Academic Journal of Polymer Science
ISSN: 2641-8282

Juniper Publishers
key to the Researchers

Mini-Review
Volume 2 Issue 1 - November 2018
DOI: 10.19080/AJOP.2018.02.555580

Academ J Polym Sci

Recent Advances in Synthetic Applications of Polyvinylpyrrolidone Supported Reagents and Catalysts

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Submission: August 23, 2018; **Published:** November 05, 2018

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Abstract

This review summarized recent progresses in the application of polyvinylpyrrolidone supported reagents and catalysts in organic synthesis.

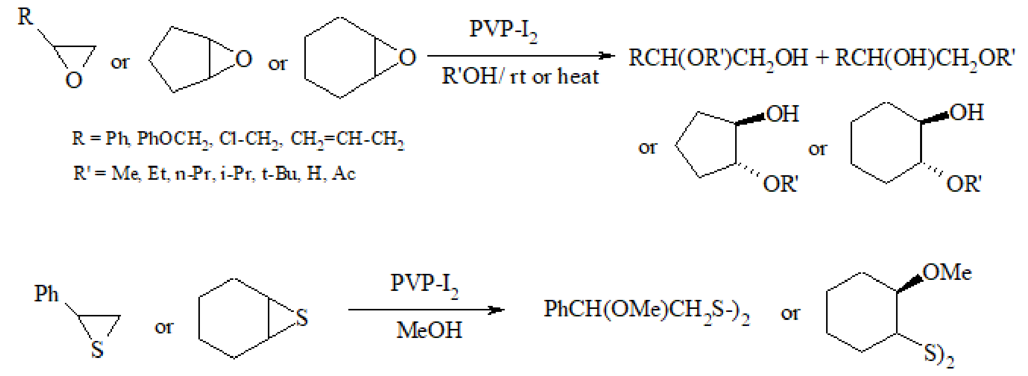
Keywords: Polyvinylpyrrolidone; Supported reagents; Supported catalysts; Multi-component reactions

Introduction

The use of solid-supported reagents and catalysts in solution-phase chemistry has emerged as a leading strategy that exploits the advantages of both solid- and solution-phase synthesis. The approach essentially combines the benefits of product isolation and purification in solid-phase synthesis with the high-speed development and flexible choice of chemistry from the vast repertoire of solution phase organic reactions. The organic molecules synthesis using polymer-supported reagents and catalysts is highly attractive because the work-up involves only simple filtration and evaporation of the solvent [1]. Polyvinylpyrrolidone (PVP) is an amorphous polymer having broad applications in biomedical field due to its special properties such as low toxicity and good solubility in water and most organic solvents, good adhesion characteristics, and great physiological compatibility [2]. Also, PVP has good biocompatibility and has been applied for many years as a biomaterial or additive to drug compositions, e.g. as a blood plasma expander [3]. Polyvinylpolypyrrolidone (PVPP, crospovidone, or crospolividone) is a highly cross-linked polyvinylpyrrolidone (PVP). The cross-linked form of polyvinylpyrrolidone is insoluble in water, though it still absorbs water and swells very rapidly generating a swelling force. This property makes PVPP useful as a disintegrant in pharmaceutical tablets [2]. Polyvinylpyrrolidone shows a strong binding affinity to small molecules. Furthermore, its iodine complex, povidon-iodine, is widely used as an anti-infective agent in clinical treatments [4].

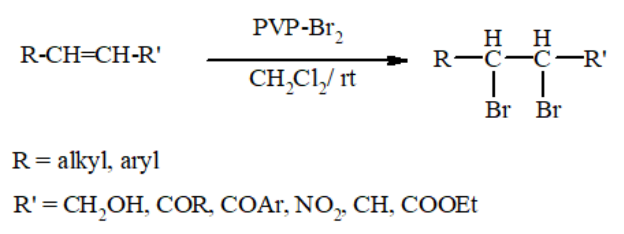
Polyvinylpyrrolidone Supported Reagents

A range of polyvinylpyrrolidone-supported reagents has been developed for applications in organic synthesis. In general, these reagents are employed in stoichiometric excess to drive the reaction to completion. Simple filtration removes the spent resin from the reaction solution and, thus, eliminates the need for any time-consuming chromatographic work-up.



Scheme 1: Ring opening of epoxides and ring opening dimerization of thiiranes by PVP-I$_2$.

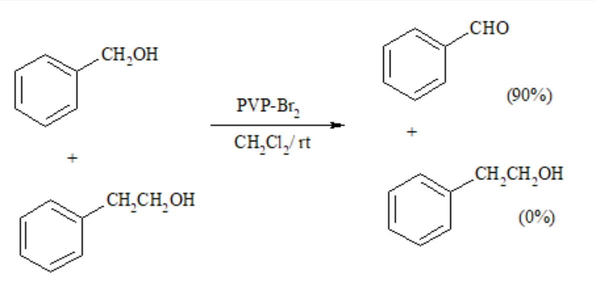
Iranpoor and coworkers [5] have prepared iodine supported on polyvinylpyrrolidone (betadine) as catalysts for ring opening of epoxides and as reagent for ring opening dimerization of thiiranes in alcohols, water and acetic acid (Scheme 1). In this report, the reaction of *R*-(+)-styrene oxide with I_2 supported on PVP in methanol was found to be very stereospecific and the product isolated in 93% ee.



Scheme 2: Bromination of alkenes using PVP-Br_2.

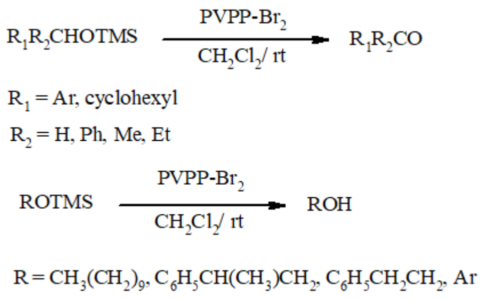
In another research, Lakouraj et al. [6] described the preparation of polyvinylpyrrolidone-bromine complex (PVP-Br_2) as a mild and convenient reagent for selective bromination of alkenes (Scheme 2) [6].

Selective oxidation of benzyl alcohol in the presence of 2-phenylethanol was also achieved at room temperature in the presence of PVP-Br_2 (Scheme 3).

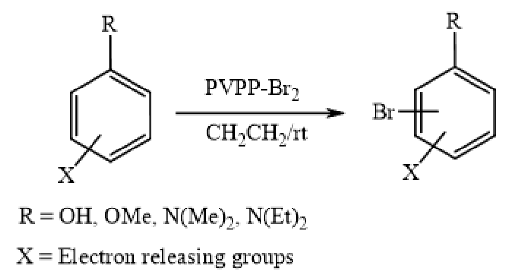


Scheme 3: Selective oxidation of benzyl alcohol in the presence of 2-phenylethanol by PVP-Br_2.

