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Catalytic Effect of 1,10-phenanthroline on the kinetic Studies of D-galactose by Cr(VI) in CPC Micellar Media

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ABSTRACT

In experimental conditions, $[\text{Cr(VI)}]_T << [\text{D-Galactose}]_T$ at various temperatures in an aqueous H_2SO_4 environment, the reductant D-Galactose is oxidized by hexavalent chromic acid with occupancy of a catalyst like 1,10-phenanthroline (phen). Cr(VI) species in monomeric form are kinetically active without phen catalysts. In contrast, the notion is that the Cr(VI)-phen complex is the vigorous oxidant with the phen catalyst. Cr(VI)-phen compound is subjected to nucleophilic onslaught by reductant to produce a ternary intricate compound that experiences an oxidation-reduction decomposition process through a couple of electron transfers, resulting in Cr(IV)-species and organic products in a catalysed route. Uncatalysed and catalysed routes depict 1st order dependency on $[\text{D-Galactose}]_T$ and also $[\text{Chromic acid}]_T$. Without a catalyst, the reaction exhibits 2nd order dependency on $[H^+]$. However, the phen route exhibits 1st order dependency on hydrogen ion concentration. The catalyzed path is a 1st order dependency on $[\text{phen}]_T$. In sight of N-cetylpyridinium chloride (CPC), these findings remain unchanged. Although CPC retards, the rate is in phen-catalysed paths. Micelle-based consequence viewings have been taken into account to determine the proposed reaction mechanism in the aqueous micellar phase.

Keywords: Catalysis, D-Galactose, Micellar, Surfactant, Oxidation.

INTRODUCTION

Diverse valency of metal ions, like chromic acid, have been used to oxidatively reduce various reducing sugars[1–12]. Such studies provide insight into the chromium chemistry in the environment where the hexavalent-charged metal ion appears as a chemical hazard due to its carcinogenic and mutagenic activity[13–15]. Biomolecules interact with intermittent chromium states to induce toxicity[15]. In vivo, reduction may occur due to the presence of reduced sugars. Hence, their survey appears to be pertinent in this context. Reports have arrived[1,16] of various ketohexoses undergoing oxidation in aqueous acid ambiance with various transition metal ions. Among diverse chelating agents[8,17–37], phen, bipy chelating agents appear to be the most elegant[38–42] chelating agents used to promote chromic acid oxidation of several organic substrates These never undergo co-oxidation along with the substrate. 1,10-phenanthroline acts as a chelating agent in micro-heterogenous reveals, similar to biological simulations, to uphold the suggested reaction path initially *in vivo* chromium reduction (VI).

EXPERIMENTAL

Reagents and Materials

D-galactose (E-Merck), 1,10-phenanthroline (E-Merck), $K_2Cr_2O_7$(AR,BDH), N-cetylpyridinium chloride (CPC) (E-Merck), and commercially available highest grade other chemicals were used. All solutions are set up by applying conductivity H_2O.

Method and Kinetic Dimensions

Necessary chemicals, acids, catalyst (phen),(i) $[\text{substrate}]_T$>>$[\text{hexavalent chromium})]_T$ and (ii) $[\text{phen}]_T$>>$[\text{hexavalent chromium}]_T$), D-galactose, i.e., familiar amounts of the substrate and oxidant solution were separately thermostated($\pm 0.1^{\circ}C$) under experimental conditions. A specific quantity of chromic acid was mingled with the reaction mixture to trigger the reaction. The titrimetric quenching technique[19] monitored hexavalent chromic acid's disappearance rate and tracked the reaction progress. The usual mathematics was used to calculate pseudo 1st order rate constants (k_{obs}). Investigation into the probable decomposition of surfactant by chromic acid revealed that the decomposition rate was insignificant. Usual estimation procedures were executed to detect errors in various rate constants and activation parameters[43].

Product Analysis and Stoichiometry

Paper chromatography[3,6,7] was used to identify the reaction product under the reaction condition such that [hexavalent chromic acid] <<$[\text{D-galactose}]_T$. Here, aldonic acid was detected as the major product. Eluant water– acetic acid– butan-1-ol (volume as in ratio 5:1:4) was used in paper chromatographic detection. A specific reagent[44], beta-anisidine, was employed specifically for D-galactose identification, along with a tri-stage dip of $AgNO_3$, NaOH, and $Na_2S_2O_3$[45]. These two developing reagents were used to visualize paper chromatography.

