



Oxidation of Perfumery Secondary Alcohols by Nicotinium Dichromate; A Kinetic Study

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Abstract: Chemical kinetics is mainly concerned with studying the factors that change the reaction rate and the response mechanism. The present paper investigates the rate law for the reaction, order of the reaction of secondary alcohols(menthol) and compares it to other secondary alcohols like Borneol and Isoborneol. Diagnose the product of the oxidation reaction. The kinetics of oxidation of perfumery secondary alcohols [menthol], [Borneol], and [Isoborneol] by NDC have been studied in the aqueous acetic acid medium at 303K. The secondary perfumery alcohols were converted to the corresponding carbonyl compounds. The order concerning [Subs], [Oxi], and [H⁺] ions concentration is found to be one. The decrease in the dielectric constant of the medium enhances the rate of the reaction. The ionic strength of the medium has no significant effect on the reactivity of the response; it does not induce the polymerization of Acrylonitrile, indicating the absence of free radical pathway. The added Mn²⁺ ions show the reaction rate was slightly increasing order. The reaction has been studied at four different temperatures Eyring equation is used to calculate the thermodynamic parameters like Gibbs free energy change ($\Delta G^\#$), entropy change ($\Delta S^\#$), and enthalpy change ($\Delta H^\#$). On increase in the temperature of the reaction, the rate of reaction increased abruptly. There was a decrease in entropy ($\Delta S^\#$) of the system indicated by the value of entropy which showed some restriction in the freedom movement of the molecules. It was observed that the reactivity follows Menthol<Isoborneol<Borneol. Based on steric factors, structure, and isomeric characteristics of the alcohols under investigation.

Keywords: Borneol, Isoborneol, Menthol, NDC

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I. INTRODUCTION

The chemical reaction may have several facets, one of which is oxidation. Oxidation processes are commonly met with everyday life. The study of such oxidation reactions through elucidation of their mechanisms and hence the examination of reaction kinetics in chemical reactions are essential aspects of chemistry. Chemical kinetics deals with the rate of chemical reactions under how fast chemical reactions proceed from reactant to product. All the chemical and biological reactions were carried out based on kinetics.

I.1. Novel studies of Chromium (VI) as an oxidant

Different varieties of Organic compounds undergo oxidation by chromium compounds in aqueous and nonaqueous media¹⁻⁴. Generally, chromium compound is considered a versatile reagent for all the organic compounds⁵. Various chromium (VI) combinations have been established to oxidize primary and

secondary alcohols to corresponding aldehyde and ketone. Highly toxic chromium (VI) compounds are converted to chromium (III). Some chromium VI reagents are well established, viz, QBC, QDC, PCC, PDC, The kinetics of oxidation of poly alcohols has been studied by QBC in aqueous sulphuric acid media⁶ as an effective oxidant under nonaqueous conditions. QBC has been used as a mild selective oxidizing reagent in synthetic organic chemistry⁷. The oxidation of nine primary aliphatic alcohols by QBC in dimethyl sulfoxide has been investigated, leading to the formation of the corresponding aldehyde.

I.2. Nicodinium dichromate [NDC] - acts as an oxidant

Chromium compounds can be used to convert alcoholic group into carbonyl group in several organic compounds. Among them chromium (VI) have been proved to be effective, oxidants recently using NDC(Fig.1). All the chromium compounds.

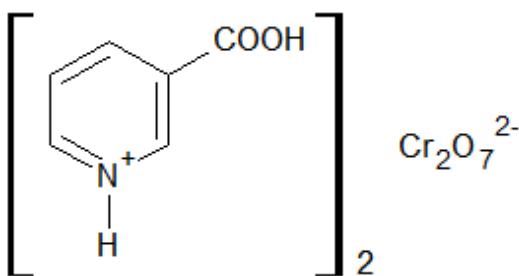


Fig.1. Nicodinium dichromate

They are soluble in water freely. Excess chromium (VI) is a deep orange-yellow solid (mp 250 - 251°C) that can be used as a mild, effective reagent for organic compounds. Due to their application in organic synthesis and kinetic investigation, NDC has been used as an alloy in leather industries. Moreover, NDC has been used for oxidation purposes. NDC is an appreciable stable salt, both aqueous and nonaqueous in the medium. NDC oxidized some organic substrates, viz. the kinetic oxidation of mandelic acid and p-chloro mandelic acid⁸, 2-pentanone⁹, para-substituted benzaldehyde¹⁰, Essential amino acid¹¹, anilines¹², etc.

