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Solvent-Free Acylation of Alcohols, Phenols, Thiols and Amines

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ABSTRACT

The hazardous and toxic nature of many solvents, in particular organic solvents that are extensively used in large scale for organic reactions have transformed a serious threat to the environment. Therefore, the design of solvent-free catalytic reaction has received considerable attention during recent times in the field of green synthesis. A solvent-free or solid state reaction may be accomplished using the reactants single or merge them in clays, zeolites, silica, alumina or other substrates. Esters, thioesters and amides are important and valuable compounds in the area of industry, medicine, pharmaceutical and heterocyclic chemistry. In this paper, various solvent-free systems to synthesize esters, thioesters and amides via the acylation of alcohols, phenols, thiols and amines are described. In this review we summarized the activities presented mainly the recent years.

GRAPHICAL ABSTRACT

1. Introduction

The chemistry of organic synthesis attracts special attention in science basically because of its great significance to life. The tremendous flexibility of bonding at carbon is suitable to a wide range of structures in order to support living systems. Organic synthesis is one of the most significant and effective disciplines of chemistry, which generally involves the formation and cleavage of carbon–carbon (C–C) and carbon–heteroatom (C–X) bond. The synthetic techniques regarding how to manufacture and cleave the above bonds represent the central issue in organic synthesis.

A common presumption with regard to organic reactions is that they are carried out in a solvent medium.[1] Solvents are very significant and effective as liquid medium for reactions to take place, and then the generation of a chemical product for extraction, isolation, purification and drying. Solvents not only are very vital and valuable for the chemical industry also play an important role in synthesis of pharmaceutical active materials and antibiotics.[2] The most of solvents are organic chemicals with harmful and toxic features, costly (part of the petrochemical industry) and part of the large waste side products of the chemical industry causing environmental problems.[3] Due to the growing concerns for the effect of the solvents on the environment as well as on human health, the performance of organic reactions without use of toxic organic solvents have attracted the great attention among organic researchers. Green Chemistry assists to shift the use of toxic solvents with greener alternatives, with replacement and synthetic strategies, isolation and purification which do not need the use of hazardous and toxic solvents.[4] One of the principles of green chemistry asks us to 'use greener solvents and auxiliaries'.

Solvent use also impacts some of the other principles and therefore, it is not surprising that in the last decades, chemistry research into the use of greener, alternative solvents has grown tremendously. If feasible, the use of solvents should be removed, or if they cannot be removed, we should try to use harmless materials instead.[5] Esters, thiol esters and amides are very valuable and effective intermediates in the biological processes, agriculture, medicine, industry, bio-chemistry and heterocyclic chemistry.[6-8] Also, these compounds play an important role in the synthesis of natural products and poly functional molecules such as nucleosides, carbohydrates, and steroids.[9-11] The acylation of alcohols, phenols, thiols and amines is one of the most common and valuable transformations in organic synthesis.[12] The strategy

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also provides a convenient and inexpensive route for protecting hydroxyl and amino groups in a multistep synthetic process.[13-14] Over the last hundred years, the acylation of alcohols, phenols, thiols, and amines have been extensively investigated by organic researchers. During recent years, a large number of protocols have been described for the synthesis of esters, thiol esters and amides.[15-35] Now, in this paper, we wish to focus on the acylation of alcohols, phenols, thiols and amines under solvent-free conditions.

1.1. The Function of a Solvent

A generic presumption with regard to organic reactions is that they are carried out in a solvent medium. The logic behind this idea is easy.[36] That is, the reactants can interact efficiently if they are in a homogeneous solution, which simplifies the stirring, shaking or other ways of agitation, whereby the reactant molecules come together quickly and continuously.[37] In addition to, uniform heating or cooling of the mixture, if needed, can be accomplished in a solution relatively readily.[38] A solvent has the power to increase or decrease the velocity of a reaction, at times tremendously. Changing of solvent of a reaction can affect the rate of that reaction and it can be strong enough to shift the reaction direction itself. This may manifest in modified yields and ratios of the products.[39] Therefore, a solvent could be profoundly and inseparably associated with the process of an organic reaction via the solvation of the reactants, products, transition-state or other interfering components.

