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Mini-Review Article

Revisiting of Boron Sulfonic Acid Applications in Organic Synthesis: Mini-Review

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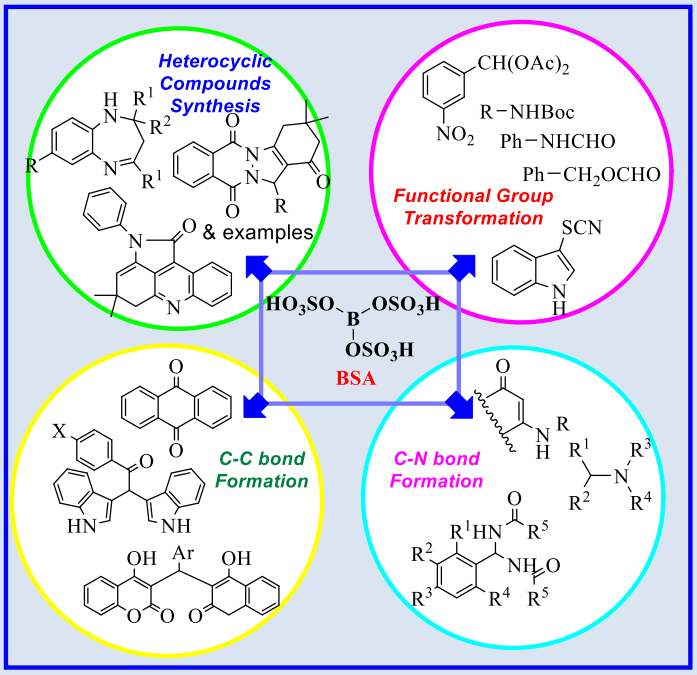
Abstract: Boron sulfonic acid (BSA) plays an important role in the improvement of many organic reaction conditions as a solid acid catalyst, which dramatically has attracted the attention of many chemists. This review covers all highlight research of literatures in the last decade from 2008 to 2018.

Key words: Boron Sulfonic Acid (BSA), Tris(hydrogensulfato)boron, Silica Boron Sulfonic Acid (SBSA), Organic Synthesis, Heterocyclic Compounds.

Graphical Abstract:



Biography:

Sami Sajjadifar was born in Malekshahi-Ilam, Iran, in **1973**. He studied chemistry at the university of Shahid chamran, Ahvaz, Iran, and recieved the M.S. degree in organic chemistry with Professor Mohammad Mehdi Baradarani at Urmia University, Urmia, Iran, in **2000** and the Ph.D degree also in organic chemistry with Professor Mohammad Ali Zolfigol at Bu-Ali Sina University, Iran, and Payame Noor of Mashhad, Iran, in **2012**. He focused his doctoral thesis on the application of Boron Sulfonic Acid (BSA) catalyst in organic synthesis. His research interests focus on the application of new reagents in organic reactions, the synthesis of different types of organic compounds, design, and study of novel solid acid catalyst and organic reaction.

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Hoda Hamidi was born in **1984** in Ramsar, Mazandaran, Iran. She obtained her B.Sc. degree in applied chemistry from Mohaghegh Ardebili University, Ardebil, Iran, in **2006** and her M.Sc. degree as well as Ph.D degree in organic chemistry from Alzahra University, Tehran, Iran, in **2010** and **2015** under the supervision of Prof. Hossein Abdi Oskooei and Prof. Majid Momahed Heravi. Her research interests are in the area of the synthesis of organic compounds particularly heterocycles and synthetic methodology.

Kaushik Pal was born in India. He received his Ph.D. from University of Kalyani, India. Most significant prestigious awards "Marie-Curie Experienced Researcher (Postdoctoral Fellowship)" offered by the European Commission in Greece and "Brain Korea-21 National Research Foundation "Visiting Scientist" in South Korea, achieved in his career. He was appointed "Senior Postdoctoral Fellow" at Wuhan University in China and promoted to the most prestigious position "Scientist & Faculty CAS Fellow" offered by Chinese Academy of Science. Most Recent, he has been working as a "Research Professor, (Group Leader & Independent Scientist)", at the Department of Nanotechnology, Bharath University (BIHER), Chennai, India. Based on his research background he selected "Editor-in-Chief" of the International peer reviewed journals and publishers 'Pan Standford', En Press, and 'InTech' as well as edited 25-book chapters. Prof. Pal is an excellent group leader as well as associate member in various scientific communities, reorganizations and professional bodies and promissing in worldwide Nanoscience research community. Several past year he organized and chairperson around 15- International events/ symposiums/ conferences/ workshops and himself contributed around 10-Pleneray, 20-Keynote, & 25-Invited lectures. Prof. Pal is an excellent group leader as well as attained distinguish Chief Guest/Visiting Professor in worldwide Nanoscience and Materials Science Research Institutes/Universities. He supervised 6- PhDs, 4- Postdocs, 8- Masters thesis under his supervision.

