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Kinetic and Mechanistic Study of Selective Oxidation Reactions by SomeCr(VI) Complexes: A Review

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Abstract

Chromium complexes of the hexavalent state are very effective and selective oxidizing agents broadly employed to oxidize many organic substrates with mild reaction conditions. This review provides basic information about the kinetics and mechanisms of selective oxidation reactions catalyzed by the various Cr(VI) complexes. Alcohols, aldehydes, ketones, phenols, amines, sulfides, and other functional group-containing molecules are the substrates covered. Some general experimental techniques used to estimate reaction rates, reaction order, isotope effects, and other kinetic parameters are discussed. Different methods, which again include DFT calculations and molecular dynamics simulations that provide information on transition states, intermediates, and reaction pathways, are also explored. Concerning the reactivity and selectivity of the process, the influence of the type of Cr(VI) complex, ligands employed, solvents, additives, and reaction conditions are described in detail. The trends in reactivity are analyzed in terms of electronic and steric factors of substrates. Possible mechanisms are outlined, along with the contemporary developments and the questions raised. In conclusion, current advancements in the use of Cr(VI)-mediated oxidation processes in organic synthesis, catalysis, and green chemistry are summarized with a focus on the future directions. This review aims to synthesize a mechanistic model for the different reactivities of Cr(VI) complexes and guide the future design of oxidation processes.

Keywords

Chromium(VI) complexes, oxidation, reaction kinetics, transition states, catalysis, green chemistry, organic synthesis

1. Introduction

Selective oxidation is one of the fundamental transformations in organic chemistry, which makes it possible to introduce oxygen-containing groups into the molecule while preserving the carbon skeleton. It is one of the essential reactions in producing fine chemicals, pharmaceuticals, Agrochemicals, and materials. The selectivity and efficiency of the oxidation reactions are critical issues, especially when using multi-functionalized substrates [1].Due to the advances in research, the chromium complexes in the highest oxidation state of +6 are efficient and selective oxidation reagents under mild conditions. Chromium(VI) complexes exhibit different reactivity and selectivity to the various organic substrates in the chemoselective and regioselective oxidation of alcohols, aldehydes, amines, sulfides, and unsaturated hydrocarbons, among others. These species are highly reactive because of their high redox potential, Lewis acidity, and ability to form highly reactive intermediates such as the chromate esters and oxo-chromium species. The oxidizing power and the selectivity of the complex can be altered by modifying the ligands around the chromium center of the complex due to the variation of its electronic and steric characteristics [2].

Understanding the kinetics and mechanisms of Cr(VI) induced oxidations helps design efficient and green oxidation processes. It can be used to establish the rate-determining step, reaction orders, as well as activation parameters in a certain reaction, while mechanism studies help one to determine intermediates, transition states, and pathways. Some recent investigations have been conducted on the mechanism of Cr(VI) oxidations with the help of both experimental and theoretical methods. These species have been identified and characterized by spectroscopic techniques such as UV-visible, EPR, and NMR spectroscopy, and the mechanism and transition state have been mapped out using kinetic isotope effects and linear free energy relationships. Density functional theory (DFT) calculations and molecular dynamics simulations have complemented experimental approaches and provide a molecular-level understanding of reaction pathways and energy profiles [3].

2. Properties and Reactivities of Cr(Vi) Oxidants

Cr(VI) can be coordinated with various ligands, counterions, and coordination numbers due to its highest oxidation state of +6. The most common Cr(VI) oxidants applied in organic synthesis are chromate and dichromate anions, CrO_3, and organochromium compounds, including PCC and PDC.The Cr(VI) complexes have high oxidizing ability due to the high redox potential of the Cr(VI)/Cr(III) couple, which is between +0.4 to +1. The later is in the range of 3V vs. NHE depending on the ligand environment and reaction conditions. The Cr(VI) center is electrophilic since it has a d^0 electronic configuration and easily gets reduced by two electrons to form Cr(IV) species that can be further reduced to form Cr(III) [4].