1.2. Liquid as a Solvent

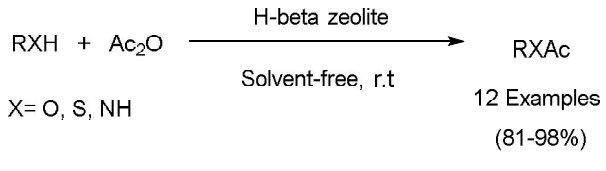
Basically, any liquid can be applied as a solvent. However, the number of generally used solvents is severely restricted.[40] They include a few ethers, esters, alcohols, amide derivatives, sulphoxides, liquid ammonia mostly applied as medium to accomplish organic reactions.[41] The selection of a solvent for the accomplishment of reaction depends on various factors such as solvent physical and chemical properties.[41] The liquid reactant occasionally acts as solvent or reaction medium. The overall, a solvent is always considered to be an indispensable component of a reaction.[41] A reaction under solvent free condition or solid state was commonly conceived to be not quite practical, or at least not quite convenient, though several solid state organic reactions have been known for a long time.[42] However, as mentioned in above, the organic researchers concern for developing environment-friendly synthetic methods has made them turns their attention to reduce or circumvent the use of solvents that are a main reason of pollution. This has led, during recent decades, to intense research activity and reinvestigation of known reactions to perform organic reactions in the absence of solvent (under solvent free-conditions).

1. 3. Advantages of Solvent-free Reactions

The performance of organic and chemical reactions in the presence single reagents or merge them in other substrates such as clays is a type of solvent-free and solid state reactions.[41] The performance of organic reactions in the absence of solvent can also accomplish using microwave and ultrasound technologies.[42] Solvent-free or solid state reactions clearly decrease pollution and reduce costs because of facilitation of experimental method, work up strategy and saving in labor.[42] These would be in particular significant during industrial production. In general, the products of solid state reactions transform to be different from those generated in solution phase reactions due to particular spatial orientation or packing of the reacting compounds in the crystalline state.[41-43]

2. Solvent-free Acylation Reactions

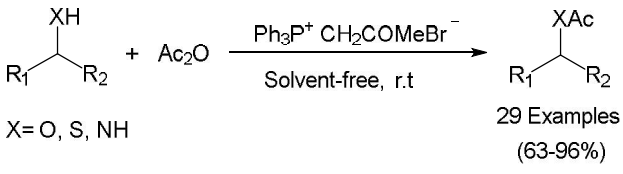
Recently, solvent free reactions have been received particular attention worldwide.[44] These are not only of valuable from an ecological point of view, but they also offer substantial synthetic benefits in terms of yields, selectivity and simplicity of the reaction protocols.[45] Therefore, some the traditional organic synthetic protocols, which have long been performed in solvents, may be modified to more modern, elegant, and safe versions.[46]



Scheme 1. Solvent-free acylation of alcohols, thiols and amines catalyzed by H-beta zeolite.

During last decade, a series of protocols were reported for the acylation alcohols, phenols, thiols and amines based on use of solvent-free or solid state conditions. For example, Bhaskar and Loganthan exploited this technique to synthesize esters, thioesters and amides in excellent yields and suitable times.[47] They used from H-beta zeolite at room temperature as a new and efficient catalyst for the performance of reactions **(Scheme 1)**.

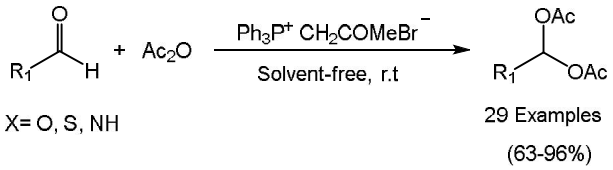
Under solvent-free conditions, a general and practical method has been extended to synthesize esters, thioesters and amides derivatives.[48] Acetonyltriphenylphosphonium bromide (ATPB; 5 mol%) at room temperature was designed as an efficient and versatile system for the acylation of alcohols, phenols, thiols and amines **(Scheme 2)**.



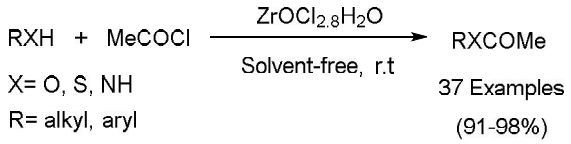
Scheme 2. Solvent-free acylation of alcohols, thiols and amines using ATPB.

