Oxidation of chemical compounds: Kinetics and mechanistic approach

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INTRODUCTION

Chemical kinetics is a branch of chemistry which deals with rate of reaction. A detail study of chemical kinetic along with other nonkinetic study will enable us to understand thoroughly mechanism of reactions. There are some reactions which takes place very fast, within fraction of second (upto femto second level). Some reactions are extremely slow for example rusting of iron. In between these two extreme ends, there are reactions which take reasonable time for completion. These reactions can be studied conveniently with suitable methods. There are several researchers who contributed in the field of chemical kinetics. Ludwig Ferdinand Wilhelmy, Wilhelm Ostwald, C F Wenzel, Louis Jacques Thenard, Pierre Eugene Marcelin Berthelot, Leon Pean de Saint-Gilles, Peter Waage and Harcourt etc. had made pioneering work in the field of chemical kinetics [1-4].

Chemical kinetics covers very wide range. It includes empirical studies of the effect of the concentration, temperature and hydrostatic pressure on reaction of various types. Such studies may be practically important in connection with technical processes. There are different types of chemical reactions and a wide variety of experimental technique has been used to investigate them. A considerable amount of efforts has been taken to study the kinetics and mechanism of the reaction in the gas phase. In many ways these investigations are useful for testing basic theories of reactions. Most of kinetic work has been done on reactions in liquid phase, especially, since it is of greatest interest to the organic and inorganic chemists. A little amount of work has been done on the reaction in the solid phase. The reaction at the gas-solid interfaces have been received more attention. Though there are various types of reactions, but oxidation reactions are widely studied. The aim of present review article is to review various oxidizing agents used to study the kinetics of oxidation process in general and to study the oxidation of medicinal drugs in particular.
Oxidation Level

Whether the organic compound is undergoing oxidation or reduction in a given reaction can be known by oxidation levels of the carbon atom of the functional group. It is used in organic chemistry. Oxidation number method is not convenient method for organic compounds, because classification of organic compounds into oxidation states is more difficult and inconsistent. To know whether organic compound is undergoing oxidation or reduction, the carbon atom of functional group is classified into five oxidation levels.

-Level zero: - The lowest oxidation level for carbon is zero. When carbon is bonded to hydrogen or carbon then & only then this state in achieved.

-Level One: - The next oxidation level is one i.e. level one is also called alcohols oxidation level. In this category, carbon atoms which have one bond to an electronegative atom such as O, N, S, X, etc.

-Level Two: - The oxidation level two contains carbon with two bonds to electronegative atoms ex.

-Level Three: -Level three contains carbon with three bonds to electronegative atoms
Level Four: - When carbon is bonded to four electronegative atoms directly, it is called level four.

\[
\begin{align*}
\text{Cl} & \text{C} \text{Cl} \\
\text{Cl} & \text{C} \text{Cl} \\
\text{Cl} & \text{C} \text{Cl} \\
\text{Cl} & \text{C} \text{Cl} \\
\text{H}_2 & \text{N} & \text{C} & \text{O} & \text{Me} \\
\text{H}_2 & \text{N} & \text{C} & \text{O} & \text{Me} \\
\text{H}_2 & \text{N} & \text{C} & \text{O} & \text{Me} \\
\text{H}_2 & \text{N} & \text{C} & \text{O} & \text{Me} \\
\end{align*}
\]

Kinetic Methods

Different methods used to study chemical kinetics. The methods are used for kinetic investigation depends on the half time period or completion time period. The extreme fast reactions are studied using molecular beam flow, stopped flow etc techniques while other reactions can be monitored by pH-metry, conductometry and colorimetry titrations etc. The different methods used are summarized in the table 1.

