Oxidomolybdenum based catalysts for sulfoxidation reactions: A brief Review

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Molybdenum-catalyzed oxygen atom transfer reactions have attracted considerable interest due to their relevance to some biological processes. In this context, some oxidomolybdenum complexes are explored as catalysts for organic transformations such as oxidation reactions. The roles of oxidomolybdenum complexes as catalysts for sulfoxidation reactions under homogeneous and heterogeneous conditions are reviewed. The utilities of oxidomolybdenum complexes have been further extended for environmentally friendly oxidation of sulfides under aqueous conditions using hydrogen peroxide as green oxidant.

Keywords: Oxidomolybdenum, catalyst, sulfoxidation, green oxidant.

Introduction

Oxidation reactions are most fundamental transformations in organic synthesis\(^1,\text{2}\) e.g. oxidation of alcohols\(^2\text{a}\), olefins\(^2\text{b}\), thiols\(^2\text{c}\) and sulfides\(^2\text{d}\) to corresponding carbonyls, epoxides, disulfides and sulfoxides/sulfones, respectively. Among the above mentioned oxidized products, many sulfoxides and sulfones are used as versatile intermediates for synthesis of natural products\(^3\). Moreover, these compounds have been proved to be active pharmaceutical ingredients for several therapeutic drug molecules\(^4\). They often play important roles due to their anti-ulcer (proton pump inhibitor), antibacterial, antifungal, anti-atherosclerotic, anthelmintic, antihypertensive, and cardiotonic activities.

Sulfoxidation of organic sulfides is a straightforward method for the selective preparation of sulfoxides or sulfones. Traditional approaches for sulfoxidation reactions involve the usage of peracids\(^5\) or halogen derivatives\(^2\text{d}\); however, they yield stoichiometric amounts of environmentally undesired by-products. In recent days, alternative approaches involving the use of transition metal based catalysts along with eco-friendly hydrogen peroxide as oxidant have been developed for oxidation of sulfides\(^6\).

Molybdenum is earth abundant and belongs to a unique family of transition metals. Various oxido-forms of molybdenum are present in many enzymes viz. xanthine oxidase, aldehyde oxidase and sulfite oxidase. Such enzymes accomplish important physiological redox functions in human body. Modeling of the molybdenum based enzymes mostly includes the study of oxygen atom transfer (OAT) reactions. Mechanistic aspects of oxidomolybdenum complexes catalyzed organic transformations, mostly oxidation reactions, are related to the understanding of functional activities of molybdenum based enzymes.

Use of oxidomolybdenum based catalysts for various organic transformations is a recent trend and reviewed by us and others\(^7\text{–11}\). The purpose of the present review is to describe and update a variety of representative oxidomolybdenum based homogeneous and heterogeneous catalysts for sulfoxidation reactions using peroxide based oxidants. Simple sulfoxidation protocols discussed in this review found practical use and hence highlighted at the last section.

1. Homogeneous catalysis

(a) Oxidomolybdenum cores with mono and/or bidentate ligands:

Oxidomolybdenum complexes having general formula Mo(O)\(_x\)(O\(_2\))\(_y\)(L)\(_z\) where L represent anionic/neutral ligands bearing one or more donor atoms are well known. However, utilization of such compounds as catalysts for sulfoxidation reactions has gained importance only in recent years (Fig. 1, Table 1).
Oxidation of thioanisole with tert-butyl hydroperoxide in benzene medium and in presence of MoO$_2$(acac)$_2$ (1) as catalyst produced methyl phenyl sulfone in 98% yield. Formation of peroxidomolybdenum species followed by electrophilic oxygen transfer to the sulfide is proposed in these reactions. Catalytic amount of MoO$_2$Cl$_2$ (2) or its solvent adducts selectively catalyzed the oxidation of sulfides to sulfoxides or sulfones by varying the amount of catalyst (1.5 or 15 mol%), oxidant i.e. H$_2$O$_2$ (1.05 or 4 equivalents) and solvent (acetone/water or acetonitrile). Importantly, the catalyst 2 could also selectively oxidize sulfides without harming sensitive substituents like olefin, acetylene, alcohol, aldehyde, ester and oxime when present in the sulfide. Whereas the use of 1 as a catalyst for sulfoxidation of an oxime functionalized sulfide provided a mixture of compounds along with the targeted sulfoxide that included the starting sulfide, over-oxidized sulfone, cleavage of oxime bonds in the products and starting material etc.

