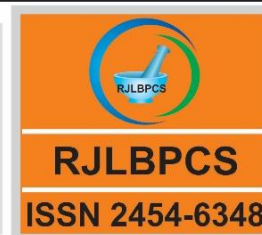




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A REVIEW OF KINETICS OF OXIDATION OF ORGANIC COMPOUNDS BY HEXACYANOFERRATE (III)

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ABSTRACT: The review surveys the development of kinetics and various oxidising agents which can be used during the study of chemical kinetics along with oxidation of some organic substrates by hexacyanoferrate (III) /HCF (III). Since HCF (III) is acting as relatively poor oxidant, it acts as a selective outer-sphere reagent applicable for most of the easily oxidisable substrates and normally it is used as free radical interceptor; because of which it converts into a species acting as an efficient one electron oxidant mainly for the octahedral complexes in comparative study.

KEYWORDS: Kinetics, Oxidation, Oxidant, Mechanism, Hexacyanoferrate (III) (HCF), Complex, Catalysis, Redox.

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1. INTRODUCTION

Chemical kinetics is a branch of chemistry which studies the rate of reaction whereby various chemical reactions take place in different conditions. Kinetics takes into consideration the time required for transformation of reactants from one state to another. Some reactions take place rapidly within fraction of seconds while some are extremely slow like rusting of iron. Reactions which take reasonable time for completion can be studied conveniently by suitable methods. Kinetics is concerned with series of all physical and chemical processes which comes during the course of chemical reactions [1-3]. Several researchers [4-6] had made pioneering work in chemical kinetics like Ludwig Ferdinand Wilhelmy, W. Ostwald, Wenzel C F, L. J. Thenard, Berthelot, Peter Waage and Harcourt, etc. Kinetic study covers the effect of concentration, temperature and pressure on different types of reactions. Based on the type of reaction wide

variety of experimental techniques had been used to investigate. Kinetics is useful in study of gas, liquid and solid phase reactions. Especially liquid phase reactions are studied the most due to higher interest of organic and inorganic chemists particularly engineers from industries [7-9]. Various factors such as concentration, catalyst, dielectric constant [10-15], salt [16-19], ionic strength, temperature [20] and free radicals [21, 22] were accounted by different researchers. Potassium hexacyanoferrate (III) was used for reading palimpsests and old manuscripts in nineteenth century. The compound has widespread use in blueprint drawing and in photography (Cyanotype process). Iron and copper toning involve the use of potassium ferricyanide. Potassium ferricyanide is used as an oxidizing agent to remove silver from negatives and positives, a process called dot etching. In color photography, potassium ferricyanide is used to reduce the size of color dots without reducing their number, as a kind of manual color correction. The compound is also used to harden iron and steel, in electroplating, dyeing wool, as a laboratory reagent and as a mild oxidizing agent in organic chemistry. It is also used in black and white photography with sodium thiosulfate (hypo) to reduce the density of a negative or gelatin silver print where the mixture is known as Farmer's reducer; this can help offset problems from over exposure of the negative or brighten the highlights in the print. Potassium ferricyanide is also one of two compounds present in ferroxyl indicator solution (along with phenolphthalein) which turns blue (Prussian blue) in the presence of Fe^{2+} ions and which can therefore be used to detect metal oxidation that will lead to rust. It is possible to calculate the number of moles of Fe^{2+} ions by using a colorimeter because of the very intense Prussian blue color of $\text{Fe}_4[\text{Fe}(\text{CN})_6]_3$. Potassium ferricyanide is often used in physiology experiments as a means of increasing a solution's redox potential ($E^\circ \sim 436 \text{ mV}$ at pH 7). As such it can oxidize reduced cytochrome C ($E^\circ \sim 247 \text{ mV}$ at pH 7) in intact isolated mitochondria. Sodium dithionite is usually used as a reducing chemical in such experiments ($E^\circ \sim -420 \text{ mV}$ at pH 7). Potassium ferricyanide is used in many amperometric biosensors as an electron transfer agent replacing an enzyme's natural electron transfer agent such as oxygen with the enzyme glucose oxidase. It is used as ingredient in many commercially available blood glucose meters for diabetics use. Potassium ferricyanide is combined with potassium hydroxide (or sodium hydroxide as a substitute) and water to formulate Murakami's etchant. This etchant is used by metallographers to provide contrast between binder and carbide phases in cemented carbides. The aim of present review article is to understand the kinetics of oxidation of various organic compounds by hexacyanoferrate (III).