Thus, the stoichiometry of the reaction is

$$3C_6H_{12}O_6 + 2Cr\,(VI) \rightarrow 3C_6H_{10}O_6 + 6[H^+] + 2\,Cr(III)$$

RESULTS AND DISCUSSION

Dependability on [Hexavalent chromic acid]$_T$

When $[\text{D-galactose}]_T$>>$[\text{1,10- phen}]_T$>> $[\text{chromic acid}]_T$ was maintained, both in occupancy and absenteeism of phen, it was noticed that the disappearing rate of hexavalent chromic acid demonstrated a 1st order dependency on chromic acid. This 1st order dependency on hexavalent chromic acid was also depicted with surfactant CPC presence. Pseudo-1st order rate constants (k_{obs}) wereassessed by graph of log $[Cr(VI)]_t$ vs. time (t),

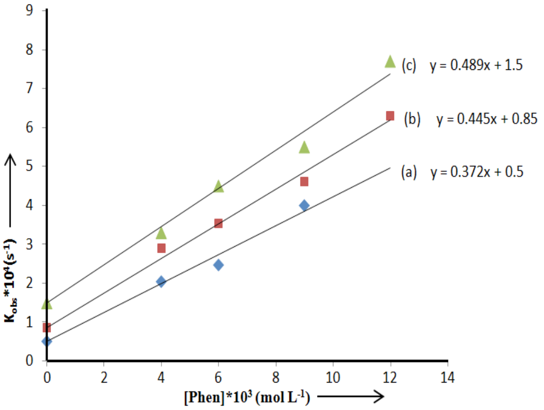


Fig. 1. $[Cr(VI)] = 60*10^{-5}$ (M), [D-galactose] = $1*10^{-2}$ (M), $[H_2S0_4] = 5*10^{-1}$ (M), $[\text{1,10-phen}]_T = (0\text{-}14)*10^{-3}$ (M), T = 22°C(a), 32°C(b), 40°C(c)

Dependability on [phen]T

The positive intercept was obtained from the liner plot (r>0.99) of k_{obs} vs. $[\text{phen}]_T$, which measures the existance of the comparatively sluggishwith uncatalysed route. There is a good agreement with the pseudo-1st order rate constants ($k_{obs(u)}$) estimated, in absenteeism of phen, it has been acquired from the intercepts of graph of $k_{obs(T)}$ vs. $[\text{phen}]_T$. These surveillancesmay be equated as:

$$k_{obs(T)} = k_{obs(c)} + k_{obs(u)}$$

$$= k_{cat}[L]_T + k_{obs(u)}\ (L=1,10\text{-phen,}) \qquad (1)$$

The above equation holds good both in occupancy and absenteeism of CPC surfactant. Table 1 provides some kcat values along with the activation parameters. In course of reaction, loss of phen occours because of generation of Cr(III)-phen inert complex compound.

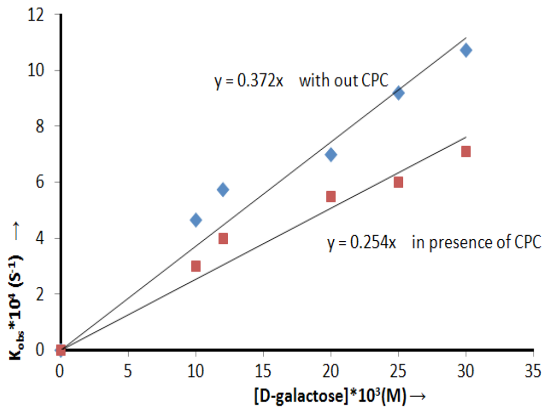


Fig. 2. $[Cr(VI)] = 6*10^{-4}$ (M), [D-galactose] = $(10-30)*10^{-3}$ (M), [phen] = $90*10^{-4}$ (M), $[H_2SO_4] = 0.5$ (M), T = 22°C, [surfactant] = 0 (M) (in absence of CPC), [surfactant] = $6*10^{-3}$ (M) (with CPC)