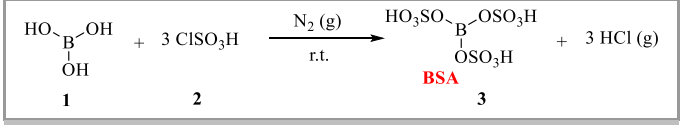
Table of Contents:

1. introduction

Over the last few years, the great potential of solid acid catalysts has been attracted in the synthesis of organic compounds. They are very interesting in wide area of chemical industries due to their supperior advantages. Solid acids as heterogeneous catalyst can be easily isolated from the reaction process and minimize the corrosion problems [1-3].

In continuation of our interests on the application of the efficient catalyst in synthesis of the organic reactions particularly in heterocyclic chemistry and name reaction synthesis [4-9], we wish to review of the BSA solid acid catalyzed reaction in the light of our previous reports. In spite of versatile promoted BSA designed up to now, we hope this report to encourage the invention of other new catalysts based on boron sulfonic acid.

BSA as a novel solid acid catalyst with dual Lewis-Bronsted acidic sites was firstly offered for a regioselective reaction at benign condition by Kiasat *et al.* (Scheme 1) [10]. Boron sulfonic acid or boric trisulfuric anhydride $B(HSO_4)_3$ is a trifunctional inorganic catalyst which has strong acidity and good solubility in water and other polar solvents.



Scheme 1. Synthesis of Boron Sulfonic Acid (BSA).

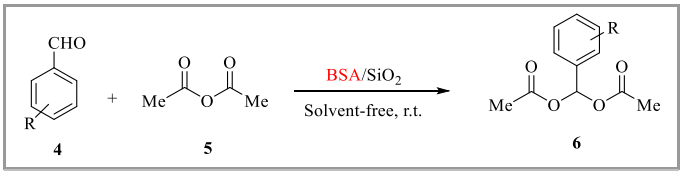
2. Application of BSA in Functional Group Transformation

BSA as a distinguished and heterogeneous catalyst has been utilized in many organic reactions. In the following parts review the details of the application of the boron sulfonic acid for functional group transformation.

2.1. Protection of Functional Groups

2.1.1. Protection of Carbonyl Groups

Preparation of 1,1-diacetates is a type of carbonyl protection which is applicable as a main step in many organic reactions. Typical studies on condensation of aldehyde (**4**) and acetic anhydride (**5**) demonstrate via various heterogenous catalysts [11,12]. We synthesized 1,1-diacetate (**6**) derivatives by Silica-supported Boron Sulfonic Acid (SBSA) as an efficient catalyst under solvent-free conditions at room temperature in high yields [13].



Scheme 2. Carbonyl protection by using BSA.

Acylals or 1,1-diacetates, as aldehyde protecting groups, are useful synthon in organic synthesis. However, the reaction of aldehyde (**7**) and acetic anhydride (**5**) in the presence of cheap and green solid acid BSA was accomplished under solvent-free conditions at room temperature by Mirjalili *et al.*(Scheme 3)**[**14**]**. In addition, some acidic catalysts such MCM-41 (MCM-SO_3H) and SiO_2-Pr-SO_3H are reported [15, 16].

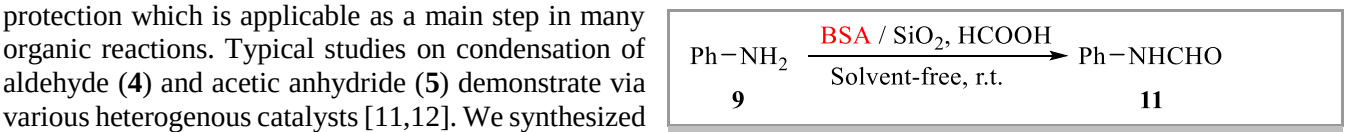
Scheme 3. Reaction of 3-nitrobenzaldehyde to acylal.

