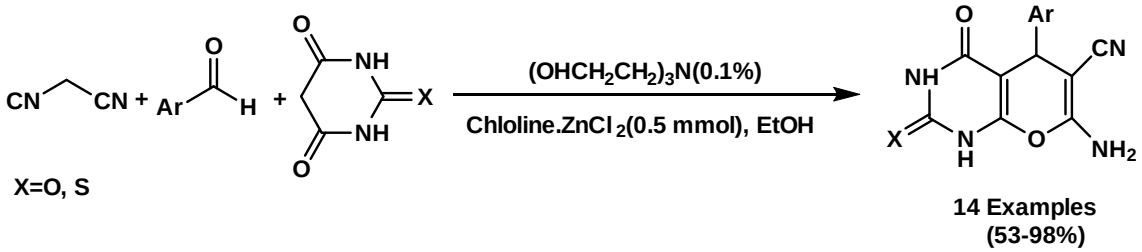
Scheme 10. Synthesis of Pyrano [2,3,d] Pyrimidines derivatives under ultrasonic irradiation.

A very efficient synthesis of Pyrano[2,3-d] pyrimidine derivatives can be achieved using cerric ammonium nitrate (CAN) in an aqueous medium under ultrasonic irradiation (Scheme 11) [70].

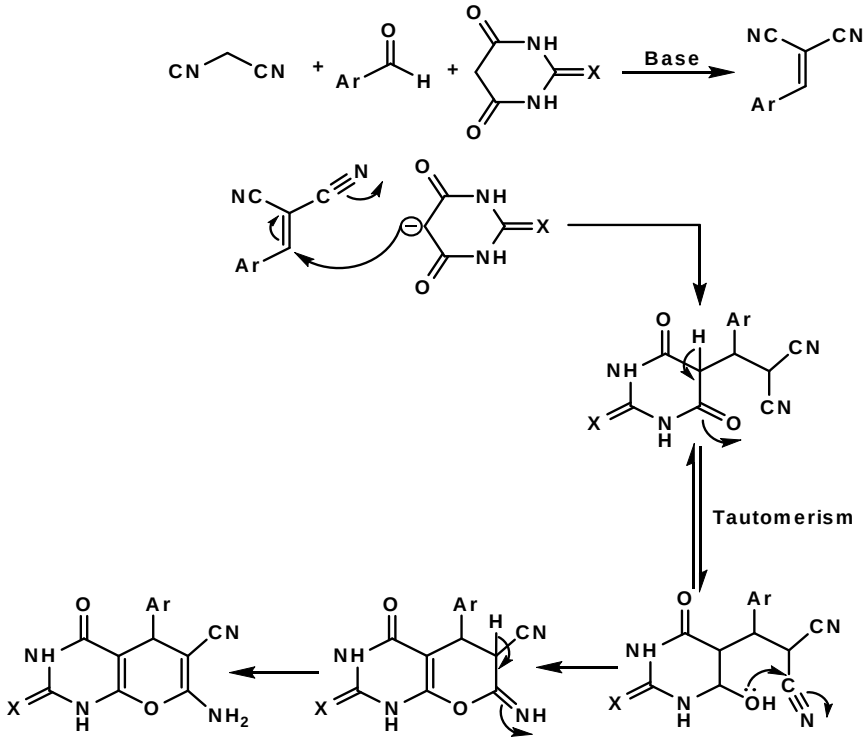


Scheme 11. Synthesis of Pyrano [2,3,d] Pyrimidines derivatives catalyzed by CAN under ultrasonic irradiation.

In one of the most valuable strategies for synthesis of Pyrano[2,3-d] pyrimidines, Choline chloride.$ZnCl_2$ was also introduced as a green and efficient reusable ionic liquid for the condensation of aromatic aldehydes, malononitrile and barbituric acid or 2-thiobarbituric acid (Scheme 12) [71]. The suggested mechanism for the synthesis of pyrano [2, 3-*d*] pyrimidines has been demonstrated in Scheme 13.



Scheme 12. Synthesis of Pyrano [2,3,d] Pyrimidines derivatives catalyzed by Choline chloride $ZnCl_2$ under ultrasonic irradiation.