The acylation of aldehydes with acetic anhydride was successfully carried out under same conditions **(Scheme 3)**. The acylation of heteroatoms (O, N and S) with acetyl colride can be achieved using $ZrOCl_2.8H_2O$ under solvent free conditions **(Scheme 4)**.[49] The yields of crude product were in the range 91–98%. Zinc oxide (ZnO) is a highly efficient and

reusable catalyst for the synthesis of esters and amides under solvent-free conditions **(Scheme 5)**.[50]

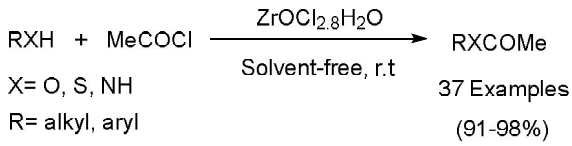


Scheme 3. Solvent-free diacylation of aldehydes using ATPB.

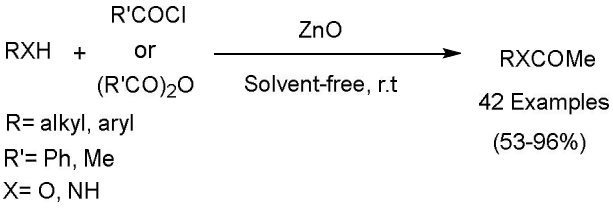


Scheme 4. Solvent-free acylation of alcohols, thiols and amines catalyzed by $ZrOCl_2.8H_2O$.

In this system, the catalyst was successfully employed for acylation of a wide range of alcohols, phenols and amines. In the case of alcohols and phenols, an acid chloride was preferred over the corresponding acidic anhydride. The reaction with acid anhydride was too slow to have practical application. Primary and secondary alcohols treat very good and tertiary alcohol is also acylated softly without formation of by-products. Under same conditions, in another reports, *O*-acylation of alcohols and phenols with acid chlorides were carried out using ZnO and nano ZnO.[51-53]

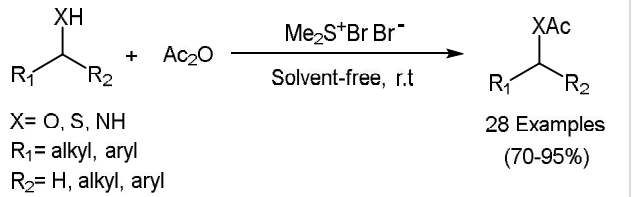


Scheme 4. Solvent-free acylation of alcohols, thiols and amines catalyzed by $ZrOCl_2.8H_2O$.



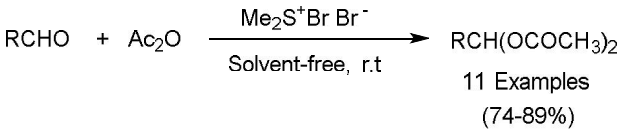
Scheme 5. Solvent-free acylation of alcohols and amines catalyzed by ZnO.

Bromodimethylsulfonium bromide is an attractive reagent in organic synthesis. In the year of 2005, Khan and his co-workers utilized from this reagent for the acylation of alcohols, phenols, thiols and amines.[54] The reactions were carried out, under solvent-free conditions, in less than 3 h at room temperature **(Scheme 6)**. Efficient acylation of aldehydes with acetic anhydride was also carried out under same conditions **(Scheme 7)**.

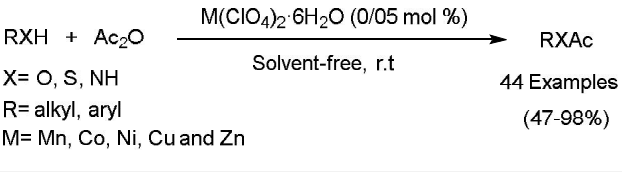


Scheme 6. Solvent-free acylation of alcohols, thiols and amines using Bromodimethylsulfonium.

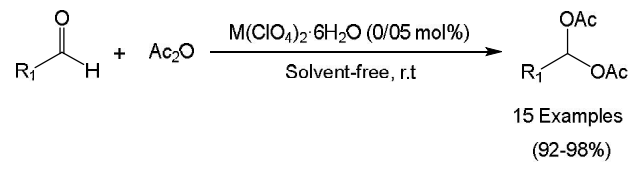
Jeyakumar and Chand surprisingly discovered that acylation of alcohols, phenols, thiols and amines could be also carried out simply in the presence of metal perchlorates.[55] Corresp-onding products were synthesized at room temperature using $M(ClO_4)_2{\cdot}6H_2O$ as catalyst where M is Mn, Co, Ni, Cu and Zn under solvent-free conditions **(Scheme 8)**. The best results were obtained when copper perchlorate was used as catalyst (93-97%). Diacyaltion of aldehydes was also performed efficiently under same conditions **(Scheme 9)**.



