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Effect of Concentration on the Rate of Production of Oxalic Acid from the Bark of Eucalyptus Camaldulensis

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Abstract – *The kinetics studies on the rate of production of Oxalic acid from the bark of Eucalyptus camaldulensis was analysized using integral and differential methods of analysis using solvent concentration of 3 and 4% H_2SO_4. 3% H_2SO_4 recorded a rate constant of 0.0389 (intergral) and 0.0375 (differential) and rate equation, $-r_A = 0.0389C_A^2$ (intergral) and $-r_A = 0.0375 C_A^2$ (differential) while 4% H_2SO_4 recorded a rate constant of 0.0446 (intergral) and 0.0478 (differential) and rate equation $-r_A = 0.0446C_A^2$ (intergral) and $-r_A = 0.0478C_A^2$ (differential). The result revealed that the rate order of reaction is the same for both percentage of H_2SO_4 and that a better yield was recorded with 4% percent H_2SO_4 concentration. Copyright © 2010 Praise Worthy Prize S.r.l. - All rights reserved.*

Keywords: *Oxalic Acid, H_2SO_4, Kinetics, Rate, Differential, Integral*

Nomenclature

C_A	Concentration of H_2SO_4 at any time t
C_{AO}	Concentration of H_2SO_4 at time $t=0$
k	Rate constant
n	Reaction order
F	Fractional conversation of H_2SO_4
t_F	Fractional conversation of H_2SO_4 at any time t
$\dfrac{d}{dt}$	Rate of change

I. Introduction

Man has, and is still trying to keep close ties with his environment by interacting with both the living and non – living components to better a lot of things in his day. Most of such interaction was limited by the level of technology available. However, technology has come to reveal some properties in plants that were hitherto unknown. One of such plants that have gain significant importance is the *Eucalyptus camaldulensis* now found in some parts of Nigeria. One of the potentialities it has for man is as a raw material base for oxalic acid production.

Oxalic acid is an organic acid ubiquitously occurring in plants, fungi, and animals, and plays several different roles in different living organisms [13]. Recently, oxalic acid application has received much attention in relation to systematic resistance and antioxidant systems ([4], [5], [15], [17] and [19]). Oxalic acid is no doubt among the most important group of organic acid. The presence of calcium oxalate in the bark of *Eucalyptus camaldulensis* makes it possible to process chemical compounds called oxalic acid.

Oxalic acid which is the first member belonging to carboxylic acid series can be used extensively in laundries as a souring agent to remove excess alkalinity in the washed fabrics. It is a constituent of cleaning solution used for the removal of ink, stains and decolourisation caused by bleaching of dyes.

Other uses of oxalic acid include a host of industrial and house-hold applications such as cleaners for rust, grease, wax, and other solid soils from printed, varnished and glass surfaces [9].

In order to produce oxalic acid from the bark of *Eucalyptus camaldulensis* for industrial purposes, there is the need to design a plant that will operate not only at a minimum cost but at conditions that will yield optimum production. The main objective of this work therefore is to study the effects of concentration of sulphuric acid on the rate of production of oxalic acid from the bark of *Eucalyptus camaldulensis*, with the aim of obtaining a satisfactory rate equation to represent the process.

I.1. Origin and Characteristic of Eucalyptus

Eucalyptus is a large genus of evergreen tree and shrubs natives to semi – tropical and warm temperate region of Australia, New Zealand, Tasmania and nearly Islands [2]. There are more than 500 species and belong to the myrtle family (myrtaceae) [2]. Eucalyptus is prominent among Australia genera, commonly known as gum trees or stingy bark trees with many trees forming shade tree or forest plantations [3].

The characteristics of the bark vary greatly. The gum has smooth bark; boxes have coarse fibrous bark; peppermints have fine fibrous stingy bark have thick loose or rough bark that may be shed in strips or flakes [3].

Economically, Eucalyptus tree constitute most valuable group within the other myrtales. The leaves are leathery and hang obliquely or vertically. The flower petals cohere to form a cap when the flower expands. The fruit is surrendered by woody cap shape receptacle and contains numerous minute seeds, possibly the largest fruit from 5 –6 centimeter are produced by E. macrocarpa [3].