<table>
<thead>
<tr>
<th>Method and time scale</th>
<th>Description</th>
</tr>
</thead>
</table>
| Conventional          | 1 Mix the reactants together in a batch reactor.  
                         2 Measure concentration Vs time. |
| ≥10^4 s               | 1 Set of continuously flow system where reactants are fed into the reactor and flow out again so quickly that there is negligible reaction.  
                         2 Stop the flow so that reactants reacts.  
                         3 Measure the conversion Vs time. |
| Pressure Jump and      | Nobel prize was given for this technique to Eigen.  
                         Temperature Jump | 1 Mix the reactants at such a low temperature that the reaction rate is negligible.  
                         ≥ 10^-6 s           | 2 Use CO_2 lasers to suddenly heat the reaction.  
                                              | 3 Measure concentration Vs time.  
                                              | 4 Measure the reactant concentration Vs time. |
| Flash photolysis      | Nobel prize was given for this technique to Porter.  
                         10^-9 - 10^-10 s | 1 Put the reactants into a vessel under conditions where reaction is negligible.  
                                              | 2 Pulse a laser flash lamp to start reaction.  
                                              | 3 Measure the reactant concentration Vs time. |
| NMR                   | 1 Initrate a change with magnetic pulse.  
                         10^-2-10^-9 s     | 2 Measure the decay of spins by NMR. |
| Conventional flow      | 1 Continuously feed reactants into a reactor.  
                         system                  | 2 Measure the steady state reaction rate. |
| ≥ 10^-3 s             | Nobel prize was given for this technique to Hersch Feld.  
                         Molecular Beam        | 1 Direct Beams of reactants towards each together in a vacuum system.  
                         10^-9-10^-13 s       | 2 Measure the steady state reaction rate. |
| Femto spectroscopy     | Nobel prize was given for this technique to Ahmed Zewel.  
                         10^-15 s             | 1. Life time reaction can be studied. |

Different oxidizing agents

The species which oxidizes other species give up oxygen or electronegative atom which accept hydrogen or any other electropositive element which gain electron are called oxidizing agents. There are large numbers of compounds which can be used as oxidizing agents in organic chemistry. Classification of these compounds is complicated and difficult. Although the
oxidation of medicinal drugs, in the present study is carried out by potassium permanganate, it is necessary to have a look into the different oxidizing agents available.

The N-halo compounds are widely used as oxidizing agents for example, N-chloronicotinamide which can be prepared by passing a slow stream of chlorine in the solution of nicotinamide in HCl. It is white precipitate with melting point 220°C [5].

Kinetics of oxidation of alpha amino acids by N-Chloronicotinamide in aqueous acetic acid medium in presence of hydrochloric acid has been investigated by Vivekanandan and Nimbi [6]. They reported first order oxidation with respect to [oxidant] and [HCl]. The rate of reaction depend on solvent, it increases with decrease in dielectric constant. They proposed that reaction takes place because of molecular chlorine, which act as strong oxidizing agent. The mechanism of reaction suggested was

\[
\begin{align*}
N&-\text{Nicotinamide} + H^+ + Cl^- \xrightarrow{k_1} \text{Nicotinamide} + Cl_2 \\
Cl_2 + \text{Amino acid} \xrightarrow{k_2} \text{Complex} \\
\text{Complex} \xrightarrow{k_3} \text{Complex (aldehyde+NH}_3^+\text{+CO}_2^-})
\end{align*}
\]

The other N-halo compounds are N-Bromophthalimide [7], N-Bromoacetamide [8], and N-Chlorobenzamide [9].

The oxidation of alpha amino acids by ethyl N-Chlorocarbamate (ECC) in aqueous acetic acid leads to the formation of the corresponding aldehydes [10]. The reaction is first order with respect to ECC. It was observed that reaction rate increases with an increase in the polarity of the medium. The reaction was susceptible to the both polar and sterric effects of the substituents. The mechanism proposed is

\[
\begin{align*}
\text{RCH(N}^+\text{H}_3\text{)}\text{COO}^- + Cl^- \xrightarrow{\text{Complex}} \\
\text{Complex} \xrightarrow{\text{RCHNH}_2^+ + CO}_2^- + HCl + \text{EtCOONH}^-}
\end{align*}
\]

The N haloamine has diverse nature of its ability to furnish halonium cations, hypo species, an N anion which acts as both bases and nucleophile. These compounds contain positive halogen and are mild oxidants [11]. The various compounds of which oxidation kinetics was investigated in the literature include. N-Chloronicotinamide which is reported to oxidized cyclohexanol [12]. N-Bromosuccinimide is a common name of 1-Bromo-2,5-pyrrolidinedione, with molar mass 177.98.