In the next research, Lakouraj and Mokhtary have developed a convenient method for deprotection and direct oxidative deprotection of silylethers to the corresponding hydroxy and carbonyl compounds using polyvinylpolypyrrolidone-bromine complex (PVPP-Br_2) (Scheme 4) [7]. Selective oxidative deprotection of benzylic silyl ethers in the presence of primary aliphatic alcohols was also achieved at room temperature.

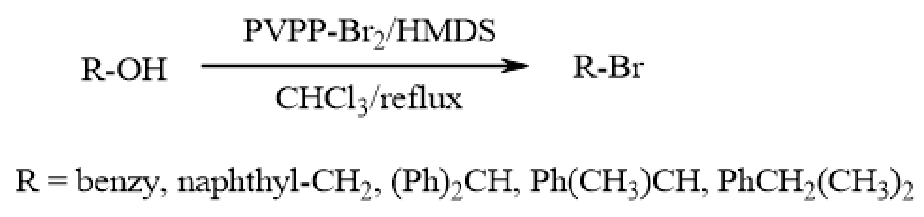


Scheme 4: Deprotection and direct oxidative deprotection of silylethers by PVPP-Br_2.



Scheme 5: bromination of electron-rich aromatic compounds by PVPP-Br_2.

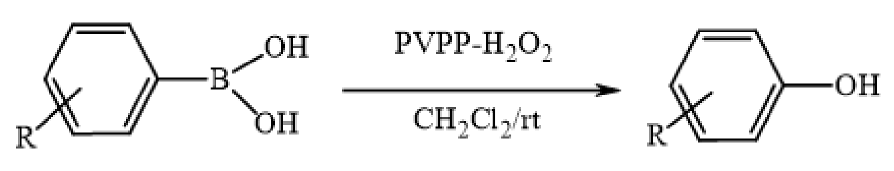
Also, PVPP-Br_2 has been used for bromination of electron-rich aromatic compounds [8]. The reaction proceeded smoothly with phenols and *N*, *N*-alkylated amines to afford the corresponding mono brominated product in good yields at ambient temperature (Scheme 5).



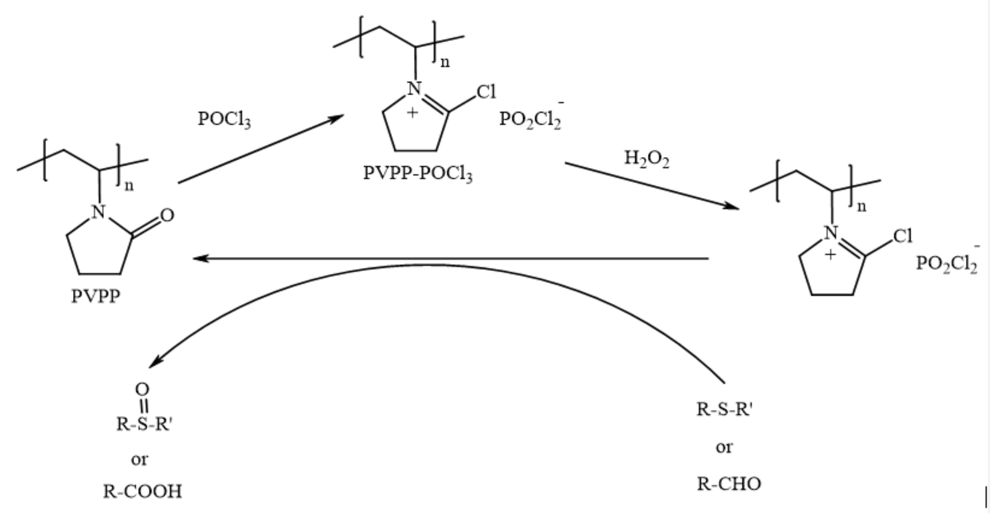
Scheme 6: Synthesis of benzylic bromides by PVPP-Br_2.

Furthermore, Mokhtary and Lakouraj synthesized benzylic bromides in high yields by the reaction of the corresponding alcohols with cross-linked polyvinylpyrrolidone-bromine complex (PVPP-Br_2) in the presence of hexamethyldisilane in chloroform at reflux condition (Scheme 6) [9]. Selective conversion of benzyl alcohol to benzyl bromide in the presence of 2-phenylethanol was also achieved.

Surya Prakash et al. have prepared solid polyvinylpyrrolidone-hydrogen peroxide complex and used as solid hydroxylating reagent [10]. This solid hydrogen peroxide is found to be much safer, convenient and efficient reagent system for the ipso-hydroxylation of arylboronic acids to the corresponding phenols in highyields at a faster rate (Scheme 7). The versatility of the reagent has been further expanded for the one-pot synthesis of halophenols.



Scheme 7: Ipso-hydroxylation of arylboronic acids by PVPP-H2O_2.



Scheme 8: Oxygenation of sulfides to sulfoxides, and oxidation of aldehydes to carboxylic acids by PVPP-POCl3 in the presence of hydrogen peroxide.

Lakouraj et al., [11] have demonstrated polyvinylpolypyrrolidone-phosphorous oxychloride as a versatile polymeric Vilsmeier reagent that exhibits excellent selectivity for oxygenation of sulfides to sulfoxides, and oxidation of aldehydes to carboxylic acids in the presence of hydrogen peroxide under mild reaction conditions (Scheme 8) [11]. This polymeric Vilsmeier reagent was found to retain its activity after months and is stable in a glass bottle at room temperature.

Iranpoor et al. [12] have reported dinitrogen tetraoxide supported on polyvinylpyrrolidone (PVP-N_2O_4) as a nitrosating and coupling agent for thiols and selective oxidation of sulfides to sulfoxides and disulfids to thiosulfonates (Scheme 9) [12].