Scheme 7. Solvent-free acylation of aldehydes using Bromodimethy-lsulfonium.

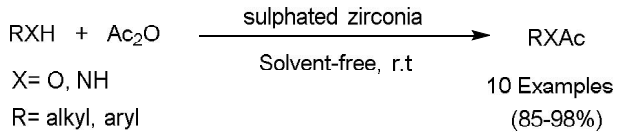


Scheme 8. Solvent-free acylation of alcohols, thiols and amines catalyzed by metal perchlorates.



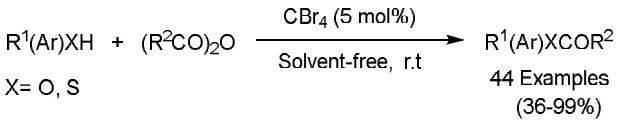
Scheme 9. Solvent-free diacylation of aldehydes catalyzed by metal perchlorates.

In recent 20 years, zirconia derivatives have received particular attention for the acylation of organic compounds. For instance, the acylation reactions of alcohols, phenols and amines with acetic anhydride (acylating agent) were accomplished readily in the presence of sulphated zirconia **(Scheme 10)**.[56] Under solvent-free conditions, 4-dimethyl-aminopyridine (DMAP) was also used for the esterification of alcohols.[57]

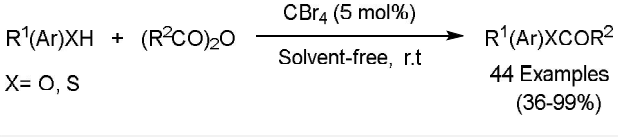


Scheme 10. Solvent-free acylation of alcohols and amines using sulphated zirconia.

Under metal and solvent-free conditions, Zhang research group reported that a convenient and valuable catalyst carbon tetrabromide (CBr_4) was found to be highly efficient for the acylation of phenols, alcohols and thiols with acetic anhydride **(Scheme 11)**.[58] A one-pot synthesis of esters and amides derivatives has been reported in high yield via the esterification of alcohols, phenols and amines under solvent-free conditions.[59] In this protocol, zinc perchlorate hexahydrate [$Zn(ClO_4)_2{\cdot}6H_2O$] was acted as an effective catalyst to perform esterification reactions **(Scheme 12)**.

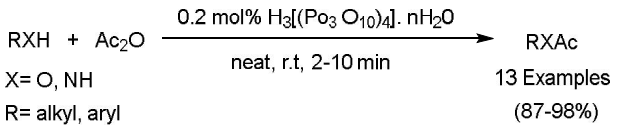


Scheme 11. Solvent-free acylation of alcohols and thiols catalyzed by carbon tetrabromide.

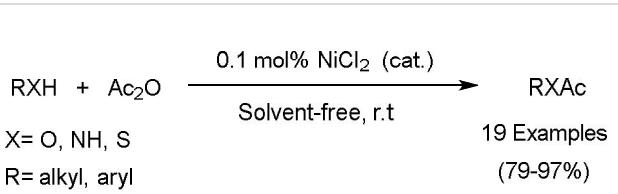


Scheme 12. Solvent-free acylation of alcohols and amines catalyzed by zinc perchlorate hexahydrate.

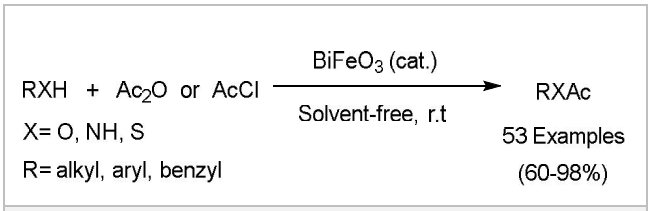
In the year of 2008, Kadam and Kim developed a useful and dependable procedure for the O and *N*-acylation of alcohols, phenols and amines in one-pot using phosphom-olybdic acid (mild and efficient catalyst) in the absence of any solvent **(Scheme 13)**.[60] The acylation of alcohols, phenols, thiols and amines with acetic anhydride has been achieved in the presence of 0.1 mol% nickel (II) chloride ($NiCl_2$) under solvent-free conditions to afford the corresponding esters, thioesters and amines derivatives in high yields **(Scheme 14)**.[61] A similar approach for the acylation of alcohols, phenols, thiols and amines has also been reported.[62-63] A series of esterification reactions of alcohols, phenols, thiols and amines was observed using bismuth ferrite ($BiFeO_3$) nanopowder in the absence of any solvent **(Scheme 15)**.[64]



Scheme 13. Solvent-free acylation of alcohols and amines catalyzed by $NiCl_2$.