Dependability on $[Substrate]_T$

Graph of k_{obsvs}. $[D\text{-Galactose}]_T$ establishes that catalyzed path leads to 1st order dependency on $[substrate]_T$, so,

$$k_{obs(c)} = k_{obs(T)} - k_{obs(u)} = k_{s(c)}[S]_T \quad (2)$$

$$k_{obs(u)} = k_{s(u)}[S]_T \quad (3)$$

In occupancy of CPC surfactant, is obligatory for maintaining 1st order dependency on $[S]_T$. Table (i) gives the conscience of $k_{s(u)}$ and $ks_{(c)}$.

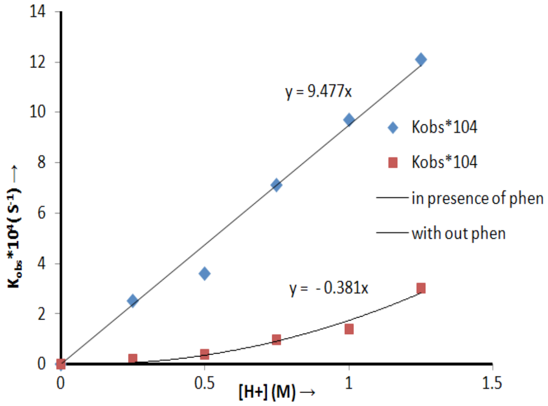


Fig. 3. $[Cr(VI)] = 6*10^{-4}$ (M), [D-galactose] = $100*10^{-4}$ (M), $[HClO_4] + [NaClO_4] = 15*10^{-1}$ (M), [phen] = $90*10^{-4}$ (M), T = 30°C

Dependability on $[H^+]$

It is observed that the acid dependency intricates for the absenteeism of catalyst and in occupancy of catalyst paths are varied. The following findings are:

$$k_{obs(u)} = k_{H(u)}[H^+]^2 \quad (4)$$

$$k_{obs(c)} = k_{H(c)}[H^+] \quad (5)$$

Surfactant Effects

From theplot, it has been perceived that reaction rate is retarded with surfactant (CPC).

Acrylonitrile polymerization Test For Free Radical

In sight of free radicals was ascertained by acrylonitrile polymerization under reaction kinetics in N_2 atmosphere .

Evaluation of Activation Parameters

By applying the Eyring equation, activation enthalpy($\Delta H^{\neq}$) and activation entropy($\Delta S^{\neq}$), have been induced.

$$-\ln(kh/k_BT) = \Delta H^{\neq}/RT - \Delta S^{\neq}/R$$

Where k_B= Boltzmann constant (1.38×10^{-23} J K^{-1}), h = Planck constant (6.62×10^{-34} J s^{-1}), R = molar gas constant (8.31 J $mol^{-1}K^{-1}$). Activation-free energy ($\Delta G^{\neq}$) and its error may be evaluatedin distinction to mathematical relations:

$$\Delta G^{\neq} = RT \ln(k_BT/hk) \text{ and } \delta\Delta G^{\neq} = RT (\delta k/k)$$