The tallest Eucalyptus may reach the height of 100metre (330ft) and a circumference of 7.5 metre. One specimen of E. Regnans is 114m (374ft) tall and is among the world's tallest trees [11]. Other tall species include E. Marginate, E gigantea, E Obligua and E Ovata. The smallest species such as E. Erythranda and E. mitrata, may be less than 2m (6.6 ft) tall [3].

The leave glands of many species contain a volatile aromatic oil known as Eucalyptus oil, its chief use is medicinal and it consistutes an active ingredient in expectorants and inhalants[7].

Also, the juvenile leaves are usually opposite, sessile (not stalked) and covered with whitish bloom. Adult leaves are alternate stalked, often suckle shaped and lack whitish bloom. The leaves margins are not nearly always smooth, although a few species have wavy or slightly toothed edges [2].

I.2. Description of Eucalyptus Plant

Eucalyptus includes the tallest hardwood trees in the world. They grow rapidly and many species attain great height. The leaves are leathery and hang obliquely or vertically. The flower petals cohere to form a cap when the flower expands. The fruits are surrounded by a woody cup-shaped receptacle and contain numerous minute seeds [7].

I.3. Classification of Eucalyptus

There are about nine (9) – genera and over 600 species of Eucalyptus plants the major Eucalyptus species include: *Eucalyptus camaldulensis, Eucalyptus citriodara, Eucalyptus Heamastima, Eucalyptus odorata,* and *Eucalyptus Ciadocalyx.* They are typically grown in well – drained soil of coastal and interior low – rainfall areas [2].

I.4. Eucalyptus in Nigeria

Eucalyptus species are known to grow on all kinds of soil except poorly drained soil, which could be water - logged. However, such soil should witness an average rainfall of between 500mm – 1800mm annually [2].

Seemingly, the Savannah zone in Nigeria (Sudan, Northern and Southern Savannah) and states like Kaduna, Kebbi, Kano, Katsina and Plateau which have a mean rainfall between 400mm-1270mm are areas with the largest plantation of *Eucalyptus Camaldulensis* and other species [11].

Earlier, plantations of Eucalyptus in Nigeria were not only seen to be successful but thriving, as compared to the same species in Australia, in these Northern soils, especially Afaka plantation in Kaduna state [3]. Presently, this species are seen in places where it was probably non – existent as at the time FAO (Food and Agricultural Organisation) study, as such, the degree of its acceptability both by people and soils can be appreciated better by a fresh census figure preceding the 1973 figure which should have been updated after 10 – 15 years [3].

Nigeria is the major African country carrying out a well organized experimental programme and substantial plantries to get the best from the genus Eucalyptus, having over 7,000 hectares nationwide [3].

I.5. Conditions for Growth of Eucalyptus

Different species require different conditions for growth which include climate, soil e.t.c. Climate conditions include temperature, for *Eucalyptus camaldulensis* mean maximum temperature of hottest month is 29-32°C, the coldest month is 5 – 6 °C and, *Eucalyptus Grandis* mean maximum temperature of hottest month is 27 – 38°C, the coldest month is 4 – 6°C and Eucalyptus camaldulensis requires summer rainfall between 500mm – 1800mm and 3 months dry season [7]. Deep free dranuling soil is also required and the plant grows best on fertile loamy or clay loamy soil. But will also perform well on the lighter sandy soils provided they are of adequate depth (1m) [11]. The use of fertilizer, have shown significant increase in growth, boron is essential to reduce debarking and improve growth.

I.6. Products Obtained from Eucalyptus and their Uses

Tannin: These are polymerization products of phenolic compounds and can be extracted from the bark of eucalyptus astringent, *eucalyptus camaldulensis*. It is an acid reaction and is very astringent. It has ability to combine with certain type of proteins e.g. animal skins, to form a strong, flexible resistant insoluble substance known as leather [2]. Tanning process converts raw hides to leather. It react with salts of iron to form dark blue or greenish black compound which form basis for inks and dyes. Since they are astringent in nature they are used as medicine. It is used in clarification of wine and beer [9].

Fuel: It is known that depending on the composition, wood can serve as a very good fuel – (energy source) for domestic and industrial purpose. Eucalyptus species are no exception here, infact they are known to be used in the generation of electricity in some part of the world [9].