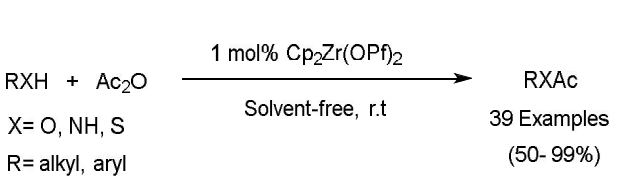


Scheme 14. Solvent-free acylation of alcohols, thiols and amines catalyzed by phosphomolybdic acid.



Scheme 15. Solvent-free acylation of alcohols, thiols and amines catalyzed by $BiFeO_3$.

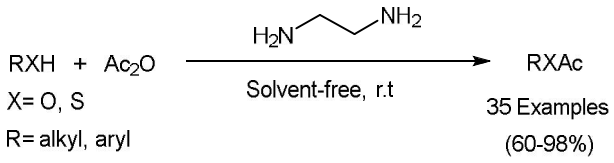
In continuation the use of zirconia derivatives to carry out acylation reactions, Qiu and his co-workers developed a practical and effective protocol for the synthesis of esters, thioesters and amides based on using zirconocene bis(per-fluorooctanesulfonate)s [$Cp_2Zr(OPf)_2$, OPf= $OSO_2C_8F_{17}$] under solvent-free condition **(Scheme 16)**.[65] Interesting advantages such as high yields, high reactivity, simple purification of the products, clean reaction medium and compatibility with solvent-free conditions demonstrated high efficiency of this protocol for the performance of esterification reactions.



Scheme 16. Solvent-free acylation of alcohols, thiols and amines catalyzed by $Cp_2Zr(OPf)_2$.

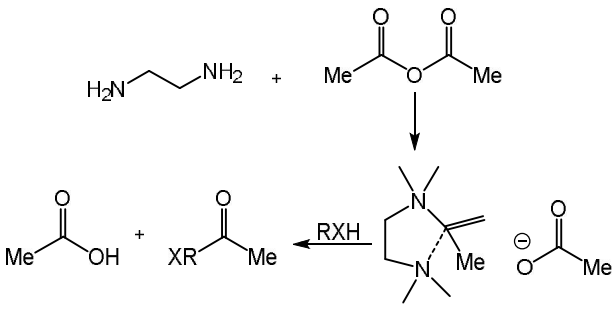
The use of *N,N,N′,N′*-tetramethylethylenediamine (TMED-A) under solvent free condition is a suitable and valuable means for the O and *S*-acylation alcohols, phenols and thiols

with acetic anhydride (Scheme 17).[66] Base on suggested mechanism, initially the reaction started with the formation of acyl TMEDA cation, from reaction of TMEDA with acetic anhydride, and followed with reaction of alcohol or thiol with acylated TMDA to form ester and thioester product (Scheme 18).

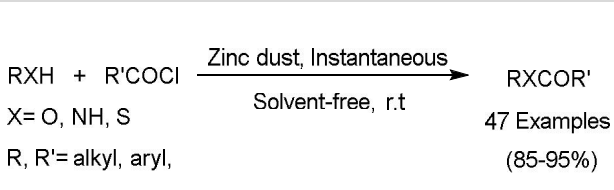


Scheme 17. Solvent-free acylation of alcohols and thiols using TMEDA.

Zinc dust is as an extremely efficient and reusable catalyst for the esterification reactions. For example, in the year of 2010, Afzal Pasha and his co-workers reported an interesting and valuable protocol for the acylation of alcohols, phenols, thiols and amines using zinc dust in the absence of any solvent (Scheme 19). The reactions were carried out in less than 2 min at room temperature.[67]

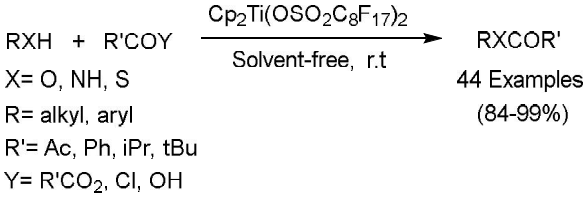


Scheme 18. Suggested mechanism for solvent-free acylation of alcohols and thiols using TMEDA.