N-Bromosuccinimide oxidation [13] of L-arginine in aqueous acidic medium is first order in [NBS], fractional order in [L-arginine] and of inverse fractional order in [H^+]. The suggested mechanism was
Complex (C) → \text{R-}[\text{C}=\text{COOH} + \text{HBr} \rightarrow \text{R-}[\text{C}=\text{COOH} + \text{NH} + \text{HBr} \rightarrow \text{R-}[\text{C}=\text{COOH} + \text{H}_2\text{O} \rightarrow \text{RCHO} + \text{NH}_3 + \text{CO}_2

N- Bromosuccinamide is successfully used to study kinetics and mechanism of oxidation of aliphatic Ketone [14], 5-aminopyrazoles [15], 1-2 diols [16]. Polyhydric alcohols [17], Lysozyme [18], norephedrine [19], D-arabinose [20], D- xylene and D- galactose [20], secondary alcohols [21]. Amines [22], acid red due [23], diamine [24], silicone [25], vanillin [26], caffeine [27] and thiols [28] are oxidized by N- chlorobenzene sulphonamide.

Oxidation of L- arginine by diperiodato nickelate (IV) (DPN), in aqueous alkaline medium was studied by Kembhavi et al. [29]. They reported that reaction is first order with respect to [DPN], fractional order in [L-arginine] and [OH\(^{-}\)]. The rate of reaction decreases with decrease in the dielectric constant. The suggested mechanism is

\[\text{[Ni(H}_3\text{IO}_6\text{)(H}_2\text{IO}_6\text{)(OH)}_2\text{]}^{3-} \rightleftharpoons \text{[Ni(OH)}_2\text{(H}_2\text{IO}_6\text{)}\text{]}^{+} \text{H}_3\text{IO}_6^{2-}
\]

\[\text{Complex} \rightarrow \text{R-}[\text{C}=\text{NH}+ \text{Ni(OH)}_2 + \text{H}_3\text{IO}_6^{2-}

Quinolinium chlorochromate is an orange/ brown crystalline solid. It is a selective oxidizing agent for the oxidation of primary alcohol in presence of secondary alcohol.

The kinetics of oxidation of aliphatic primary alcohol by quinolinium chlorochromate (QCC) to the corresponding aldehydes has been studied by Abdul Jameel [30]. He observed that the reaction is first order in [QCC], [alcohol] and [H\(^{+}\)]. Based on kinetic measurement the proposed mechanism is

\[\text{Alcohol} + \text{QCC} \rightleftharpoons \text{Complex}\]
Quinolinium dichromate (QDC) is also used as oxidant for organic and inorganic compounds [31]. It is prepared by adding a known quantity of quinoline to cold solution of chromium trioxide in water with stirring. After 30 minutes, the solution is diluted with acetone and cooled to -20 °C for about 15 hours. A orange solid is obtained (melting point-160 °C). It is recrystallised from water.

Similar oxidants used are zinc chlorochromate, pyridinium chlorochromate, magnesium and potassium chlorochromate [32]. Tetraethyl ammonium chlorochromate is a mild and selective oxidizing reagents used for the oxidation of primary aliphatic alcohols [33].

The potassium nitrosodisulphonate (PNDS) (Fremy’s radical) can be used as oxidizing agent, Baloji Kawale [34] et al studied oxidation of alpha amino acids by Fremy’s radical in aqueous borate buffer medium. They reported that oxidation is first order in [PNDS] and [alpha amino acid]

\[
\text{RCH(N}^+\text{H}_3\text{)COOH} + \text{OH}^- \xrightarrow{\text{H}^+} \text{RCH(N}^+\text{H}_3\text{)COO}^- + \text{H}_2\text{O}
\]

\[
\text{RCH(N}^+\text{H}_3\text{)COO}^- + \text{N(SO}_3^-\text{)}_2 \xrightarrow{\text{H}^+} \text{RCH(N}^+\text{H}_3\text{)COOH} + \text{N(SO}_3^-\text{)}_2
\]

\[
\text{RCH(N}^+\text{H}_3\text{)COOH} + \text{OH}^- \xrightarrow{\text{H}^+} \text{RCH(N}^+\text{H}_3\text{)COO}^- + \text{H}_2\text{O}
\]

\[
\text{RCH(N}^+\text{H}_3\text{)COO}^- + \text{N(SO}_3^-\text{)}_2 \xrightarrow{\text{H}^+} \text{RCH(N}^+\text{H}_3\text{)COOH} + \text{N(SO}_3^-\text{)}_2
\]