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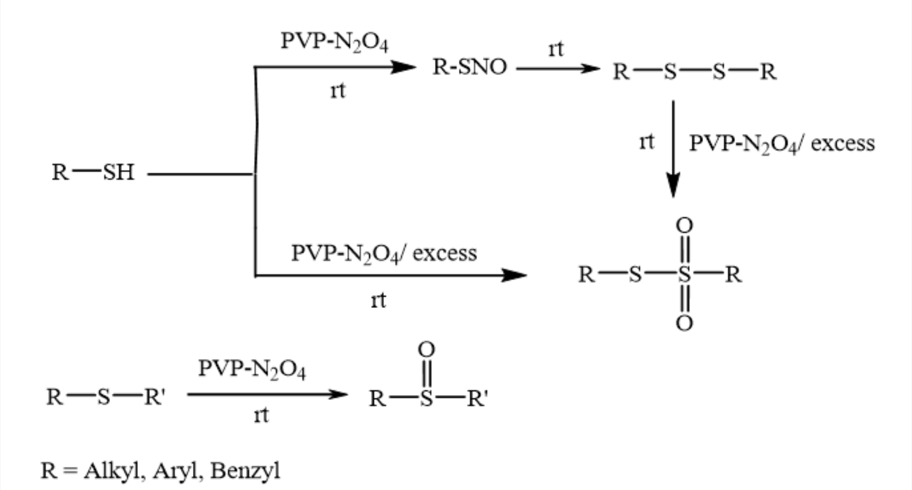
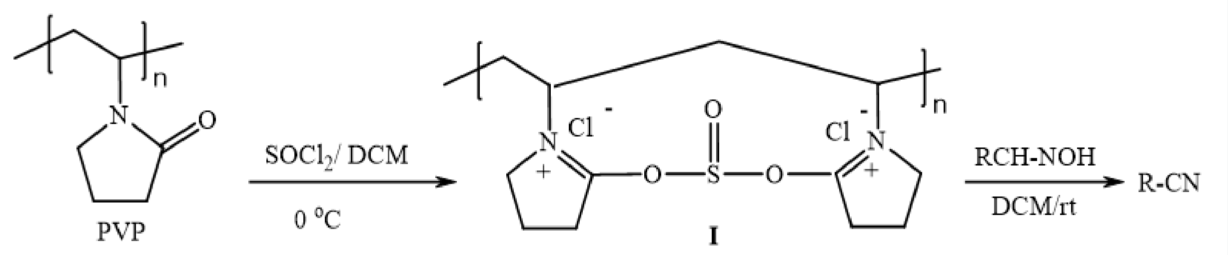
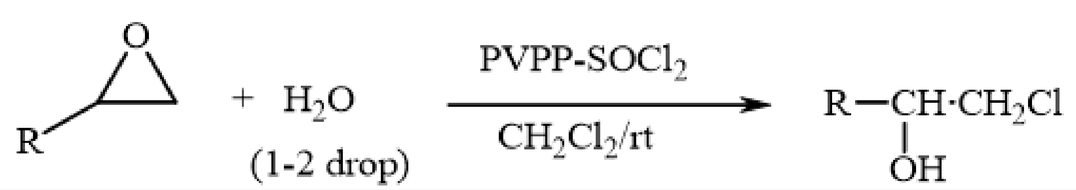


Scheme 9: Coupling of thiols and selective oxidation of sulfides to sulfoxides and disulfids to thiosulfonates by PVP-N_2O_4.



Scheme 10: Dehydration of a variety of aldoximes by PVP-$SOCl_2$.

Tamami and Kiasat have reported [13] a polyvinylpyrrolidone-thionyl chloride complex by the reaction of thionyl chloride with two equivalents of polyvinylpyrrolidone in dichloromethane at 0°C. The polymer-bound complex I was used for the rapid dehydration of a variety of aldoximes to produce the corresponding nitriles in high yields (Scheme 10).



Scheme 11: Ring opening of epoxides to afford β-chlorohydrins using PVP-$SOCl_2$.

Also, Tamami et al. [14] have reported rapid ring opening of epoxides to afford *β*-chlorohydrins with cross-linked polyvinylpyrrolidone/thionyl chloride complex (PVP-$SOCl_2$), under mild reaction condition in high yields (Scheme 11).

Polyvinylpyrrolidone Supported Lewis Acidic Catalysts

Replacement of conventional, toxic and unstable Lewis acidic catalysts with eco-friendly reusable solid acid catalysts is an essential requirement in the development of green chemistry. For example, boron trifluoride is widely used in organic syntheses as a Lewis acid. However, boron trifluoride is highly water sensitive, irritant, and has to be used in a carefully dried apparatus. Moreover, all work must be carried out in an efficient fume hood, and its recovery from the reaction mixture results in a main source of waste, which on an industrial scale is environmentally unacceptable.

Mokhtary et al. have reported several papers on the application of the polyvinylpolypyrrolidone-bound boron trifluoride (PVPP-BF_3) as a stable polymeric Lewis acid catalyst in some organic reactions such as synthesis of amides by the reaction of nitriles with benzhydrol and tertiary alcohols or *tert*-butyl acetate *via* Ritter reaction (Scheme 12) [15, 16], the acylation of alcohols, phenols and trimethylsilyl ethers with acetic anhydride (Scheme 13) [17], the synthesis of coumarins *via* Pechmann condensations of phenols with ethyl acetoacetate (Scheme 14) [18], the synthesis of 14-aryl-14*H*-dibenzo [a,j] xanthenes and bis(naphthalen-2-yl-sulfane) derivatives (Scheme 15) [19], the synthesis of 1,8-dioxooctahydroxanthenes and 1,8-dioxodecahydroacridines via condensation of aromatic aldehydes and dimedone in

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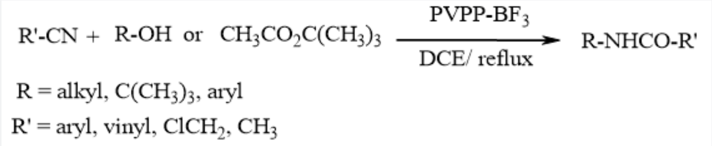
acetonitrile at room temperature, and aromatic aldehydes, dimedone, and aromatic amines in acetonitrile at 80 °C respectively (Scheme 16) [20], the oxidation of aldehydes to carboxylic acids and oxidative esterification of benzaldehydes in the presence of 35% hydrogen peroxide (Scheme 17) [21] and the oxidation of sulfides to sulfones in the presence of 35% hydrogen peroxide at room temperature (Scheme 18) [22]. Excellent yields, easy work-up and reusability and stability of the catalyst are some advantages of these methods.

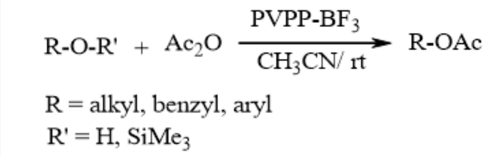


$$\text{R'-CN} + \text{R-OH or } CH_3CO_2C(CH_3)_3 \xrightarrow[\text{DCE/ reflux}]{\text{PVPP-BF}_3} \text{R-NHCO-R'}$$

R = alkyl, $C(CH_3)_3$, aryl

R' = aryl, vinyl, $ClCH_2$, CH_3

Scheme 12: Synthesis of amides by the reaction of nitriles with benzhydrol and tertiary alcohols or tert-butyl acetate using PVPP-BF_3.

