# ODUCTION OF OXALIC ACID FROM THE BARK OF EUCALYPTUS CAMALDULENSIS

BY

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finally, my special gratitude to Allah for the special gift of life and the wonderful miracle of my Being. Thank you God for everything in and out.

# DEDICATION

This project work is specially dedicated to my parents the two people who gave me everything they had to offer for me to be what I am, with love and to three people who, each in his own way, made me to be me and made my studies possible;

Alhaji Abubakar Danladi - Garkuwan Zazzau Suleja
Mr. P.B. Dara - Wazirin Bwari and
Alhaji Mohammed Mudi - Danmadami,

They left a very deep impression in my life.

# CERTIFICATION

I, hereby certify that I have supervised, read and approved this project work which is adequate in scope and quality for the partial fulfilment of the Award of Bachelors degree in Chemical Engineering.

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|---|---|
| ----------------------- | ----------------------- |
| PROJECT SUPERVISOR<br>(DR. K.R. ONIFADE) | HEAD OF DEPARTMENT<br>(DR.K.R. ONIFADE) |
| ----------------------- | ----------------------- |
| EXTERNAL SUPERVISOR | STUDENT'S SIGNATURE. |

# DECLARATION

This work "Production of Oxalic acid from the bark of eucalyptus camaldulensis", was carried out by me under the careful supervision of Dr. K.R. Onifade, the head of department chemical Engineering Department, Federal University of Technology, Minna. All documents and text used have been noted in the reference.

AWWAL BISALLAH.

JANUARY, 1998.

# ABSTRACT

**The** production of oxalic acid from the bark of eucalyptus camaldulensis was carried out with reaction time of 3 and 6 hours at a temperature of 50°C using 2% dilute $H_2SO_4$. The experiment indicate, that an increase in particle size of sample results in decrease in the quantity of oxalic acid produced and also an increase in the volume of the reacting solvent i.e. 2% dilute $H_2SO_4$ results into an increase in the yield of oxalic acid.

The work showed that for 250ml of solvent and reaction time of 3 hours, the smallest particle size range of sample of 0.250–0.500mm gave an oxalic acid yield of 1.476g while the largest particle size range of 1000 - 1400 mm gave a yield of 0.546g. For 500ml of reacting solvent (2% dil.$H_2SO_4$) and reaction time of 3 hours, 250-500mm particle size range sample gave an oxalic acid yeild of 2.149g. For 750ml of 2% dil. $H_2SO_4$, and reaction time of 3 hours 0.250 - 0,500mm particle size range sample gave 2.739g oxalic acid. Similar trend of result was obtained for the reaction time of 6 hours. The percentage purify of the oxalic acid obtained was found to be between 78 and 84%.

# CHAPTER ONE

## 1:0 INTRODUCTION:

The project, "Production of oxalic acid from the bark of eucalyptus camaldulensis" is basically to highlight the utilization of eucalyptus camaldulensis for the production of exalic acid. At present, the bark of eucalyptus camaldulensis is not utilized for the production of any useful substance hence the attempt to determine the uptimum condition necessary for the production of oxalic acid from the bark.

The plantation of eucalytus is scattered on a reasonably large scale in the Savanna region of Nigeria, thus, it is evident that several thousand tonnes of the bark will be available.

The bark of this tree (eucalyptus camaldulensis) contain calcium oxalate salt which reacts with sulphuric acid ($H_2SO_4$) to produce Oxalic acid. The reaction that occurs between the salt and the sulphuric acid is represented as follows:-

$$CaC_2O_4 + H_2SO_4 === Ca\ SO_4 + CaSO_4 + C_2H_2O_4$$

(calcium oxalate) (sulphuric Acid). (calcium sulphate)(oxalic acid).

The oxalic acid is used industrially and in the laboratory. A list of some major areas where oxalic acid is employed is discussed on a later page.

## 1.1 OBJECTIVE OF THE PROJECT:

Basically the objective of this experiment are:-

(1) To investigate the relationship between particle size of sample and the quantity of oxalic acid obtained.

(2) To investigate the effect of agitation time on the oxalic acid yield and purity.

(3) To find out the effect of increase in solvent (2%$H_2SO_4$)

on the yield and purity of oxalic acid

(4) To obtain a general result that wil be compared with other results already available (based on other project works) in order to determine that will give an uptimum yield and uptimum purity of oxalic acid

(5) To find the most suitable parameters to be considered in the design of a small scale plant(a pilot plant) for the production of oxalic acid from the bark of eucalyptus camaldulensis and

(6) to provide a basis for the comparison of the yield and purity of oxalic acid from eucalyptus camaldulensis and oxalic acid from other sources (e.g. wood sorrel) to find out the most economical means of production.

# CHAPTER TWO

## 2.0 LITERATURE REVIEW:

### 2.1 ORIGIN AND CHARACTERISTICS OF EUCALYPTUS:

Eucalyptus is a large genus of evergreen trees of the myrtle family (Myrtaceae) with more than 600 species and varieties. It is a native of Australia and with a few species in eastern Malaysia, Tasmania and New Zealand. They are also known as 'gum trees' and 'stringy bark tree'. Eucalyplus species have been introduced into North and South Africa, India, mediterranean countries, California (where they have become the distintive vegetation of previously treeless areas) and most other tropical and warm Countries. The most commonly planted species of Eucalyptus are Eucalyptus globulus (also called Labill) and Eucalyptus Saligna (also called Sm) which are very quick growing. The genus as a whole tolerates a wide range of climatic and soil conditions, but they are not commonly grown in very wet tropics at low altitudes, although the New Guinea and allied species may succeed under these conditions.