Methods of kinetic study

Various methods are used to study chemical kinetics based on half life period or time period of completion of reactions. Extremely fast reactions can be studied by using stopped flow, molecular beam flow, etc techniques whereas other can be examined by conductometry, pH-metry and colorimetric titrations, etc. Different kinetic methods are as tabulated below.

Method	Time scale	Illustration
Conventional	≥ 10 s	The reactants are mixed together in a batch reactor and Conc. Vs time is measured
Stopped flow	$\geq 10^{-1}$ s	Set of continuously flow system in which reactants are fed into the reactor and flow out again so quickly that there is negligible reaction followed by stopping the flow for reactant to react. Finally, conversion Vs time calculated
Conventional flow system	$\geq 10^{-3}$ s	Continuously reactants are feed into a reactor and the steady state reaction rate was measured
Pressure Jump and Temperature Jump	$\geq 10^{-6}$ s	The reactants are mixed at such a low temperature that the reaction rate is negligible. CO ₂ lasers used suddenly to heat the reaction, conc. Vs time measured then the reactant conc. Vs time measured. Eigen got Nobel prize for this technique.
NMR	10^{-2} - 10^{-9} s	Initiate a change with magnetic pulse and measure the decay of spins by NMR.
Flash photolysis	10^{-9} - 10^{-10} s	Reactants are placed into a vessel under conditions where reaction is negligible. Pulse a laser flash lamp to start reaction. The reactant conc. Vs time was measured. Porter got Nobel prize for this technique.
Molecular Beam	10^{-9} - 10^{-13} s	Direct Beams of reactants towards each other in a vacuum system and the steady state reaction rate was measured. Hersch Feld got Nobel prize for this technique to.
Femto spectroscopy	10^{-15} s	Life time reaction can be studied. Ahmed Zewel got Nobel prize for this technique.

Various Oxidizing Agents

The species which oxidizes other species, give up oxygen or electronegative atom, which accepts hydrogen or any other electropositive element, which gain electron named as oxidizing agent. Numerous compounds functions as oxidizing agents in chemistry. Oxidations of various organic compounds by hexacyanoferrate (III) were considered in our study but it is essential to have a look into the other oxidizing agents also. The N-halo compounds are widely used as oxidizing agents

for oxidations of various organic compounds like N-chloronicotinamide [23] where molecular chlorine is generated within the reaction acts as oxidizing agent. Other N-halo compounds are N-Bromophthalimide, N-Bromoacetamide and N-Chlorobenzamide. Ethyl N-Chlorocarbamate (ECC) [24] works as efficient oxidant for α -amino acid. N-haloamine furnishes halonium ion and a hypo species which works as nucleophile as well as base. Positive halogen containing compounds works as mild oxidants. N-Bromosuccinamide oxidizes L-arginine in acidic medium to corresponding aldehyde. Survey of literature reveals that N-Bromosuccinamide oxidizes various organic compounds [25-31]. Diperoxidatonickelate (IV) in basic medium oxidizes L-arginine to aldehyde [32]. Quinolinium dichromate (QDC) acts as selective oxidant for oxidising primary alcohol in presence of secondary alcohol. Quinolinium dichromate works as an efficient oxidant for both organic and inorganic compounds [33]. Other similar oxidants are pyridinium chlorochromate, zinc chlorochromate, magnesium chlorochromate and potassium dichromate [34]. A mild and selective oxidant tetraethyl ammonium chlorochromate oxidizes primary aliphatic alcohol [35]. Fremy's radical i.e. potassium nitrosodisulphonate oxidizes α -amino acid to corresponding α -keto acid. Pyridinium hydrobromide perbromide oxidizes α -amino acid to corresponding aldehyde in aqueous acetic acid. Chloramine-T i.e. 4-chloro-4-methyl benzenesulfonamide oxidizes various organic compounds [36-41]. It yields a hypochlorite and act as a source of electrophilic chlorine in organic synthesis. Various other important oxidising agents are octacyanomolybdate, periodate ion [42], hexamethylene diammine tetraacetatocobaltate (II) complex, t-butyl hydroperoxide, potassium dichromate [43], persulphate, hydrogen peroxide, N-chlorosaccharin, pyridinium chlorochromate, tris(benzhydro oxalate) iron (III), benzimidazoliumfluorochromate [44], diperoxidatoargentate (III) [45], Imidazolium dichromate [46], methylene blue [47], diperoxidatocuprate (II), tetrakis (pyridine) silver dichromate, benzyl dimethyl ammonium chlorobromate, morpholinium chlorochromate, trichloroisocyanuric acid [48], cetyl trimethyl ammonium dichromate [49]. A well known strong oxidant potassium permanganate acts as an oxidant in acidic, basic and in neutral medium. Literature reveals oxidation of various compounds by potassium permanganate [50-62]. The most significant characteristic of an oxidation-reduction process is the charge transfer between the reactants. The electron transfer means that changes must take place at the atomic level in the orbital populations of the two species, thus influencing the other bonds in which the individual atoms are involved [63]. This work move toward mixed redox reactions, taking as reducing agents several organic substrates and as oxidizing agent as complexes of different transitional metals ion in their highest oxidation state.

