

Kinetic and mechanistic aspects of cerium(IV) as an oxidant in ruthenium catalysed oxidation reaction: A mini review

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Abstract

Purpose of Study: The review article explores the kinetic and mechanistic aspects of ruthenium(III) chloride catalysis in the oxidation of organic and inorganic substrates using cerium(IV) as the oxidizing agent in an aqueous acid medium. The reactions have been classified based on the type of metal ion catalyst used. The article highlights that the nature of the mechanism of catalytic oxidation of alcohols by Ce(IV) depends on the specific metal ion catalyst used and the nature of the acidic medium employed.

Methodology: The article sheds light on the purpose and scope of catalytic oxidation of alcohols by Ce(IV). The oxidation of alcohols is an essential reaction in organic chemistry and has numerous applications in the synthesis of various organic compounds. The use of Ce(IV) as an oxidizing agent in the presence of a suitable catalyst can provide an efficient and selective method for the oxidation of alcohols.

Principal Findings: The article discusses the role of ruthenium(III) chloride as a catalyst in the oxidation of alcohols in the presence of Ce(IV) and provides insights into the reaction mechanism. The review also highlights the importance of the nature of the acidic medium used in the reaction and its effect on the reaction kinetics and mechanism.

Implications: Overall, the review article provides a comprehensive overview of the use of ruthenium(III) chloride as a catalyst in the oxidation of alcohols using Ce(IV) as an oxidizing agent in an aqueous acid medium. The insights provided in this article can be valuable for researchers working in the area of organic and inorganic chemistry.

INTRODUCTION

The kinetics of the oxidation of organic compounds is highly useful in determining the different factors that influence the rate of reaction and in understanding the mechanisms of chemical reactions. Various oxidations that do not proceed to completion even when carried out for an infinite period of time get completed in a matter of a few minutes when a specific catalyst is added. Oxidation of organic and inorganic compounds to give specific products by certain selective catalysts under certain conditions has received much attention from an academic as well as an industrial point of view.

A large number of oxidants till now have been used in the oxidation of organic and inorganic compounds like perchloric acid ([Wordsworth et.al. 1957](#)), trivalent manganese ([Drummond et.al. 1955](#)), hexavalent chromium ([Vishavandham., 1943](#)), lead tetraacetate, sodium bismuthate, heptavalent manganese ([Wiberg et.al. 1965](#)), osmium tetroxide ([Singh, H.S. 1987](#)), potassium hexacyanoferrate(III) ([Singh, M.P. 1957](#)), hydrogen peroxide ([Tandon, et.al. 2007](#)), cerium(IV) ([Tandon, et.al. 1984](#)), selenium dioxide, ceric ammonium nitrate, peroxymonosulphate, peroxydisulphate, N – bromothalamide etc [Verma et.al. 1985]. Out of these cerium(IV) and peroxymonosulphate oxidations are of particular importance due to their eco-friendly nature and their capability to oxidize a variety of organic and inorganic compounds catalyzed by different transition metal catalysts.

Ce(IV) as an oxidant

Cerium is a member of the lanthanide series, whose $[Xe]5d^1 5d^1 6s^2$ electronic configuration permits its existence in tri $[Xe]5d^1 5d^1 6s^0$ and tetra $[Xe]5d^0 5d^0 6s^0$ positive states. Cerium(IV) is a good oxidizing agent since the ion converts into Ce^{3+} which is a quite stable ion. Cerium(IV) is obtained by the oxidation of cerium(II) species with peroxydisulphate, or bismuthate in nitric acid. As a result of their high charge, cerium(IV) ions tend to hydrate and very frequently co-ordinate with counter ions. This last aspect provides a rationale for Ce(IV)/Ce(III) potential dependence on the nature of the acid used as a medium. An increase in sulphuric acid or nitric acid concentration decreases the oxidation potential due to the complexing of cerium(IV) ions. On the other hand, potential increases with increasing concentrations of perchloric acid, partly due to the hydrolysis of cerium(IV) ions. A confirmation of the complexation of cerium(IV) with sulphate ([Marroco, 1983](#); [Moore, 1945](#), [McCurdy, 1960](#)) and nitrate [[Jones, 1935](#); [Blaustein, 1957](#)] ions has been reported. Cerium(IV) nitrate equilibria are complicated by dimerization, and hydrolysis by association with cerium(III), as shown in Table 1. Cerium (IV) is a very powerful equivalent oxidant.