Timber: Eucalyptus trees are grown to serve as timbers used as house hold furniture, car parts. Because of its height, it can also be used as electric and telephone

poles. The trees are also used in making canoes. The Eucalyptus employed here include: *Eucalyptus Maculata* and *Eucalyptus citriodora* [3].

Medicine: The combination of Eucalyptus with other plants cure coughs and cold for its large content of menthol – oil (containing cineole) distilled from fresh leaves especially of Eucalyptus globules is used as antiseptic and ingredient for gargles ointment, liniment and to relieve bronchitis and asthma [9].

Essential oil: This is gotten from bark wood and leaves *of E. citriodora, E. Smithii, E. globules* and *E. robusta* [2]. The oil finds a wide application in our day – to – day life. Among the uses are in the manufacturing of perfumes and soaps. The terpene and ketone derivative in the oil are used as stain remover [2]. Piperitone is used in manufacturing synthetic thymols and menthol [9].

Papers, Plastics, Cellophane, and Carboxylic Cellulose: Many paper industries use the bark of eucalyptus species as raw material for making papers, plastics, cellophane, carboxyl methyl and cellulose. The world production of pulp from eucalyptus is over hundred million tonnes per year [2].

Viscose (Rayon Fibre), Acetate and Triacetate in Textiles: These are also gotten from processing wood pulp in the presence of caustic potash, carbon disulphate and H_2SO_4. The pulp used is sourced from *E. paniculate, E. Fastigata, E. grandis* and *E. globules* [9].

Honey: Within Australia, the greater part of honey consumed is Eucalyptus honey. Eucalyptus honey is a useful export for Australia, a total of around 17,000,000kg being exported annually [2].

Resin: These are natural or synthetic organic compound consisting of a non – crystalline or viscous liquid substance [9]. Synthetic resins are sometimes referred to as plastics.

I.7.   Oxalic Acid (Ethanedioc Acid)

Oxalic acid is a strong dicarboxylic acid found in many vegetables and other plants. It exists usually as crystal hydrate, HOO – COO. $2H_2O$, whose melting point is 101^0C and anhydrous acid, melts at 189.5^0C. Oxalic acids are found naturally as oxalates in free state [9]. Calcium oxalate is contained in all plants, while potassium acid KOO-COOH in dock, oxalates of alkaline metal dissolves in water, while calcium oxalate practically does not dissolve in cold water. When metabolism is upset in human organism, calcium oxalates accumulates, causing the formation of stones in the liver, kidneys and urinary tracts [6].

II.   Experimental Methodology

II.1.   Equipments, Apparatus, Chemicals and Materials Used

Equipments:

1.   Vibrating shake sieve

2.   Electronic weighing balance
3.   Water bath

Apparatus:

1.   Measuring cylinder
2.   Beakers
3.   Test tubes
4.   Thermometer
5.   Filter papers
6.   Burette
7.   Pipette
8.   Conical flask
9.   Funnels
10.   Mortar and pestle
11.   Retort stand
12.   Timer

Chemicals/materials used

1.   Bark of Eucalyptus camaldulensis
2.   Sulphuric acid
3.   Potassium hydroxide
4.   Distilled water
5.   Phenolphthalein

II.2.   Sample Preparation

Fresh sample of *eucalyptus camaldulensis* was obtained from the National Forestry Department of the Federal Ministry of Agricultural, Minna, Niger State, where there are large collections of eucalyptus species. The fresh bark was dried. During drying it was ensured that the constant humidity of the eucalyptus bark was obtained. This was made possible by monitoring the weight of eucalyptus bark after each drying day until a constant mass was obtained for repeat measurements.

After drying, the sample was grinded into fine particles using mortar and pestle; this is done so as to increase the surface area of the sample. The grinded sample was then sieved into different mesh size of 500pm, 250pm.

II.3.   Experimental Procedure

50g of the sieved sample of 250pm was measured into a beaker and $500cm^3$ of 3% H_2SO_4 added. The beaker was placed inside the water bath set at 60^0C and stirred continuously. The thermometer was used to monitor the temperature rise. Twenty (20) minutes from the time the temperature becomes stable at 60^0C, about $15cm^3$ of the reacting mixture was drawn for analysis; $15cm^3$ of the reacting mixture was filtered to obtain a clear filtrate. $10cm^3$ of prepared potassium hydroxide (KOH) was then reacted with $10cm^3$ of the filtrate. This is to stop the reaction between the calcium oxalate present in the bark and H_2SO_4 producing the oxalic acid (quenching). The resulting mixture was titrated with part of the prepared 3% H_2SO_4 using phenolphthalein as an indicator. The

volume of acid used was recorded. The experiment was run for 140 minutes and after each 3 minutes; samples were drawn for analysis as shown in tables.