The Larger species of Eucalyptus are among the giants of the vegetable world. The tallest on reliable record is an Australian specimen of the Mountain ash (Eucalyptus Amygdalina regnans)in Victoria; this was about 100 meters high and measured about 8 meters round near the base. Another claimants, in the valley of styx river in southern Tasmania, stands 98 meters high. Among the Smaller eucalypts is the coolibah tree of song and legend. It grows in the scrup on the borders of the Australian deserts. The smallest of these class may be less than 2 meters tall .

The eucalypts have entire, generally lanceolate, leathery leaves, opposite in the young tress but alternate in the mature trees. in many species the leaves turn their edges towards the sun, following it across the sky to avoid too rapid evaporated of their moisture. The solitary or umbellate flowers shed part of the calyx and corolla when the blossom opens, and they owe their beauty to their long stamens which are very numerous and in some species richly coloured.

The flower petals of the eucalyptus cohere to form a cap whenthe flower expands. The clustering of eucalyptus flowers could either be recemose or cymose. In the cymose form, both the main axis and the pairs of lateral branches from the axis are terminated by flowers.

They are usually of odd numbers of flowers and the arrangement is known as dichasium. In the recemose form, there is development of flowers which are in succession on a main axis.

Each flower of the encalyptus consist of two parts Viz:- the operculum or cap and the ovary. The operculum is the upper portion which falls off along with the fixed petals to reveal the stamens inside. This portion can be off different shapes such as hemisphare-shaped, long and narrow cylindrical or cone - shaped. The filaments of the stamen also have various colours like pink, yellow, red, cream and scarlet or white coloured. The ovary is the lower portion. The fruit is a woody capsule containing very small seeds. The capsule is topped by a disc that breaks up into to seven valves, depending on the species.

Some seeds are prominently winged while others are smoothed or sculptured.

## 2.2 EUCALYPTUS IN NIGERIA:

Nigeria is a major African nation carrying out a well-organised experimental programme and substantial planting to get the best use of eucalyptus plant. It is mostly grown in the savanna region of the country. At the end of 1973 the planting of Eucalyptus totalled about 4,000 hectres at the rate of 800 hectres per year. Major areas in Nigeria that have eucalyptus plantation includes Jos in Plateau state, Kano state, Katsina State and Kaduna State.

Eucalyptus camaldulensis specie was introduced to Nigeria in 1916 (Jackson Ojo 1973) and was planted bare-rooted in the northern parts without protection against termites and it grew fairly successful.

The eucalyptus distribution among the major vegetation zones in Nigeria is given below:

### (1) THE NORTHERN GUINEA ZONE:_

In this zone, the mean annual rainfall varies from 1016mm to 1270mm, with only about three rainless months. The soil here is basically reddish brown sandy loam and the natural vegetation is affected by family activities and bush fires over a long period. Eucalyptus specie most suitable for this zone are E. Camaldulensis, E. Citriodora, E. Cloeziana E. Pilularis, E. Propingna, E. Grandis/Saligna and E. Tece-ticornis.

### (2) THE SOUTHERN GUINEA ZONE:-

This zone receives about the same amount of rain as the northern Guinea zone but the wet season is more prolonged and the dry season less severe. Many eucalyptus in this zone are found promising with eucalyptus camaldulensis being generally the most suitable, but others produce good poles more quickly on the best sites. These include E. Cloeziana, E. Propinqua, E. Punctata, E. Rubusta and E. Saligan.

(3) SUDAN ZONE:-

Climate condition in this zone are dominated by the severely dry hamattan wind from the north-east direction. The total rainfall varies between 500mm to 1,000mm but for seven consecutive months the rainfall is either nil or less than 25mm. It is common to have 275 consecutive days with out rain. On deep dry sands both E. Camaldulensis and E. Microtheca are promising.
Ferguson (1973) compiled detailed costing for the establishment of eucalyptus plantations in the savanna region aimed primarily at the production of fuel wood and poles. Cost are projected to cover the first four rotations, assuming an annual planting of 200 hectres at 3m x 3m spacing and a year cropping rotation. Overhead costs include allowance for a modest increase in provincial staff.

## 2.2 IMPORTANCE OF EUCALYPTUS SPECIES:

Eucalyptus tree is important and of great economic potential due to its various uses. It is considered as a important economic tree. In Nigeria, eucalyptus are basically grown for poles, used in Electric Power and Telecommunication lines, for fuel and as wind breakers in the far northern part of the country.

All around the world the purpose of eucalyptus cultivation varies from nation. Also the yield from countries growing eucalyptus vary from a minimum of 4km2 to a maximum

35Km2 or more per hectre per year. The yield here refers to utilisable under bark volume. For instance, in austrilia, it is cultivated mainly for commercial, environmental domestic and industrial purposes. In united state it is grown for wind break, shelter belts, fuel wood, and as a low-cost source of hardwood fibre. In Algeria, Burundi, Chad and Niger, it is cultivated for timber and shelter belt. In Argentina, Bolivia and Kenya it is grown for pipes, railway sleepers, heavy construction and for paper making

Basically, the major uses of eucalyptus specie are listed below:-

(a) TANNIN:- This is a generic term for a widely occurring group of water soluble substances which can be extracted from the bark of some eucalyptus, particularly E. Wandoo and E. astringens. The bark of E. astringens is sufficiently rish in tannin to warrant export in its crude state. Tannin is of economic importance because of their ability to unite with certain type of protein, such as those in animal skin, by converting the galatin of hides into insoluble non-putrefying materials, thus changing the hide into leather. Tannin also react with salt of iron to form dark-blue or greenish-black compound which for the basis of our common inks and dyes.