I.3. Menthol act as a substrate

Menthol is an organic covalent compound and a mono-terpene secondary alcohol. It is collected from perfumery oil like peppermint oil etc. The boiling point of menthol is 212°C, a waxy white crystalline substance and solid at room temperature. Potassium bromates¹³ oxidize it in an acetic solution to form Menthone.

I.4. Medicinal application of menthol

Menthol is used in pharmaceutical¹⁴ and cosmetic industries¹⁵. Various extracts from peppermint contain menthol as a major active constituent. They have been used for centuries as historical medicines for several ailments, including infection, insomnia, irritable bowel syndrome, and on insect repellant. This medication treats minor aches and pain in the muscles/joints. Menthol works by causing the skin to feel cool and then warm. These senses on the skin divert you from feeling the aches/ pain deeper in your muscles and joints. Menthol is presented at skin curative activity by modulating the anti-

accident system of the cells and the intensification response in addition to stimulatory epithelialization.

I.5. Novel trends in chemical kinetics

For example, chemical kinetics have been used daily, and food is kept at room temperature. However, present in the atmosphere that damages food. Hence cooked food is preserved in refrigerators under lesser conditions. The modern trend is kinetics in analytical chemistry, and aniline blue acetic chloride reaction has been used to determine ruthenium (III). Hydropower plants, windmills, roller coasters, moving cars, bullets from a gun, flying airplanes, etc., are based on kinetic energy.

I.6. Scope and importance of the present investigation

The present work is mainly concerned with the kinetics of oxidation of menthol (perfumery 2° alcohol) by Nicotine dichromate. The work is analyzed systematically. Reaction parameters such as the effect of substrate, the development of oxidant, the result of temperature, the impact of varying dielectric constant of the medium effect of perchloric, etc., have been used to investigate the oxidation reaction product analysis has also been carried out. Menthol yield menthone on oxidation. Some physical techniques confirmed the product. The mechanism of the oxidation reaction has been proposed based on the result obtained from the kinetic reaction. The development of the oxidation reaction of menthol was compared to that of Isoborneol and Borneol (perfumery 2° alcohol). The product obtained from this oxidation reaction is camphor (ketone) and has been confirmed by physical techniques. The rate law has been applied to all three types of alcohol. The rate of reaction of all three alcohols was first-

order kinetics. The kinetics of oxidation of three alcohols follow the order.

borneol > isoborneol > menthol

Chemical kinetics play a role in the oxidation process, framing some important areas covered there, like drug design, development, and manufacturing, to ensure the stability of trucks and by physical and chemical pathways. Oxidation kinetics study of the rate and the mechanism of the chemical reaction is used to predict the number of product conservations.

2. EXPERIMENTAL AND METHODS

2.1. Synthesis of nicotinism Dichromate (NDC)

Nicotinic acid (7.38g, 60mM) was added to chromium trioxide (12g, 120mM) dissolved in water (12 ml) at 0.5° C (ice water bath) with stirring. After 15 minutes, acetone (100 ml) at 0.5° C was added to the resulting red-orange suspension, and the mixture was stirred at 0.5° C for 15 minutes. Finally, the product was filtered off and washed with acetone (4 x 50ml) and dichloromethane (25 ml), affording nicotinism dichromate¹⁶ (6 g) as an orange-yellow solid, m.pt 250 to 251° C.