The electron transfer mechanism: Outer-sphere and inner-sphere mechanism

Henry Taube [64] (Nobel Prize 1983) on the basis of electron transfer reaction and structural changes involving in it classified redox processes into two types as outer-sphere and inner-sphere. Outer-sphere mechanisms have a direct transfer of electron between reducing and oxidizing agent

with minimum of interaction. The coordination spheres do not undergo any modifications to their compositions, only a little change of ligand-metal ion distances and solvation interaction occurs. The electron moves by tunneling and follows the Franck-Condon restriction. The electron moves faster (10^{-15} s), while nuclei remains stationary in their definite positions, the time of their movement being significantly longer (10^{-13} s). Taking into consideration the outer-sphere mechanism and self-exchange reactions (without chemical transformation) R.A. Marcus (Nobel Prize 1992) set up and developed a theoretical model to determine rate constants for the electron-transfer [65], [66]. Many inorganic reactions obeyed this model [67-69]. The extension of this to organic reactions led to additional explanation of the theory by several other authors [70-73]. Inner-sphere mechanisms, on the converse require a close interaction between the coordination spheres of the reactants by joining them together in a complex having at least one ligand in common. This would lead to the electron-transfer in a substitution process. The electron is shifted through this ligand functioning as a bridge. These mechanisms include three steps: a) formation of the complex with a common ligand as a bridge, b) transfer of electron c) breaking up of the complex into the reaction products [74]. While, in principle, any of these steps might manage the rate, in majority of conditions the electron transfer takes place in the rate-determining step [63], [75], [76]. Movement of the common ligand from the oxidizing species to the oxidized form of the reducing agent is a concrete argument for the inner-sphere mechanism [77]. An alteration of the rate by changing the ligand having the role of bridge is an additional support of such a mechanism. On the converse, the lack of such influence argues for an outer-sphere mechanism. Usually, systems which are inert to substitution will react by an outer-sphere mechanism. For systems labile to substitution and with a redox rate equivalent to the substitution rate indicates an inner-sphere interaction. The activation parameters give additional information of the way by which the electron transfer takes place. A lower value of activation enthalpy suggests an outer-sphere mechanism.

Proof of the involvement of an intermediate

When the rate law suggests the participation of an intermediate, the main direct support is established by measurement of its physico-chemical properties. This approach needs the existence of an experimental technique for its recognition. Frequently, it is essential that the intermediate is formed in fairly high concentration (equivalent to the reactants in the case of a rapid pre-equilibrium). Based on the life-time of the intermediate, the detection can use classical techniques or some arrangements intended for rapid reactions.

Proof for rejection of intermediate existence

When there is large difference between electronic spectra (UV-VIS) of reactants and products, the consecutive recordings of spectra throughout the reaction can convey information on the involvement of intermediate. The existence of isobestic points is a proof of a linear correlation

[78] of the concentrations of the species, suggesting that the reactant is converted directly into the product without any intermediate. If this is not observed or appears only within a short period at the start of the reaction and the intersection position is changed, it suggests some intermediate of significant concentration is formed.