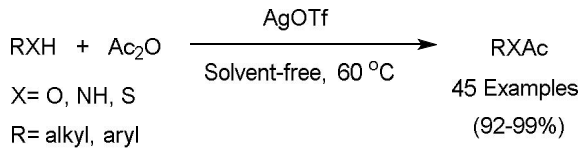


Scheme 19. Solvent-free acylation of alcohols, thiols and amines catalyzed by Zinc dust.

The reactions were carried out in less than 2 min at room temperature. Titanocene bis(perfluorooctanesulfonate) [$Cp_2Ti(OSO_2C_8F_{17})_2$] in the absence of solvent is also tremendous and popular system to synthesize esters, thioesters and amines in excellent yields (Scheme 20).[68] Silver triflate catalysis under solvent-free conditions was accomplished to achieve the one-pot synthesis of esters, thioesters and amines (Scheme 21). This protocol is an improvement on the existing protocol because products were obtained in very excellent yields and suitable times.[69]

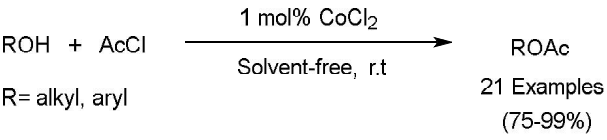


Scheme 20. Solvent-free acylation of alcohols, thiols and amines catalyzed by $Cp_2Ti(OSO_2C_8F_{17})_2$.



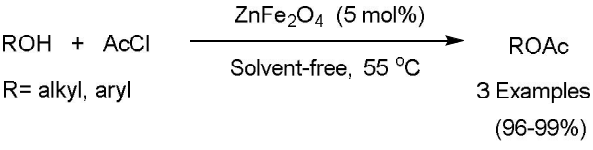
Scheme 21. Solvent-free acylation of alcohols, thiols and amines catalyzed by silver triflate.

Cobalt (II) chloride is an attractive Lewis acid for the acylation of alcohols. In this regard, Mulla research group exploited this reagent under solvent-free conditions to synthesize various ester derivatives (Scheme 22).[70]



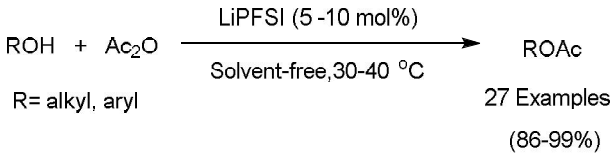
Scheme 22. Solvent-free acylation of alcohols catalyzed by Cobalt (II) chloride.

Nanoparticles are one of the most interesting and efficient reagents in chemistry science in particular organic synthesis.[71-74] $ZnFe_2O_4$ nanoparticles are valuable reagents in synthesis of organic compounds. In the year of 2012, a good and useful protocol was reported for the synthesis of esters based on using $ZnFe_2O_4$ nanoparticles in the absence of solvent (Scheme 23).[75]



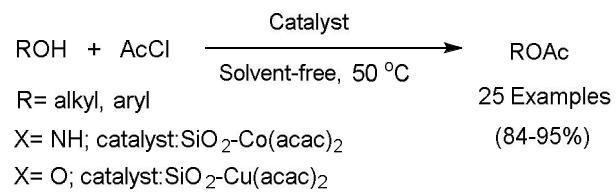
Scheme 23. Solvent-free acylation of alcohols catalyzed by $ZnFe_2O_4$.

An convenient method with the same reagents and in the presence of lithium bis(perfluoroalkylsulfonyl)-imide (LiPFSI) has also been developed **(Scheme 24)**.[76]



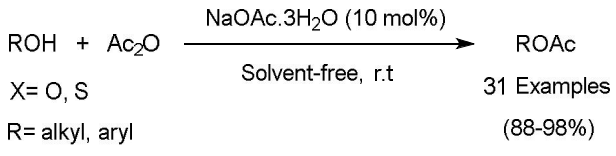
Scheme 24. Solvent-free acylation of alcohols using LiPFSI.