Tannin are also used because of their astrinment nature. This astringent nature makes tannin to be used mechanically in the treatment of diseases and as antidote for metallic, alkaloidal, and glycosidic poinsons, with which it forms insoluble precipitates.

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Tannin material are also used in oil drilling to reduce the viscosity of the drill without reducing the specific gravity.

(b) Essential oil:- This is another important product from eucalyptus species which distilled basically the leaves, blossoms, roots, twigs, fruit and bank of E. citriodora, E. Smithil, E. globulus and E. rubusta. the oil is colourless spicy and pungent. Among the terpene and ketone derivatives in the oil are cinoele used as stain removers and in veterinary medicine and piperitone used in the manufacture of perfumes, soaps, inhalers, nasal sprays, and as antiseptic. Another derivative is eudensmyl acetate which is used as a substitute for bergamot essence, it mixes well with lavender essence and phellandrene oil which is used in industry as a solvent and floatation for metals.

(c) TIMBER:- Eucalyptus species are important sources of timber for domestic and export purposes. A lot~~s~~ of eucalyptus species are used as hard wood in building construction, for railways sleepers, furniture manufacturing pulp wood, pegs, drawing board, telephone and electric poles.

(d) APICULTURE:- This basically is the keeping of bees for honey and flowering which is important for bee pasturage. Many tropical eucalyptus grown in plantations, including Grandis, E. dewglupta and E. Camaldulensis, flower profusely, often in most months of the year , and can yeild much In south Africa, it is estimated that the honey potential of eucalyptus is between one and four percent of its timber value.

(e) FUEL WOOD:- Eucalyptus was first introduced into several countries to supplement fuel wood supplies, and this function was fulfilled during the earlier years of their introduction. The wood of most eucalyptus burns well when air-dried and leaves little ash and relatively little smoke. Eucalyptus planatations and other forest resources can supply a substantial part of the house hold fuel needed in middle and low classes. The oil crisis in 1993 and 1994 also led to a return to wood for fuel in many household.

(f) ORNAMENTAL :- Eucalyptus are grown for their attractive form and foliage, colourful flowers or decorative bark. The scarlet - flowering gums (E. Focifolia and E. calohpylla) are two widely planted trees with brightly coloured flowers. attractive form species with decorative barks include the red iron bark (E. sederoxylon) which has black furrowed bark, the spotted gum (E. maculatal), which has smooth mottled bark in shade gray and reddish brown, and the ribbon gum (E. viminals) which has smooth white bark, that trails in long ribbons from the branches.

(g) PULP AND PAPER:- eucalyptus are used extensively in the pulp and paper industry. Plantation eucalyptus are already in use in countries like Brazil, portugal, Spain and south Africa to furnish the raw materials for the paper and pulp industry. The world production of pulp from eucalyptus species is over one million tonnes annually. All types of paper grade pulps are produced. chemical-mechanical, semi-chemical and mechanical of this one million tonnes about 3/4 are produced in Australia and Portugal.

(h) CHARCOAL :- eucalyptus provide good commercial charcoal that yield more colories per kg than the raw wood. The charcoal does not smoke and it is relatively safe to use. It is used in charcoal-iron industries which maintain their place within the overall steel industry. charcoal from eucalyptus is popular in Brazil, India, Madagascar, Spain and south Africa. The charcoal can be used to make domestic gas or as a gas fuel for motor vehicles. It is also an important industrial chemical used in pigments, fire works, gun powder, rubber production, drawing, poultry and animal feeds and cyanide carbide and disulphide manufacture.

(i) ALKALOIDS :- these are basic or alkaline chemical substances that neutralice acids. The physiological activitiy of alkaloids is important in medicine e.g. as analgesics. Rutin is a very important alkaloid obtained from the bark or leaves. The substance (Rutin) is a vitamin P-like which has therapeutic property and is used to affect the permeability of the extremities of blood veins.

(j) PIPETONE:- this is a chemical substance obtained from the seeds of eucalyptus. This substance is used in the manufacture of medicinal thymol and menthol.

(k) RESINS:- These are natural or synthetic organic compounds consisting of a non-crystalline or viscous liquid substance. Resins from eucalyptus tree (mainly E. longirostics) are used medicinally as astringent and in the treatment of diarrhea. they are also used in the production of vanishes. They are insoluble in water but soluble in alcohol, turpentine and spirit, when present in the wood, resin add to its strength and durability.

(l) OXALIC ACID:- This is a dicarboxylic acid that can be produced from the oxalate salt (calcium oxalate) present in the bark of eucalyptus tree (mainly E. camaldulensis). This project work is based on this.

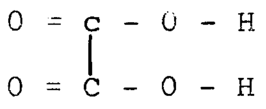
(m) OTHER USES:- Due to its height, eucalyptus species are used as shelter belt against cold, as shade trees, as sawn wood on farms. they can also be used as forest trees for wind break, for making handles for tools e.g. hammer, axe in railway construction.

# CHAPTER THREE:

## 3.0 OXALIC ACID:

This is an organic compound with the chemical formula $C_2H_2O_4$. it was first discovered by K.W. sheele in 1976 by heating sugar with nitric acid and later obtained from a plant called oxalis acetosella (wood sorrel) from which it takes its name. The acid also occurs naturally in other plants including spinach, rhubarb, tomatoes, potatoes, eucalyptus e.t.c.