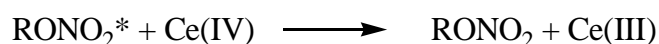
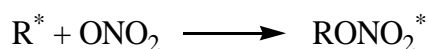
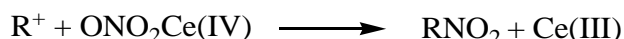
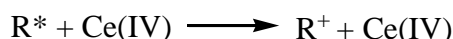
Table 1: The oxidation potential of the Ce(IV) ion in various acids

$\text{Ce}^{4+} + \text{e}^- \rightarrow \text{Ce}^{3+}$		
Electrolyte	Concentration (M)	$E^0(\text{V})$ Vs (SCE)
H_2SO_4	1	1.20
	4	1.19
	8	1.18
CF_3COOH	1	1.36
	3	1.33
	6	1.31
HNO_3	1	1.37
	4	1.37
	8	1.32
CH_3SO_3	1	1.40
	3	1.41
	6	1.39
	1	1.46
	4	1.51
	6	1.57
	8	1.63

Oxidation of organic compounds with cerium(IV) sulphate was initially studied in relation to analysis (cerimetry) (Dorfman, 1962). Later on, other aspects of cerium(IV) oxidations were developed (Smith, 1942). More results have been incorporated into two articles. One (Ogata, 1958) of these emphasizes the reaction mechanism, while the other (Richardson, 1965) focuses on the synthetic applications of cerium(IV) ion to functional group oxidation. The first review of the systematic study of cerium(IV) as an oxidant was published by G.F Smith in 1942 followed by two more reviews in 1958 (Ogata, 1958) and then by W. H Richardson in 1965. On the basis of these excellent reviews, a selective review was published by Bernson, in 1976). A detailed study of cerium(IV) oxidations of organic compounds has been published by TSE-LOK HO [Ho, 1973].

Ce(IV) is a typical one equivalent oxidant that removes one electron at a time from the substrate. In this regard, Ce(IV) shares certain similarities in the reaction pattern with manganese(III), cobalt(III), and vanadium(V), although the three oxidants are ions of transition metals. On the other hand, different behavior is expected from that of chromate and permanganate, which have reactive oxy-anions.

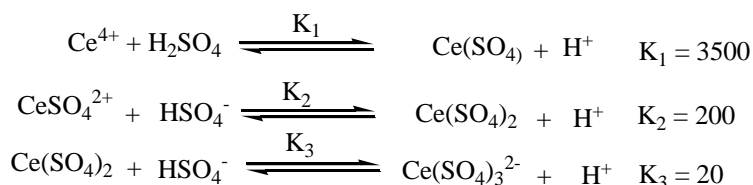
Cations, or free radicals, are generated in one equivalent oxidation of neutral or anionic organic species. Through outer sphere mechanisms, these intermediates generally undergo rapid oxidation to give neutral products by electron transfer or by ligand transfer via inner sphere mechanisms. Alternatively, oxidation occurs after its combinations with counter ions such as nitrate in CAN oxidations.



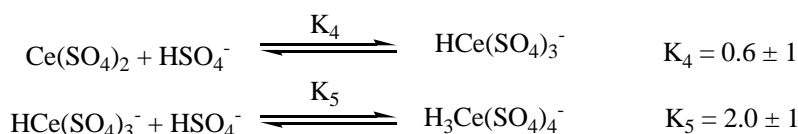
Cerium(IV) oxidations deal most frequently with neutral organic compounds; therefore, radical cation intermediates are encountered most of the time. In general, cerium(IV) oxidations are conducted in acetic acid, acetonitrile, and their aqueous mixtures because these solvents are quite resistant to destruction by strong oxidants while compromising solvent power for both the oxidant and most of the organic compounds. The most common cerium(IV) reagents are cerium(IV) sulphate, cerium(IV) perchlorate, and especially the double salt ceric ammonium nitrate.