2.1.2. Protection of Amine Groups

Another important protection in organic strategies specially in multistep reactions is amine protection in which the *N*-tert-butoxycarbonyl (*N*-Boc) group due to some advantages is commonly used [17-21]. Herein, an efficient and time-consuming protocol was carried out by Hamadi *et al.* using silica-supported BSA for the *tert*-butoxycarbonylation of various amines (**9**) under solvent free and room temperature conditions which illustrate in Scheme 4 [22, 23]**.**

Scheme 4. Protection of amines by using BSA.

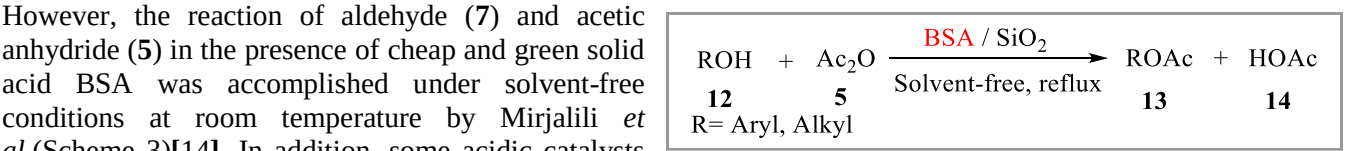
The importance of *N*-formylation reactions have been obscured in the synthesis of pharmaceutically organic compounds containing the cores such as fluoroquinolones, imidazoles, 1,2-dihydroquinolines, formamidines and isocyanides [24-28]. Recently Hamadi *et al.* reported a chemoselective *N*-formylation of amines (**9**) by catalytic activity of BSA in mild conditions which illustrated in Scheme 5 [23].



Scheme 5. BSA catalyzed N-formylation reaction.

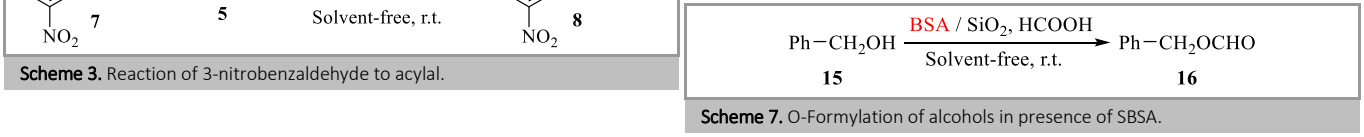
2.1.3. Protection of Hydroxyl Groups

Acylation of alcohols and phenols are the important methods for protection of hydroxyl group, providing a powerful tool in different areas of synthesis, which are often performed in acid catalyzed conditions and are finally removed easily [29,30]. Sedighinia *et al.* reported the acetylation of aliphatic and aromatic alcohols (**12**) with acetic anhydride (**5**) by the aid of silica supported boron sulfonic acid (SBSA) under reflux conditions. The esters (**13**) were prepared at short times and in good to high yields (Scheme 6) [31].



Scheme 6. Acetylation of alcohols by using SBSA.

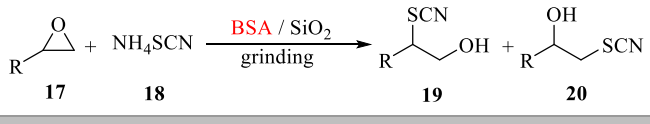
Also, Hamadi *et al.* proposed a green O-formylation of alcohols (15) which catalyzed by SBSA in mild conditions with excellent yields [23].



Scheme 7. O-Formylation of alcohols in presence of SBSA.

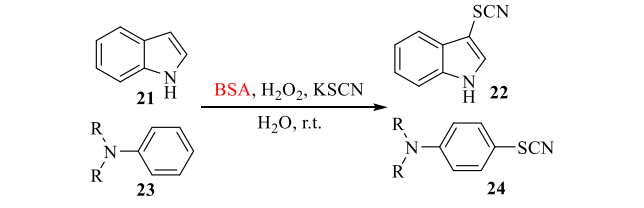
2.2. Thiocyanation

The high transformation ability of thiocyanates to other functional groups including, sulfides, aryl nitriles, thiocarbamates, and thionitriles converted them to the benefit synthon [32-35]. A regioselective synthesis of *β*-hydroxy thiocyanates through the reaction of epoxides (**17**) and NH_4SCN in the presence of BSA under solvent-free condition which has high isolated yields was described by Kiasat *et al.* (Scheme 8) [10].