2.2. Isolation and Purification of reagents

The selective secondary alcohols used were purified by distillation, and their physical constant of menthol 97° C and Borneol 213° C. The other reagents are perchloric acid, sodium perchlorate, sodium thio sulphate, Acrylonitrile, and manganous sulphate were all of Anala R grade and were used as such. Acetic acid was purified¹⁷ by the standard method, and the fraction distilled at 118° C was collected. The solutions were prepared in triple distilled water.

2.3. Product analysis of perfumery alcohol by NDC

Menthol (0.1M) and NDC (0.1M) were mixed in 50% acetic acid water and kept at room temperature for two days under conditions employed for the kinetic runs. Then the product was extracted with chloroform. The extraction was dried over anhydrous sodium sulfate, and the chloroform layer was

evaporated. The pure product obtained was the corresponding carbonyl compound, and its derivative test identified it.

2.4. Kinetic measurements studies

Measurements were made at 30° C, and the temperature was controlled using a thermostat to an accuracy of $\pm 1^{\circ}$ C. The desired solution concentration was prepared, like menthol in acetic acid, isoborneol in acetic acid, borneol in acetic acid, NDC in water. Other reagents in doubly distilled water, now the volume of subtracting perchloric water acid and acetic acid, were mixed to bring the percentage of acetic acid to the desired value and thermostated. The reaction was started when half of the oxidant solution had been delivered. Aliquots (2ml) were removed at definite intervals and the liberated iodine was titrated against standard sodium thiosulphate solution to starch (1%) endpoint. Reactions were generally followed to 70 to 80% completion. The kinetic investigation was carried out under pseudo-first-order conditions keeping the subtract concentration in excess. The pseudo 1st order rate constant (K) was calculated from the slope of linear plots by log titer against time by least squares method. Throughout the kinetic studies, the Menthol concentration was kept ten times greater than that of the NDC.

3. RESULTS AND DISCUSSION

The complex of chromium carries out the conversion of hydroxy into the carbonyl group in selective secondary alcohols. The investigation of kinetics and mechanism of oxidation of selective secondary alcohols by NDC has been studied. In this paper, the kinetic reaction was Examined under pseudo-first-order conditions in the presence of perchloric acid using (1:1) solvent mixture. Kinetic results have been encapsulated.

3.1. Effect of oxidant [NDC]

The oxidation of menthol by NDC has been examined by changing the oxidant; keeping other reactant concentrations as constant (Table I) showed the observed rate constant. The method of least square assesses the reaction rate was pseudo first order dependence w.r.to concentration of NDC¹⁸.

Table I. Dependence of the reaction rate on oxidant	
10^2 [NDC] (mol dm ⁻³)	10^4 kobs (s ⁻¹)
0.20	9.22
0.25	9.30
0.30	9.36
0.35	9.67
0.40	9.71

[Alcohols] 1.5×10^2 mol dm⁻³, [H⁺] 0.75×10^{-2} mol dm⁻³
solvent AcOH :H₂O 1:1 [v/v], Temp 303K

3.1.1. Oxidation of menthol by NDC

Different concentrations carried the reaction rate on the concentration of menthol (in the range 1.0 to 3.0×10^{-2} mol dm⁻³) and kept the attention of other reactants as constant at

303K. The reaction rate is found to be first-order dependence^{13,19}. A plot of log (Menthol) versus log Kobs (Fig.1a), is linear (B=0.647; r=0.97 : sd = 0017) (Table 2).