H S Singh et al [35] reported that the rate of oxidation of ethyl glycol, n- mannitol and n-sorbitol by hexacyanoferrate (III) ion in aqueous alkaline medium is directly proportional to [substrate] and [OH⁻]. The dependence of rate on hexacyanoferrate ion is nearly first order at lower concentration and tends towards zero order at high concentration. The mechanism involved is

\[
\text{S} + \text{OH}^- \xrightarrow{k_1} \text{S}^- + \text{H}_2\text{O}
\]

\[
\text{S}^- + [\text{Fe(CN)}_6]^{3-} \xrightarrow{\text{C (complex)}} \text{C} \xrightarrow{k_{-1}} \text{S}^- + [\text{Fe(CN)}_6]^{3-}
\]

\[
\text{S}^- + [\text{Fe(CN)}_6]^{3-} \xrightarrow{\text{Product} + [\text{Fe(CN)}_6]^{3-}}
\]

The kinetics of oxidation of Disaccharides such as lactose, maltose, cellobiose and melibiose has been studied by hexacyanoferrate in the presence of ammonia [36]. The other compounds for
which oxidation by hexacyanoferrate is studied includes aromatic amines [37], glutathione [38], trithanolamine [39], diol [40], thiols [41], and phenylhydrazinium chloride [42].

K K Banerji et al [43] reported that oxidation of alpha amino acids by pyridinium hydrobromide perbromide (PHPB) in aqueous acetic acid leads to the formation of the corresponding aldehydes. The reaction is first order with respect to PHPB. The proposed mechanism was

\[
2\text{RCH}(\text{N}^+\text{H}_3)\text{COO}^- + \text{PyH}^+\text{Br}_3^- \rightarrow \text{Complex}
\]

\[
\text{Complex} \rightarrow \text{RCH} \rightarrow \text{N}^+\text{H}_2 + \text{CO}_2 + \text{Br}^- + \text{HBr} + \text{PyHBr} + \text{RCH}(\text{N}^+\text{H}_3)\text{COO}^-
\]

Chloramine- T: - The IUPAC name of chloramines T is 4-chloro-4 methyl benzenesulfonamide. Its molar mass is 227.64. In water it breaks to yield a hypochlorite. It can be used as a source of electrophilic chlorine in organic synthesis.

Chloramine-T is another oxidant; literature shows oxidation of methyl vinyl ketone and isopropyl methyl ketone [44], ketoglutaric acids [45], formic acids [46], substituted trans cinnamic acids [47], methionine [48], substituted and unsubstituted imidazole and benzimidazoles [49].

The kinetics of oxidation of ascorbic acid has been reported by using Octacyanomolybdate (V) [50]. Periodate ion is also used as oxidizing agent, the substrate used was Crotonic acid [51], Hexamethylenediamine tetraacetocobaltate (II) complex [52]. t-butyl hydro peroxide is reported to study kinetics and mechanism of oxidation of anisole [53]. Solvent free oxidation of primary alcohol [54] by potassium dichromate and oxidation of arsenius acid [55] by persulphate is also reported.

The other oxidants used in for the kinetic and mechanistic study of organic and inorganic compounds include hydrogen peroxide [56], N-Chloros accharin [57,58], Pyridinium chloro chromate[59], Tris(benzhydro xamato)iron(III) [60], Benzimidazidiumfluorochromate [61], Benzimidazidium bromochromate [62], Diperiodatoargentate(III) [63], Imidazolium dichromate [64], methylene blue [65], diperiodatocuprate (III) [66], tetrakis (pyridine) silver dichromate [67], benzyl trimethylammonium chlorobromate [68], morpholinium chlorochromate [69], trichloro isocyanuric acid [70], Cetyl trimethylammonium dichromate [71].