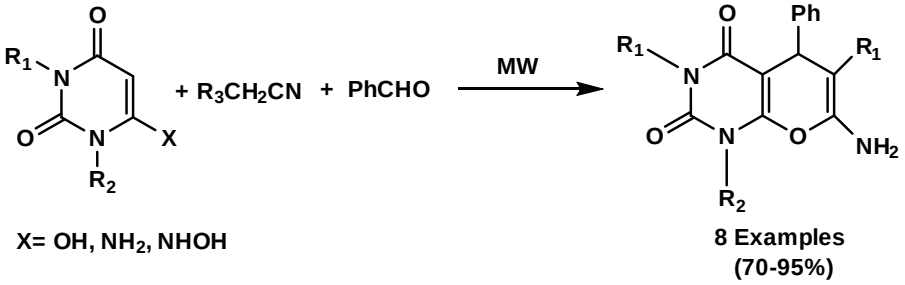


Scheme 13. Plausible mechanism for the synthesis of Pyrano [2,3,d] Pyrimidines under microwave conditions.

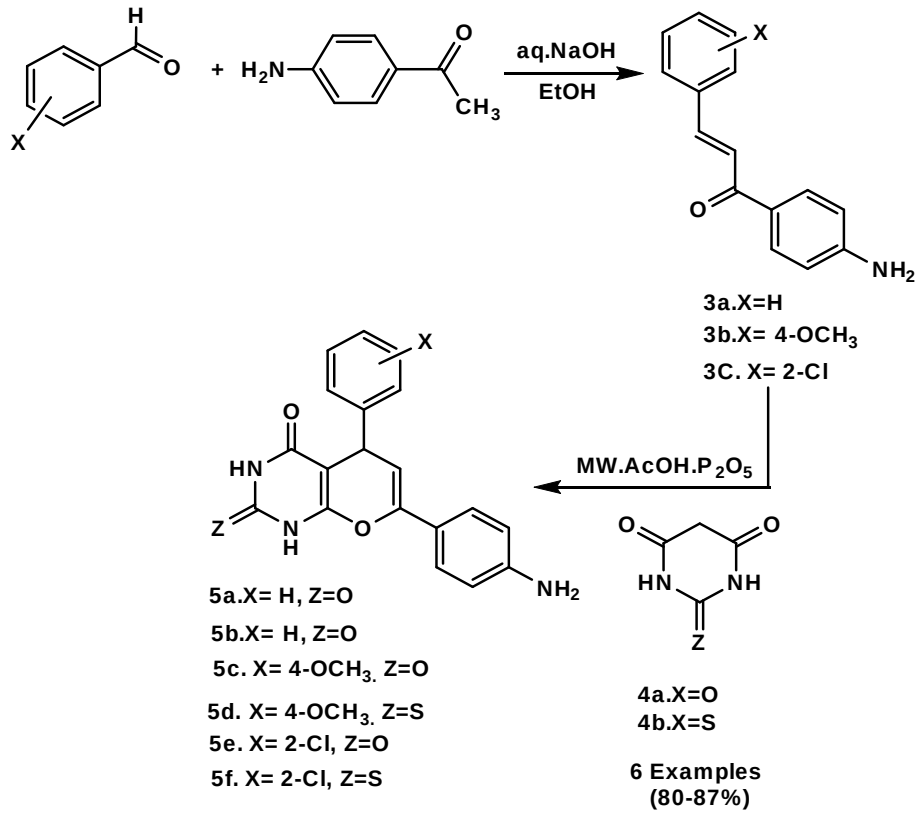
2.5. Microwave-assisted reactions

In the last few decades, many important advances in practical aspects of organic chemistry have included modern synthetic strategies and methods as well as advent of an extensive array of analytical strategies. In these environmentally conscious days, the advances in the technology are conducted towards environmentally sound and cleaner protocols. Therefore, the present day chemists are no longer restricted to using only thermal energy for driving chemical reactions [72]. The use of microwave technology in the performance of organic reactions, for the first time was reported in the mid-1980s [73]. For this purpose, Green chemistry concentrates on the principle of microwave activation, various types of microwave technology and its characteristics [74]. The microwave technique suggests easy, clean, rapid, efficient, and economic for the synthesis of a large number of organic compounds, have presented the momentum for chemistry researchers to change from traditional heating procedure to microwave assisted chemistry [75]. During recent years, microwave technologies have been widely applied for accomplishing chemical reactions and have become a beneficial non-conventional energy source for performing organic synthesis [76]. For example, Devi and his co-workers reported a new and convenient multicomponent reaction in the solid state to synthesize pyrano[2,3-*d*]pyrimidines in excellent yields [77]. In this protocol, microwave irritation was successfully employed in a polar medium for the cyclocondensation of