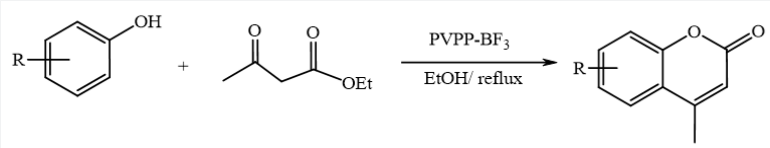


$$\text{R-O-R'} + Ac_2O \xrightarrow[CH_3CN/\text{ rt}]{\text{PVPP-BF}_3} \text{R-OAc}$$

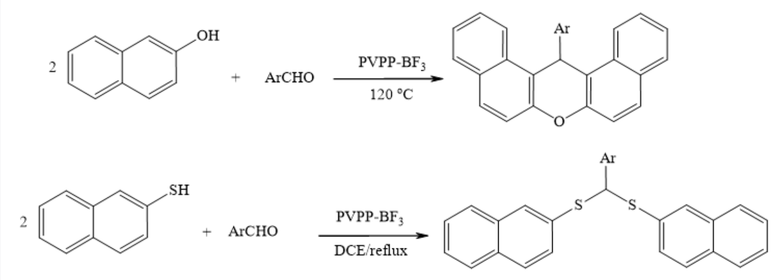
R = alkyl, benzyl, aryl

R' = H, $SiMe_3$

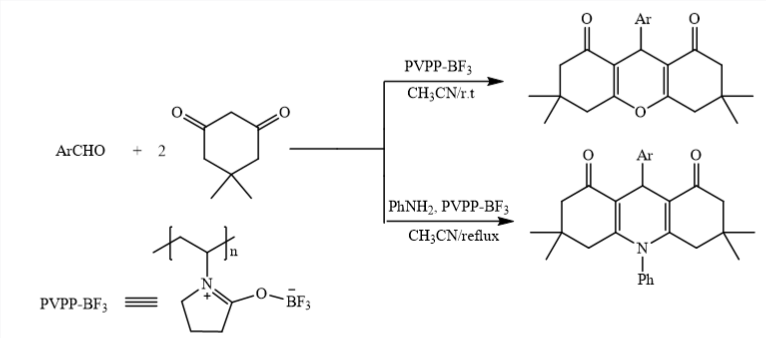
Scheme 13: Acylation of alcohols, phenols and trimethylsilyl ethers using PVPP-BF_3.



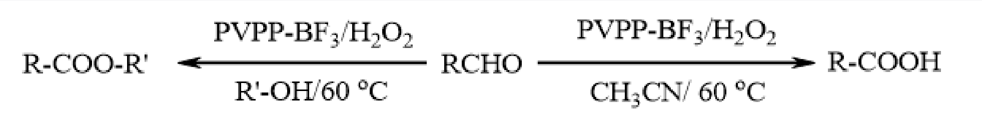
Scheme 14: Condensations of phenols with ethyl acetoacetate by PVPP-BF_3.



Scheme 15: The synthesis of 14-aryl-14H-dibenzo[a,j]xanthenes and bis(naphthalen-2-yl-sulfane) derivatives by PVPP-BF_3.

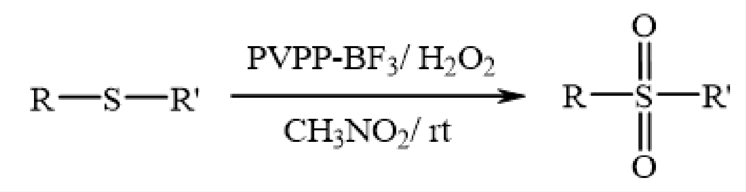


Scheme 16: synthesis of 1,8-dioxooctahydroxanthenes and 1,8-dioxodecahydroacridines using PVPP-BF_3.

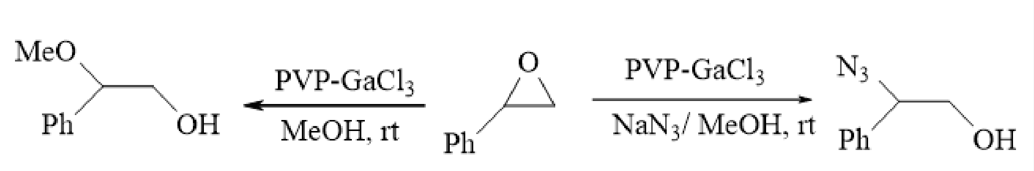


$$\text{R-COO-R'} \xleftarrow[\text{R'-OH/60 °C}]{\text{PVPP-BF}_3/H_2O_2} \text{RCHO} \xrightarrow[CH_3CN/\text{ 60 °C}]{\text{PVPP-BF}_3/H_2O_2} \text{R-COOH}$$

Scheme 17: Oxidation of aldehydes to carboxylic acids and oxidative esterification of benzaldehydes by PVPP-BF_3/H_2O_2.

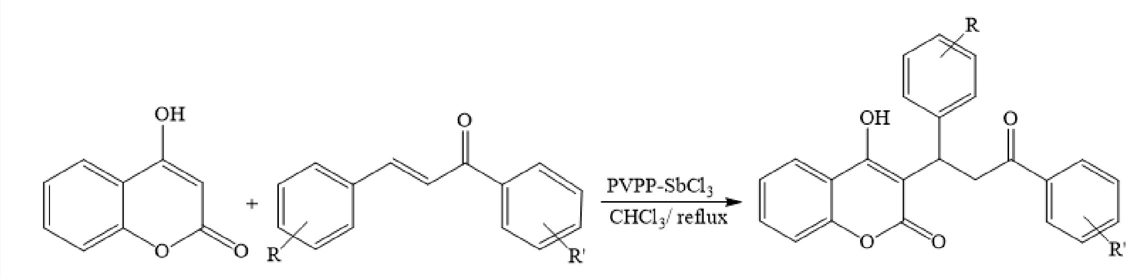


Scheme 18: Oxidation of sulfides to sulfones in the presence of PVPP-BF_3/H_2O_2.



Scheme 19: Ring-opening of epoxides by various alcohols and regioselective conversion of epoxides to β-azidohydrines by PVP-$GaCl_3$.