The reactivity of Cr(VI) complexes can be significantly affected by the nature of ligands bound to the metal ion. Ligands can change some physical and chemical characteristics of the complex and, as a result, its ability to oxidize, its selectivity, and its stability. For example, chromate and dichromate anions, which are electron definition ligands, enhance the electrophilicity of the Cr(VI) center and its oxidative ability, while the steric demanding ligands like pyridine, quaternary ammonium cations help in stabilizing the complex [5]. The part played by solvents cannot be taken lightly when discussing the reactivity and selectivity of Cr(VI) oxidations. The solvent can influence the stability of reactive intermediates, the formation of reactant complexes, and the solvation of transition states through its dielectric constant, polarity, and hydrogen bonding ability. Therefore, polar aprotic solvents such as DMF, DMSO, and acetonitrile are generally used in Cr(VI) oxidation reactions because they can stabilize charged species and transitory species best, while polar protic solvents such as water and alcohols are nucleophilic or can act as proton donors [6].

There are also the species distribution and reactivity of Cr(VI) species in the reaction medium, which is dependent on the pH. The Cr(VI) is more stable in the acidic medium. The species present is the chromate ion CrO_4^{2-} which is a stronger oxidizing agent than the dichromate ion $Cr_2O_7^{2-}$ which is formed in a neutral to mildly acidic medium. Under very acidic conditions, the chromate ion will react with protons to produce very aggressive species like H_2CrO_4 and $HCrO_4^-$ On the other hand, in alkaline conditions, $Cr(OH)_6^-$ species are formed, which reduces the Cr(VI)complex's ability to oxidize. Reduction of Cr(VI) complexes generally includes the transfer of an oxygen atom to the substrate to form Cr(IV) or Cr(V) species, which further reacts with the substrate or disproportionate to give Cr(III) and Cr(VI). Some of the reactive oxochromium species, such as Cr(V)=O and Cr(IV)=O, have been proposed to be involved in the Cr(VI) dependent oxidation processes, and their participation has been proved by spectroscopic and kinetic methods [7].

3. Oxidation of Alcohol

Conversion of alcohol to carbonyl compounds is one of the most well-researched reactions catalyzed by Cr(VI) complexes. Alcohols are good substrates for Cr(VI) oxidation due to the ease with which chromate esters can be formed and subsequently oxidized to aldehydes or ketones. This reaction is generally selective for the primary and secondary alcohols, but tertiary alcohols are less readily oxidized due to steric hindrance. Many workers have investigated the oxidation of alcohol by Cr(VI), both with respect to rate and spectral data. The mechanism that has been suggested is the following: the alcohol attacks the Cr(VI) center to form a chromate ester intermediate, and the ester is then slowly hydrolyzed to form the carbonyl compound and a Cr(IV) species. The Cr(IV) intermediate can then be reduced to Cr(III) or disproportionate to give Cr(VI) and Cr(V) [8].

This approach is for the microwave-assisted oxidation of alcohols to carbonyl compounds utilizing wet alumina-supported ammonium chlorochromate in a solvent less system. Mohammadi et al. delineated an effective and gentle approach for the oxidation of alcohols and polyarenes utilizing TMAFC under microwave irradiation conditions.

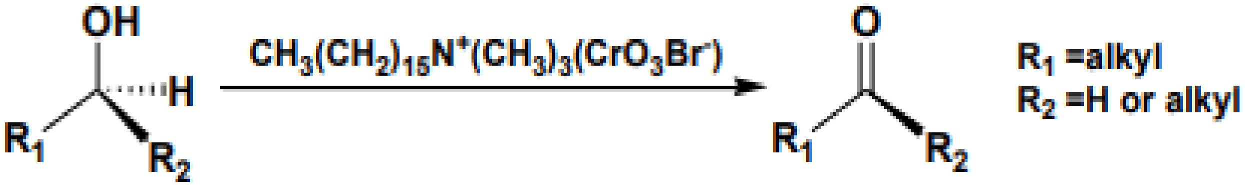

Figure 1. Oxidation of alcohol using TMAFC

The kinetics of the reaction between alcohol and Cr(VI) has been determined to be first-order in the alcohol and Cr(VI) complex, implying that the rate-determining step is a second-order process. The reaction is also first order in acid concentration; that is to say, to decompose the chromate ester, it must be protonated. Hammett's studies have shown that the rate of oxidation increases with electron-withdrawing groups at the alcohol and decreases with electron-donating groups, and this correlates with the formation of a chromate ester intermediate, which is stabilized by the electrons from the alcohol. The isotope labeling studies have also provided more support to the chromate ester mechanism of the reaction. The primary KIE is observed in the Cr(VI) oxidation of α-deuterated alcohols, and this indicates that the C-H bond cleavage is a part of the rate-determining step. Contrary to the above-mentioned reaction, the oxidation of β-deuterated alcohols has a much smaller KIE, which implies that the rate-determining step is not the cleavage of the C-H bond [9].