Oxalic acid (also called ethanedioic acid) is the first member of the dicarboxylic acids. Its salts (oxalates) are prevalent in nature, for example, $K\ H\ C_2O_4$ in plants of the oxalis family and $CaC_2O_4$ in eucalyptus bark. The structure of the acid is

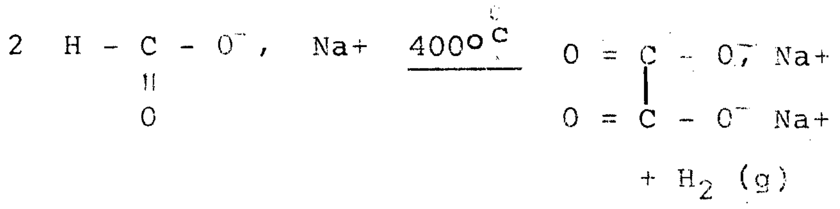


$$\begin{array}{c} O = C - O - H \\ | \\ O = C - O - H \end{array}$$

The acid exists in two forms, one as a dehydrate and the other as anhydrous. The dehydrate which melts at 101.5°C, loses water when heated at 100-105°C , and at about 200°C it decomposes into carbon dioxide, carbon monoxide, formic acid and water.

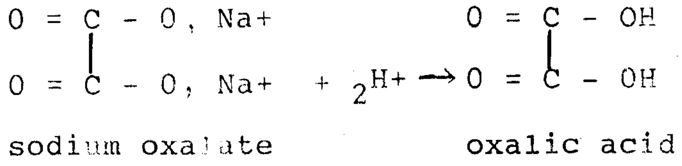
The anhydrous form is odourless, hygrosscopic and white to colourless and melts with decomposition at 189.5°C . It is obtained by careful drying of the dihydrate.

Commercially, oxalic acid is made by the thermal decomposition of sodium formate :-



$$2\ H - \underset{\overset{\|}{O}}{C} - O^-,\ Na+ \xrightarrow{400^\circ C} \begin{array}{c} O = C - O^-\ Na+ \\ | \\ O = C - O^-\ Na+ \end{array} + H_2\ (g)$$

Followed by liberation of the free acid, using $H_2SO_4$



$$\begin{array}{l} O = C - O,\ Na+ \\ \quad\ \ | \\ O = C - O,\ Na+ \end{array} + 2H+ \rightarrow \begin{array}{l} O = C - OH \\ \quad\ \ | \\ O = C - OH \end{array}$$

## 3.1 PROPERTIES OF OXALIC ACID:

### PHYSICAL PROPERTIES:

(i) The anhydrous acid is a white powder, The hydrated form consists of transparent crystals with 2 - molecules of water of crystallization.

(ii) The acid is odourless, poi sonous and soluble in water and ethanol but almost insoluble in either.

(iii) Melting point of the hydrated oxalic acid is 101.5°C while for the anhydrous is 189.5°C

(iv) the dehydrate loses water when heated to 100°C - 105°C, when hearted to 200°C, it decomposes to $CO_2$ and water.

(v) It has specific gravity of 1.9

(vi) It has solubility of 15°C.

### CHEMICAL PROPERTIES:

(i) Its chemical formula is $CO_2H - CO_2H$

(ii) It is a reducing agent. It is readily oxidised to CO2 and $H_2O$ with warm acidified solution of $KmnO_4$ i.e. $\begin{array}{l} CO_2H \\ \ \ | \\ CO_2H \end{array} \xrightarrow{Kmn\ O_4}$ $2\ CO_2 + H_2O$

(iii) It is a stronger acid than ethanoic acid but weaker than inorganic acids.

(iv) With concentrated $H_2SO_4$, it is dehydrated giving $CO_2$, CO and water

(v) It is undergoes esterification - When anhydrous oxalic acid is reflixed with excess methonol or ethanol, it yield esters with need of stronger acid as catalyst;-

$CO_2H - CO_2H + 2E \quad OH \qquad (CO_2EE)_2 + 2H_2O$

(vi) It reacts with phosphorus pentachloride and similar regents to give acid chloride e.g. oxalyl chloride

(Dip 64°C) i.e. $(CO_2H)_2 + 2 PCL_5 \quad (COCL)_2 + _2POCL_3 + 2 HCL$

(vii) On heating with concentrated H SO Oxalic acid and oxalates are dehydrated to an equi molar mixture of carbonmonoxide and dioxide:-

$(CO_2H)_2 + H_2SO_4 \quad CO + CO_2$

## 3.2 HEALTH HAZARD ASSOCIATED WITH OXALIC ACID:

Oxalic acid poisoning results from ingestion of solid or solution of the acid. There is a marked corrosion of the mouth, esophagus and stomach, with symptoms of vomiting, burning abdominal pain, collapse and sometimes convulsions. Death may follow quickly. The systemic effects are attributed to the removal by the oxalic acid of the calcium in the blood. The renal tubules become obstructed by the insoluble calcium oxalate and there is profound kidney disturbance which could result in a genetic disorder known as primary oxaluria, (resulting in kidney stone).

The inhalation of the dust or vapour of the acid may cause symptoms of Irritation of the upper respiratory tracts, gastro-intestinal disturbance, albuminuriam, gradual loss of weight, increasing weakness and nervou s system complaints. Oxalic acid has a caustic action on the skin and may cause dermatitis, a case of early gangrene of the fingers resembling that caused by phenol.

The chief effects of inhalation of the dusts or vapour are irritation of the eyes and upper respiratory tracts, ulceration of the mucous membrane of the nose and throat, epistaxis, headache, irritability and nervourness. More severe cases may show albulminuria, chronic caugh, vomiting, pain in the back and gradual emaciatiuon and weakness. The skin lesions are chracterised by cracking and fissuring of the skin and the development of slow healing ulcers. the skin may be bluish in colour, and the nails brittle and yellow.