Scheme 8. Regioselective thiocyanation of epoxides by using BSA.

In the light of our studies, the thiocyanation of aromatic and heteroaromatic compounds is proceeded in water using catalytic activity of BSA. This green protocol carried out in the benign conditions with high yields (Scheme 9) [36-39].

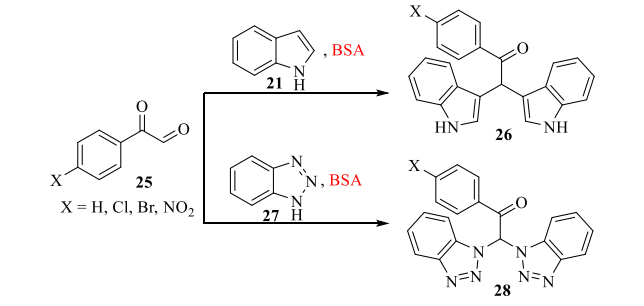


Scheme 9. Thiocyanation of amines in presence of BSA.

3. Application of BSA in C-C Bond Formation

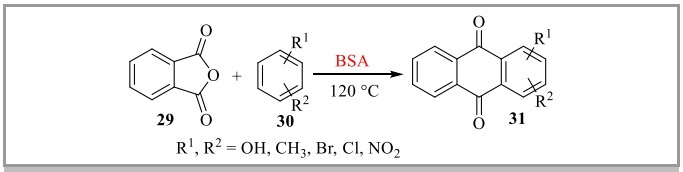
According to the recent research studies, no other type of reaction can provide such large accommodation in synthetic chemistry as much as carbon-carbon bond formation reactions. Synthesis of bisindoles by Friedel-Crafts reaction between indoles and carbonyl compounds can occure in the presence of acid or base [40].

Hence, Mosslemin *et al.* reported synthesis of bisindoles (**26**) and bisbenzotriazoles (**28**) as potentially pharmacologically active structures by a solvent free carbon-carbon bond formation reaction of indole (**21**) and benzotriazole (**27**) in the presence of silica supported boronsulfonic acid in aqueous media with excellent isolated yields (Scheme 10) [41,42].



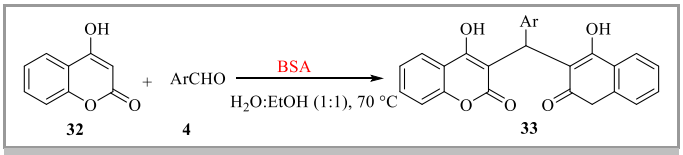
Scheme 10. Synthesis of bisindoles and bisbenzotriazoles via BSA catalyst.

Madje *et al.* reported a Friedel-Crafts reaction between phthalic anhydride (**29**) and substituted benzenes (**30**) involving worthwhile carbon-carbon bond formation for synthesis of anthraquinones (**31**) under solvent free condition using BSA as catalyst (Scheme 11) [43].



Scheme 11. Friedel-Crafts reaction to anthraquinones synthesis in presence of BSA.

Some of biscoumarins derivatives are successful urease and HIV inhibitors [44, 45]. Typically, α,α'-benzylidene bis(4-hydroxycoumarin) (**33**) derivatives was prepared with good yields from the reaction of aromatic aldehyde (**4**) and 4-hydroxycoumarin(**32**) by the aid of catalytic amount of tris(hydrogensulfato) boron [$B(HSO_4)_3$] at short times, and in aqueous media [46].

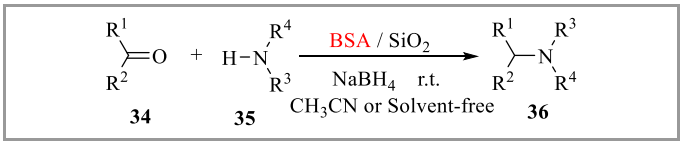


Scheme 12. BSA catalyzed preparation of biscoumarins derivatives.