Similarly, catalytic amount of MoO$_3$ (3) oxidized sulfides to sulfoxides and sulfones with 1.3 and 2.6 equivalents of 30% H$_2$O$_2$, respectively as an oxidant. Moreover MoO$_3$ (3) selectively oxidized the sulfides in the presence of alcohol and acetate functional groups. The catalyst MoO$_2$(S-BINOL)(THF)$_2$ (4) successfully oxidized methyl phenyl sulfide to respective sulfoxide and sulfone in dichloromethane with 1 and 2 equivalents of 5–6 M TBHP in n-decane, respectively. Unfortunately, the catalyst showed less enantioselectivity. The related complexes MoO$_2$Cl$_2$(L)$_2$...
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[where \( L = (R)\)\(^{-}\)methyl-\( p \)-tolylsulfoxide, \( 5 \) or \((S,S)\)\(^{-}\)bis\((p\)-tolyl sulfinyl)methane, \( 6 \) were used as catalyst for the oxidation of methyl-\( p \)-tolylsulfide to corresponding sulfoxide in dry THF using 1.00 equivalent of 70% aq. TBHP as oxidant. The use of these catalysts could not give rise to any significant enantiomeric excess in the resulting sulfoxides\(^{17} \).

The molybdenum(VI) oxido-peroxido complex \( \text{Mo}(\text{O})(\text{O}_2)(\text{phox}) \), \( 7 \) has been found to be an excellent catalyst for oxidation of various aryl and aliphatic sulfides to sulfoxides in acetonitrile using TBHP as an oxidant\(^{18} \). A variety of Mo\(^{VI} \) complexes i.e. \( \text{MoO}_2X_2L_2 \) (\( X = \) halide or \( \text{Me, L neutral ligand} \) behaved as catalysts for sulfoxidation reactions using \( \text{H}_2\text{O}_2 \) or TBHP as oxidant while the peroxide coordinated to molybdenum center transferred oxygen to the sulfides\(^{19} \). The oxidomolybdenum complex, \( \text{MoO}_2\text{Cl}_2(4,4'\text{-di tert-buty l}-2,2'-\text{bipyridine}) \), \( 10 \) has been used for catalytic oxidation reaction of sulfides using urea hydrogen peroxide (UHP) as a green oxidant. The complex \( 10 \) oxidized various aliphatic and aryl sulfides to corresponding sulfoxides and sulfones using 1 and 5 equivalents of UHP, respectively\(^{23} \). Similar kind of oxidomolybdenum complex comprising of \( \text{C}_3 \) symmetric amino tris-\( \text{tert} \)-butyl phenolate ligand oxidized \( \text{p} \)-tolyl methyl sulfide to corresponding sulfoxide using 0.1 M aq. \( \text{H}_2\text{O}_2 \) as an oxidant\(^{24} \). A series of cis-oxidomolybdenum(VI) complexes with different chiral ONO-type ligands including \( 12 \) were synthesized and screened for oxidation reaction of aryl sulfides with 30% aq. \( \text{H}_2\text{O}_2 \) as an oxidant\(^{25} \). A variety of \([\text{MoO}_2(\text{L})(\text{D})]\) type oxidomolybdenum complexes where \( \text{L stands for tridentate ligand and \"D\" for additional monodentate ligand are known in literature}\(^{22} \). The focus of this part of the review is to describe catalytic activities of such complexes and related complexes containing or ONOO type tetradeinate ligand (\( 10-13 \)) for oxidation of sulfides (Fig. 2, Table 2).