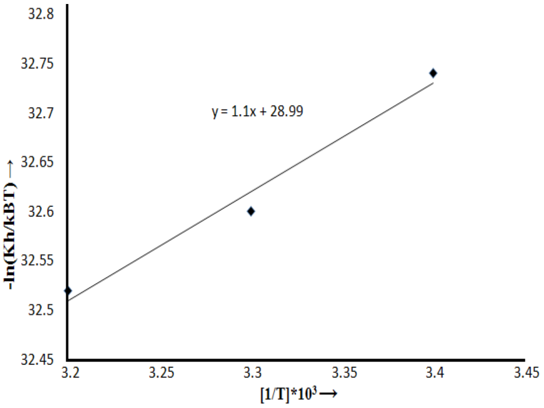


Fig. 4. Graph for calculation of activation parameter

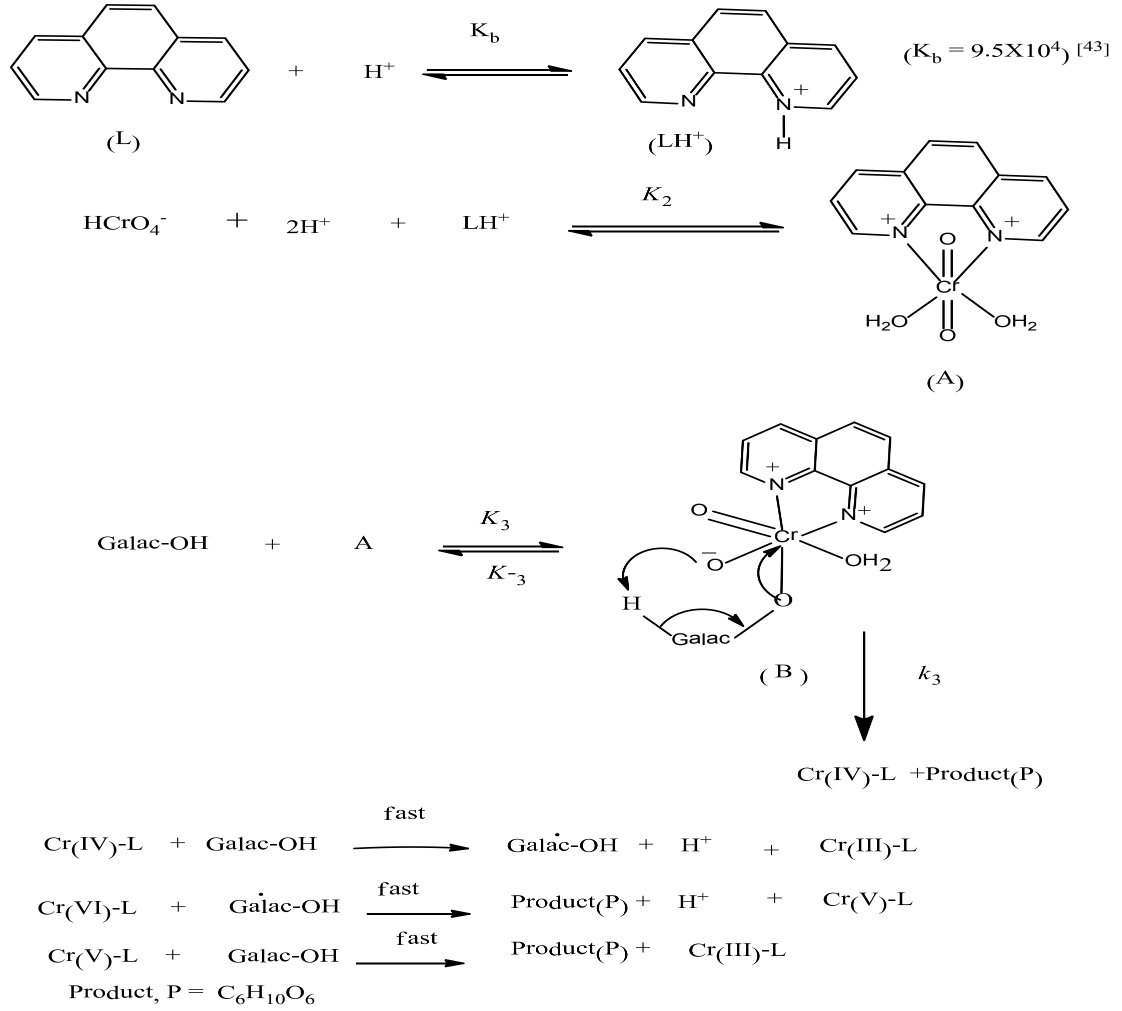
Kinetics aspects and mechanism of the reaction

Scheme 1 depicts the reaction mechanism for D-galactose for the phen-catalysed paths and can explain the experimental results leading to the rate laws thus:

$$k_{obs(c)} = (2/3)k_2K_3K_4\, [S]_T[L]_T[H^+] \quad (6)$$

Ligand phen be subjected to complexation with chromium's far up labile oxidation states, resulting in Cr(III)-phen in the catalyzed path.

