



e-ISSN: 2319-8753 | p-ISSN: 2347-6710

# IJIRSET

International Journal of Innovative Research in  
**SCIENCE | ENGINEERING | TECHNOLOGY**

# INTERNATIONAL JOURNAL OF INNOVATIVE RESEARCH

IN SCIENCE | ENGINEERING | TECHNOLOGY

Volume 12, Issue 5, May 2023

**ISSN** INTERNATIONAL  
STANDARD  
SERIAL  
NUMBER  
INDIA

Impact Factor: 8.423

9940 572 462

6381 907 438

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# Kinetics and Mechanism of Oxidation of Aromatic Carbonyl Compounds by Dichromate in Aqueous Acetic Acid

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**ABSTRACT:** The kinetics of oxidation of benzaldehyde (BA) and para-substituted benzaldehydes by imidazolium dichromate (IDC) has been studied in aqueous acetic acid medium in the presence of perchloric acid. The reaction is first order each in [IDC], [Substrate] and  $[H^+]$ . The reaction rates have been determined at different temperatures and the activation parameters calculated. Electron withdrawing substituents are found to increase the reaction and electron releasing substituents are found to retard the rate of the reaction and the rate data obey the Hammett relationship. The products of the oxidation are the corresponding acids. The rate decreases with the increase in the water content of the medium. A suitable mechanism is proposed.

**KEYWORDS**-kinetics, oxidation, aromatic, carbonyl, acetic, acid

## I. INTRODUCTION

Kinetics of the oxidation of benzyl alcohol and some of its para substituted derivatives by acidified dichromate has been studied in acetic acid – water medium. The reaction showed first order dependence on both [substrate] and [oxidant]. The reaction failed to induce the polymerisation of added acrylonitrile and this rules out the involvement of any radical intermediate in the reaction. The reaction was found to be acid catalysed and showed fractional dependence on [acid]. The order of reactivity among the studied benzyl alcohols is  $p - OCH_3 > p - CH_3 > - H > p - Cl > p - NO_2$ . Plots of  $\log k_2$  versus Hammett's substituent constant ( $\sigma$ ) has been found to be curve shaped and this suggests that there should be a continuous change in transition state with changes in substituent present in the substrate from electron donating to electron withdrawing. Thermodynamic parameters were determined and a suitable mechanism is proposed in concordance with the obtained results.[1,2,3]

Hexavalent chromium in various forms has been widely used as mild and selective oxidizing agent in synthetic organic chemistry<sup>1-2</sup> and is mainly applied in the oxidation of primary and secondary alcohols to corresponding carbonyl compounds. There are many reports on such type of oxidations for synthesis of carbonyl compounds and on their kinetic aspects.<sup>3-7</sup> But studies on structure – activity relationship are scanty and hence the present work is reported.

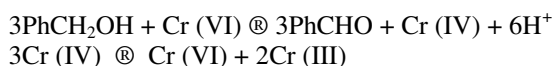
The present report deals with the kinetics of the oxidation of benzyl alcohol and substituted benzyl alcohols by acidified dichromate using acidified dichromate in aqueous acetic acid medium. Stoichiometry of the reaction, product analysis, effect of concentration of added acid, salt, substrate and oxidant, effect of polarity of the medium, effect of substituent and effect of temperature on the oxidation were carried out. Based on the results obtained activation parameters were computed. Structure – activity relationship was also studied by plotting the Hammett plot. A suitable mechanism is proposed based on the experimental observations.

## II. MATERIALS AND METHODS

Analar grade potassium dichromate (Merck, India) was used and its solution was prepared in doubly distilled water. Analar Benzyl alcohol and acetic acid (Merck, India) were purified by reported methods.<sup>8</sup> 4 – methoxybenzyl alcohol, 4 – methylbenzyl alcohol, 4 – chlorobenzyl alcohol and 4 – nitrobenzyl alcohol (Lancaster, England) were used as such.