The same procedure was used for 4% concentration and chemical equations representing the reaction and thus:

$$CaC_2O_4 + H_2SO_4 \longrightarrow CaSO_4 + C_2H_2O_4$$
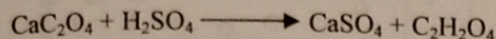
Quenching reaction:

$$2KOH + H_2SO_4 \longrightarrow K_2SO_4 + H_2O$$
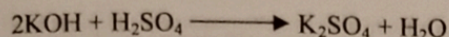
II.4. Integral Method of Analysis

Using the nth order kinetics as suggested by [8] and [12]:

$$\frac{dC_A}{dt} = kC_A^{\,n} \tag{1}$$

On integrating the above expression, it yields:

$$C_A^{1-n} - C_{AO}^{1-n} = k(n-1)t \tag{2}$$

For any fractional conversion in any time t_F, the above equation can be re-written as:

$$t_F = \frac{\left(F^{1-n} - 1\right)C_{AO}^{1-n}}{k(n-1)} \tag{3}$$

where:

$$F = \frac{C_A}{C_{AO}} \tag{4}$$

Taking logarithm of both sides of equation (3):

$$Log\,t_F = Log\left(\frac{F^{1-n}-1}{k(n-1)}\right) + (1-n)Log\,C_{AO} \tag{5}$$

This expression shows that a plot of $Log\,t_F$ against $Log\,C_{AO}$ should give a straight unit of slope $(1-n)$ and the intercept equals $Log\left(\dfrac{F^{1-n}-1}{k(n-1)}\right)$. Using fractional conversion of 0.9, the equation can be rewritten as:

$$Log\,t_F = Log\left(\frac{0.9^{1-n}-1}{k(n-1)}\right) + (1-n)Log\,C_{AO} \tag{6}$$

II.5. Differential Method of Analysis

Using n^{th} order kinetics:

$$\frac{-dC_A}{dt} = kC_A^{\,n} \tag{7}$$

Taking logarithm of both sides of the above, we have:

$$Log\left(\frac{-dC_A}{dt}\right) = Log\,k + nLog\,C_A \tag{8}$$

This expression show that a plot $Log\left(\dfrac{-dC_A}{dt}\right)$ against $Log\,C_A$ should give a straight line with a slope equals n and intercept of the graph is equal to log k.

III. Result and Discussion

The experiments were carried out under constant reaction temperature of 50°C and at dilute acid concentration of 3 and 4%. The curve of concentration against time was plotted so as to obtain the slope at various times and corresponding concentrations of acid. Also, the graphs of logarithms of various times obtained were plotted against logarithm of the initial acid concentration (i.e. log t_F against log C_{Ao}) which gave a straight line graph. Integral method of analysis was used to calculate the rate constant and reaction order using the relation described in chapter section II. Furthermore, a plot of slope $(d(-C_A)/dt)$ against C_A obtained from the curve above were also plotted against logarithm of acid concentration, this also gave a straight line graph and differential method of analysis were also used to calculate the rate constant and reaction order for both 3% and 4% acid concentration.

Figure 1 show that both the starting weight and stopping weight exhibited the same trend with time: They both decreased as the number of days increased. However, the moisture content decreased from about 1.57 to 0.186 kg and then increased to about 1.01 before finally decreasing to 0 kg.

From the results shown in Tables II and V and Figures 2 and 5, it can be observed that there is a high rate of conversion between the time interval 0 to 140 minutes, the concentration of acid decreased from (0.5633 to 0.2625) and (0.7510 to 0.3613) mol/dm^3 for 3 and 4% concentrations respectively. It can also be deduced that the high conversion rate is due to the effect of concentration and temperature. That is, the higher the concentration of acid, the faster the reaction rate, and thus the higher the temperature will be. This will trigger a faster conversion rate and vice versa.