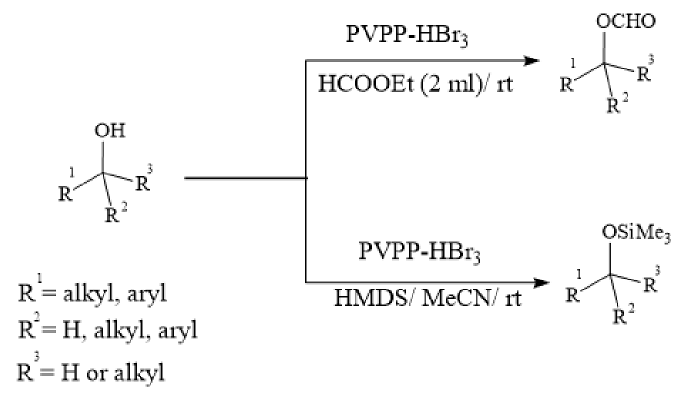
Pourali and et al. prepared cross-linked polyvinylpyrrolidone supported $GaCl_3$ for efficient and regioselective ring-opening reaction of epoxides by various alcohols under solvent-free conditions at room temperature (Scheme 19) [23]. Furthermore, regioselective conversion of epoxides to *β*-azidohydrines was accomplished by sodium azide in MeOH in the presence of $GaCl_3$/PVP at room temperature. $GaCl_3$/PVPP is a non-hygroscopic and recoverable catalyst and is easily separated from reaction mixture by a simple filtration and reused repeatedly.



Scheme 20: Synthesis of chromenylphenylpropanone derivatives using PVPP-$SbCl_3$.

In the next research, an efficient synthesis of chromenylphenylpropanone derivatives as warfarine-like analogues was developed by the Michael addition of 4-hydroxycoumarin to *α*, *β*-unsaturated compounds in the presence of polyvinylpolypyrrolidone supported antimony (III) chloride (PVPP-$SbCl_3$) as a new polymeric Lewis acid catalyst in chloroform at reflux conditions (Scheme 20) [24].

Polyvinylpyrrolidone Supported Brönsted Acidic Catalysts



Scheme 21: Trimethylsilylation and formylation of alcohols by PVPP-HBr_3.

Polymer-supported Brönsted acidic catalysts have gained considerable importance due to their low cost, high efficiency, easy work-up, and reusability. Ghorbani-Choghamarani et al. reported trimethylsilylation and formylation of alcohols in the presence of

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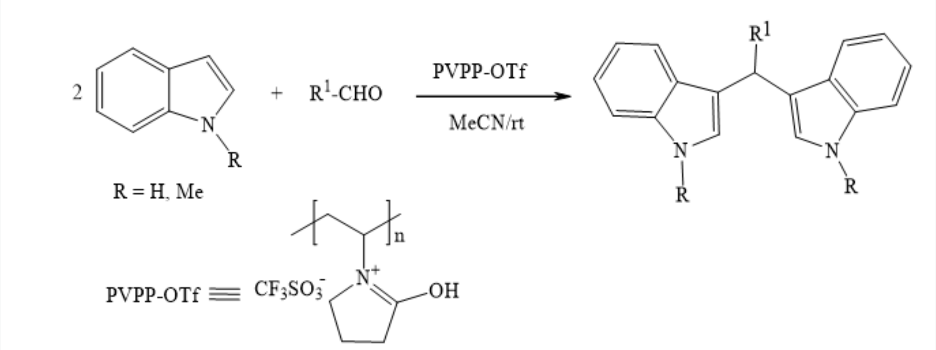
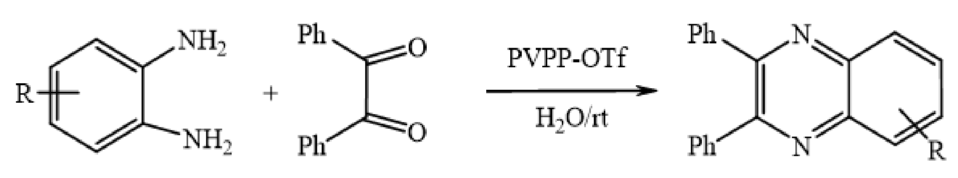
polyvinylpolypyrrolidoniume tribromide in acetonitrile at room temperature (Scheme 21) [25].

Khaksar et al. have prepared polyvinylpolypyrrolidone-supported triflic acid (PVPP.OTf) as an environmentally friendly and efficient catalyst for the synthesis of bis-indolyl methane derivatives by the reaction of indole or *N*-methyl indole with aldehydes (Scheme 22) [26].



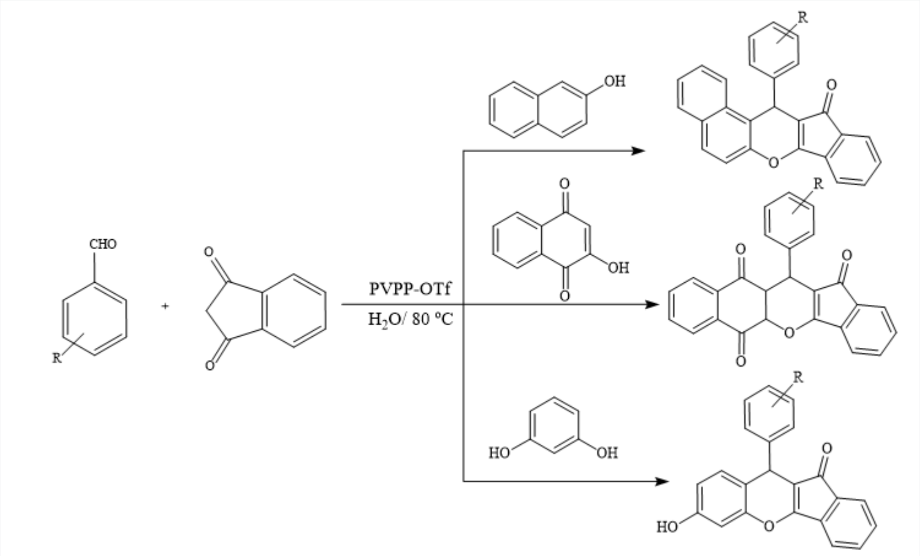
Scheme 22: synthesis of bis-indolyl methane derivatives by PVPP-OTf.



Scheme 23: Synthesis of quinoxaline derivatives using PVPP-OTf.

Furthermore, PVPP.OTf was found to be useful as a recyclable heterogeneous catalyst for the rapid synthesis of quinoxaline derivatives (Scheme 23) [27].