Kinetic measurements were carried out under conditions where  $[PhCH_2OH] \gg [K_2Cr_2O_7]$ . The progress of the reaction was followed by titrating aliquots of the reaction mixture with standard sodium thiosulphate at regular intervals of time. The experiments were repeated and pseudo-first order rate constants,  $k_{obs}$  were computed from the linear least square plots of  $\log$  [oxidant] versus time.

Stoichiometry of the oxidation was established by equilibrating known excess concentration of potassium dichromate with known amount of benzyl alcohol. It was found that one mole of dichromate is equivalent to three moles of benzyl alcohol.



The product, corresponding benzaldehyde was identified as its 2,4 – dinitrophenylhydrazone (DNP) and is characterized by melting point and by various spectral techniques. The yield of the product was found to be above 85% and no traces of benzoic acid formed were detected. The oxidation of benzyl alcohol by potassium dichromate in an atmosphere of nitrogen failed to induce the polymerization of acrylonitrile and rules out the involvement of any radical intermediate in the reaction.[4,5,6]

The effect of  $[\text{H}^+]$  on the rate of oxidation of benzyl alcohol was studied by varying  $[\text{H}_2\text{SO}_4]$  under pseudo-first order condition and is given. The plot of  $\log k_{\text{obs}}$  versus  $\log [\text{H}^+]$  was linear with a positive slope and the order with respect to  $[\text{H}^+]$  is found to be fractional. The linear increase in the oxidation rate with acidity suggests the involvement of a protonated Cr (VI) species in the rate-determining step.

The oxidation of benzyl alcohol was carried out with different initial concentrations of the oxidant and substrate in 20% aq. acetic acid medium with  $1.0 \times 10^{-2} \text{ mol dm}^{-3} \text{ H}_2\text{SO}_4$  at 308 K and rates were measured. The effect of [oxidant] and [substrate] on oxidation is given. The plot of  $\log [\text{oxidant}]$  versus time was found to be linear at various [oxidant]. This proved that the reaction is first order with respect to [oxidant]. This was further confirmed from the constancy in the values of specific rates ( $k_{\text{obs}}$ ) for the different [oxidant] for a given concentration of the substrate. The effect [substrate] on the rate of oxidation was studied with different initial concentrations of the substrate and the observed rate constant increased linearly with the increase in [benzyl alcohol]. Further, the second order rate constants  $k_2$ , were found to be constant indicating first order dependence of the reaction with respect to [substrate]. The first order dependence on [substrate] is further confirmed by the plot of  $\log k_{\text{obs}}$  versus  $\log [\text{substrate}]$  which is linear with a slope of unity. The reaction was found to show first order kinetics with respect to [oxidant] and [substrate]. The effect of  $[\text{H}^+]$  and [salt] on the rate of oxidation was studied. The effect of polarity of the medium on the rate of oxidation of benzyl alcohol was investigated by varying the acetic acid percentage in the reaction mixture. It has been observed that the reaction rate increases with increase in percentage of acetic acid (decrease in dielectric constant of the medium) suggesting more polar solvents may require larger reaction time. The plot of  $\log k_2$  versus  $1/D$ , where  $D$  is the dielectric constant of the medium is linear ( $r = 0.9978$ ) with positive slope. This implies the occurrence of an interaction between a dipole and a positive ion and indicates the probable involvement of protonated Cr (VI) species in the reaction.

The reaction mixture failed to induce the polymerization of added acrylonitrile which rules out the involvement of any radical intermediate. This indicated that a one electron oxidation is not possible and supports the interaction between an ion and a dipole. The influence of substituents at the para position of the benzene ring of benzyl alcohol was studied and found that electron-releasing substituents accelerate the oxidation process while the electron-withdrawing groups retard the process. The plot of  $\log k_2$  with  $s$ , where  $s$  is the substituent constant gave a smooth curve. The curved shape of Hammett's plot may be due to gradual change in mechanism on passing from electron donating to electron withdrawing substituents or may be due to the change in rate determining step on changing substituents or due to change in the nature of transition state.