barbituric acids, benzaldehyde and alkyl nitriles (Scheme 14).A series of pyrano[2,3-*d*]pyrimidines derivatives was also prepared in aqueous medium under microwave conditions (Scheme 15) [78]. Products were isolated in high yields (81-87%) and in less than 8 min. To synthesize pyrano[2,3-*d*]pyrimidines derivatives, a valuable and practical method under same condition has also been described [51].

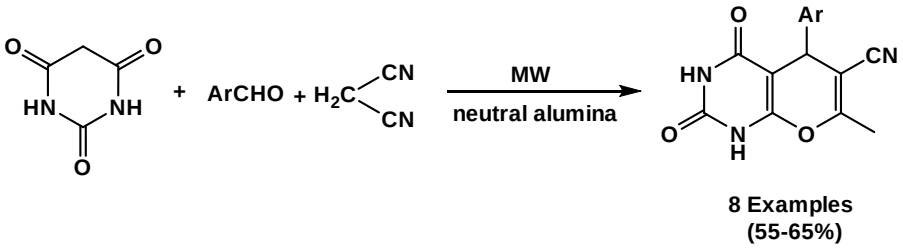


Scheme 14. Synthesis of Pyrano [2,3,d] Pyrimidines derivatives under microwave conditions.

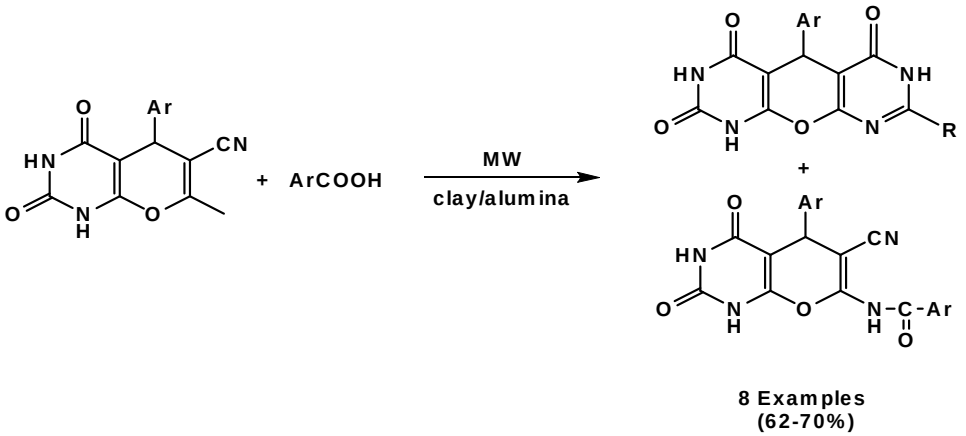


Scheme 15. Synthesis of Pyrano[2,3,d]Pyrimidine derivatives in aqueous medium under microwave conditions.

Neutral alumina is also an interesting reagent in condensation reactions. In the year of 2007, An efficient procedure was reported for the synthesis of pyrano[2,3-*d*]pyrimidines based on using neutral alumina under microwave irritations (Scheme 16) [79]. In this strategy, pyranodipyrimidine derivatives were synthesized by reaction of pyrano[2,3-*d*]pyrimidines with various aromatic carboxylic acids (Scheme 17).



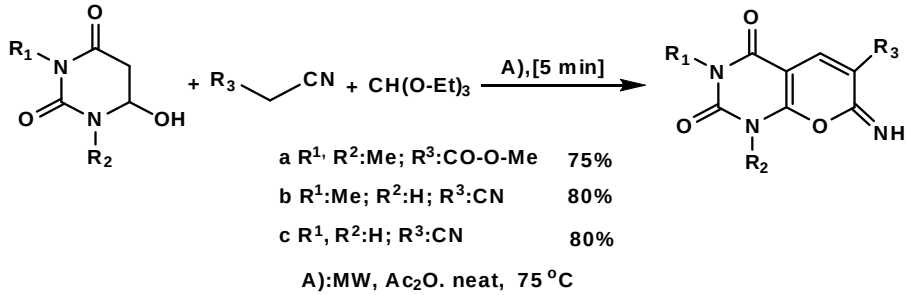
Scheme 16. Synthesis of Pyrano [2,3,d] Pyrimidines derivatives using neutral alumina under microwave conditions.