From the analysis of the results shown in Table VIII, it can be concluded that the production of Oxalic acid from the bark of Eucalyptus Camaldulensis has different rate constants, (0.0389, 0.0446)min^{-1} and (0.0375, 0.0478)min^{-1} for 3 and 4% H$_2$SO$_4$ concentration using both Integral and differential methods respectively. This is represented in graphical form in Figures 3 and 6, 4 and

7, for integral and differential methods, respectively. The plots therefore show approximately the same value of rate constants using both methods of analysis. Figures 2 and 5 on the other hand show that concentration decreases with time.

It can be observed that an approximately the same value of order of reaction, second order of reaction is obtained for both integral and differential method of analysis.

Also by comparing the rate equations obtained from (equations (6) and (8)) which are obtained by using integral and differential methods of analysis at 3% and 4% concentration of H_2SO_4 respectively, show that the rates are higher at 4% compared to the rates at 3% concentration. It can also be observed that the rate equations can better be represented as proportional to the concentration of solvent used.

However, from Tables IV and VII, it can be observed that the gradient gets less as the reaction proceeds, also, the rate of reaction was observed to be decreasing with time with the highest rate at the initial concentration where the concentration was high and similarly, it shows that there was low reactivity as reaction proceeds and this may due to the fact that at the early stages of the reaction there are high contact between the reactants, since the frequency of collision is concentration dependent, therefore, as the reaction proceeds and the concentration of the reactant decreases, there tends to be reduction in the frequency of collision of reactants and therefore the reduction in the concentration of the reactant consumed.

However, it was shown that both the differential and Integral method of analysis provide a satisfactory method for evaluating the rate equation for the production of oxalic acid from the bark of Eucalyptus Camaldunesis in which Integral method has advantage for a few numbers of data over that of differential method which requires large numbers of data.

IV. Recommendations

1. The effect of operating variables such as particle size, reaction time has being investigated; therefore more work should be done on the effect of concentration on the rate of the reaction.
2. Also, further investigation on the effect of temperature on the rate of reaction should be carried out.
3. The time interval for this work, which was 20 minutes, should be lessened in order to obtained accurate data.
4. And the experiment should be carried out in a more controlled environmental condition to reduce the effect of external disturbances.
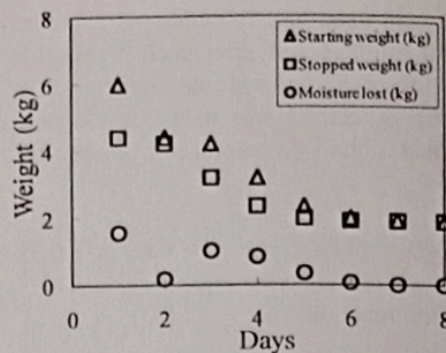
Fig. 1. Variation of starting weight, stopped weight and moisture lost with time. The figure shows the drying rate of the bark of eucalyptus camaldulensis
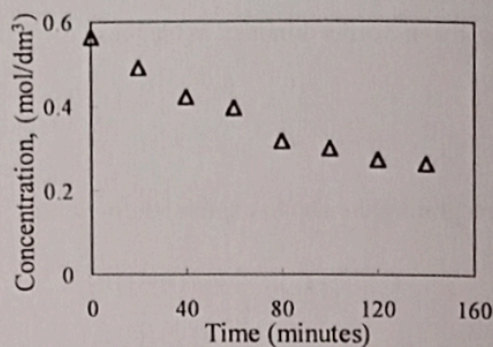
Fig. 2. Variation of concentration with time from experiment for 3% concentration at 50°C
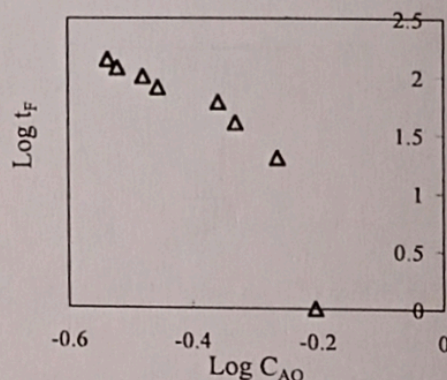
Fig. 3. A plot of Log t_F versus Log C_{AO} based on integral method of analysis for 3% concentration at 50°C
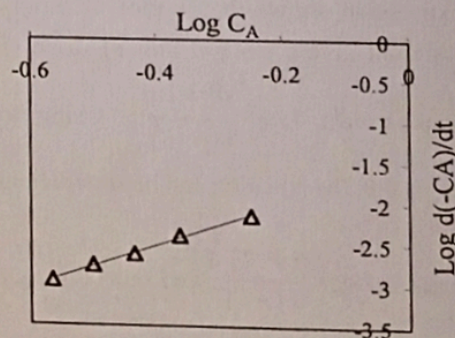
Fig. 4. A plot of Log ($-dC_A/dt$) versus Log C_A based on differentialmethod of analysis for 3% concentration at 50°C