4. Application of BSA in C-N Bond Formation

A reliable approach for the C–N bond formation is reductive amination which can be provided for primary, secondary and tertiary amines without over-alkylation problems in comparison to direct alkylation of amines with alkyl halides [47-49].

On the basis of recent studies, an efficient protocol for the direct reductive amination of aromatic and aliphatic aldehydes or ketones (**34**) has been accomplished in the presence of sodium borohydride accompanied with the catalytic activity of SBSA in acetonitrile under solvent-free conditions [50].

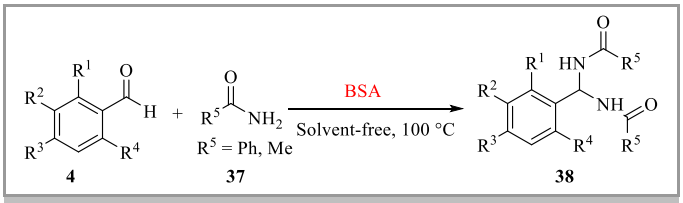


Scheme 13. Reductive amination of aldehydes or ketones to synthesis of amines.

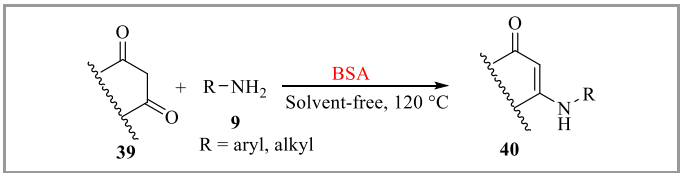
Bisamides containing two C-N bonds are significant targets in the synthetic methodologies of pharmacologically active compounds such as pseudopeptides [51, 52]. A green and one-pot reaction of aldehydes and amides in the presence of BSA led to the desired *N,N'*-arylidene bisamides (**38**) under solvent-free conditions at 100 ˚C. In this protocol, all of various examined aromatic aldehydes (**4**), acetamide or

benzamide (**37**) reacted well to reach high yields of bisamides (83-96%) [53].

Enaminones and enamino esters which often observed in alkaloids and pharmaceuticals are useful precursor in organic synthesis [54-56]. Synthesis of β-enaminones by the reaction of 1,3-dicarbonyl compounds and amines in the presence of catalytic amount of tris(hydrogensulfato)boron (BSA) was performed to produce products in good yields within short reaction times (Scheme 15) [57].



Scheme 14. BSA catalyzed synthesis of bisamides.



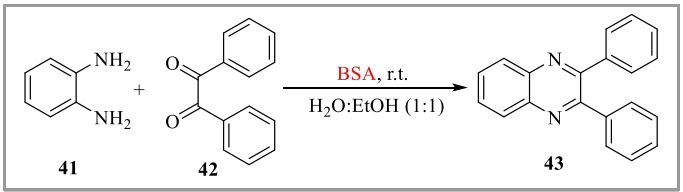
Scheme 15. Synthesis of β-enaminones and β-enamino esters.

5. Application of BSA in Heterocyclic Compounds Synthesis

5.1. Quinoxaline Synthesis

Quinoxalines are interesting heterocycles in pharmaceutical chemistry which demonstrate biological activities [58-60].

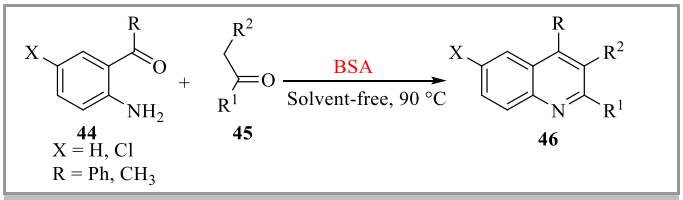
In continuation of our work, BSA successfully applied for green quinoxalines synthesis. A green method for conversion of 1,2-phenylendiamine (**41**) and 1,2-dicabonyl (**42**) to quinoxaline (**43**) in aqueous media in the presence of catalytic amount of BSA was achieved (Scheme 16) [61-63].



Scheme 16. Synthsis of quinoxaline by using BSA.