The oxidomolybdenum complex, \([\text{MoO}_2(\text{L})(\text{CH}_3\text{OH})]\), \( 10 \) has been used for catalytic oxidation reaction of sulfides using urea hydrogen peroxide (UHP) as a green oxidant. The complex \( 10 \) oxidized various aliphatic and aryl sulfides to corresponding sulfoxides and sulfones using 1 and 5 equivalents of UHP, respectively\(^{23} \). Similar kind of oxidomolybdenum complex \( 11 \) comprising of \( \text{C}_3 \) symmetric amino tris-\( \text{tert} \)-butyl phenolate ligand oxidized \( \text{p} \)-tolyl methyl sulfide to corresponding sulfoxide using 0.1 M aq. \( \text{H}_2\text{O}_2 \) as an oxidant\(^{24} \). A series of cis-oxidomolybdenum(VI) complexes with different chiral ONO-type ligands including \( 12 \) were synthesized and screened for oxidation reaction of aryl sulfides with 30% aq. \( \text{H}_2\text{O}_2 \) as an oxidant\(^{25} \). A series of related molybdenum(VI) complexes containing \( \text{4,6-O-ethylidene-\( \beta \)-D-glucopyranosylamine derived ligands\,(e.g.} \) \( 13 \) have been employed for selective oxidation of sulfides

(b) Oxidomolybdenum core with tri or tetradeinate ligands:

A variety of \([\text{MoO}_2(\text{L})(\text{D})]\) type oxidomolybdenum complexes where \( \text{L stands for tridentate ligand and \"D\" for additional monodentate ligand are known in literature}\(^{22} \). The focus of this part of the review is to describe catalytic activities of such complexes and related complexes containing or ONOO type tetradeinate ligand (\( 10-13 \)) for oxidation of sulfides (Fig. 2, Table 2).

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Fig. 2. Oxidomolybdenum(VI) compounds \( 10-13 \) used for sulfoxidation reactions.

Table 2. Oxidation of sulfides using the catalysts \( 10-13 \)

<table>
<thead>
<tr>
<th>Sl. No.</th>
<th>Catalyst</th>
<th>Oxidant</th>
<th>Sulfide</th>
<th>Selectivity</th>
<th>Ref. No.</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>( 10 )</td>
<td>UHP</td>
<td>Aliphatic and aryl sulfides</td>
<td>Sulfoxides or sulfones</td>
<td>23</td>
</tr>
<tr>
<td>2.</td>
<td>( 11 )</td>
<td>( \text{H}_2\text{O}_2 )</td>
<td>( \text{p} )-Tolyl methyl sulfide</td>
<td>Sulfoxide</td>
<td>24</td>
</tr>
<tr>
<td>3.</td>
<td>( 12 )</td>
<td>( \text{H}_2\text{O}_2 )</td>
<td>Aryl sulfides</td>
<td>Sulfoxides</td>
<td>25</td>
</tr>
<tr>
<td>4.</td>
<td>( 13 )</td>
<td>( \text{H}_2\text{O}_2 )</td>
<td>Aryl sulfides</td>
<td>Sulfoxides</td>
<td>26</td>
</tr>
</tbody>
</table>
to the corresponding sulfoxides using 1 equivalent UHP as an oxidant. All molybdenum complexes related to 13 showed good activities towards sulfides and corresponding sulfoxides are obtained in good to excellent yields 26.

(c) Polyoxidomolybdate cores:

Polyoxidometalates including polyoxido molybdates are known for a variety of organic transformations 27. The major focus of this part of review is oxidation of various sulfides using polyoxidomolybdate as catalyst and peroxide as oxidant (Table 3). A simple and efficient method for the oxidation of sulfides to sulfoxides at room temperature is developed using ammonium heptamolybdate, (NH4)6Mo7O24·4H2O (14) as catalyst with 4 equivalents of 30% H2O2 as an oxidant. The reactions provided excellent yields of sulfoxides and sensitive functional groups such as allyl, vinyl, propargyl, alcohol, ketone, ester, pyridine and nitrile were found to be tolerated 28. Tetra-(tetraalkylammonium)octamolybdate, [(n-C8H17)4N]+(α-Mo9O26) (15) was successfully demonstrated for selective oxidation of various sulfides to sulfoxides with 30% aqueous hydrogen peroxide as oxidant under mild reaction conditions. The catalyst 15 tolerated active functional groups like hydroxyl and C=C bond while doing oxidation of sulfides under optimized condition 29. The selective oxidation of sulfides to corresponding sulfoxides are achieved by an Anderson-type catalyst sodium hexamolybdocromate(III) (16) with 30% H2O2 in acetonitrile at 60°C. The catalyst 16 successfully oxidized several diaryl, alkyl-aryl and dialkyl sulfides to respective sulfoxides with high yields 30. Heteropolycompounds were screened for controlled oxidation of sulfides to sulfoxides and sulfoxides using 35% H2O2 as an oxidant. An acidic salt of heteropolycompound, (PyH)(H3PMo11 VO40), 17 was found to be an effective catalyst for oxidation of various aliphatic and aryl sulfides with oxidant (35% H2O2) to sulfoxides and sulfones in acetonitrile at 20°C and 40°C, respectively 31. The catalytic activity of three polyoxidometalates, Mo72M30 (M = V, Cr, Fe) are screened for oxidation of various sulfides to sulfoxides in water using 30% H2O2 as an oxidant. Among three catalysts, Mo72Cr30 (18) showed high catalytic activities towards environmentally friendly oxidation of various allyl and aryl sulfoxides with aqueous 30% H2O2 32. The four alkyl imidazolium and pyridinium salts based octamolybdates were screened for controlled oxidation of sulfides to sulfoxides and sulfoxides with 1.5 and 3 equivalence 30% H2O2, respectively. Among the four octamolybdates, [Bmim]4Mo9O26 (Bmim = 1-butyl-3-methylimidazolium), 19 showed good catalytic activity towards oxidation of sulfides in acetonitrile. Moreover the catalyst 19 showed excellent reusability up to 8 cycles 33. A chiral bisguanidinium dinuclear complex oxidodiperoxido-molybdosulfate ([μ-SO4]Mo2O2(μ-O2)2(O2)2)32− (9) is an ion pair and acted as a catalyst for enantioselective sulfoxidation using aqueous H2O2 as terminal oxidant 31,34.

2. Heterogeneous catalysis

A number of heterogeneous molybdenum based catalysts have been exploited for different kind of oxidation reactions 35. In this part of review, oxidation of sulfides to sulfoxides and/or sulfoxides is discussed (Fig. 3, Table 4).

The polymeric catalyst [MoO3(2,2’-bipy)]n (20) is employed for the oxidation of sulfides to sulfoxides and sulfoxides, using tert-butylhydroperoxide as the stoichiometric oxidant. The catalyst possesses several advantages because of its stability and possibilities of recycling 36. Enantioselective oxidation of thioanisole in water was explored by Mo VI supramo-