Another practical and highly efficient strategy was also described with SiO_2-$Co(acac)_2$ and SiO_2-$Cu(acac)_2$ under solvent-free conditions **(Scheme 25)**.[77] The acylation of alcohols, phenols and amines with acetyl chloride was carried out in less than 60 min at 50 ^{0}C. The esterification of alcohols and amines was performed respectively with SiO_2-$Cu(acac)_2$ and SiO_2-$Co(acac)_2$.



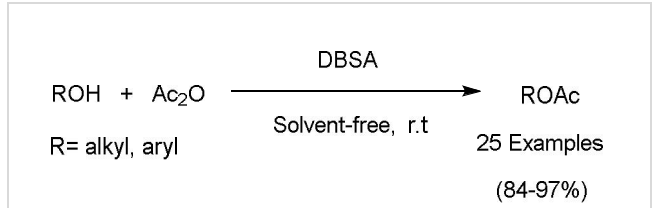
Scheme 25. Solvent-free acylation of alcohols and amines using SiO_2-$Co(acac)_2$ and SiO_2-$Cu(acac)_2$.

A synthesis of esters and thioesters derivatives has been described using sodium acetate trihydrate as efficient catalyst, from esterification of alcohols, phenols and thiols under solvent-free conditions **(Scheme 26)**.[78] It is noteworthy that esters and thioesters were synthesized in high yields and short times. The transformation of tertiary alcohols to corresponding esters was the most important advantage this system.



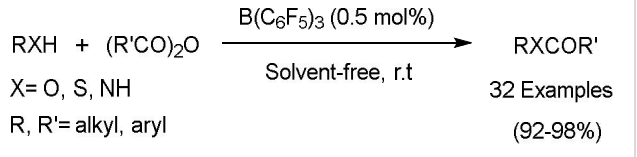
Scheme 26. Solvent-free acylation of alcohols and thiols catalyzed by sodium acetate trihydrate.

Recently, Dodecylbenzenesulfonic acid (DBSA) was introduced as an effective, inexpensive and chemoselective and stable Bronsted catalyst to synthesize esters via for O-acylation of alcohols and phenols and under solvent-free conditions **(Scheme 27)**.[79] The reactions were performed in various solvents, such as CH_2Cl_2, H_2O, $CHCl_3$, CH_3CN and CH_3CO_2Et but the best results were obtained under solvent-free conditions. The catalyst can be readily separated and is also recyclable.

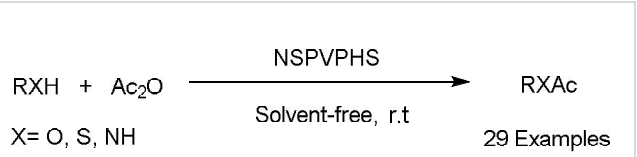


Scheme 27. Solvent-free acylation of alcohols and thiols catalyzed by sodium acetate trihydrate.

Boron derivatives have been frequently used to carry out acylation reactions. For instance, a solvent-free acylation of alcohols, phenols, thiols and amines by tris (pentafluorophenyl)borane [$B(C_6F_5)_3$] as catalyst has been developed in high yields **(Scheme 28)**.[80] Very recently, Khaligh and Ghasem-Abadi described the use of *n*-Sulfonic acid poly4-vinylpyridinum) hydrogen sulfate as recyclable acid catalyst for the acylation of alcohols, thiols and amines under solvent-free conditions **(Scheme 29)**.[81] Under same condition, efficient acylation of aldehydes was also observed **(Scheme 30)**.



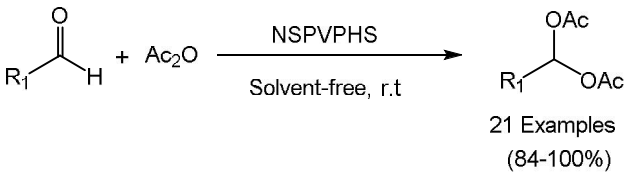
Scheme 28. Solvent-free acylation of alcohols, thiols and amines catalyzed by [$B(C_6F_5)_3$].



Scheme 29. Solvent-free acylation of alcohols, thiols and amines catalyzed by *n*-Sulfonic acid poly4-vinylpyridinum) hydrogen sulfate.