5.2. Quinoline Synthesis

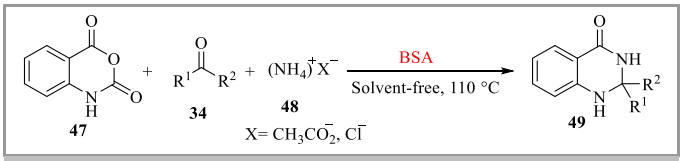
Quinoline is a main core for organic chemists with owning broad vast biological activities such as anti-inflammatory, antimalarial, antihypertensive, and antibacterial agents (properties) [64, 65]. Friedländer annulations of 2-aminobenzophenone (**44**) with acetylacetone (**45**) established quinoline (**46**) derivatives using $B(HSO_4)_3$ as catalyst in solvent free condition which is reported by Saghanezhad *et al.* (Scheme 17) [66].



Scheme 17. Preparation of Quinoline compounds via BSA catalyst.

5.3. Quinazoline Synthesis

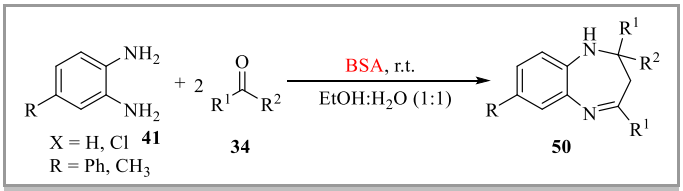
There are many consideration of the 2,3-dihydroquinazolin-4(1*H*)-ones synthesis due to the biological and pharmacological properties including, anticancer, antiinflammatory, and anticonvulsant activities [67-70]. The convenient conversion of the aldehydes or ketones (**34**), isatoic anhydride (**47**), and ammonium salts (**48**) in the presence of tris-(hydrogensulfato)boron leading to corresponding 2,3-dihydroquinazolin-4(1*H*)-ones (**49**) in high yields under solvent-free conditions [71].



Scheme 18. Tree-component synthesis of 2,3-dihydroquinazolin-4(1H)-ones.

5.4. Benzodiazepine Synthesis

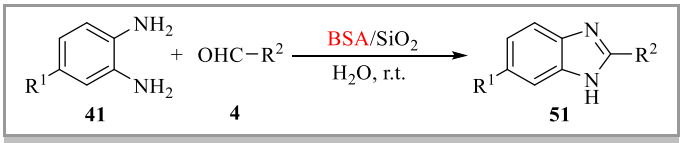
Benzodiazepine framework with various biological activities is main structural motif in medicinal chemistry [72]. Therefore developing methods of benzodiazepine synthesis is important in heterocyclic synthesis. We found that boron sulfonic acid is a useful catalyst for 1,5-benzodiazepines (**50**) synthesis at room tempreture as illustrated in Scheme 19 [73].



Scheme 19. 1,5-Benzodiazepines synthesis by using BSA.

5.5. Benzimidazole Synthesis

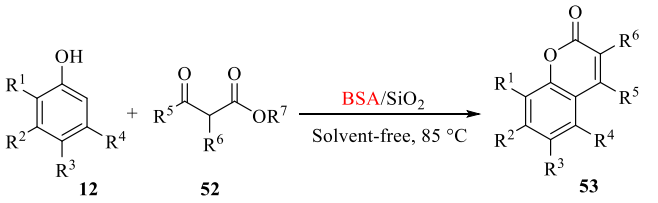
Benzimidazole which exhibit anti-HIV, anti-virus, anti-tumor and inhibitor properties [74-77] is a valuable heterocycle. So, we reported a BSA catalyzed reaction of 1,2-phenylendiamine(**41**) and aldehydes (**4**) to prepare benzimidazole (**51**) derivatives in water at room temperature and with high yields (Scheme 20) [78-80].



Scheme 20. Synthesis of benzimidazole derivatives.

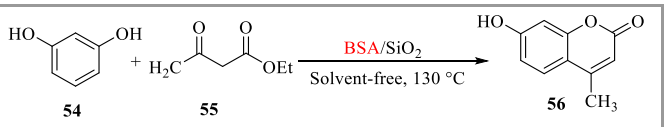
5.6. Coumarin Synthesis

Substituted coumarins as significant building block of pharmaceuticals have many biological activities. They are also originated from naturally occurring compounds. The Pechmann reaction is commonly used to synthesis of coumarin analogues [81]. Silica supported BSA catalyzed Pechman reaction between phenols (**12**) and *β*-ketoesters (**52**) was described by Parhami *et al*. in a one-pot procedure. As illustrated in Scheme 21, this synthetic strategy was occurred in mild and solvent-free conditions [82].