### Table 3. Oxidation of sulfides using the catalysts 14-19

<table>
<thead>
<tr>
<th>Sl. No.</th>
<th>Catalyst</th>
<th>Oxidant</th>
<th>Sulfide</th>
<th>Selectivity</th>
<th>Ref.</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>(NH4)6Mo7O24·4H2O, 14</td>
<td>H2O2</td>
<td>Aliphatic and aryl sulfides</td>
<td>Sulfoxones</td>
<td>28</td>
</tr>
<tr>
<td>2.</td>
<td>[(n-C8H17)4N]4(α-Mo9O26), 15</td>
<td>H2O2</td>
<td>Aliphatic and aryl sulfides</td>
<td>Sulfoxides</td>
<td>29</td>
</tr>
<tr>
<td>3.</td>
<td>Na3[CrMo7O24H16]8H2O, 16</td>
<td>H2O2</td>
<td>Aliphatic and aryl sulfides</td>
<td>Sulfoxides</td>
<td>30</td>
</tr>
<tr>
<td>4.</td>
<td>(PyH)(H3PMo11 VO40), 17</td>
<td>H2O2</td>
<td>Aliphatic and aryl sulfides</td>
<td>Sulfoxides or sulfoxones</td>
<td>31</td>
</tr>
<tr>
<td>5.</td>
<td>Mo72Cr30, 18</td>
<td>H2O2</td>
<td>Aliphatic and aryl sulfides</td>
<td>Sulfoxides</td>
<td>32</td>
</tr>
<tr>
<td>6.</td>
<td>[Bmim]4Mo9O26, 19</td>
<td>H2O2</td>
<td>Aliphatic and aryl sulfides</td>
<td>Sulfoxides or sulfoxones</td>
<td>33</td>
</tr>
</tbody>
</table>
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Molecular catalysts with β-cyclodextrin-based ligands using 30% $\text{H}_2\text{O}_2$ as an oxidant. The MoVI complex, 21 showed moderate enantioselectivity towards oxidation of thioanisole to methyl phenyl sulfoxide. Selective catalytic oxidation of thioethers to sulfoxides by using a special type of Mo-silicate sample on an organic template with Si/Mo ratio of 80, 22 and 30% $\text{H}_2\text{O}_2$ as an oxidant was explored. The catalyst 22 oxidized various types of alkyl and aryl sulfides to corresponding sulfoxides. Oxidodiperoxidomolybdenum complex [MoO($\text{O}_2$)$_2$(OPyr)($\text{H}_2\text{O}$)] was coated with silica gel to prepare the material, 23 that was explored for the oxidation of sulfides to sulfoxides selectively. Equimolar amount of

Table 4. Oxidation of sulfides using the catalysts 20-28

<table>
<thead>
<tr>
<th>Sl. No.</th>
<th>Catalyst</th>
<th>Oxidant</th>
<th>Sulfide</th>
<th>Selectivity</th>
<th>Ref. No.</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>[MoO$_2$(2,2'-bipy)]$_n$. 20</td>
<td>TBHP</td>
<td>Aliphatic and aryl sulfides</td>
<td>Sulfoxides or sulfones</td>
<td>36</td>
</tr>
<tr>
<td>2.</td>
<td>β-CD-MoVO, 21</td>
<td>H$_2$O$_2$</td>
<td>Thioanisole</td>
<td>Sulfoxide</td>
<td>37</td>
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<tr>
<td>3.</td>
<td>Mo-Si sample, 22</td>
<td>H$_2$O$_2$</td>
<td>Aliphatic and aryl sulfides</td>
<td>Sulfoxides or sulfones</td>
<td>38</td>
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<tr>
<td>4.</td>
<td>Silica coated-MoO(O$_2$)$_2$(Opyr), 23</td>
<td>–</td>
<td>Aliphatic and aryl sulfides</td>
<td>Sulfoxides or sulfones</td>
<td>39</td>
</tr>
<tr>
<td>5.</td>
<td>β-CD-MoVI, 24</td>
<td>H$_2$O$_2$</td>
<td>Aryl sulfides</td>
<td>Sulfoxides</td>
<td>40</td>
</tr>
<tr>
<td>6.</td>
<td>Supported-PMo$<em>{12}$O$</em>{40}$, 25</td>
<td>H$_2$O$_2$</td>
<td>Aliphatic and aryl sulfides</td>
<td>Sulfoxides</td>
<td>41</td>
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<tr>
<td>7.</td>
<td>Supported-MoO$_2$Cl$_2$, 26</td>
<td>H$_2$O$_2$</td>
<td>Aliphatic and aryl sulfides</td>
<td>Sulfoxides</td>
<td>42</td>
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<tr>
<td>8.</td>
<td>Supported-[Mo(O)$_2$(O)$_2$(L)$_2$]$^{2-}$, 27a/b</td>
<td>H$_2$O$_2$</td>
<td>Aliphatic and aryl sulfides</td>
<td>Sulfoxides or sulfones</td>
<td>43</td>
</tr>
<tr>
<td>9.</td>
<td>(CTA)$_2$[MoO(O$_2$)$_2$(C$_2$O$_4$)$_2$]$\cdot$H$_2$O, 28</td>
<td>H$_2$O$_2$</td>
<td>Aliphatic and aryl sulfides</td>
<td>Sulfoxides or sulfones</td>
<td>45</td>
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</tbody>
</table>
corresponding uncoated $\text{Mo}^{\text{VI}}$ complex however oxidized aliphatic and aryl sulfides to sulfones\(^{38}\). The modified $\beta$-cyclodextrin ($\beta$-CD) derivatives having catechol-type ligand (2,3- and 3,4-dihydroxy groups on the benzoate ring) are synthesized and corresponding $\text{Mo}^{\text{VI}}$ complexes are used for enantioselective oxidation of sulfides to sulfoxides. The molybdenum complex 24 oxidized different aryl sulfides to sulfoxides in good yields with moderate enantioselectivity\(^{40}\). The molybdenum based heteropolyacid ($\text{H}_3\text{PMo}_{12}\text{O}_{40}$), immobilized on aminopropyl-functionalized silica catalyst, 25 could catalyze the oxidation of aliphatic and aromatic sulfides to corresponding sulfoxides. The catalyst shows good catalytic activity towards various sulfides with 35% $\text{H}_2\text{O}_2$ as an oxidant and corresponding sulfones are isolated in good yields\(^{41}\).