Also, polyvinylpolypyrrolidone-supported triflic acid has been used as a recyclable catalyst for synthesis of a series of 7-hydroxy-10-aryl-10*H*indeno[1,2-b]chromen-11-one derivatives, 13-phenyl indeno[1,2-b]naphtho[1,2-e]pyran-12(13H)-one and 12-phenyl-12*H*-indeno[1,2-*b*]naphtho[3,2-e]pyran-5,11, 13-triones (Scheme 24) [28].



Scheme 24: Synthesis of 7-hydroxy-10-aryl-10Hindeno[1,2-b]chromen-11-one derivatives, 13-Phenyl indeno[1,2-b]naphtho[1,2-e]pyran-12(13H)-one and 12-phenyl-12H-indeno[1,2-b]naphtho[3,2-e]pyran-5,11, 13-triones by PVPP-OTf.

Nitration of the substituted phenols (Scheme 25) [29] and oxidation of urazoles and bis-urazoles to the corresponding triazolinediones (Scheme 26) [30] were reported by Nikoorazm et al. and Ghorbani-Choghamarani et al. respectively in dichloromethane at room temperature using supported nitric acid on polyvinylpyrrolidone as an efficient, environmentally friendly, mild catalyst.

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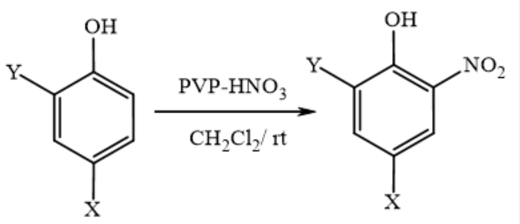
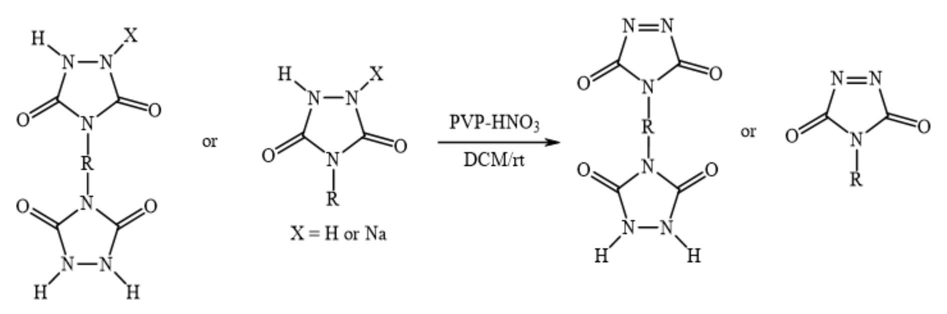
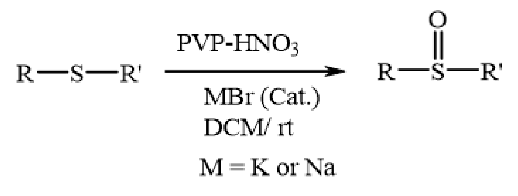


Scheme 25: Nitration of the substituted phenols by PVP-HNO$_3$.



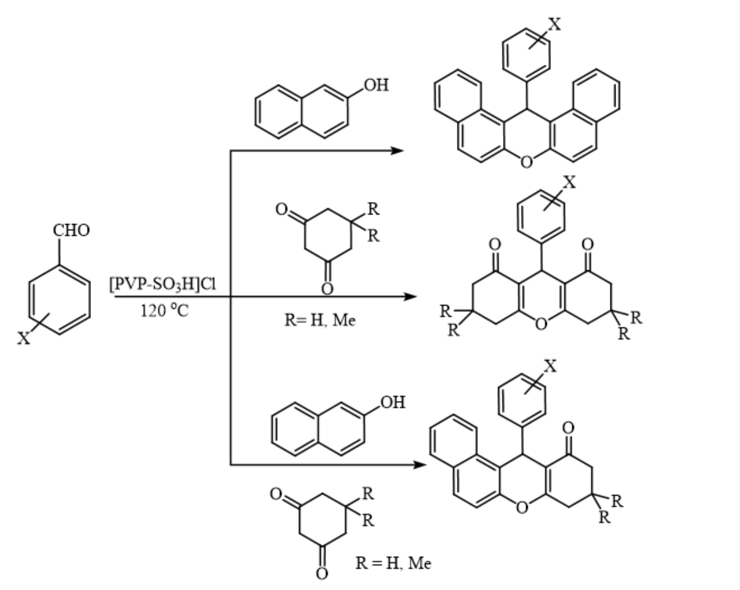
Scheme 26: Oxidation of urazoles and bis-urazoles using PVP-HNO$_3$.



Scheme 27: Chemo selective oxidation of sulfides to sulfoxides using PVP-HNO$_3$.

Furthermore, Zolfigol et al. was described chemo selective oxidation of sulfides to sulfoxides using PVP-HNO$_3$ in the presence of a catalytic amount of KBr or NaBr (Scheme 27) [31].

The synthesis of xanthenes derivatives including 1,8-dioxooctahydroxanthenes, 14-aryl-14*H*-dibenzo[a,j] xanthenes, and 12-aryl-8,9,10,12-tetrahydrobenzo[a]xanthen-11-ones reported by Shirini et al, using *O*-sulfonated poly(4-vinylpyrrolidonium) chloride [PVP-SO$_3$H]Cl as a polymeric solid acid catalyst (Scheme 28) [32].



Scheme 28: Synthesis of xanthenes derivatives including 1,8-dioxooctahydroxanthenes, 14-aryl-14H-dibenzo[a,j] xanthenes, and 12-aryl-8,9,10,12-tetrahydrobenzo[a]xanthen-11-ones by [PVP-SO$_3$H]Cl.