Catalysis in redox reactions

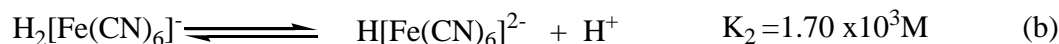
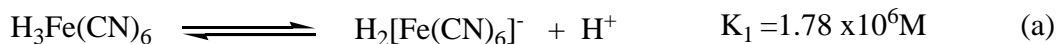
Catalysis using another redox couple phenomenon occurs when a redox couple mediates uni-equivalent or bi-equivalent electron transfer. An example is the oxidation of Cr (III) to Cr (VI) by $S_2O_8^{2-}$, which is catalyzed by Ag (I). The latter is able to form Ag (III) with the bi-equivalent peroxydisulfate. This then forms Ag (II) in a reaction with Ag (I). Ag (II) will react uni-equivalently with Cr (III), or another intermediate species of Cr to finally form Cr (VI). Many oxidations of organic compounds are catalyzed by redox couples Such as OsO_4 catalysis via the formation of double bridged intermediates [79-81] or Ce (IV) catalysis via Ce (III) intermediates. A review of the literature illustrates oxidation of many organic and inorganic compounds by potassium hexacyanoferrate (III). Oxidation of methyl cellulose polysaccharide a natural polymer by alkaline potassium ferricyanide was carried by R.M. Hassan et al [82]. Interaction of L-phenyl alanine with potassium hexacyanoferrate (III) catalyzed by Iridium (III) in aqueous alkaline medium have been studied by Goel and Sharma [83].

Characteristics of $K_3[Fe(CN)_6]$ and oxidation states of Iron

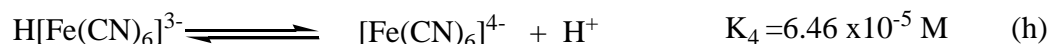
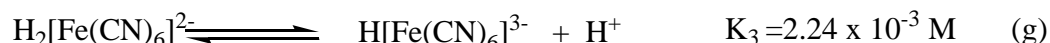
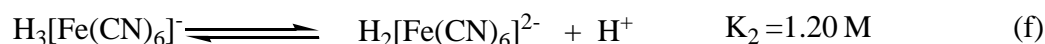
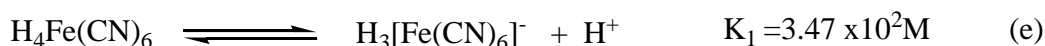
Molecular formula- $C_6N_6FeK_3$, Molar mass- 329.24 g/mol, Appearance-deep red crystals, Density 1.89 g/cm³, Melting point 300°C, Solubility- slightly soluble in alcohol, soluble in acid, soluble in water 464 g/L (20°C), Crystal structure- monoclinic, Coordination geometry- octahedral at Fe, Flash point-Non-flammable. Iron shows various oxidation states +6, +5, +4, +3, +2, +1, 0, -1, and -2, but out of these oxidation states +2 and +3 states are stable states. Potassium hexacyanoferrate (III), $K_3[Fe(CN)_6]$ is basically a substitution-inert transition metal complex [84]. It does not substitute its ligands at a rate fast enough to compete with rapid electron transfer. As a result, oxidations by hexacyanoferrate (III) ion takes place by way of a non-bonded electron transfer or outer sphere process, in which an electron is transferred from substrate to the metal ion through the cyano ligand. In acidic medium, potassium hexacyanoferrate (III) has been employed for the oxidation of sulphur containing compounds [85-89] and also for oxidation of toluene and substituted toluenes [90-92], diphenylmethane and triphenyl methane [93], unsaturated systems [94] and polynuclear systems [95]. In neutral medium, potassium hexacyanoferrate (III) has been utilized for the oxidation of aliphatic amines [96]. In basic medium, potassium hexacyanoferrate (III) has been widely used for the oxidation of various types of organic compounds like aldehydes [97-101], amines [102-108], alcohols and diols [109-115], sulphur compounds [116-122], acids [123-127], sugars [128,129], hydrazines, amino acids [130] [131], acyloins, As(III) [132,133], hypophosphite [134], hydrocarbons [135] and phenols [136,137].

Ferri- and ferro-cyanic complexes

From many oxidation states possible for iron, only the very usual +2 and +3 states form hexacyanoferric complexes, extremely stable species, inert to ligand substitution. The hexacyanoferrate (III) ion $[\text{Fe}^{\text{III}}(\text{CN})_6]^{3-}$ also known as ferricyanide is the completely deprotonated anion of ferricyanic acid $\text{H}_3[\text{Fe}(\text{CN})_6]$, a strong acid in all its dissociation steps [138].



Thus, on a large pH range, completely dissociated form is the only one present in solution. Similarly, hexacyanoferrate (II) ion $[\text{Fe}^{\text{II}}(\text{CN})_6]^{4-}$ also known as ferrocyanide, is the completely deprotonated form of the ferrocyanic acid ($\text{H}_4\text{Fe}(\text{CN})_6$), which is also strong in its first and second dissociation steps, but weaker in its last two [139].