The oxidomolybdenum complex $\text{Mo}_2\text{O}_2\text{Cl}_2(\text{DMF})_2$ immobilized on different polystyrene resins as ligands and resulting $\text{Mo}^{\text{VI}}$ complexes are used for oxidation of sulfides. The catalyst 26 shows good catalytic activity towards various aliphatic and aryl sulfides to sulfoxides with good yields\(^{42}\). The heterogeneous catalysts supported-[MoO$_2$(O$_2$L)$_2$][Cl]$_2$- 27a-b [where, L = valine or alanine] immobilized on Merrifield resin, which exhibit excellent activity, stability and selectivity for the oxidation of sulfides to the corresponding sulfoxides and sulfones with $\text{H}_2\text{O}_2$ at ambient temperature\(^{43}\). Catalysis using metalloamic acid compounds under aqueous conditions is considered versatile and ecofriendly for organic transformations\(^{44}\). A new molybdenum based metalloamic acid catalyst ($\text{C}_{19}\text{H}_{42}\text{N}_2\text{MoO}_3\text{O}_{10}$)$_2$($\text{C}_2\text{O}_4$)$_2$-$\text{H}_2\text{O}$, 28 was developed and utilized for oxidation of various sulfides to sulfoxides and sulfones using 1 and 3 equivalents of 30% $\text{H}_2\text{O}_2$, respectively as oxidant. The micro heterogeneous metalloamic acid catalyst 28 depicted good recyclability in water\(^{45}\).

3. Practical applications

Literature search on the actual use of molybdenum complexes considered in this review revealed noteworthy points. The catalyst 1 ($\text{MoO}_2$(acac)$_2$) is known for deprotection of acetals\(^{46}\) and found to be useful in organic synthesis\(^{47}\). So far as sulfoxidation reactions are concerned, the catalyst 2 ($\text{MoO}_2\text{Cl}_2$) behaved profoundly. Selective and controlled oxidation of sulfides to corresponding sulfoxides or sulfones using a mild catalyst system $\text{MoO}_2\text{Cl}_2$. 2 is presented in Table 1, entry 3. This catalyst also tolerated a variety of sensitive functional groups present in the substrate and hence attracted wide attention. The protocols found practical uses in different laboratories for synthesis of targeted sulfoxides/sulfones\(^{48}\).

Conclusion

In recent years, oxidomolybdenum based catalysts are being established for various oxidation reactions. A notable progress has been made especially for oxidation of sulfides to sulfoxides and sulfones. Sulfide oxidation using peroxide (ROOH) as terminal oxidant and carried out in organic media is usually practiced. A definite outlook in this field would be to develop workable oxidomolybdenum complexes as catalyst that could be utilized under aqueous conditions. This would allow fine chemical production of sulfoxides and sulfones in industrial scale while taking care of the environment.

Acknowledgements

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