It has been reported that the active species responsible for the oxidation of primary and secondary alcohols using Cr (VI) compound is  $\text{HCrO}_4^-$ .<sup>14-16</sup> A hydrogen abstraction mechanism leading to the formation of free radicals is ruled out in view of the failure to induce the polymerization of acrylonitrile. The reaction is first order with respect to [oxidant] and [substrate]. The observations suggest the formation of an intermediate in a pre-equilibrium step and its subsequent disproportionation in the rate-determining step. It has been already established that oxidation of primary alcohols exhibits a substantial primary kinetic isotopic effect<sup>17-18</sup>. This confirmed the cleavage of a C–H bond in the rate-determining step. The negative value of the polar reaction constant together with substantial deuterium isotope effect indicates that the transition state approaches a carbocation in character. Hence the transfer of hydride ion from the alcohol to the oxidant is suggested. The linear increase in the rate of oxidation with acidity suggests the involvement of protonated Cr (VI) species in the rate-determining step. The negative  $\text{DS}^\ddagger$  values are in agreement with the formation of a chromate ester. Bordwell<sup>19</sup> has documented cogent evidence against the occurrence of concerted one step bimolecular process by hydrogen transfer. It is well established that intrinsically concerted sigmatropic reactions, characterised by transfer of hydrogen in a cyclic transition state are the only truly symmetrical processes involving a linear hydrogen transfer.<sup>20</sup> Littler has also shown that a cyclic hydride transfer, in the oxidation of alcohols by Cr (VI), involves six electrons and being a Huckel type system, is an allowed process.<sup>21</sup> The overall mechanism is proposed to involve the



formation of a chromate ester in a fast pre-equilibrium step and then a disproportionation of the ester in a subsequent slow step via a cyclic concerted symmetrical transition state leading to the product (Scheme 1). The observed hydrogen ion dependence can be explained by assuming a rapid reversible protonation of the chromate ester and the protonated ester decomposing at a faster rate than the chromate ester.[7,8,9]

### III.DISCUSSION

Among oxo derivatives of variable valence metals, Chromium compound play the most important role, in oxidative reaction. A number of Chromium reagents are readily available. Almost every oxidisable functional group may undergo Chromium oxidation. Chromium (VI) containing reagents include Chromium acid, dichromate ion, Chromyl chloride, chromyl acetate, *t*-butyl Chromate, Chromyl nitrate and Co-ordination complexes of Chromium trioxide. 1-2 Chromium (VI) oxidations are usually performed under acidic condition. Co-solvents like (Jones's reagent) benzene, methylenechloride (Two Phase system) are often added in order to deal with water insoluble organic complexes. The oxidation rate is generally high under acid catalysed conditions, however the low pH of the reaction medium and the presence of water favouring hydrolytic condition exclude the use of this class of reagents for the oxidation of molecules containing acid sensitive groups.[1] Polymer supported Chromium (VI) reagents have also been developed. These reagents offer the advantage of reducing the work of procedure to more filtration. Recently some neutral or almost neutral Chromium (VI) reagents have been developed to effect oxidation under mild conditions. Pyridinium Chlorochromate (PCC) introduced by Covey et al<sup>3</sup> is widely used in the oxidation of alcohols. Pyridinium fluorochromate has a less pronounced acidity and is an effective agent for the oxidation of Polycyclic organic substitutes.[2] PFC was developed by Bhattachargee and coworker. 4 In 1986, Narayanan and Balasuramaniam<sup>5</sup> introduced Pyridinium bromochromate. This is an efficient oxidant for alcohols and a brominating agent as well. Firouzahadi and Coworkers used Zinc chromates. This is a very useful reagent for the oxidation of variety of organic compounds, including alcohols, oximes, olefins and aromatic hydrocarbons.[5] They have also shown chromium peroxide complexes as versatile, mild and efficient oxidants in organic synthesis. Chlorotrimethyl Silone, Imidazolium dichromate, 2,2'-Bipyridyl chlorochromate (BPCC) and the corresponding chromate (BPC) have been proposed as oxidants for the oxidation of the hydroxyl group to the carbonyl group. 4-(dimethyl amino)-pyridinium chlorochromate and chromyl nitrate have been shown to be efficient and mild oxidant in aprotic media. Naphthyridium chlorochromate, Pyrazinium chlorochromate, tetrabutyl ammonium chlorochromate, dimethylpyrazole chromate, tetrabutyl ammonium dichromate, Pyridine complex of oxidiperoxochromium and the dipyridine complex of chromium trioxide are some of the Chromium (VI) oxidants introduced in the last twenty years.[6] Quinolinium Chlorochromate was used by Bhavani et al. to oxidize methyl phenyl sulphoxides. Quinolinium dichromate was introduced by Balasubramanian and Prathiba<sup>6</sup> as an effective oxidant under non-aqueous conditions.[7] Pyridium Chlorochromate (PCC) is irreplaceable in the generation of functional groups in highly unsaturated carbinols and pyrrols. yet mildly acidic character of Pyridinium chlorochromate precludes its use with acid sensitive substrates or products. Pyridium Chlorochromate oxidation has been investigated under various condition on different substrates. Despite the introduction of numerous oxidants based on chromium (VI) not much work has been done to investigate the mechanism of oxidation in these cases.[10,11,12]