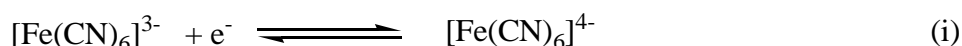


Therefore, $[\text{Fe}(\text{CN})_6]^{4-}$ is protonated easier than $[\text{Fe}(\text{CN})_6]^{3-}$ in the media of the same acidity.

2. CONCLUSION

Oxidations with hexacyanoferrate (III)

Hexacyanoferrate (III) i.e. $[\text{Fe}(\text{CN})_6]^{3-}$ acts as a competent oxidant [140] for diverse organic substrates due to the fact that the electron transfer in the hexacyanoferrate complexes takes place between the metallic centers, the redox couple $[\text{Fe}(\text{CN})_6]^{3-}/[\text{Fe}(\text{CN})_6]^{4-}$ is a monoelectronic system. It is also very stable, due to the fact that both complex ions are inert to ligand substitution, which means that almost without exception the oxidations with hexacyanoferrate (III) proceed by outer-sphere mechanisms [141]. A rate constant of $5.54 \times 10^4 \text{M}^{-1}\text{s}^{-1}$ has been determined for the electron transfer.



On the other hand, since H^+ ions are not involved in the redox equation, the standard potential of the couple is independent on acidity [142] over a large pH range (4-13). Its relatively small value (0.41 V for $[\text{K}^+] = 0.1 \text{M}$), makes hexacyanoferrate a weak oxidant. It acts, therefore, as selective oxidizing agent, only appropriate for substrates which are highly susceptible to oxidation. Also, for

substrates with more possible oxidation steps, it will be a mild oxidant, generally leading to the first such step. The key step in any of its redox processes is an electron transfer. For this to take place, an ion pair, oxidant-substrate intermediate should be formed in a rapid pre-equilibrium. The electron transfer and rate determining step takes place inside the outer-sphere type of precursor. The presence of cationic species in solution, particularly alkali-metal ions plays crucial role in kinetic reactions in acidic media, since the high negative charge on $[\text{Fe}(\text{CN})_6]^{3-}$ favors the formation of ion-pair. Larger cations increase the reaction rate due to formation of cation bridge of $[\text{Fe}(\text{CN})_6]^{3-}$. The use of suitable hexacyanoferrate salt generally existing $\text{K}_3\text{Fe}(\text{CN})_6$ is advisable. The electronically favourable CN^- ligand reduces the barrier for an electronic flow from highest occupied molecular orbital of substrate to the lowest unoccupied molecular orbital of metal complex. The reactions order for both the oxidant and the reducing agent are usually one. The oxidations using hexacyanoferrate (III) are expected to be quite simple and free from any secondary processes due to their outer-sphere mechanisms, thus recommending it as an oxidant. One limitation is the sensitivity to catalysis by trace ionic impurities, especially copper, brought in with the chemicals. This aspect needs to be either quantified, eliminated or at least minimized by using highly pure chemicals and if possible also from the same batch. To monitor all oxidations absorption band at 420nm using spectrophotometry or stopped flow techniques is appropriate. With [substrate] reactions are first order as it is always in rate determining step but the basic substrate is more reactive than neutral or protonated species due to transfer of an electron from an electron rich species to substrate. Hexacyanoferrate oxidizes variety of compounds having diverse functionalities like phenols, alcohols, aldehydes, ketones, acyloins, sugars, enediols, α -hydroxy acids, α -ketoacids, α,β -unsaturated acids, amines, hydrazines, heterocyclic cations, hydroxylamines, hydroxamic acids, aminoacids, thiols, thioamides, arylalkanes, aromatic hydrocarbons, olefins, metalloproteins, radicals and chelating agents. Hexacyanoferrate(III) can be used as oxidant for oxidation of those organic substrates which are susceptible to withdrawal of one electron from electron rich site similar to other available oxidants having specificity and selectivity in action such as SeO_2 , $\text{Pb}(\text{OAc})_4$, OsO_4 , t-butyl chromate, CrO_3 -pyridine complex, peroxiacids, periodic acid, peroxitrifluoroacetic acid. These oxidants are versatile and selective for the introduction or attack by functional groups either on simple or complex molecules. It is important to note that hexacyanoferrate (III) fails to oxidise many organic substrates because of its outer-sphere tendency of application for easily oxidisable substrates and thus making it as selective and useful oxidant.

CONFLICT OF INTERST

Authors have no any conflict of interest.

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