## 2.3 SAFETY MEASURES FOR OXALIC ACID HAZARDS:

When handling oxalic acid, rubber gloves, aprons, safety boots and goggles should be used. Adequate ventilation should also be provided in raeas where oxalic acid dust fumes are present and proper respiratory devices should be worn. Prolonged skin contact with oxalic acid and oxalates should be avoided. Individua with peripheral vascular disease of the hands should avoid exposure to oxalic acid or oxalates. the antidole of oxalic acid is non-poinsonous calcium chalk that is available e.g. chalk, white wash e.t.c.

The TLV (threshold limited value) for exalic acid is 1mg/m of air.

## 3.4 USES OF OXALIC ACID:

(i) The most well-known application of oxalic acid is in automobile radiator cleaners, where it dissolves rust in the radiator without presumably dissolving the radiator at the same time. the reaction is :-

$\mathrm{Fe\ O\ (s)\ F + 6\ H_2C_2O_4\ (aq) \quad 2\ Fe_2(C_2O_4)_3^{3-}\ (aq) + 3H_2O + 6H^{+} + (aq)}$

(ii) it is used in laundry rinse water where it serves to remove rust and ink stains without damaging the fabric.

(iii) it is used in the preparation of other oxalate salts such as sodium oxalate, which is used in the manufacture of pyrotechnic blue printing ink, diethyl oxalate which is used in plastic manufacturing industries as solvent.

(iv) it is used in the laboratory as a very important reagent.

(v) The acid is also used as one of the raw materials in the production of varnishes, paints and reagents for condensation varnishes, paints and **reagents for** condensation and dehydration

(vi) it is used in textile and leather industries and as a manoglyceryl oxalate in the manufacture of allyl alcohol and formic acid.

## 3.5 REACTION OF CALCIUM OXALATE WITH DIL. $H_2SO_4$

Present in the bark of E. camaldulensis is calcium oxalate ($CaC_2O_4$) it is this oxalate which reacts with the dilute H SO to give the oxalic acid ($C_2H_2O_4$). The agitation of the medium during reaction bring about increase in the rate of reaction and also promote uniform temperature distribution within the reacting is given below:

$H_2SO_4$ + Ca $C_2O_4$ ---- $Ca_2SO_4$ + $H_2C_2O_4$

Dil, sulphuric acid+calcium oxalate→calciumsulphate+oxalic acid

# CHAPTER FOUR:

## 4.0 EXPERIMENTAL WORK:

## 4.1 APPARATUS/EQUIPMENTS USED:

(i) Mortar and pestle (ii) Electronic weighing balance (iii) sieve shaker (electronic vibrator) (iv) 1000ml volum-etric flask (v) filter papers (vi) Beaker (of various size ranges) (vii) Funnels (plastic and glass) (viii) conical Flasks (ix) Hot plate (x) Electro mantle (xi) 1000ml measuring cylinder (xii) Burette and pipette (xiii) Retort stand (xiv) Oven (xv) Refrigerator.

## 4.2 PRELIMINARY PROCESSES:

(a) Collection of bark:- This is the first stage in the experimental work. the bark is collected from the tree using a sharp marchet.

(b) Soaking on water overnight. the soaking is done to remove water soluble substance from the bark. Such substance include tannin, rutin e.t.c. After soaking the bark is sun-dried to a constant weight.

(c) Crushing:- When the bark is dried, it is then crushed using wooden pestle and mortar. Crushing is basically the reduction is size of the bark. the next proces after crushing is screening (screen analysis)

(d) Screen Analysis;- Screening is basically the separation of a mixture of various sizes of grains into two or more portions by means of screening surfa the screening surface acting as a multiple go-no-go gaugo and the final portion consisting of grains of more uniform size than those of the original mixture.

Materials that remain on a given screening surface are the oversize or plus material, meterials passing through the screening surface is the undersize or minus material, and materials passing one screening surface and retained on a subsequent surface are the intermediate materials.

The screening surface may consist of woven-wire, silk or plastic cloth, perforated or punched plate, grizly bars or wedge wires sections.

for this experiment, the crushed bark was put on the top screen and stock shaken mechanically with the aid of an electrical vibrator (or sieve shaker) for five minutes. After shaken, the samples retained on each screen was collected and weighed. The result of the screen analysis is shown on table 5.2.

### 4.3 EXPERIMENTAL PROCEDURE:

50 grammes sample of the first particle size range was weighed into 600ml beaker. 250ml 2% dilute $H_2SO_4$ was weighed separately and the two results tabulated. the acid was then mixed with the sample in the beaker. the resulting mixture was weighed and recorded. The mixture was placed on a magnetic hot plate and stirred for three hours at a temperature of 50 of 50°C.

After agitation for three hours the hot mixture was filtered into a conical flask and allowed to cool at room temperature and the weight of the first filtrate was taken and recorded. The calcium sulphate precipitate formed was thenfiltered off. The weight of the second filtrate was taken. The second filtrate was concentrated to not less than one third/its original volume at 70°C in moisture extraction oven, till the colour of the filtrate turns wine red. This was referred to as concentrated filtrate.