The role of chromate ester intermediates has also been backed up by spectroscopic data. The formation of chromate esters in the reaction of alcohols with Cr(VI) has been confirmed by UV-visible spectroscopy with absorption bands at wavelengths of 350−400 nm. Other techniques like NMR spectroscopy have also been used in chromate esters where the downfield shift of the carbinol carbon and hydrogen signals have been observed upon coordination with the Cr(VI) center. The nature of the Cr(VI) complex and the reaction conditions used in the oxidation process can control the rate of the alcohol oxidation and the selectivity of the process. For example, the use of Pyridinium chlorochromate (PCC) gives better reaction rates and yields than chromium trioxide because the chromate ester, which is an intermediate, is stabilized by the pyridinium cation. The oxidation of alcohol can be made faster and more selective when biphasic conditions are used with a phase-transfer catalyst to facilitate the formation and decomposition of the chromate ester at the organic/aqueous interface [10].

4. Oxidation of Aldehydes and Ketones

Another important reaction that takes place in organic synthesis is the oxidation of aldehydes and ketones by Cr(VI) complexes in this case, carboxylic acids and esters are formed, respectively. This reaction is generally less selective than alcohol oxidation because the carbonyl group is more reactive, and over-oxidation to CO_2 and other compounds is possible. Although the mechanism of aldehyde and ketone oxidation by Cr(VI) is less certain than that of alcohol oxidation, several mechanisms have been proposed on the basis of kinetics and spectroscopy. A possible mechanism seems to be the formation of chromate ester via nucleophilic attack of the enolized carbonyl carbon of the aldehyde or ketone on the Cr(VI) and then the oxidation of the ester product into the carboxylic acid or the ester [11].

It has been synthesized the kinetics of oxidation of six aliphatic aldehydes by QFC in dimethylsulphoxide (DMSO) as the solvent. The oxidation of aldehydes resulted in the formation of corresponding carboxylic acids.

$$\mathrm{RCHO + CrO_2FOQH \longrightarrow RCOOH + CrOFOQH}$$

The reactions are of first order with respect to QFC. Michaelis-Menten type kinetics are observed with respect to the aldehydes. This leads to the postulation of following overall mechanism and rate law.

$$\text{Aldehyde} + \text{QFC} \overset{K}{\rightleftarrows} [\text{complex}]$$

$$\text{Complex} \xrightarrow{k_2} \text{Products}$$

$$\text{Rate} = k_2\,K\,[\text{aldehyde}]\,[\text{QFC}] \,/\, (\,1 + K\,[\text{aldehyde}])$$

Another process is the one-electron reduction of the carbonyl compound by the Cr(VI) center to form a Cr(V) species followed by a two-electron reduction to give the product and a Cr(III) species. This mechanism is in accord with the observation of Cr(V) intermediate species in the reaction of aldehydes and ketones with Cr(VI) by EPR spectroscopy. It has been found that phase-transfer catalysts and crown ethers improved the oxidation rate and selectivity of aldehyde and ketone using Cr(VI). For example, when using 18-crown-6 as a phase-transfer catalyst, the oxidation of benzaldehyde with pyridinium chlorochromate (PCC) in a two-phase system; the rate and the yield of the formation of benzoic acid were considerably enhanced [12].

5. Oxidation of Phenols and Aromatic Alcohols

Chromium (VI) complexes mediated oxidation of phenols and aromatic alcohols to produce quinones, aromatic aldehydes and carboxylic acids is a useful oxidative reaction. The reaction is normally more complex than the oxidation of aliphatic alcohols and carbonyl compounds since it involves the benzene ring, and many pathways of oxidation are possible [13].

Phenol has been reacted with Cr(VI) for various techniques, namely kinetic, spectroscopic, and computational. This mechanism involves the generation of a chromate ester intermediate through the nucleophilic attack of the phenoxide ion at the Cr(VI) center, and the rate-determining step is the decomposition of the ester to form quinone and a Cr(IV) species. The Cr(IV) intermediate can be further reduced to Cr(III) or can be disproportionate to form Cr(VI) and Cr(V) species [14].