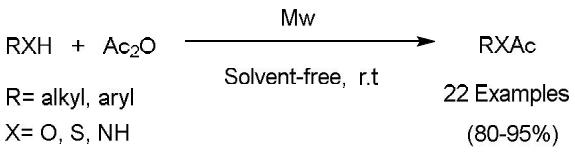
The microwave strategy offers easy, clean, rapid, efficient, and economic for the synthesis of a large number of organic compounds, have presented the momentum for chemistry researchers to change from traditional heating procedure to microwave assisted chemistry.[82-83]

The performance of reactions without solvent under microwave conditions is a powerful tool in organic synthesis. For example, the acylation of alcohols, thiols and amines was successfully performed in the absence of solvent and catalyst under microwave conditions **(Scheme 31)**.[84] It is considerable that products were generated in high yields and suitable times.

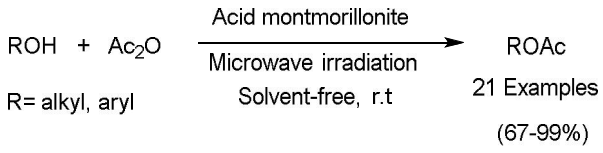


Scheme 30. Solvent-free diacylation of aldehydes catalyzed by *n*-Sulfonic acid poly4-vinylpyridinum) hydrogen sulfate.

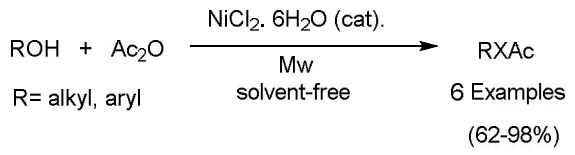
In the year of 2004, the synthesis of acetates by rapid acylation of alcohols, phenols and amines an in the presence of acid montmorillonite using microwave technology under solvent-free condition has been reported **(Scheme 32)**.[85] In the presence of nickel (II) chloride, the esterification of alcohols and phenols leads to the formation of esters upon exposure to microwave irradiation **(Scheme 33)**.[86] It was also mentioned that O and *N*-acylation of alcohols and amines readily accomplished in the presence of zeolite using microwave irradiation under solvent free-conditions **(Scheme 34)**.[87] Without use of solvent, the acylation of cellulose was also described in the presence of iodine as catalyst under microwave conditions.[88]



Scheme 31. Microwave assisted solvent-free acylation of alcohols, thiols and amines.

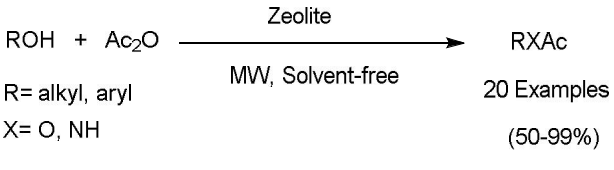


Scheme 32. Microwave assisted solvent-free acylation of alcohols in the presence of acid montmorillonite.

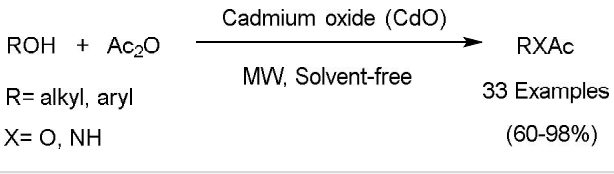


Scheme 33. Microwave assisted solvent-free acylation of alcohols in the presence of nickel (II) chloride.

Finally, Asghari and his co-workers introduced nanosized cadmium oxide as a modern and highly efficient catalyst for t-he solvent-free acylation of amines and alcohols **(Scheme 35)**.[89] The reactions were carried out in less than 20 min at 80 ^{0}C under microwave irradiation.



Scheme 34. Microwave assisted solvent-free acylation of alcohols and amines in the presence of zeolite.



Scheme 35. Microwave assisted solvent-free acylation of alcohols and amines in the presence of cadmium oxide.

3. Conclusion

As mentioned in above, the acylation of alcohols, thiols and amines is one of the most common and important reactions in organic synthesis because esters, thioesters and amines play a vital role in the synthesis of medicine, pharmaceutical and natural products. The foregoing short discussion demonstrates that a wide range of acylation reactions, which are traditionnally performed in solvent media, can be accomplished more profitably in the absence of solvents. This paper summarizes the recent advances and eco-friendly advantages of the solvent-free reactions in the acylation of alcohols, thiols and amines. The organic researchers will surely continue his effort to bring more and more reactions into the fold of the solvent-free synthetic methodology. As described presently, the solvent-free reactions can be performing not only at different temperatures, but also by exposing the reactants to microwave irradiation. We anticipate the development of main applications of solvent-free synthetic methodology for performance of organic reactions.

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