Reactive species of cerium(IV) in aqueous sulphuric acid

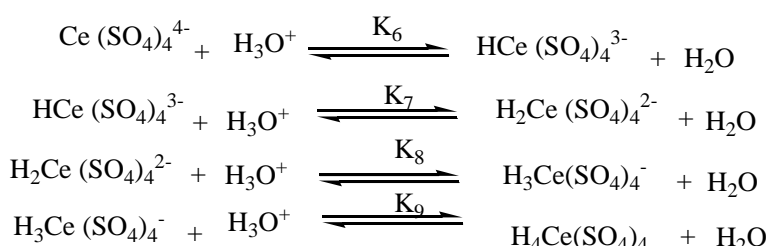
According to Hardwick and Robertson, (Harwick, 1951) cerium(IV) exists in various forms in sulphuric acid solution (2 molar) and at constant ionic strength, the following equilibrium exists.



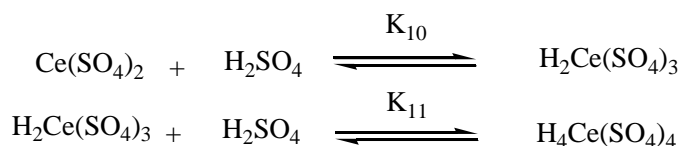
Without keeping the ionic strength constant, Buganko and Kaun Lin investigated the nature of cerium(IV) spectrophotometrically in 0.1 to 17.6 M sulphuric acid. The equilibrium at $20 \pm 2^\circ\text{C}$ is shown below. ([Buganko et.al. 1963](#))



They have suggested that $\text{Ce}(\text{SO}_4)_2$ and $\text{HCe}(\text{SO}_4)_3^-$ are the prominent species up to 2M and $\text{H}_3\text{Ce}(\text{SO}_4)_4^-$ exists above 2M H_2SO_4 . Hargreaves and Sutcliffe have suggested in addition the following equilibrium to explain mechanisms of certain reactions. ([Hargreaves et.al. 1955](#))



But the equilibrium constants were not calculated, though they may be useful in some mechanisms. In addition to the above equilibrium, the following were also proposed.



[Sant et.al. 1972](#); and Mehrotra and Sankhla, 1972 have suggested reactive species of cerium as cerium(IV) sulphate in the oxidation of ethylene glycol with cerium(IV) sulphate. On the other hand Mac Auley and Brubaker, 1996; Mehrotra and Sakhala, 1973; and Guilbail and Mc Curdy, 1963, suggested reactive species as $\text{Ce}(\text{SO}_4)_4^{2+}$. While in the oxidation of pinacol by cerium(IV), Mino has suggested $\text{Ce}(\text{SO}_4)_3^{2-}$ as the existing species. ([Mino et.al. 1959](#))

Ruthenium(III) as catalyst

It has been reported that in hydrochloric acid medium ruthenium(III) chloride forms RuCl_6^{3-} species. Using the ion-exchange technique, it has been confirmed that the half-life of the loss of chloride from $[\text{RuCl}_5(\text{H}_2\text{O})]^{2-}$ is of the order of a year, while the aquation of RuCl_6^{3-} to $[\text{RuCl}_5(\text{H}_2\text{O})]^{2-}$ takes only a few seconds ([Cady, 1958](#); [Connik, 1960](#); Griffith, 1968). This equilibrium, which is also the case in the present study, may be written as



During the heterolytic splitting of hydrogen and the hydration of acetylene, it has been confirmed that $[\text{RuCl}_5(\text{H}_2\text{O})]^{2-}$ is the actual reacting species ([Halperon, 1961](#); [Harrod, 1961](#)) of ruthenium(III) chloride. Depending on the effect of chloride ions on the reaction velocity, many other workers [[Singh, 1980](#); [Tandon, 1985](#)] also considered $[\text{RuCl}_5(\text{H}_2\text{O})]^{2-}$ as the only reactive species of ruthenium(III) chloride in an aqueous acidic medium. Thus, one might safely assume that $[\text{RuCl}_5(\text{H}_2\text{O})]^{2-}$ is the actual reacting species of ruthenium(III) chloride in acidic medium for the oxidation of benzyl alcohol and 2-phenyl ethanol in our study.