The oxidation of benzyl alcohol by dichromate and Cr(VI) complexes exhibits identical kinetic orders; the oxidation conforms to the isokinetic and Exner relationships, by the UV–visible spectrum of the reaction solution after completion of the reaction (578 and 397 nm), were identified as the reaction products of Cr(VI) – benzyl alcohol reaction. Hence the reaction has to be as:

$$Cr_2O_7^{2-} + 8H^+ + 3PhCH_2OH \rightarrow 3\ PhCHO + 2Cr^{3+} + 7H_2O$$

This was further confirmed by kinetic study; kinetic investigation under identical conditions with benzaldehyde as substrate reveals that Cr(VI) oxidation of benzaldehyde does not occur under the experimental conditions. The reaction rate of phenol oxidation by Cr(VI) was determined to be first order with respect to both the phenol and the Cr(VI) complex, meaning that the rate-determining step is a bimolecular process. It is also a first-order reaction with respect to base concentration, meaning that phenol must first be deprotonated to form the phenoxide ion before it can form the chromate ester. With regard to Hammett studies, it has been noticed that electron-withdrawing groups on the phenol increase the rate of oxidation while electron-donating groups have the opposite effect, which is in line with chromate esterintermediate that is stabilized by the electron donation from the phenoxide anion [15].

6. Oxidation of Amines and Other Nitrogen-Containing Compounds

Complexation of amines and other nitrogen-containing compounds with Cr(VI) complexes is a good approach to synthesizing imines, nitriles, amides, and other nitrogen heterocycles. The reaction is often more complex than the oxidation of alcohols and carbonyls due to the presence of nitrogen and the possible formation of several products. This work reports on the interaction of Cr(VI) with amines using kinetic, spectroscopic, and computational techniques. The general mechanism involves the formation of a Cr(VI)-amine complex via the donation of the nitrogen's lone pair to the Cr(VI) center and then a transfer of an α-hydrogen atom from the amine to the Cr(VI) center leading to the formation of an imine intermediate and a Cr(IV) species. The imine intermediate can then be further oxidized or hydrolyzed to the final product, and the Cr(IV) species can be further reduced or disproportionate to regenerate Cr(VI) [16].

The potent and expeditious oxidative deamination of various α-aminophosphonates which allows the synthesis of α-ketophosphonates using $ZnCr_2O_7.3H_2O$ under solvent free conditions. This method can also be applied to the rapid and highly selective oxidation of various amines to form aldehydes and ketones in very good yields.

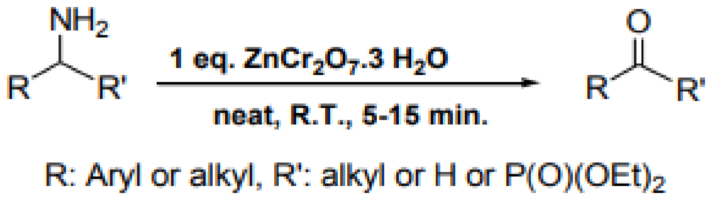

Figure 2. Synthesis of α-ketophosphonates

From the results obtained, it has been found that the reaction between the amine and Cr(VI) complex is of first order with respect to the amine and Cr(VI) complex, which is in accordance with a bimolecular rate-controlling step. The reaction is also usually zero-order with reference to the acid concentration, which indicates that the free base kind of the amine participates in the reaction. However, there are situations where the order of the reaction concerning the concentration of acid has been observed to be fractional, which suggests that the protonation of the amine or the Cr(VI) complex might be part of the reaction [17].

The presented work establishes the dependencies of the rate and selectivity of amine oxidation by Cr(VI) on the structure of the amine and the reaction conditions. For example, the oxidation of primary amines with PCC in dichloromethane gives nitriles as the major products, while that of secondary amines gives imines. The above discussion reveals that the oxidation of amines can also be improved if biphasic conditions are used in combination with phase-transfer catalysis because the formation of Cr(VI)-amine complex and its reaction can be effectively facilitated at the organic/aqueous interface [18].

7. Factors Affecting Reactivity and Selectivity

The effectiveness of Cr(VI) based oxidations is governed by the Cr(VI) complex, reaction conditions, and substrate. The awareness of these factors is crucial for the development of the proper and efficient selective oxidation processes. The reactivity of Cr(VI) complexes is related to the type of ligands that are bound to the Cr(VI) ion. Ligands can affect the Cr(VI) complex's electronic and steric environment, which in turn will affect the complex's activity and selectivity. For example, ligands that are electron-withdrawing in nature, like chloride and acetate, may increase the electrophilicity of the Cr(VI) center and its tendency to attack nucleophilic substrates. On the other hand, large ligands like pyridine and bipyridine can also have steric hindrance, which reduces the Cr(VI) complex reactivity [19].