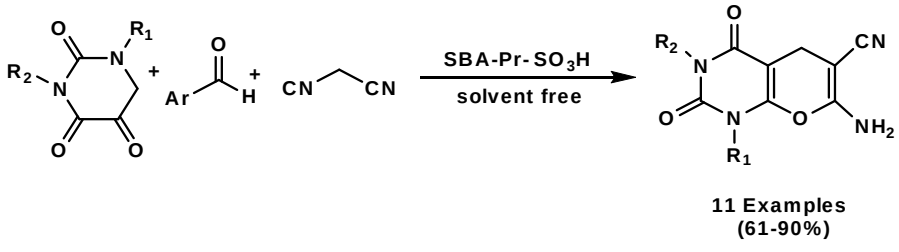
Scheme 17. Synthesis of Pyrano [2,3,d] Pyrimidines derivatives under microwave conditions.

2.6. Solvent-Free Synthesis

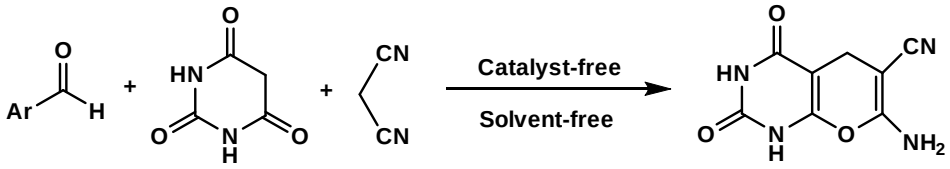
In general, Changing of solvent of a reaction can affect the rate of that reaction, and it can be strong enough to change the reaction direction itself. Although a series of new solvents, likeionic liquids and water have been widely recently, not using a solvent at all is certainly the best selection [80-81]. Solvent-free synthesis is obtaining prominence as a means for the preparation of a wide range of beneficial and important compounds, with the number of reactions directed under these conditions increasing [82]. Recently remarkable attention has been paid to the solvent free reactions. These are not only of attractive from an environmental point of view, but in many cases they also suggest significant synthetic features in terms of yields, selectivity and simplicity of the reaction strategies [83-84]. These factors are in particular valuable in industry field. Therefore, some the traditional organic synthetic procedures, which have long been accomplished in solvents, may be amended to more modern, efficient, and safe versions [85-86]. In the last 20 years, a large number of protocols have been reported based on using microwave technique under solvent-free conditions. For example, a diverse range of pyrano[2,3-*d*]pyrimidines were prepared in good yield using acetic anhydride under neat conditions (Scheme 18) [87]. Sulfonic acid nanoporous silica (SBA-Pr-SO_3H) under solvent-free condition is an efficient and popular catlytic system for the condensation of aromatic aldehydes, *malononitrile*, and barbituric acid (Scheme 19) [88]. SBA-Pr-SO_3H was used as an efficient, recycable and active nano-reactor to synthesize pyrano[2,3-*d*]pyrimidines. An industrial and valuable process was also developed to synthesize pyrano[2,3-*d*]pyrimidine derivatives in the presence of ball-milling under without use of any organic or basic catalyst[89].The condensation reactions of aromatic aldehydes, malononitrile, and barbituric acid were carried out in less than 90 min under solvent-free condition (Scheme 20).Products were isolated in excellent yields (94-99%).



Scheme 18. Synthesis of Pyrano [2,3,d] Pyrimidines derivatives under solvent-free conditions.

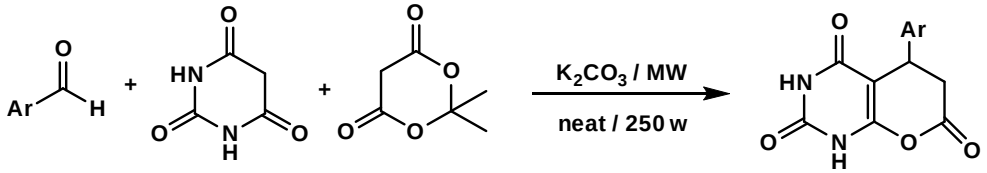


Scheme 19. Synthesis of Pyrano [2,3,d] Pyrimidine derivatives by using SBA-Pr-SO_3H under solvent-free condition.