**Permanganic oxidation:-**

MnO₄⁻ with Mn (VII) oxidation state is the inorganic oxidant. Formerly known as permanganate of potash or condy’s crystals, it is a strong oxidizing agent. It dissolves in water to give intense purple solutions, the evaporation of which gives prismatic purplish – black, glistening crystals, potassium permanganate decomposes when exposed to light:

\[
2\text{KMnO}_4 \rightarrow \text{K}_2\text{MnO}_4 + \text{MnO}_2 + \text{O}_2
\]

It forms orthorhombic crystals with constants: a=910.5 pm, b=572.0 pm, c=742.5 pm.

The MnO₂ is fused with KOH and heated in air or with KNO₃ (a source of oxygen). This process gives potassium manganate, which upon electrolytic oxidation in alkaline solution gives potassium Permanganate, permanganates can also be generated by treating a solution of Mn²⁺ ions with strong oxidants such as PbO₂ (lead oxide), or sodium bismuthate (NaBiO₃). These reactions used the vivid violet colors of permanganate as a test for the presence of manganese. Almost all applications of KMnO₄ exploit its oxidizing properties. As a strong oxidant that does
not generate toxic byproducts, KMnO₄ has many niche uses. KMnO₄ is one of the principal chemicals utilized in the film and television industries to ‘age’ props and set dressing. The Mn-containing products from redox reactions depend on the pH. Acidic solutions of permanganate are reduced to the faintly pink manganese (II) ion (Mn²⁺) and water. In neutral solution, permanganate is only reduced by three electrons to give MnO₂, wherein Mn is in a 4+ oxidation state. Mn shows variable oxidation state from 7+ to 2+. The most stable oxidation state for Mn are 2+, 4+, and 7+.

Manganese (III) ions exist in strong concentration acidic media. It undergoes disproportionation to give Mn²⁺ & Mn¹⁺. Trivalent manganese is known as mangenic ion. The Mn is stable in 4+ forms as MnO₂. It is grey to grey white black solid. In fuming sulphuric acid, it dissolves to give clear blue solution. In potassium hypomanganate (K₃MnO₄) manganese is in 5+ states which is unstable and decomposes to give Mn⁴⁺ or Mn³⁺. It slowly decomposes to MnO₂. The Mn in +6 states exists only in basic solution as deep green manganese ion. The permanganate i.e. Mn in 7+ oxidation state and Intense purple colour.

In Visible range, the solution of potassium permanganate shows different $\lambda_{\text{max}}$ which indicates mostly Mn⁷⁺, Mn⁶⁺ & Mn⁵⁺. The absorption maxima data for these species is given in the table 2. The visible spectrum of permanganate is unaltered by changes in solvents and temperature.

<table>
<thead>
<tr>
<th>Species</th>
<th>Maximum wavelength (Mµ) &amp; molar extinction coefficients×10⁻³</th>
</tr>
</thead>
<tbody>
<tr>
<td>MnO₄⁻</td>
<td>546(ε = 2.38); 526(ε = 2.40); 311(ε = 1.80)</td>
</tr>
<tr>
<td>MnO₄²⁻</td>
<td>606(ε = 1.71); 439(ε = 1.38); 347(ε = 1.63); 299(ε = 1.66)</td>
</tr>
<tr>
<td>MnO₃⁺</td>
<td>667(ε = 0.90); 313(ε = 3.90)</td>
</tr>
</tbody>
</table>

Alkaline KMnO₄ is also used [72] to oxidize simultaneously Pb²⁺ and Ti⁺. The other inorganic species which are studied by acid or alkaline KMnO₄ includes sulphite [73], arsenic (III) [74]. KMnO₄ is used for the oxidation of methylated humic acids [75], thymidine and thymidylic acids [76], DNA [77], 4-thiouracil derivatives [78], primary alcohol [79], aldehydes [80], catechol [81], silymarin [82], L-valine [83], mandelic [84], pyrimidines [85], 3 and 5 formylsalicylic [86], L-valin [87], substituted mandelic acids [88] and pyrimidines [89].