3.1.2. Oxidation of Isoborneol NDC

Table 2: Dependence of the reaction rate on substrate concentration

SI.No	10^2 substrate] mol dm $^{-3}$	10^4 Menthol] K obs s $^{-1}$	10^4 Isoborneol] K obs s $^{-1}$	borneol] K obs s $^{-1}$
1	1.0	7.50	7.50	7.82
2	1.5	9.36	9.60	9.75
3	2.0	10.90	11.49	9.78
4	2.5	12.39	13.57	18.42
5	3.0	13.56	14.50	26.82

[NDC] 0.30×10^{-2} mol dm $^{-3}$, $[H^+] 0.75 \times 10^{-2}$ mol dm $^{-3}$
solvent AcOH:H₂O 1:1 [v/v], Temp 303K

The reaction was carried out with varying concentration of Isoborneol, keeping all the other factors as constant the rate constants were found to increase with the increase in the concentration of Isoborneol¹⁹ (Table 2). The order of the

reaction was found to be nearby unity (0.837) with respect to Isoborneol] as evidenced by the linear plot of log Kobs versus Isoborneol] with slope near to unity (Fig.1) ,(r=0.996; S=0.044).

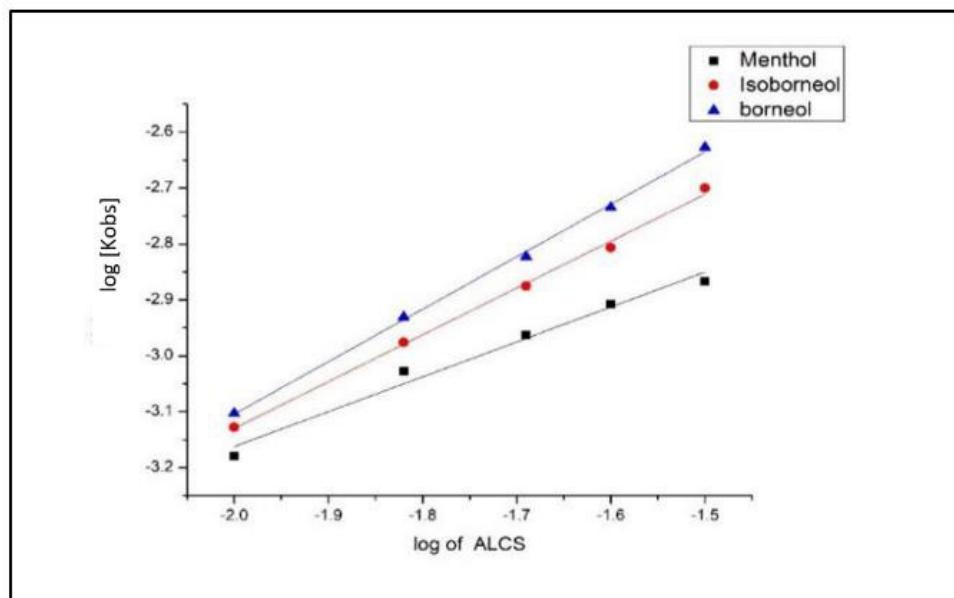


Fig 1a: The plot of log of Kobs versus log [menthol]

3.1.3. Effect of varying Borneol¹⁹

As described earlier, the reaction was carried out with varying concentration of Borneol. The reaction rate constant Kobs was found to increase with the increase in the [Borneol] (Table 2). The plot of log Kobs versus log [Borneol] was found to be linear with a slope [0.938] of near to unity indicating the unit (Fig.1a), (r=0.997; s=0.0368) order dependence on the [Borneol].

3.2. Effect of (HClO₄)²⁰ concentration

The Kinetic reaction was carried out with varying concentrations of perchloric acid. Keeping the concentration of other reactants at a constant rate was measured with the increase in the concentration of perchloric acid there will be enhancement the rate of Oxidation reaction (Table 3). The hydrogen ions provided by solvent molecular had protonated effect on the rate of oxidation reaction. The order of the reaction with respect to [HClO₄] is one.

Table 3: Dependence of the reaction rate on [HClO₄]

10^2 [NDC] (mol dm $^{-3}$)	10^4 kobs (s $^{-1}$)
0.25	5.10
0.50	7.50
0.75	9.36
1.00	11.01
1.25	12.90

[NDC] 0.30×10^{-2} mol dm $^{-3}$, [Alcohols] 1.5×10^{-2} mol dm $^{-3}$
solvent AcOH:H₂O 1:1 [v/v], Temp 303K

3.3. Effect of solvent composition

The reaction was conducted at different solvent composition by varying the percentage of acetic acid in the reaction mixture. The rate of reaction has been found to increase with decrease in the dielectric constant of the medium (Table 4).