Idleness of Cr(III)(t_2g^3) inhibits entry of the catalyst into the inmost coordination domain of Cr(III), which is initiated upon the reduction of Cr(IV). In Pre-equilibrium state the active oxidant[38,39] is the complex (C1) formed by the rapid reaction of phen with the labile Cr(VI) center to produce the instinctive cyclic Cr(VI)-L. Over the span of $[L]_T$ used, $[phen]_T$ shows a primary dependency. Thus, it is pertinent to assume that the equilibrium constant for cyclic Cr(VI)-L complex (C1) is of a low magnitude. Hence, there is no kinetic affirmation for C1 composite generation. In addition to phen, there is no change in the chromic acid spectrum, thus negating any spectroscopic evidence. However, this does not obliterate the possibility of such complex formation[46–49]. In oxidation-reduction reactions, cyclic form of sugar get involved[6,7,3]. Arguably, better exposure of hemiacetal hydroxy groups result in profound interaction with Cr(VI). Hence, it is justified to conclude that the cyclic and the acyclic forms contribute additively to the pseudo 1st order rate constants while being in a potent equilibrium. Subsequently, The interaction of the substrate with Cr(VI)-L complex results in a ternary compound (C2) (*cf.* Scheme 1) that undergoes redox decomposition to finally yield the organic product before passing through a cyclic transition state. The Cr(IV)-phen composite compound might later take part in more swift reactions, i.e., the Rocek mechanism[50]. $\Delta S^{\neq}$ (entropy of activation, *cf.* Table 1) with a large negative magnitude of the complex rate constant (k_{cat}) indicates a cyclic transition state. The final product is obtained when the Cr(IV)-species, caused in the rate-determining step, takes part in the forthcoming faster paces.



Scheme 1. Mechanistic aspects of Cr(VI) oxidation of D-galactose in presence of 1,10-phenanthroline

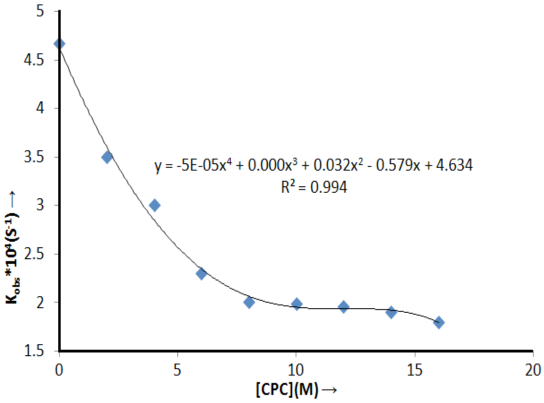
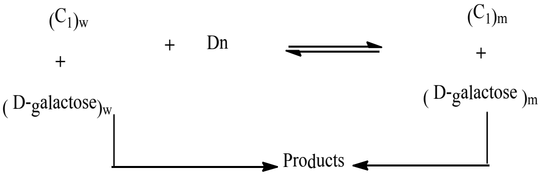


Fig. 5. [Cr(VI)] = 6*10^{-4}(M), [D-galactose] = 10* 10^{-3}(M), [phen] = 9*10^{-3}(M), [H_2SO_4] = 0.5(M), T = 32°C, [CPC] = (2-16)* 10^{-3}(M)

Effect of surfactant(CPC)

CPC, is a typical cationic surface active agent, acting as an inhibitor of the catalysed paths. An incessant diminution and, ultimately, the ease off at larger CPC concentrations was observed in $k_{obs(T)}$ vs. $[CPC]_T$ (*cf.* Fig. 5) profile. It matches the observation of Cerichelli and Bunton[51] in the ferrocene oxidation using ferric salts in surfactant like CTAB (cetyltrimethyl ammonium bromide) media. Panigrahi and Sahu[52] reported very similar observations in acetophenone oxidation by Ce(IV) in accompanied by NDPC (N-dodecyl pyridinium chloride). Reddi and Sarada[34] observed similar oxidation of aromatic azo compounds using chromic acid with anionic surfactant SDS. It was found that in the catalysed route, CPC thwarts electropositive Cr(VI)-catalyst composite compound (C1 is the agile oxidizing agent) in water phase, thereby resulting in cumulation of neutral substrate in the stern layer of micellar phase which is impotent to take part in the reaction. Thus, in catalyzed process, this reaction is primarily constrained in the water media, where substrate concentration is exhausted owing to its portioning in stern layer of micellar phase. Scheme 2 depicts the segregation of reactants between micellar and aqueous phases, where Dn appears for micelles surface active agents with ‘n’ signifying accumulation number.