Scheme 21. Preparation of coumarins by using BSA catalyzed Pechmann condensation.

Another efficient method for coumarin synthesis is including the interaction of resorcinol (**54**) and ethyl acetoacetate (**55**) by Pechmann condensation using catalytic activity of BSA (Scheme 22) [83].

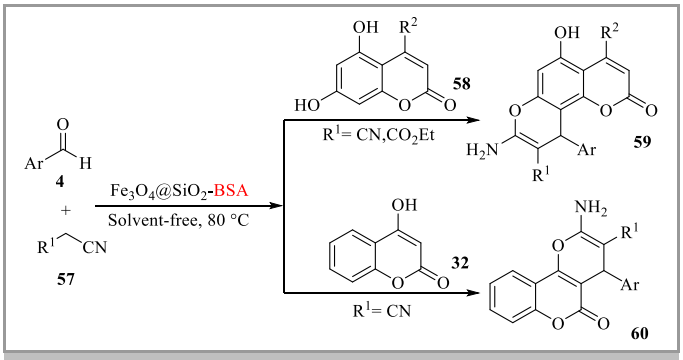


Scheme 22. BSA catalyzed Pechmann reaction.

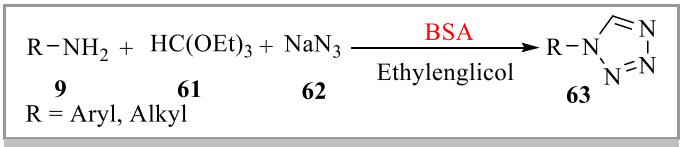
Recently, an attractive modified BSA catalyst such as Fe_3O_4@SiO_2-BSA was utilized to provide pyrano coumarin derivaties with good yield and atom-economy. Farahi and co-workers successfully reported a novel magnetically heterogeneous catalyst by promotion of boron sulfonic acid with Fe_3O_4@SiO_2 nanoparticles. The three-component simple procedure condensation reaction of malononitrile/ethyl cyanoacetate (**57**), aryl aldehyde (**4**), and 5,7-dihydroxy-4-substituted coumarin (**59**)/4-hydroxycoumarin (**31**) was performed without any hazardous organic solvents (Scheme 23) [84].

5.7. Aryltetrazole Synthesis

The tetrazole groups with widespread applications which can be used as carboxylic acids substitutes play an important role in medicinal chemistry [85]. Hence, various methodologies in synthesis of tetrazoles are investigated in recent years [86]. We prepare a synthetic reaction for renovation of arylamines (**9**), triethyl orthoformate (**61**), and sodium azide (**62**) to aryltetrazoles (**64**) using boron sulfonic acid in ethylenglicol at high temperature (Scheme 24) [87].



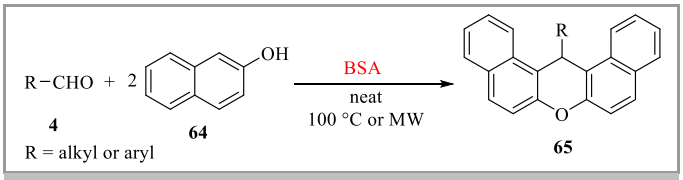
Scheme 23. Preparation of pyrano coumarins.



Scheme 24. BSA catalyzed synthesis of aryltetrazoles.

5.8. 14H-Dibenzoxanthenes Synthesis

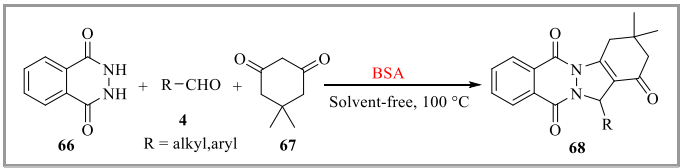
Xanthene derivatives as main structural heterocycle have vast range of application from fluorescent materials and laser technologies to biological properties such as antiviral, antibacterial, antiflammatory agents [88-91]. BSA as a solid acid catalyst play an essential role in the reaction of 2-naphthol (**64**) with aldehydes (**4**) due to 14-alkyl or aryl 14*H*-dibenzoxanthenes (**65**) synthesis which was reported by Borah and co-worker (Scheme 25) [92].