Table 4: Values of dielectric constant for various proportion of acetic acid

ACEH: H ₂ O (v/v)	D	$10^2 \times 1/D$	$(D-1)/(2D+1)$	$10^4 [\text{Menthol}]$	$K_1 \text{ obs s}^{-1}$	$10^4 [\text{Isoborneol}]$	$10^4 [\text{borneol}]$
					$K_1 \text{ obs s}^{-1}$	$K_1 \text{ obs s}^{-1}$	$K_1 \text{ obs s}^{-1}$
40-60	49.28	2.09	0.4889		8.17	8.50	8.70
50-50	42.10	2.375	0.4835		9.36	9.60	9.75
60-40	34.92	2.863	0.4788		10.77	10.85	10.95
70-30	27.74	3.603	0.4734		12.07	12.50	13.90
80-20	20.56	4.863	0.4643		13.46	13.80	16.85

[Alcohols] $1.5 \times 10^2 \text{ mol dm}^{-3}$, $[\text{H}^+] 0.75 \times 10^{-2} \text{ mol dm}^{-3}$, $[\text{NDC}] 0.30 \times 10^{-2} \text{ mol dm}^{-3}$ Temperature 303K
 3.4.1 Solvent reactivity correlation relationship of perfumery alcohol by NDC different solvent composition: A Amis plot