Scheme 2. Partitioning of the reactive species between the aqueous and micellar phases

Table 1: Rate constants and kinetic parameters like $\Delta H^{\neq}$ and$\Delta S^{\neq}$ for D-galactose oxidation by CrIVI) with 1,10-phen catalyst in aqueous-surfactant environment

Temperature(^{0}C)	$10^4k_{obs(u)(w)}$a(s^{-1})	$k_{cat(w)}{}^a$(L $mol^{-1}s^{-1}$)	$k_{s(c)(w)}$(L $mol^{-1}s^{-1}$)	$k_{s(c)(CPC)}{}^b$(L $mol^{-1}s^{-1}$)	$10^4k_{H(c)(w)}{}^d$(L $mol^{-1}s^{-1}$)	$k_{eff(w)\ (10)}$
22	0.50	0.0372	0.0372	0.0254	9.477	7.400000
32	0.85	0.0445				5.235294
40	1.50	0.0489				3.066667
	$\Delta H^{\neq}$ (KJ/mole)				9.24	
	$\Delta S^{\neq}$ (J/K/mole)				-203.71	

Depicts uncatalyzed path (u);catalzsed route (c);aqueous media (w); value in the presence of N-cetylpyridininium chloride media (CPC).

$K_{obs(u)(w)}$ = rate constants of uncatalysed reaction in aqueous medium.

$K_{obs(c)(w)}$ = rate constants in catalysed reaction in non-surfactantphase.

$K_{cat(w)}$ = slope of the plot of $k_{obs(c)(w)}$ vs. [1,10-phen] of phen catalysed reaction in water environment.

$K_{eff(w)} = (k_{obs(c)(w)}-k_{obs(u)(w)})/k_{obs(u)(w)}$, determined at [phen] = 10*10^{-3}(M); [Cr(VI)] = 60*10^{-5}(M); [D-galactose] = 1*10^{-2} (M); [H_2SO_4] = 5* 10^{-1}(M).

(a) [Cr(VI)] = 60*10^{-5}(M); [D-galactose] = 1*10^{-2}(M); [1,10 phen] = (2-12) *10^{-3}(M);[H_2SO_4] = 5* 10^{-1}(M).

(b) [Cr(VI)] = 60*10^{-5}(M); [D-galactose] = (10-30) *10^{-3}(M); [1,10-phen] = 40*10^{-4}(M); [H_2SO_4] = 5* 10^{-1}(M); [CPC] = 60*10^{-4}(M).

(c) [Cr(VI)] = 60*10^{-5}(M); [D-galactose] = 1*10^{-2}(M); [H^+] = (25*10^{-2}-125*10^{-2}) (M).

(d) [Cr(VI)] = 60*10^{-5}(M); [D-galactose] = 10*10^{-3} (M); [1,10-phen] = 12*10^{-3}(M), [H^+] = (25*10^{-2}-125*10^{-2}) (M).

CONCLUSION

Reduction of D-galactose by chromic acid in a phen-catalysed path to give the lactone as the oxidized product. The active oxidizing agent has been identified as the cationic species as the Cr(VI)-phen composite compound. This active oxidant combines with the reductant D-galactose to produce a ternary complex that undergoes 2-electron transport oxidation-reduction reactions. The reactions were executed using an aqueous micelle-infused environment. CPC is a cationic surface reagent acting as an inhibitor of the catalysed paths. An incessant diminish and, ultimately, the ease off at larger CPC concentrations has been observed. The observed micellar effects were rationalized in the aqueous-micellar phase to uphold the suggested reaction mechanism.

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Conflict of interest

The author declared no potential conflicts of interest with respect to the research, authorship and/or publication of this article.

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