Kinetic investigations on the oxidation of organic and inorganic substrates with the well known oxidant, peroxodisulphate (PDS), have been carried out by many research workers.<sup>3</sup> Recently the corresponding monoperoxo compound, namely peroxomonosulphate (PMS), has attracted the attention of many research workers. Peroxomonosulphate and peroxodisulphate bear relation in their structures as the derivatives of H<sub>2</sub>O<sub>2</sub>, replacing one or both the hydrogen atoms in H<sub>2</sub>O<sub>2</sub> by an SO<sub>3</sub> group. Simple metal-ion-catalysed<sup>4</sup> as well as the dual system Ag<sup>+</sup>-S<sub>2</sub>O<sub>8</sub><sup>2-</sup>-catalysed<sup>46</sup> and uncatalysed thermal decompositions of peroxomonosulphate (PMS) and oxidation of chloride: bromide, *i*-dide, nitrite, chlorite, and hypophosphite<sup>10</sup> ions have been carried out. Recently, radiolytic chain decomposition,<sup>11</sup> in situ photolysis-e.s.r.,<sup>12</sup> flash photolysis<sup>13</sup> of PMS, and photochemical chain decomposition of peroxomonosulphate in the presence and absence of propan-2-ol<sup>14</sup> have been carried out. Oxidation of organic substrates like aldehydes,<sup>15</sup> dimethyl sulphoxide,<sup>16</sup> pyridine,<sup>17</sup> *o*-benzoquinone,<sup>18</sup> glycine,<sup>19</sup> cyclopentanone,<sup>20</sup> cyclohexanone,<sup>21</sup> and anilines<sup>22</sup> by peroxomonosulphate have been carried out. Peroxomonosulphate is found to undergo reactions by different types of mechanisms (free radical, nucleophilic, or electrophilic) in the investigations mentioned above. It has been also observed that peroxomonosulphate is a better oxidant than peroxodisulphate in the oxidation of halide ions.<sup>6</sup> The present study involves the oxidation of benzaldehyde and substituted benzaldehyde by peroxomonosulphate. In this study, it is proposed to find out: (i) whether the reactions follow free-radical pathways or polar mechanisms, (ii) the effect of [H<sup>+</sup>] since the reactions of peroxides are prone to acid catalysis, (iii) the effect of substituents on the reactivities of benzaldehydes and product distribution, (iv) the mechanistic aspects resulting from the kinetic and thermodynamic data, and (v) the relationship of the kinetic parameters and the reactivity with the