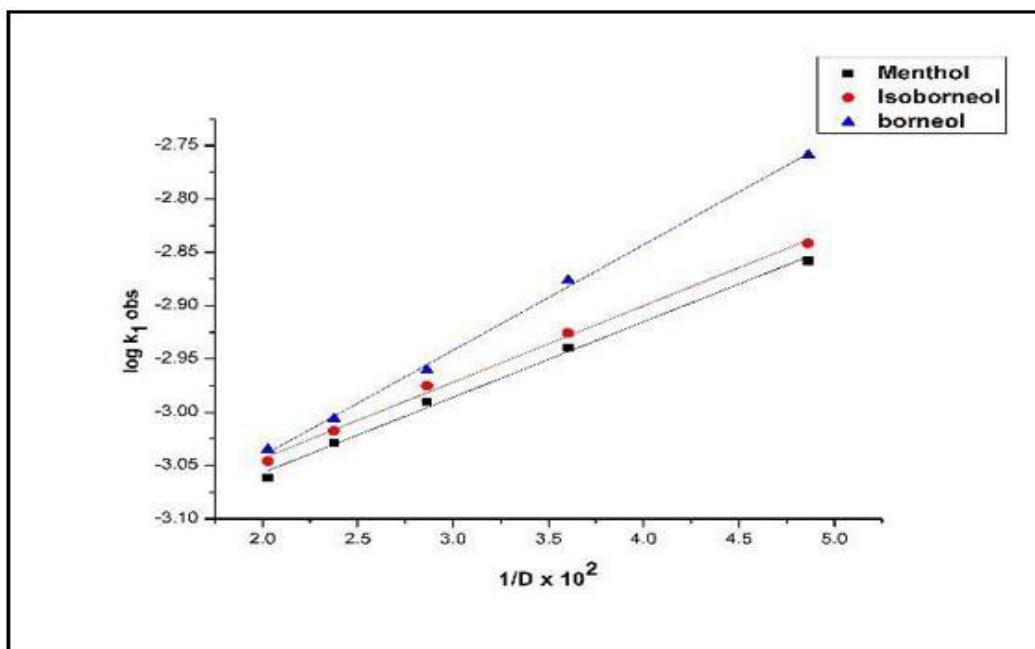


Fig 2: Solvent effect – Amis plot

The percentage of concentration of solvent was commuted, by increasing the concentration of acetic acid like 40%, 50%, 60% etc., While decreasing the concentration of water like 60%, 50%, 40%, etc., and was noted for change. The rate of reaction increases with decrease in the dielectric constant of the medium. (Table 4) The Amis plot of $\log k_1$ versus $1/D$ and the linear plot of $\log k_1$ versus $(D-1)/(2D+1)$ are linear shows in the fig 2. It shows the dipole-dipole and ion-dipole interaction in the reaction. This announces the required excess reaction times for the polar solvents to carry on the oxidation reactions. It also announces that interaction between on ion and dipole transpired in the rate determining step²¹. Thus exhibiting the occurrence of protonated Cr(VI) species during the reaction.

3.4. Effect of $[\text{NaClO}_4]$, acrylonitrile, $[\text{MnSO}_4]$

In order to know the effect of ionic strength on the reaction rate, the reaction was carried out with varying concentration of added perchlorate (Table.5). It has been observed that the addition of perchlorate ion no remarkable effect the reaction takes place. Hence the reaction may be between an ion and neutral molecule or between neutral molecule^{22,23}. The reaction was studied by adding different concentrations of acrylonitrile solutions to the reaction mixture, while keeping the concentrations of all other reactants constant (Table.5). Adding Acrylonitrile to the reaction was not having the free radical pathway and also the polymerization step^{24,25}.

Table 5: Reaction rate for the effect of added $[\text{NaClO}_4]$, [Acrylonitrile] and $[\text{MnSO}_4]$

$10^4 [\text{NaClO}_4] \text{ mol dm}^{-3}$	$10^4 k_1 \text{ obs s}^{-1}$	$10^4 [\text{Acrylonitrile}] \text{ mol dm}^{-3}$	$10^4 k_1 \text{ obs s}^{-1}$	$10^4 [\text{MnSO}_4] \text{ mol dm}^{-3}$	$10^4 k_1 \text{ obs s}^{-1}$
0.00	9.36	0.00	9.36	0.00	9.36
0.57	9.47	9.10	9.33	4.50	9.47
1.14	9.48	18.10	9.32	9.00	9.85
1.71	9.51	27.20	9.29	13.50	10.10
2.28		36.20	9.32	18.00	10.48

[Alcohols] $1.5 \times 10^2 \text{ mol dm}^{-3}$, $[\text{H}^+] 0.75 \times 10^{-2} \text{ mol dm}^{-3}$, $[\text{NDC}] 0.30 \times 10^{-2} \text{ mol dm}^{-3}$ solvent AcOH: H₂O 1:1 [v/v], Temp 303K

The reaction was studied by adding different concentrations of Manganese Sulphate Solution to the reaction mixture while keeping the concentration of all other reactant constant (Table.5). The rate of reaction slightly increases by adding the addition of Mn^{2+} salt. This is shown in the rate determining step^{26,27}.

3.5. Dependence of rate of the temperature

The rate constants were measured at four different temperatures viz., 298,303,308,313 K (Table 6) with an accuracy of $\pm 0.1\text{K}$. from the Eyring's plot $\ln k_{\text{obs}}/T$ in versus $1/T$ ($r > 0.988$; $s <$) the activation parameters $\Delta H^\#$, $\Delta S^\#$ and $\Delta G^\#$ were calculated from the slope and intercept from the isokinetic plot [Fig.3].

Table 6: Variation of rate with temperature and activation parameters of 2° perfumery alcohols

Sl.No	Substrates $10^{-2} \text{ mol dm}^{-3}$	Order with respect to Substrate	$K_{\text{obs}}\text{S}^{-1}$				$\Delta H^\#$ kJ mol^{-1}	$\Delta S^\#$ $\text{JK}^{-1}\text{mol}^{-1}$	$\Delta G^\#$ (303K) kJmol^{-1}	r	S
			298K	303K	308K	313K					
1	Menthol	0.647	8.50	9.36	10.80	11.00	34.14	-197.63	-25.74	0.999	0.0036
2	Isoborneol	0.837	9.52	12.40	13.00	13.30	34.41	-197.55	-25.44	0.995	0.0057
3	Borneol	0.938	10.40	13.40	14.14	14.50	34.99	-197.58	-25.22	0.999	0.00127

$$[\text{NDC}] = 0.30 \times 10^{-2} \text{ mol dm}^{-3}; [\text{HClO}_4] = 0.75 \times 10^{-2} \text{ mol dm}^{-3}$$