Oxidation of alcohols by cerium(IV) catalyzed by ruthenium(III)

Several researchers ([Meyers, 1901](#); [Duke, 1940](#); [Ardon, 1957](#)) have studied the mechanism of oxidation of aliphatic alcohols by cerium(IV) in an acidic medium, proposing a 1:1 intermediate complex formation between cerium(IV) and organic substrate, which was confirmed by the appearance of a red shift in the visible spectrum on the addition of ethanol to cerium(IV). Littler, and Waters, also proposed the mechanism of ceric sulphate oxidation of cyclohexanol into acetaldehyde, involving the formation of a Ru(III)-substrate complex. ([Littler, 1959](#); Waters, 1960). Young & Trahanovsky in the oxidation of primary, secondary, and tertiary alcohols, cyclic alcohols, glycols, and beta substituted ethanols by ceric ammonium nitrate have reported the complex formation between cerium(IV) and alcohol ([Young](#)

&Trahanovsky 1969). In solvents such as 70% aqueous acetonitrile, the steric requirements of salvation seem to be greater than those of complexation. Since the formation constants of cerium(IV)-alcohol complexes increase as the hydroxyl group of a series of alcohols becomes more sterically crowded, other factors remain more or less constant.

The empirical formulae of cerium(IV) complexes with n-secondary and tertiary butanols were determined from spectrophotometric measurements (Offner, 1965) and in all cases, a 1:1 ratio of cerium and alcohol was observed in the complexes. Oxidation of 1, 2-glycol and related compounds and their mechanisms were reported by (Conant and Aston, 1928). Oxidation of -o-, -p-, and -m cresols by cerium(IV) in sulphuric acid medium was studied by J. P. Singh et.al. 1990; and Rao and Mohammad, 1963. They have also proposed a 1:1 intermediate complex formation. Substituent effect (Subramanian, et.al. 1970) in the oxidation of benzyl alcohol by cerium(IV) perchlorate and complexation between benzyl alcohol and cerium(IV) in perchloric acid medium (Paquette, 1968) have also been reported. The formation of complexes between cerium(IV) and diols (Sankhala, et.al. 1973; Prakash, et.al. 1972) and the presence of dimeric cerium(IV) and its precipitations in the reaction mechanism have been reported with the proposed rate law as-

$$\frac{-d\text{Ce(IV)}}{dt} = \frac{2(k+k_1k_2[\text{H}_2\text{SO}_4][\text{H}^+]^{-1}[\text{Ce(IV)}][\text{diol}])}{1+k_1[\text{HSO}_4^-][\text{H}^+]^{-1} + k_1k_2[\text{HSO}_4^-][\text{H}^+]^{-1}[\text{diol}]}$$

Ruthenium(III) and ruthenium(VIII) catalyzed oxidation of cycloalcohol (Tandon, et.al. 1985) by cerium(IV) sulphate in sulphuric acid medium, and the oxidation of acetyl acetone by cerium(IV) perchlorate using stopped flow spectrophotometric technique has also been reported [Singh, H.S. 1998]. Oxidation of benzaldehyde to benzoic acid by cerium(IV) perchlorate has been reported by Wiberg and Ford and 1:1 intermediate complex formation between Ce (IV) and benzaldehyde was proposed. (Wiberg and Ford 1969)

Oxidation of aliphatic ketones, aliphatic alcohols, cyclic ketones, and aromatic aldehydes, has been reported from our laboratory (Tandon, et.al. 2005; 2006; 2008; 2004; 2006; 2008). Isobutyl alcohol, methanol, and tetrahydrofurfuryl alcohol oxidation has recently been reported, with complex formation between cerium (IV), organic substrate, and iridium (III), which decomposes in the slow step (Song, et.al. 2004; 2007; 2005). Un-catalyzed conversion of the side chains in aromatic hydrocarbons into aldehydes (Syper, 1966) and conversion of aromatic alcohols into aromatic aldehydes (Trahanovsky, 1965) have been reported from the synthetic point of view.

CONCLUSIONS

Cerium(IV) is a typical one equivalent oxidant that removes one electron at a time from the substrate. In this regard, cerium(IV) shares certain similarities in the reaction pattern with manganese(III), cobalt(III), and vanadium(V), although the three oxidants are ions of transition metals. On the other hand, different behavior is expected from that of chromate and permanganate, which have reactive oxy-anions. Several kinetic studies have been carried out to explore the mechanistic aspects of different metal ion catalysis in the Ce(IV) oxidation of different types of substrates. This review has correlated the different factors that control the mechanistic paths of catalysis in the oxidation reactions of organic substrates.

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