corresponding reactions of similar peroxides, peroxodisulphate,  $S_2O_8^{2-}$  and peroxomonophosphate,  $H_2PO_5^-$ . Experimental All the chemicals used were of AnalaR grade. Potassium peroxomonosulphate was a gift from du Pont de Nemours under the trade name ~xone.~ The purity of this salt was estimated by cerimetry using ferroin indicator, as well as by iodometry and was found to be 96% pure. Tests with permanganate showed the absence of free hydrogen peroxide. No attempt was made to purify the compound further because of previous unsuccessful attempts." From the methods of preparation of this compound, it is assumed that that  $KHSO_5$ , or  $K_2S_2O_8$ , or both might account for the 4% difference between the formula and analysis results. Benzaldehyde (B.D.H. AnalaR) was used after distillation, b.p. 179-179.5 °C. All the other aromatic aldehydes, o-chlorobenzaldehyde, o-methoxybenzaldehyde, o-nitrobenzaldehyde, m-bromobenzaldehyde, m-chlorobenzaldehyde, m-methoxybenzaldehyde, m-nitrobenzaldehyde, p-bromobenzaldehyde, p-methoxybenzaldehyde, p-nitrobenzaldehyde, and p-tolualdehyde were the purest samples available from Fluka and were used as such. Acetic acid used as solvent was purified (to 99.9%) before use. Other chemicals were analytical grade samples from B.D.H., and sodium perchlorate was from E. Merck. Experiments were carried out in aqueous acetic acid medium (30-700%, v/v) at a constant ionic strength of 1.20 mol dm<sup>-3</sup> maintained by the addition of NaClO<sub>4</sub>. The effect of [H<sup>+</sup>] on the rate was studied by carrying out the kinetic runs with [H<sup>+</sup>] 0.10--1.00 mol dm<sup>-3</sup>. Under the present experimental conditions, no spontaneous self-decomposition - as well as carbonyl-assisted decomposition - of the oxidant could be detected. The kinetics of the reaction was followed by monitoring the disappearance of peroxomonosulphate by iodometry at different time intervals. The rate constants were obtained by integral methods. The stoichiometry of the reaction was established by estimating unchanged PMS by iodometry and the aldehydes by the 2,4-dinitrophenylhydrazone method.<sup>26</sup> Product analysis was carried out under the condition [aldehyde]  $\gg$  [PMS], as in the kinetic runs. After the complete disappearance of PMS, the mixture was extracted with ether. The ether extract was dried with (Na<sub>2</sub>SO<sub>4</sub>) and evaporated and the residue containing the product and unchanged substrate was then analysed by g.c. after converting the acid products into methyl ester by reaction with diazomethane, and phenols into methyl ether by reaction with dimethyl sulphate. The g.c. measurements were carried out with Varian model 3700 gas chromatograph attached to a Varian CDS 401 microprocessor. Identification of the products was made by comparison of retention time data of authentic samples. Duplicate experiments were always carried out to estimate the reproducibility of the results (&5%). The least-squares method was employed to estimate the uncertainties in the values of the kinetic and thermodynamic parameters [16,17,18]