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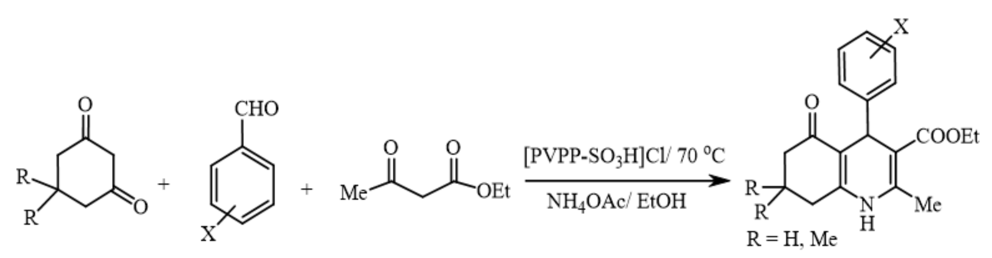
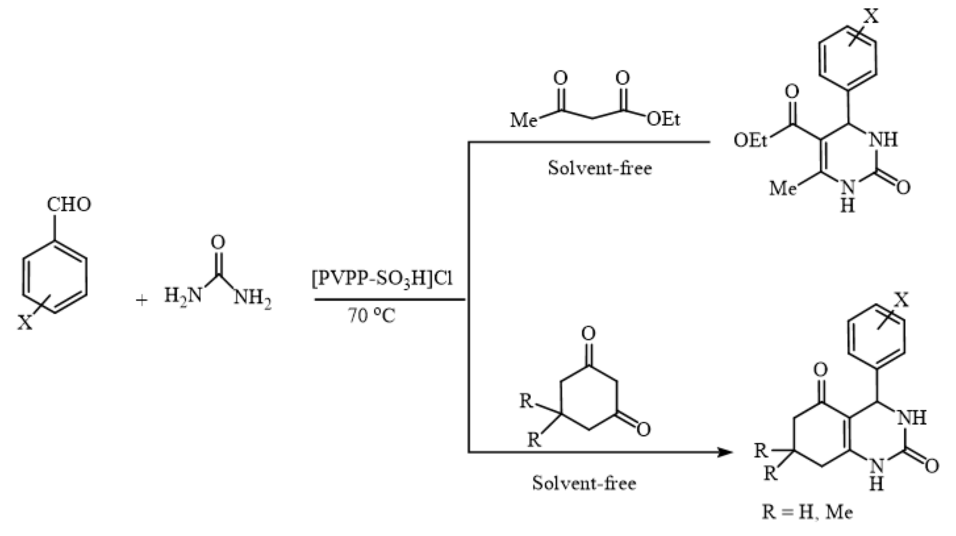


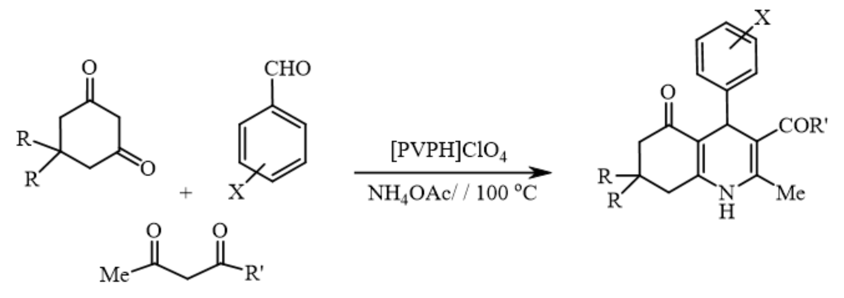
Scheme 29: Synthesis of hexahydroquinolines by [PVPP-SO_3H]Cl.



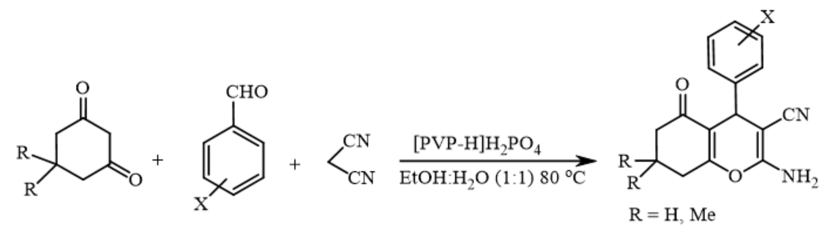
Scheme 30: Synthesis of dihydropyrimidinones and octahydroquinazolin-2,5-diones by [PVPP-SO_3H]Cl.

Also, polyvinylpolypyrrolidone supported chlorosulfonic acid ([PVPP-SO_3H]Cl) was evaluated by Mokhtary et al, as a recoverable catalyst for the one-pot synthesis of hexahydroquinolines (Scheme 29) [33], dihydropyrimidinones and octahydroquinazolin-2,5-diones (Scheme 30) [34].

In another research, Abedini et al. have described a green approach for the promotion of the synthesis of Hantzsch products using polyvinylpyrrolidinium perchlorate ([PVPH]ClO_4) as a new modified polymeric catalyst (Scheme 31) [35].



Scheme 31: Synthesis of Hantzsch products using [PVPH]ClO_4.



Scheme 32: Synthesis of 2-amino-3-cyano-tetrahydro-4H-benzopyran derivatives by PVP-H]H_2PO_4.

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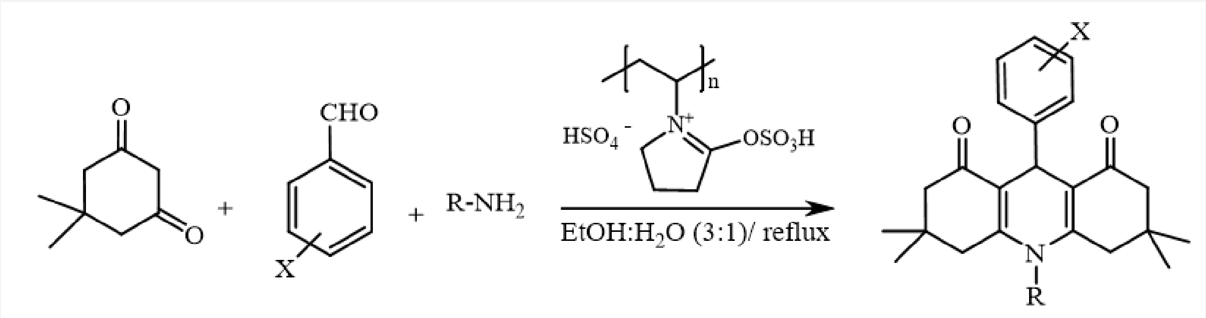
In the next research, Shirini et al., have introduced polyvinylpyrrolidonium hydrogen phosphate ([PVP-H]H_2PO_4) as a heterogeneous, and reusable catalyst for the synthesis of 2-amino-3-cyano-5-oxo-5,6,7,8-tetrahydro-4*H*-benzopyrans (Scheme 32) [36].

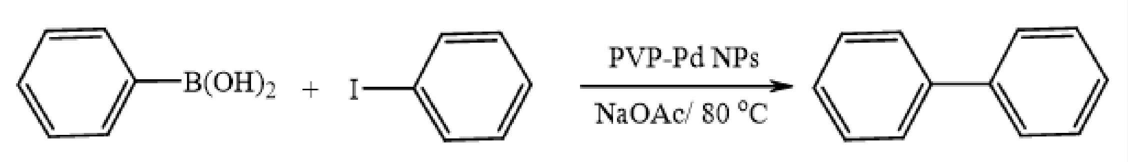
In another study, sulfuric acid-modified polyvinylpyrrolidone ([PVP-SO_3H]HSO_4) was prepared by Safaei et al. as an efficient reusable polymeric catalyst for the one-pot multi component synthesis of acridinedione derivatives as an important class of heterocyclic compounds (Scheme 33) [37].