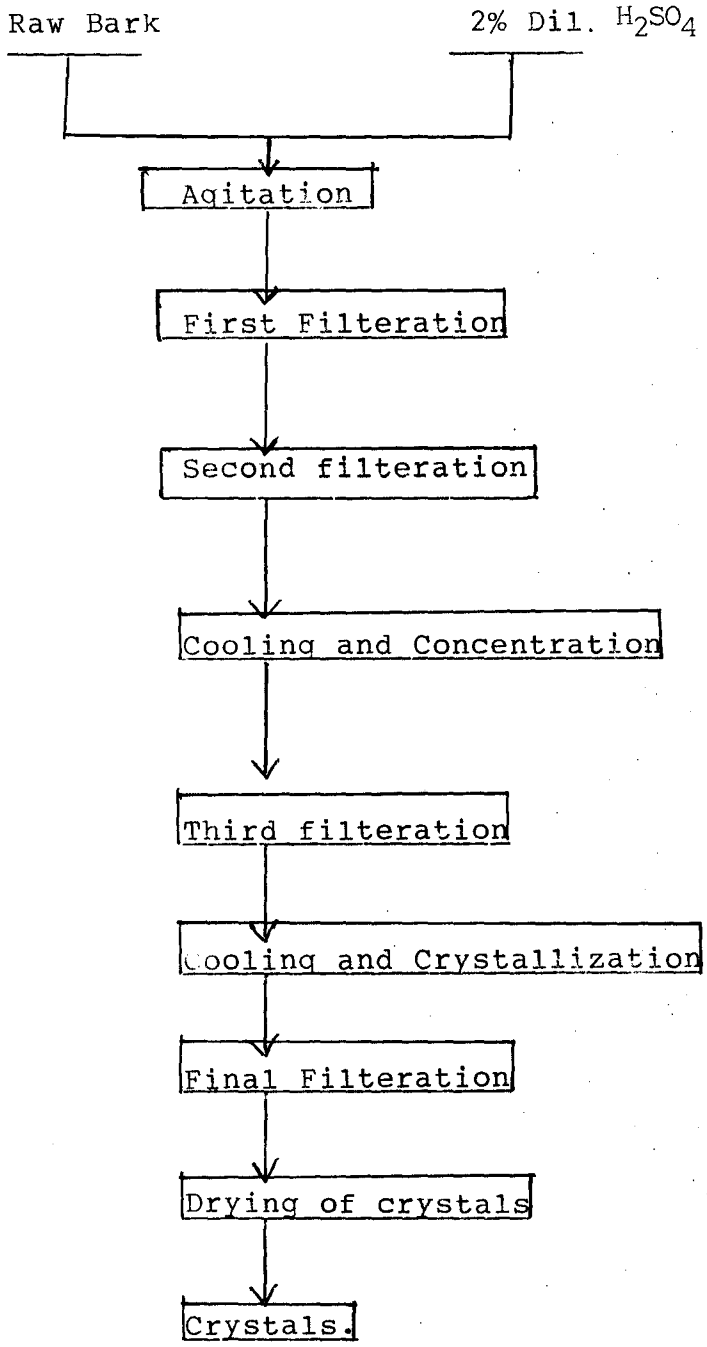
This filtrate was allowed to cool at room temperature and weighed. Another precipitation of calcium sulphate was formed The sulphate was filtered to obtain the final filtrate. the final filtrate was then weighed and kept in a refrigerator overnight for crystallization of oxalic to occur.

the supernatant liquid was separated from the crystals by filtration. The oxalic acid crystals formed was then carefully rinsed with cold water to remove traces of the solvent (i.e. $H_2SO_4$) remaining in the medium. The oxalic acid crystals obtained was kept in a moisture extraction oven in order to dry the oxalic acid.

From the same particle size range, same quantity of the sample was taken and mixed with 250ml of 2% $H_2SO_4$ and the procedure above was repeated with agitation for 6 hours (as against 3 hours for the first experiment).

Same procedure was used for the other particle size ranges. Using size range .250-.500mm, the experiment was repeated with 500ml solvent (for the first experiment) and 750ml (for the second experiment). The general flow chart for the production of oxalic acid from the bark of eucalyptus camaldulensis is shown on diagram 4.3.

## 4.3 FLOW CHART FOR THE BATCH PRODUCTION OF OXALIC ACID FROM THE BARK OF E. CAMALDU LENSIS.



## 4.4 PRODUCTION PROCESSES:

Various operational processes for the production of materials are available. The three major operational processes are:-

1. BATCH PROCESS:-

This is a process whereby some quality of feed is charged into an equipment for a period of time and after the completion of the reaction the product is discharge. There is no flow in or out there may be relative motion within the system. the batch process is an time but it is uniform throughout the reacting medium. This process is normally employed in a small scale production of materials or substances.

## 2. SEMI BATCH PROCESS:-

This process is an intermediate between the batch and continous. This occurs in different ways, Viz:-

i) The volume and composition changes with time, this is a situation whereby another reactant B is charged into a system that has reactant A already in it.

ii) The volume changes but composition is unchanged, as time increases, here both the reactant A and B are charged into the system at the same time.

iii) The volume is constant but composition chanrges, in this aspect there is both inflow and outflow.. Reactant into it and product is being formed at thesame time.

## 3. CONTINUOUS PROCESS:-

In a continuous process the composition at a point is unchanged with time. The feed is charged into the equipment and this is processed continuously. This occurs mainly in a large scale production.

the operational process employed for this project work is the batch process.