Other factors that may also affect the reactivity and the selectivity of the Cr(VI)-mediated oxidations are the solvent used, temperature, and the pH of the reaction. DMF and DMSO are used frequently to oxidize Cr(VI) because the polar aprotic solvents are able to stabilize the charged intermediates and transition states. Higher temperatures may increase the rate of the reaction but, at the same time, may also lead to side reactions and, hence, decreased selectivity. The reaction medium's pH can influence the Cr(VI) complex speciation and the substrate's protonation, which can influence the reaction rate and selectivity [20].

The nature and structure of the substrate are other factors that affect the reactivity and selectivity of the Cr(VI) mediated oxidation reactions. Electron donating or drawing groups on the substrate may influence the reactivity of the substrate with Cr(VI) and the stability of the intermediate species formed in the reaction. For example, substrates that have electron-donating groups such as alkoxy and amino on them are likely to react more with Cr(VI) than those with electron-withdrawing groups like nitro and cyano. Steric hindrance at the active site also affects the reaction and selectivity [21]; bulky groups decrease the rate of the reaction and increase the selectivity. Crown ethers, phase transfer catalysts, and surfactants are also used to regulate the reactivity and selectivity of the Cr(VI) mediated oxidation processes. The use of phase-transfer catalysts such as quaternary ammonium salts assists in the transportation of the Cr(VI) complex from the aqueous phase to the organic phase, which in turn increases the reaction rate and selectivity. Crown ethers can chelate metal cations and act as catalysts to form reactive intermediates and hence amplify the rate and selectivity of reactions. Surfactants can make micelles, and those can provide a hydrophobic environment for the reaction; this is helpful in increasing the rate and selectivity of some reactions [22].

8. Experimental Techniques for Mechanistic Studies

Some of the experimental techniques that can be used to study the Cr(VI) catalyzed oxidation reactions include information about the rates of the reaction, intermediates, and transition states. These methods include spectroscopy, KIE, LFER, and product analysis. Some of the most frequently used spectroscopic methods for the study of Cr(VI) oxidations intermediates and transition states are UV-visible, NMR, and EPR spectroscopy [23]. UV-visible spectroscopy will be useful in determining the electronic transitions and the absorption bands of the Cr(VI) complex and the intermediate species formed in the reaction and thus aid in identifying the species involved in the reaction as well as the stage of the reaction. The rate constants of the formation and the decomposition of the intermediates and products of the reaction can be obtained in NMR spectroscopy. Spectroscopy, such as EPR, is useful in the identification of paramagnetic species such as Cr(V) and Cr(III), which are often involved in the oxidation of Cr(VI) [24].

Another technique employed further to elucidate the mechanism of Cr(VI) oxidation is the kinetic isotope effect (KIE). KIEs rely on the differentiation between the rates of the reaction involving labeled and unlabeled substrates and can provide information on bond-breaking and bond-making processes in the reaction. For instance, identifying a primary KIE for C-H bond cleavage in the rate-determining step of alcohol oxidation suggests that the hydride transfers from the substrate to the Cr(VI) center in the mechanism. The Secondary KIEs that arise from the change in vibrational frequencies of bonds in the vicinity of the reaction center are very relevant in the determination of the transition state and the degree of bond cleavage in the rate-limiting step [25].

Also, linear free energy relationships (LFERs) can be used to predict the mechanisms of Cr(VI) oxidations. These are linear free energy relationships that show how the rates or equilibrium constants of a number of reactions are related to the substituent constants or other parameters characterizing the electron and steric effects of the substrates. Based on the Cr(VI) oxidations research LFERs most often used are the Hammett equation, which shows the correlation between rates or equilibration constants of reactions of substituted aromatic compounds and the substituent constants of the substituents and the Taft equation, which shows correlation between the rates or equilibration constants of reactions In this way, one can determine the influence of the substituents on the rates and selectivity's of Cr(VI) oxidations and, thus, gain insight into the nature of the transition state and the factors that govern the substrate reactivity [26].

Besides the above-mentioned methods, product analysis has also been applied to investigate the mechanisms of Cr(VI) oxidation. Therefore, analysis of products and their yields allows one to determine the nature of the reactive intermediates and the methods for their generation. For instance, if the products of the reaction are observed to have different oxidation states or regiochemistry, it may suggest that more than one intermediate species is involved in the reaction or that the reaction occurs through different pathways. One can also obtain some information on the intermediates of the reaction and the processes that lead to their formation using isotopically labeled substrates or trapping experiments [27].