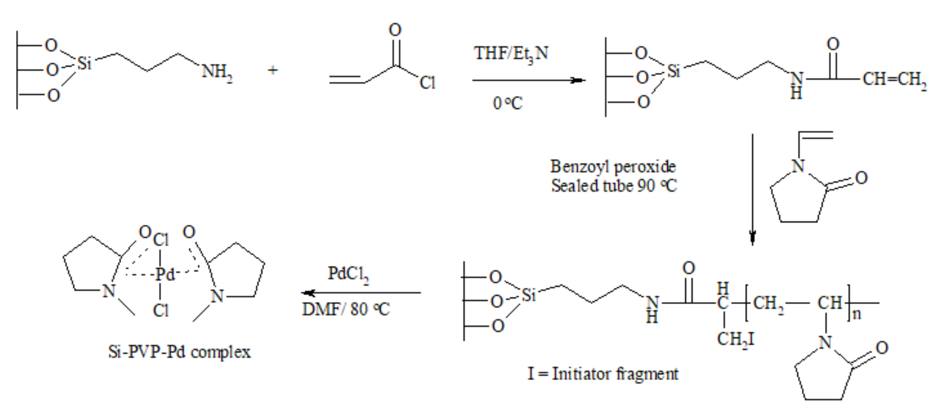
Scheme 33: Synthesis of acridinedione derivatives by [PVP-SO_3H]HSO_4.

Polyvinylpyrrolidone Supported Pd Catalyst

A series of poly(*N*-vinyl-2-pyrrolidone) immobilized Pd nanoparticles (PVP-Pd) with varying particle size have prepared by Li et al., [38] using the stepwise growth reaction. The effect of Pd particle size on the Suzuki reaction between phenylboronic acid and iodobenzene was investigated by the use of four Pd catalysts (Scheme 34). The catalytic activity of the Pd nanoparticles expressed in terms of the initial turnover frequency (moles of the biphenyl product per mole of total surface Pd atoms per min) was found to be in the order of Pd (3.9nm) > Pd (3.0nm) ~ Pd (5.2nm) > Pd (6.6nm), indicating that surface Pd atoms do not all have the same reactivity in this reaction. The general trend of increased catalytic activity with the decrease in the particle size suggests that the low-coordination number vertex and edge atoms on the particle surface are active sites for the Suzuki reaction. The lower catalytic activity for the smallest Pd nanoparticles may be due to stronger adsorption of the reaction intermediates on the particle surface, in which the strongly adsorbed species act as a poison to the reaction thereby decreasing the rate of the reaction.



Scheme 34: Reaction between phenylboronic acid and iodobenzene by PVP-Pd NPs.



Scheme 35: Synthetic strategy for the preparation of PVP-grafted silica with $PdCl_2$ catalyst.

In another work, Tamami et al., [39] was prepared a catalytic system based on palladium nanoparticles supported on poly(*N*-vinylpyrrolidone) grafted silica (Scheme 35).

The complexation of PVP-grafted silica with $PdCl_2$ was carried out to obtain the heterogeneous catalytic system. Transmission electron microscopy (TEM) image showed that palladium dispersed through the support in nanometer size (Figure 1).

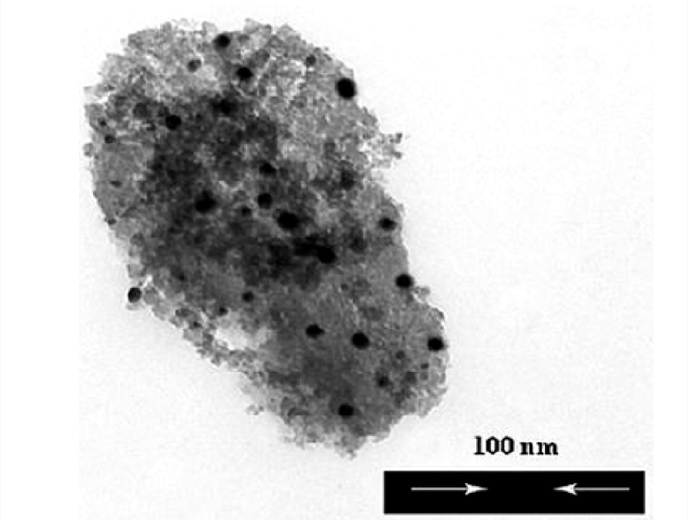
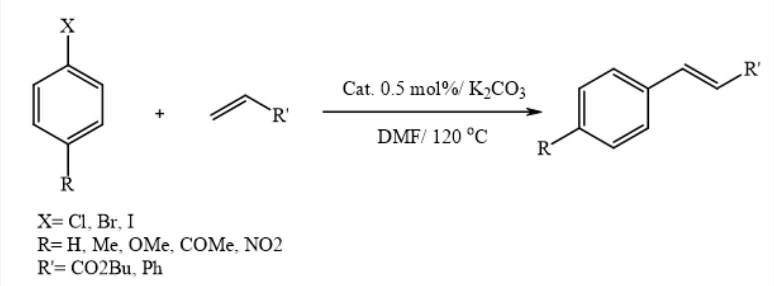


Figure 1: Transmission electron microscopy (TEM) image of palladium supported on polyN-vinylpyrrolidone)-grafted silica [39].



Scheme 36: Cross-coupling reactions of aryl iodides, bromides and also chlorides with olefinic compounds by PVP-grafted silica with $PdCl_2$ catalyst.

This catalytic system exhibited excellent activity in cross-coupling reactions of aryl iodides, bromides and also chlorides with olefinic compounds in Heck-Mizoraki reactions in short reaction time and high yields (Scheme 36).

Conclusion

Polymer-supported reagents and catalysts have emerged as important tools for the rapid preparation of chemical compounds in solution-phase. Clean methodologies, easy preparation of the catalysts, simple work-up procedures, good to high yields, environmentally friendly and reusable catalysts are some advantages of polyvinylpyrrolidone supported catalysts and reagents in organic multi-step synthesis. Further progresses in the development of new PVP-bound reagents, and catalysts will continue to attract innovative application of this strategy in multi-component synthesis.

Acknowledgements

Financial support by Rasht Branch, Islamic Azad University is gratefully acknowledged.

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