Scheme 25. Synthesis of dibenzoxanthenes in presence of BSA.

5.9. 2H-indazolo[1,2-b] Phthalazine-Trione Synthesis

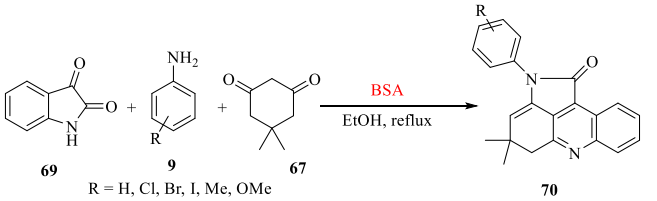
Compounds containing phthalazine were found as inhibitors and shown pharmacological properties such as cytotoxic, antimicrobial, anticonvulsant, antifungal, anticancer, and anti-inflammatory activities in medicinal chemistry [93-100]. As shown in Scheme 26, three-component condensation reaction of dimedone, phthalhydrazide, and aromatic aldehydes afforded 2*H*-indazolo[1, 2-*b*] phthalazine-trione derivatives in high yields using catalytic effect of boron sulfonic acid under solvent-free conditions [100, 101].



Scheme 26. 2H-Indazolo[1,2-b] phthalazine-triones synthesis by using BSA.

5.10. Pyrroloacridone Synthesis

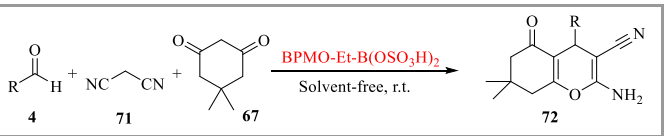
Pyrroloacridone derivatives with pharmacological and biological activities which attracted chemist's attention [102-104]. The efficient synthesis of 2-arylpyrroloacridin-1(2*H*)-ones (**70**) *via* a three-component reaction between dimedone(**67**), anilines (**9**) and isatin(**67**) in the presence of tris(hydrogensulfato) boron [$B(HSO_4)_3$] with refluxing in ethanol has been described by Karimi-Jaberi and co worker (Scheme 27) [105].



Scheme 27. Synthesis of pyrroloacridine derivatives.

5.11. Tetrahydrobenzo[b]pyrans Synthesis

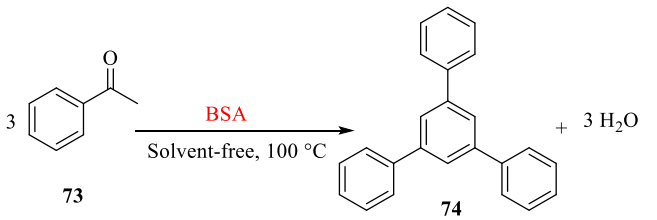
Tetrahydrobenzo[*b*]pyrans due to their pharmacological and therapeutic properties such as anticancer, spasmolytic, anticoagulant and anti-anaphylacticagents are significant target in synthetic organic chemistry [106-108]. A novel BSA modified nanocatalyst as ethyl-based PMO supported boron sulfonic acid was desined by Norouzi *et al*. benzaldehyde (**4**), dimedone (**67**) and malononitrile (**71**) reacted by using [BPMO-Et-$B(OSO_3H)_2$] under solvent-free conditions at room temperature in high to excellent tetrahydrobenzo[*b*]pyran yields [109].



Scheme 28. Synthesis of tetrahydrobenzo[b]pyran in presence of BSA modified nanocatalyst.

6. Miscellaneous

A cyclotrimerization reaction of alkyl and aryl ketones was described in the presence of catalytic amounts of boron sulfonic acid [$B(HSO_4)_3$] under solvent-free conditions without any by-product. Cyclotrimerization of acetophenone obviously depicted in Scheme 29 [110].



Scheme 29. Cyclotrimerization of acetophenone by using BSA.

7. Conclusion

This review presented an overview of typical applications of boron sulfonic acids in multicomponent reactions. In the light of our studies in many literatures we found BSAs as worthwhile green catalysts due to their stability, non toxicity, reusability, ease of handling, inexpensiveness, and availability which are able to catalyze various transformations in the organic chemistry.

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