Potassium permanganate is also used to carry out induced oxidation of carboxylic acid. There are several reactions which can be carried out simultaneously. Some of them are spontaneous and others nonspontaneous. When a system consists of two reactions one is spontaneous and another nonspontaneous, the spontaneous reactions cause a nonspontaneous processes. This is called chemical induction. The induced oxidation of various carboxylic acids indicates involvement of Mn (VII) species [90].

\[
\begin{align*}
\text{Mn (VII)} + \text{As (III)} & \rightarrow \text{As (V)} + \text{Mn (V)} \\
\text{Mn (V)} + \text{As (III)} & \rightarrow \text{As (V)} + \text{Mn (III)} \\
\text{Mn (III)} + \text{CH}_2(\text{COOH}) & \rightarrow \text{Mn (II)} + \text{oxidation product}
\end{align*}
\]

**Oxidation of Medicinal drugs**

Since in the present study, oxidation of medicinal drug is carried out, a literature survey regarding oxidation of various drugs by different oxidizing agents has been made. It was observed that the medicinal drug was removed from water by oxidation process or it would be helpful to understand the metabolism of the medicinal drugs, in pharmacokinetics study.
The gabapentin is a potent drug used for the prevention of seizure. It is prescribed for the management of neuralgia, mood disorder, anxiety and tardive dyskinesia. Mohan and Jagdeesh [91] studied kinetics of oxidation of gabapentin by chloramines–T and by N-Bromosuccinimide in perchloric acid medium [92]. Chloramines–T is considered to be antiseptic has been used in the oxidation of vitamin B₁ and B₆ [93].

Metronidazole and tinidazole are antibacterial and anti-protozoal drugs. They are used in the treatment of amoebiasis. In the presence of an anaerobic electron transport system, the nitrogen group of these compounds is reduced to a series of transiently reactive intermediates that are thought to cause DNA damage. N-Bromosuccinimide in acid medium is also reported for the oxidation of these drugs [94]. The oxidation of Sulfacetamide, a Sulfonamide drug by alkaline diperiodatocuprate (III) has been studied by Naik et al [95]. They reported that, the reaction is first order in oxidant and has less than unit order in drug concentration. They also observed that rate constants increase with increase in alkali concentration and decrease with increase in periodate concentration.

Atenolol is a drug used to monitor blood pressure because of its cardio selective action as a beta blocker. Puttaswamy and Suresha [96] studied oxidation of Atenolol by chloramine-T in aqueous perchloric acid medium. They reported that reaction is first order with respect to [oxidant], a zero order with respect to [drug] and an inverse fractional order with respect to [H⁺]. They also used N-chloro-p-toulene sulfonamide (chloramines-T) for the oxidation of Atenolol in Alkaline medium [97].

Sulfamethoxazole (SMZ) is a antibacterial drug. Its removal from waste water by environmental friendly oxidant, potassium ferrate (VI) was studied by Sharma and other [98]. They observed that the oxidation is first order with respect to SMZ and Fe (VI) respectively. The second order rate constant decreases non linearly with increase in pH. The results of their study reveal that K₂FeO₄ has the potential to sence as an oxidative treatment for removing SMZ from water. Williams [99] has also included SMZ in the list of essential drugs.

Chloroamphenicol is a antibiotic drug. It is reported that, the kinetics of oxidation of chloroamphenicol by 1- chlorobenzotriazole (CBT) in HClO₄ medium over the temperature range 293-323 K exhibits first order with respect to [oxidant]₀ and zero order with respect [substrate]₀ respectively. The fractional order dependence on [H⁺] indicates formation of protonated complex between oxidant and [H⁺] [100].

Mephenesin and Guaifenesin are used for the relief of skeletal muscle spasm and have mild sedative property. Puttaswamy and Sukhdev [101] studied oxidation of these drugs using chloramine-B in hydrochloric acid medium. The oxidation behavior is similar for both the drugs. The rate shows a first order dependence on both [CAB]₀ and [HCl]₀ respectively and is fractional in [sub]₀.

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