## CHAPTER FIVE

### 5.0 EXPERIMENTAL RESULTS:

The general results for the experiment are presented on the following tables:-

**TABLE 5.1 DRYING RESULT:**

| DAY | DRYING PERIOD | INITIAL WEIGHT(kg) | FINAL WEIGHT(kg) | MOISTURE. LOSS (kg) |
|---|---|---|---|---|
| 1 | 6 hours | 7.305 | 5.530 | 1.775 |
| 2. | " | 5.530 | 3.835 | 1.695 |
| 3. | " | 3.835 | 3.432 | 0.403 |
| 4 | " | 3.438 | 3.287 | 0.151 |
| 5. | " | 3.291 | 3.190 | 0.101 |
| 6. | " | 3.190 | 3.179 | 0.011 |
| 7. | " | 3.179 | 3.175 | 0.004 |
| 8. | " | 3.175 | 3.173 | 0.002 |
| 9. | " | 3.173 | 3.173 | 0.000 |

**TABLE 5.2 PARTICLE SIZE ANALYSIS:**

| sample particle size | Mass of Particle(g) | Mass fraction of particles | Commulative mass fraction. |
|---|---|---|---|
| 0·250 - ·500mm | 380 | 0.221 | 0.221 |
| 0·500 - ·710mm | 380 | 0.221 | 0.442 |
| 0·710 - ·850mm | 310 | 0.180 | 0.622 |
| 0·850 - 1·000mm | 300 | 0.174 | 0.796 |
| 1·000 - 1·400mm | 350 | 0.203 | 1.000 |

## EXPERIMENT ONE

**TABLE 5.3 PARTICLE SIZE RANGE: 250 m - 500 m**

| REACTION TIME | 3 HOURS | | 6 HOURS | |
|---|---|---|---|---|
| | WEIGHT OF SAMPLE(g) | | WEIGHT OF SAMPLE(g) | |
| | A | B | A | B |
| Mass of sample | 50 | 50 | 50 | 50 |
| Mass of acid | 256.027 | 257.121 | 256.173 | 256.382 |
| Mass of sample + acid | 306.027 | 307.121 | 306.173 | 256.382 |
| Mass of first filtrate | 107.385 | 113.434 | 109.427 | 112.651 |
| Mass of sample filtrate | 91.076 | 105.184 | 97.266 | 99.714 |
| Mass of conc.filtrate | 86.401 | 98.362 | 89.543 | 91.858 |
| Mass of final filtrate | 82.492 | 91.710 | 81.489 | 86.116 |
| Mass of oxalic acid | 1.541 | 1.410 | 0.956 | 1.535 |

## EXPERIMENT TWO

**TABLE 5.4 PARTICLE SIZE RANGE: 500 m - 710 m**

| REACTION TIME | 3 HOURS | | 6 HOURS | |
|---|---|---|---|---|
| | WEIGHT OF SAMPLE(g) | | WEIGHT OF SAMPLE (g) | |
| | A | B | A | B |
| Mass of sample | 50 | 50 | 50 | 50 |
| Mass of acid | 256.301 | 256.911 | 257.172 | 257.013 |
| Mass of sample + acid | 306.301 | 306.911 | 307.172 | 307.013 |
| Mass of first filtrate | 115.434 | 117.712 | 113.765 | 119.239 |
| Mass of second filtrate | 102.028 | 106.611 | 103.699 | 112.301 |
| Mass of conc.filtrate | 91.531 | 100.023 | 93.549 | 104.101 |
| Mass of final filtrate | 87.063 | 94.428 | 88.145 | 89.730 |
| Mass of oxalic acid | 1. 394 | 1.400 | 1.072 | 1.201 |

## EXPRIMENT THREE

TABLE 5.5 PARTICLE SIZE RANGE: 0.710mm - 0.850mm

| REACTION TIME | 3 HOURS | | 6 HOURS | |
|---|---|---|---|---|
| | WEIGHT OF SAMPLE(g) | | WEIGHT OF SAMPLE(g) | |
| | A | B | A | B |
| Mass of sample | 50 | 50 | 50 | 50 |
| Mass of acid | 256.136 | 256.011 | 256.317 | 257.812 |
| Mass of sample+acid | 306.136 | 306.011 | 306.317 | 307.812 |
| Mass of first filtrate | 119.433 | 117.219 | 112.900 | 116.751 |
| Mass of second filtrate | 110.901 | 106.173 | 100.311 | 105.633 |
| Mass of conc.filtrate(g) | 1.2751 | 100.187 | 97.215 | 97.172 |
| Mass of final filtrate | 99.171 | 91.341 | 92.367 | 90.501 |
| Mass of axalic acid | 0.984 | 0.911 | 0.851 | 0.859 |

## EXPERIMENT FOUR

TABLE 5.6 PARTICLE SIZE RANGE: 0.850mm - 1.000mm

| REACTION | 3 HOURS | | 6 HOURS | |
|---|---|---|---|---|
| | WEGIHT OF SAMPLE(g) | | WEIGHT OF SAMPLE(g) | |
| | A | B | A | B |
| Mass of sample | 50 | 50 | 50 | 50 |
| Mass of acid | 257.013 | 256.117 | 256.930 | 257.161 |
| Mass of sample+ acid | 307.013 | 306.117 | 306.930 | 307.161 |
| Mass of first filtrate | 120.175 | 118.912 | 117.313 | 113.400 |
| Mass of second filtrate | 112.611 | 109.317 | 110.202 | 107.661 |
| Mass of conc.filtrate | 107.222 | 100.277 | 98.160 | 95.173 |
| Mass of final filtrate | 93.126 | 89.133 | 93.390 | 89.240 |
| Mass of oxalic acid | 0.553 | 0.627 | 0.614 | 0.583 |