Solvent: Acetic acid – water (1:1 v/v)

- Structure and reactivity of secondary perfumery alcohols

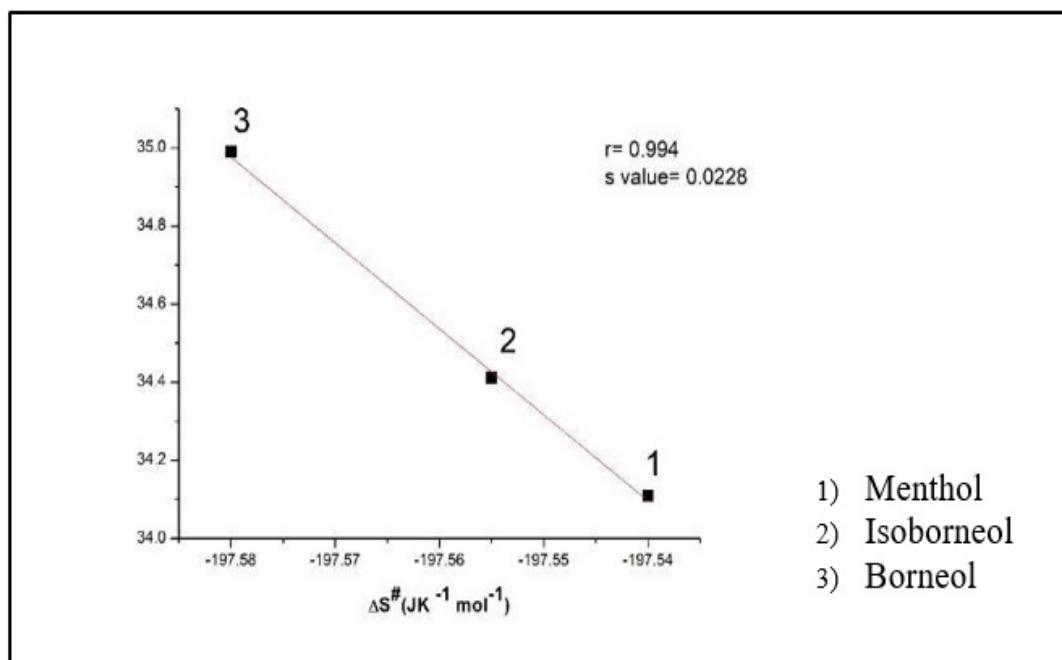


Fig 3: Iso kinetic plots 2° perfumery alcohols

On oxidation all the alcohol under investigation the reaction rates are same with respect to steric factors, in the case of menthol it contained α -hydrogen has more hindred, therefore menthol is susceptible to oxidation, but in the case of Borneol, oxidation is more rapid due to most hindred α - hydrogen¹³.

- Exner plots of perfumery secondary alcohols

Exner plot between $\log k_{\text{obs}}$ at 303K and $\log k_{\text{obs}}$ at 313K

was linear. The value of isokinetic temperature is estimated from Exner's plot at ($r; 0.9786$, $b; 0.7443$). The linear isokinetic correlation implies that all the alcohols are oxidized by the same mechanism, and the changes in the rate are conducted by changes in both the enthalpy and entropy of activation. (Fig 4)