Fig. 5. Variation of concentration with time from experiment for 4% concentration at 50°C

However, a rate equation that links the rate of reaction to the concentration of solvent was established.

Fig. 7. A plot of Log (-dC$_A$/dt) versus Log C$_A$ based on differential method of analysis for 4% concentration at 50°C

Fig. 6. A plot of Log t$_F$ versus Log C$_{AO}$ based on integral method of analysis for 4% concentration at 50°C
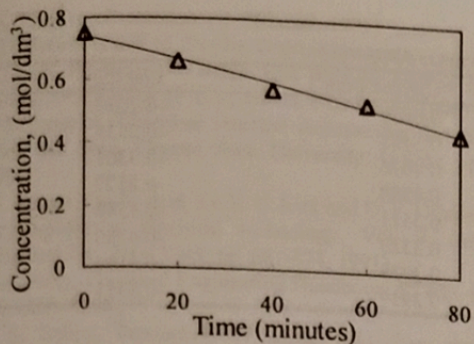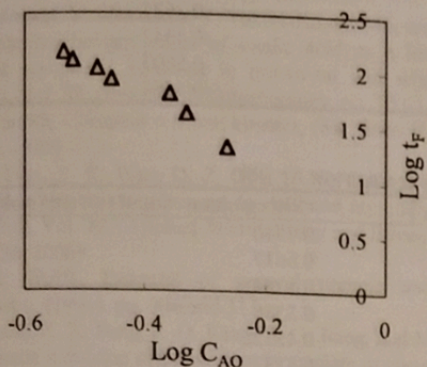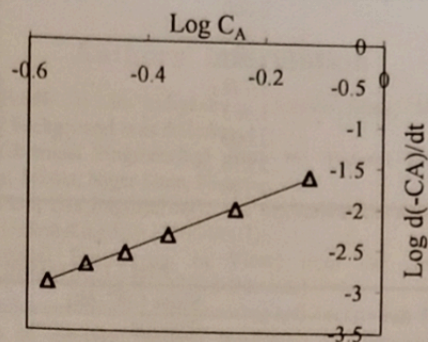
The rate of reaction was shown to be proportional to the concentration of solvent, which means that the rate of reaction was found to increased when the solvent concentration also increased. Also the rate constants were found not to be the same because different solvent concentrations were used. That is at 3% H$_2$SO$_4$ concentration, rate constant obtained were (0.0389 and 0.0375) min^{-1} for Integral and Differential method of analysis respectively. While at 4% H$_2$SO$_4$ concentration, (0.0446 and 0.0478) min^{-1} were obtained for Integral and Differential method respectively. Therefore, it can be concluded that the mechanism of production of this Oxalic acid involved one-step reaction; its rate is affected by both reactants and has second order of reaction. Also, that oxalic acid can be extracted from the bark of Eucalyptus Camaldunesis with better yield at 4% concentration of the solvent.

V. Conclusion

From the results of the experiment carried out, Oxalic acid can be obtained from the bark of Eucalyptus Camaldunesis due to the Calcium oxalate salt contained in the bark.

Appendix

Tables I to VIII present experiment results obtained from experiments.