Scheme 20. Synthesis of Pyrano [2,3,d] Pyrimidines derivatives under catalyst and solvent-free conditions.

Recently, attractive and excellent protocol reported by Pasha in which involved potassium carbonate under microwave condition for the condensation of aromatic aldehydes, meldrums acid, and barbituric acid (Scheme 21) [90]. The reactions were performed at 100 ^{0}C under neat condition. Several valuable indices such as short reaction times, exxcellent yields, simple reaction conditions and environmentally friendly demonstrated the excellent efficiency of this methodology for the synthesis of pyrano[2,3-*d*]pyrimidines.



Scheme 21. Synthesis of Pyrano [2,3,d] Pyrimidines derivatives by using k_2CO_3 under solvent-free conditions.

Table 1. The comparison of efficiency of various protocols in the synthesis of Pyrano [2,3,d] Pyrimidines derivatives.

Entry	Conditions	Examples	Time(min)	Yield
1	DAHP (10 mol%),aq. EtOH, r.t.	16	120	80-90
2	*L*- Proline (5 mol%),aq. EtOH, r.t.	16	30-150	68-88
3	TBAB(10%mol)H_2O,Reflux	8	25-35	80-90
4	DABCO 5mol%,EtOH,H_2O	12	30-50	84-97
5	Triethylamine (2-3drops) EtOH:H_2O, r.t	10	43-110	69-94
6	Alum (10% mol),H_2O, 80 ^{0}C	8	30-45	80-90
7	NaOAc (5mol%),EtOH, H_2O, 50◦C	1	100	98
8	Ionic Liquids, 90 ^{0}C	5	60-600	46-84
9	Ultrasound,H_2O, 90 ^{0}C	5	60-180	62-87
10	CAN/H_2O	8	29-40	70-95
11	$(OHCH_2CH_2)_3N$(0.1%).$ZnCl_2$(0.5 mmol), EtOH	14	1-8	53-98
12	Mw, DMF, Et_3N	8	4-8	70-95
13	NaOHEtOH aq. AcOH, P_2O_5, Mw	6	5-8	80-87
14	Mw,Neutral alumina	8	4-5	55-65
15	Mw,Clay/alumina	8	4-5	62-70
16	Mw,Ac_2O.neat, 75 ^{0}C	3	5	75-80
17	SBA-Pr-SO_3H,Solvent-free	11	4-45	61-90
18	Catalyst and solvent-free	8	15-90	94-99
19	K_2CO_3/MW, Neat/250 W	12	1-3	95-97

The obtained results in the synthesis of Pyrano [2,3,d] Pyrimidines derivatives was listed in Table 1. The best results (in terms of number of examples, product yields, reaction time and conditions) were obtained under microwave and solvent-free conditions.

Conclusion

As described in this paper, pyrano [2,3,d] pyrimidine derivatives are very important, effective and valuable compounds in medicine and pharmaceutical industries. Therefore, synthesis of these compounds has received much attention among organic researchers. During recent years, to synthesize pyrano [2,3,d] pyrimidine derivatives, various traditional protocol have been developed. Despite great significance of these protocols in organic synthesis, unfortunately, most of them have main disadvantage such as unsatisfactory yields, formation of side product, harsh reaction conditions, use of strong bases, and environmentally unfriendly due to use of an hazardous and toxic organic solvent as reaction medium.

Green chemistry promotes a modern idea for accomplishing chemical research and productions. With enhancing public concerns on environmental protection, chemistry science researchers are asked to maximize the advantages of chemical products and minimize the side effects that could be harmful to the environment and humans. Organic reactions conducting under nontraditional conditions (green synthetic strategies) are obtaining in popularity, primarily to decrease environmental concerns. The advantages of green synthetic strategies facilitated (assisted) organic transformations, namely the selectivity, simplicity of experimental manipulation, and increased reaction rates, were highlighted. The advantage of these strategies is obvious (clear); it is not only useful for the world economy, but also acts toward making the world more environmentally friendly. We hope more and more green examples will become available in the future.

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