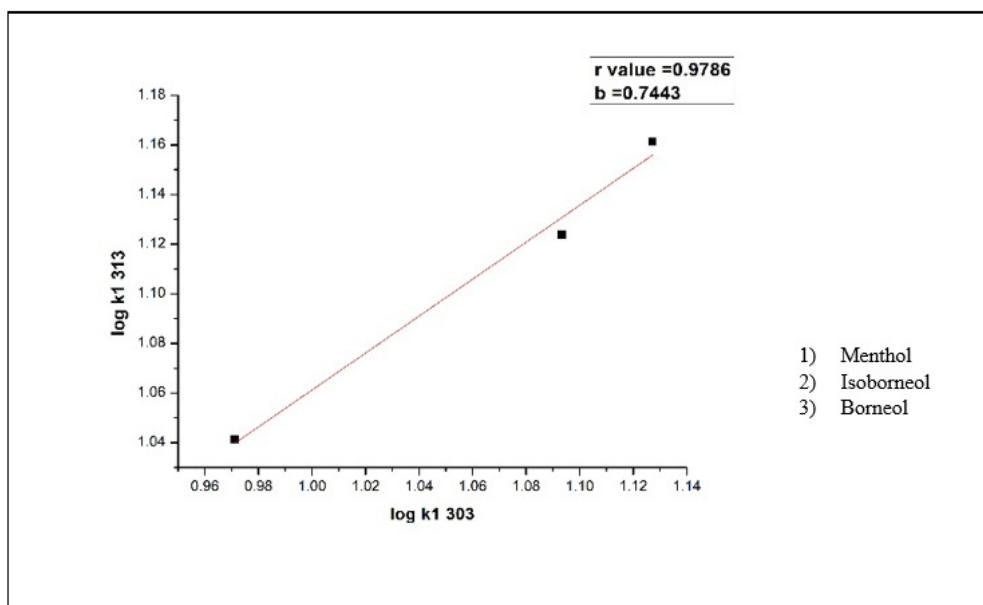
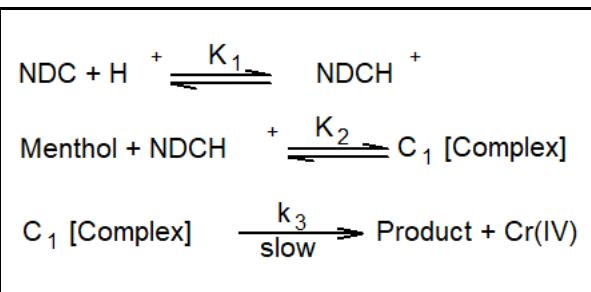


Fig 4: Exner plot of 2° perfumery alcohols

3.6. The rate law for the above mechanism

In this case, the concentration of chromium (VI) is very much lower, under this conditions the active oxidising species is HC_6O_4 ²⁸⁻³⁰. Products analysis clearly indicating that the Menthone. From these observations the following mechanism and rate law were proposed.



Rate Law

$$\text{Rate} = k_3[\text{Complex}]$$

$$k_3 K_2 K_1 [\text{Menthol}] [\text{NDCH}]$$

$$k_3 K_2 K_1 [\text{Menthol}] [\text{NDC}] [\text{H}^+]$$

$$-\frac{d[\text{NDC}]}{dt} = \text{Kobs} [\text{oxidant}] [\text{Menthol}] [\text{H}^+]$$

The proposed mechanism and suitable rate law support all the observations made including the effect of solvent polarity and the negative entropy of activation.

4. CONCLUSION

The oxidation of secondary alcohols by NDC is catalyzed reaction the oxidation of alcohols yields corresponding carbonyl compounds. The reaction source first order with respect to [substrates], [Oxi] and $[\text{H}^+]$ ion concentration. The order of reactivity was found to be menthol < Isoborneol < Borneol

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5. AUTHORS CONTRIBUTION STATEMENT

Mr.C.Surulivel conceptualized and gathered the data with regard to this work. Dr.J.Dharmaraja, Dr.S.Srinivasan and Dr.S.Srinivasan analyzed these data and necessary inputs were given towards the designing of the manuscript. All authors discussed the methodology and results and contributed to the final manuscript.

6. CONFLICT OF INTEREST

Conflict of interest declared none.

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