TABLE I
DRYING RATE OF THE BARK OF EUCALYPTUS CAMALDULENSIS

Day	Time started (am)	Time stopped (pm)	Duration (hr)	Starting weight (kg)	Stopped weight (kg)	Moisture lost (kg)
1.	10.30	5.30	7	5.964	4.388	1.567
2.	10.30	5.30	7	4.388	4.202	0.186
3.	10.30	5.30	7	4.202	3.190	1.012
4.	10.30	5.30	7	3.190	2.348	0.842
5.	10.30	5.30	7	2.348	1.998	0.350
6.	10.30	5.30	7	1.998	1.911	0.087
7.	10.30	5.30	7	1.911	1.896	0.015
8.	10.30	5.30	7	1.896	1.896	0.000

TABLE II
RESULTS OBTAINED FROM THE EXPERIMENT FOR 3% CONCENTRATION AT 50°C

Time(min)	Titre value (cm^3)	Volume of Acid reacted (cm^3)	Concentration of acid reacted (Mol/dm^3)
0	0.00	0.00	0.5633
20	5.01	0.80	0.4906
40	6.55	2.04	0.4208
60	7.01	2.50	0.3949
80	8.41	3.90	0.3160
100	8.71	4.20	0.2991
120	9.19	4.68	0.2721
140	9.36	4.85	0.2625

TABLE III
RESULT OBTAINED USING INTEGRAL METHOD OF ANALYSIS

T (min)	Log (t)	C_A (mol/dm³)	C_{AO} (mol/dm³)	Log C_{AO}
0	0.000	0.5633	0.6259	-0.2035
20	1.300	0.4906	0.5451	-0.2635
40	1.602	0.4208	0.4676	-0.3301
60	1.778	0.3949	0.4388	-0.3577
80	1.903	0.3160	0.3511	-0.4546
100	2.000	0.2991	0.3323	-0.4785
120	2.074	0.2721	0.3023	-0.5196
140	2.146	0.2625	0.2917	-0.5351

TABLE IV
RESULT OBTAINED USING THE DIFFERENT METHOD OF ANALYSIS

C_A(mol/dm³)	Slope (-dC_A/dt)	Log (-dC_A/dt)	Log C_A
0.5633	0.00708	-2.15	-0.2493
0.434	0.00398	-2.40	-0.3625
0.368	0.00234	-2.63	-0.4342
0.316	0.00170	-2.77	-0.5003
0.272	0.00110	-2.96	-0.5654

TABLE V
RESULTS OBTAINED FROM EXPERIMENT FOR 4% CONCENTRATION AT 50°C

Time (min)	Titre value (cm³)	Volume of acid reacted (cm³)	Concentration of acid reacted (mol/dm³)
0	0.00	0.00	0.7510
20	5.21	0.70	0.6617
40	6.36	1.85	0.5754
60	6.85	2.34	0.5386
80	7.96	3.45	0.4552
100	8.52	4.01	0.4132
120	8.95	4.44	0.3809
140	9.21	4.70	0.3613

TABLE VI
RESULT OBTAINED USING THE INTEGRAL METHOD OF ANALYSIS

T (min)	Log (t)	C_{AO} (mol/dm³)	Log C_{AO}
20	1.300	0.5117	-0.291
40	1.602	0.4169	-0.380
60	1.778	0.3707	-0.431
80	1.903	0.3273	-0.485
100	2.000	0.3076	-0.512
120	2.079	0.3020	-0.520
140	2.146	0.2818	-0.552

TABLE VII
RESULT OBTAINED USING THE DIFFERENTIAL METHOD OF ANALYSIS

C_A(mol/dm³)	Slope (-dC_A/dt)	Log (-dC_A/dt)	Log C_A
0.7510	0.00239	-1.62	-0.1244
0.5633	0.00955	-2.02	-0.2493
0.434	0.00457	-2.34	-0.3625
0.368	0.00275	-2.56	-0.4342
0.316	0.00199	-2.70	-0.5003
0.272	0.00120	-2.92	-0.5654

TABLE VIII
THE SUMMARY OF RESULTS

Used Concentration at 50°C	Methods of Analysis	Rate Constant (min⁻¹)	Order of Reaction	Rate equation (mol/dm⁻³min⁻¹)
3% H₂SO₄	Integral	0.0389	2	$-r_A = 0.0389\, C_A^{2.21}$
	Differential	0.0375	2	$-r_A = 0.0375\, C_A^{2.22}$
4% H₂SO₄	Integral	0.0446	2	$-r_A = 0.0446\, C_A^{2.12}$
	Differential	0.0478	2	$-r_A = 0.0478\, C_A^{2.12}$

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