#### IV.RESULTS

It is reported that benzaldehyde and most of its substituted derivatives are not hydrated to any appreciable extent. Moreover, no reaction is known in which nucleophilic attack on tetrahedral carbon as in an aldehyde hydrate is faster than that on the free aldehyde " ArCHO. So it is fair to assume that the free HC=O group of the aldehyde acts as the active species of the substrate as in the case of oxidation of aromatic aldehydes by peroxomonophosphate.~ The acid dependence observed is attributed to the acid-catalysed decomposition of the adduct formed by the fast addition of peroxomonosulphate to the aldehydes. Two kinds of products are found to be formed depending upon the migration of hydride and/or aryl group. Taking p-methoxybenzaldehyde as an example, the products formation by aryl and hydride migration may be represented by the Scheme. In the case of benzaldehyde oxidation, only benzoic acid was identified which is due to hydride migration and there was no phenol formed. The rate constant for o-methoxybenzaldehyde is at a maximum and is twice as high as that for p-methoxybenzaldehyde revealing the relative unimportance of steric effects. The electron-withdrawing substituents o-Cl and o-NO<sub>2</sub> exhibit lower reactivity than those at the meta- and para positions. Generally, the electron-withdrawing groups are found to favour hydride migration and electron-donating groups favour aryl migration. Among the electron-donating groups, CH<sub>3</sub> and OCH<sub>3</sub>, the reactivity trend is in the order p-CH<sub>3</sub> < m-OCH<sub>3</sub> < p-OCH<sub>3</sub> < o-OCH<sub>3</sub>. Among the electron-withdrawing substituents, the reactivity trend observed is p-NO<sub>2</sub> > m-NO<sub>2</sub> > p-Br > m-Br > m-Cl > o-NO<sub>2</sub> > o-Cl. Multiplying the values of k<sub>2</sub> (overall second-order rate constant) with the product selectivity, the rate constants for hydride and aryl migrations are calculated.~ From the plot of log k<sub>H</sub> versus  $\sigma$ , the p value for hydride migration is calculated and found to be in good agreement with the p (+1.74) observed for the corresponding reaction of peroxomonophosphate (PMP) with benzaldehydes. However, for aryl migration, from the plot of log k<sub>A</sub>, versus  $\sigma$  the observed value of p in the present investigation is less negative than that observed in the PMP oxidation (-2.88).<sup>27</sup> This discrepancy may be due to the difference in the reaction conditions, the PMP reactions being carried out at pH 1.3 and above whereas the present work involves [H<sup>+</sup>] 0.40 mol dm<sup>-3</sup>. Similar values of p, as exhibited in the present investigation, have been observed in Baeyer-Villiger oxidations involving the slow and rate-determining migration. It is also known that the rate of the Baeyer-Villiger reaction changes with substrate and pH for various peroxide reactions.<sup>27</sup> The AS: values are found to be highly negative indicating the formation of a severely structured transition state. The values of ASs observed in this investigation are very similar to the values reported by Panigrahi and Panda in the reactions of para-substituted benzaldehydes with peroxomonophosphate <sup>33</sup> at



0.5 mol dm<sup>-3</sup>, H'. The similar values of AG' observed in the present investigation may indicate that the mechanism operative is similar for all the reactions[19,20,21]

## V.CONCLUSION

peroxomonosulphate is found to undergo nucleophilic reactions with aromatic aldehyde, whereas the available literature on peroxodisulphate (PDS) oxidation of benzaldehyde shows that the reaction follows a free-radical mechanism involving SO<sub>2</sub><sup>-</sup> as the reaction intermediate. Comparing the results of oxidation of benzaldehydes by peroxomonophosphate 27\*33 in acidic medium with those of the present investigation, it is found that these two oxidants behave similarly: (i) the reactions exhibit non-radical overall second order kinetics, (ii) the mechanisms involve fast addition of the peroxide with aldehydes followed by the rate-determining hydride or aryl migration, and (iii) they exhibit product selectivities and correlation of substituent constants (σ) with the rate constants of hydride and aryl migration steps. The behaviour of peroxomonosulphate is also similar to the other organic peroxides 35~36 undergoing nucleophilic reactions with benzaldehydes.[22,23] However, the product distribution and reactivity differ due to the better oxidising power of, and the better leaving group (SO<sub>2</sub><sup>-</sup> or €+SO<sub>4</sub><sup>-</sup>) from, peroxomonosulphate.[24]

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