9. Applications in Organic Synthesis and Catalysis

Cr(VI) complexes have been found to be very effective in the selective oxidation of organic substrates in organic synthesis and catalysis to give valuable products with high efficiency and minimal pollution to the environment. Further, the application of the Cr(VI) oxidations in the synthesis of complex natural products and bioactive compounds is one of the most important fields of application, mainly when the selective oxidation of some functional groups is required for the proper structure and stereochemistry of the compound. For example, Cr(VI) reagents such as pyridinium chlorochromate (PCC) and pyridinium dichromate (PDC) have been used in the synthesis of terpenes, steroids and alkaloids in which the reagents oxidize alcohols and alkenes into aldehydes, ketones, and enones respectively [28].

Another major field that applies Cr(VI) oxidations is the development of sustainable and green chemistry. Using stoichiometric Cr(VI) reagents such as PCC and PDC generates a large amount of chromium waste, which are toxic to the environment and human beings. Thus, to solve this issue, Cr(VI) complexes have been employed for the catalytic oxidation of organic substrates in the presence of environmentally friendly oxidizing agents such as hydrogen peroxide or oxygen. For instance, chiral Cr(VI) complexes have been used in the asymmetric epoxidation of alkenes with hydrogen peroxide as the oxidants, affording optically active epoxides in a green and efficient way [27, 28].

Cr(VI) complexes have also been applied in sequential and domino reactions where several oxidative transformations are conducted in a single pot to build up complex structures. For example, the oxidation of alcohols using Cr(VI) has been combined with other transformations like aldol condensation, Knoevenagel condensation, and Michael addition to get α,β-unsaturated carbonyl compounds and other valuable synthetic intermediates. Also, the oxidation of amines to imines by Cr(VI) has been achieved, followed by the reduction of the formed imines with hydrogen transfer catalysts for the selective synthesis of secondary amines [29].

Cr(VI) complexes have also served as catalysts in water oxidation to produce hydrogen fuel, which is one of the best strategies for the production of hydro energy for a sustainable energy supply. The use of Cr(VI) complexes in these systems makes it possible to carry out efficient and selective water decomposition into hydrogen and oxygen, which can be viewed as a possible alternative to the energy produced from fossil fuels. There is also ongoing work on the design of better and more stable Cr(VI) based catalysts for water oxidation with the purpose of raising the turnover numbers and the lifetimes of the catalysts.

10. Conclusions and Future Directions

The area of selective oxidation of organic substrates using Cr(VI) complexes has been one of the most popular and actively investigated areas of organic chemistry for several years now and has resulted in the development of many efficient and selective oxidants and catalysts. Starting from the first publications on chromate and dichromate reagents to the present chiral and non-toxic chromium-based reagents, Cr(VI) oxidations are a key player in the establishment of the field of selective oxidation reactions and its application in synthesis and catalytic processes.

This review, therefore, shows that Cr(VI) induced oxidations are not a simple and direct process but a complex one that involves intermediates, transition states, and pathways. Thus, the use of contemporary experimental approaches like spectroscopy, kinetic isotope effects, and LFERs and computational methods, including density functional theory and other theoretical model systems, has advanced the understanding of the electronic structure and energy of the species participating in these reactions. These studies have identified the major factors that control the selectivity and the rate of Cr(VI) oxidations: The structure of the substrate, the arrangement of the ligand around the chromium (VI) center, solvent, temperature, and pH of the reaction also affects the reduction process.

The selective oxidation reactions that are mediated by Cr(VI) complexes have become a vivid and productive area of organic chemistry, which offers numerous effective and universal oxidants and catalysts. Despite the current research gaps and possibilities for further development in this area, the necessity of green chemistry and environmentally friendly research has been conducted in the past decade to determine the mechanisms of chemical reactions, leading to the development of new selective oxidation methods that will set the trends for future organic synthesis and catalysis. Based on the information collected on experimental and computational research, employing the principles of green chemistry and sustainable design, it is possible to define new and improved methods to accomplish the selective oxidation of organic substrates without involving hazardous Cr(VI) reagents. This will require an interdisciplinary approach to the problem whereby chemists, biologists, engineers, and other experts will be required to address various issues that are associated with selective oxidation and to develop new and sustainable processes in the future.

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