## EXPERIMENT SEVEN.

USING 750ml of 2% $H_2SO_4$

TABLE 5.9 PARTICLE SIZE RANGE: 0.250mm - 0.500mm

| REACTION | 3 HOURS | | 6 HOURS | |
|---|---|---|---|---|
| | WEIGHT OF SAMPLE(g) | | WEIGHT OF SAMPLE(g) | |
| | A | B | A | B |
| Mass of sample | 50 | 50 | 50 | 50 |
| Mass of acid | 768.288 | 765.914 | 769.012 | 765.101 |
| Mass of sample + acid | 818.288 | 815.914 | 819.012 | 815.101 |
| Mass of first filtrate | 603.767 | 601.632 | 578.493 | 580.910 |
| Mass of second filtrate | 589.688 | 585.621 | 559.947 | 563.871 |
| Mass of conc.filtrate | 567.911 | 571.001 | 545.385 | 548.177 |
| Mass of final filtrate | 548.172 | 553.097 | 539.172 | 542.631 |
| Mass of oxalic acid | 2.624 | 2.853 | 2.563 | 2.485 |

## EXPERIMENT ONE

## 5.1 RESULTS ANALYSIS:

USING 250ml of 2% $H_2SO_4$

TABLE 6.0 EXPERIMENT ONE:

PARTICLE SIZE RANGE 0.250mm - 0.500mm

| REACTION TIME | 3 HOURS | | 6 HOURS | |
|---|---|---|---|---|
| | A | B | A | B |
| Mass of sample used(g) | 50 | 50 | 50 | 50 |
| Mass of oxalic acid obt(g) | 1.541 | 1.410 | 0.956 | 1.535 |
| percentage yield of O.acid | 3.082 | 2.820 | 1.912 | 3.070 |
| Conc.of impure oxalic acid | 0.5922 | 0.5899 | 0.5701 | 0.5699 |
| Conc.of pure O. acid($g/dm^3$) | 0.7056 | 0.7056 | 0.7056 | 0.7056 |
| Percentagepurity of O.acid | 83.93 | 83.60 | 80.80 | 80.77 |

## EXPERIMENT TWO

TABLE 6.1

PARTICLE RANGE : 0.500mm - 0.710mm

| REACTION TIME | 3 HOURS | | 6 HOURS | |
|---|---|---|---|---|
| | A | B | A | B |
| Mass of sample used(g) | 50 | 50 | 50 | 50 |
| Mass of oxalic acid (g) | 1.394 | 1.400 | 1.072 | 1.201 |
| Percentage yield of O. acid(%) | 2.788 | 2.800 | 2.144 | 2.402 |
| Conc.of impure O.acid (g/dm$^3$) | 0.593 | 0.5213 | 0.5471 | 0.5800 |
| Conc. of pure acid (g/dm$^3$) | 0.7056 | 0.7056 | 0.7056 | 0.7056 |
| Percentage purity of O acid(%) | 84.06 | 73.88 | 77.54 | 82.20 |

**TABLE 6.2** EXPERIMENT THREE

PARTICLE SIZE RANGE: 0.710mm - 0.850mm

| REACTION TIME | 3 HOURS | | 6 HOURS | |
|---|---|---|---|---|
| | A | B | A | B |
| Mass of sample (g) | 50 | 50 | 50 | 50 |
| Mass of oxalic acid obtained(g) | 0.9840 | 0.9110 | 0.851 | 0.859 |
| percentage yield of O acid (%) | 1.968 | 1.822 | 1.702 | 1.718 |
| Conc.of impure O.acid (g/dm$^3$) | 0.5798 | 0.5970 | 0.5810 | 0.5800 |
| Con .of pure O acid (g/dm$^3$) | 0.7056 | 0.7056 | 0.7056 | 0.7056 |
| Percentage purity of O.acid(%) | 82.17 | 84.61 | 82.34 | 82.20 |

## TABLE 6.3 EXPERIMENT FOUR:

PARTICLE SIZE RANGE: 0.850mm - 1.000mm

| REACTION TIME | 3 HOURS | | 6 HOURS | |
|---|---|---|---|---|
| | A | B | A | B |
| Mass of sample used(g) | 50 | 50 | 50 | 50 |
| Mass of oxalic acid(%) | 1.106 | 1.254 | 1.228 | 1.166 |
| Mass of oxalic acid obtained(g) | 0.5530 | 0.6270 | 0.6140 | 0.5830 |
| Conc. of impure acid(g/dm$^3$) | 0.5861 | 0.5797 | 0.6591 | 0.5900 |
| Conc.of pure oxalic acid(g/dm$^3$) | 0.7056 | 0.7056 | 0.7056 | 0.7056 |
| Percenatage purity of O.acid(%) | 83.06 | 82.16 | 80.65 | 83.62 |

## TABLE 6.4 EXPERIMENT FIVE:

PARTICLE SIZE RANGE: 1.000 m - 1.400 m

| REACTION TIME | 3 HOURS | | 6 HOURS | |
|---|---|---|---|---|
| | A | B | A | B |
| Mass of sample used (g) | 50 | 50 | 50 | 50 |
| Mass of oxalic acid obtained(g) | 0.4910 | 0.6010 | 0.5930 | 0.2000 |
| Percentage yield of O.acid (%) | 0.9820 | 1.2032 | 1.1860 | 0.4000 |
| Conc.of umpure O. acid(g/dm ) | 0.5916 | 0.5910 | 0.5799 | 0.5887 |
| Conc.of pure oxalic acid(g/dm ) | 0.7056 | 0.7056 | 0.7056 | 0.7056 |
| Percentage purity of O. acid obtained (%) | 83.70 | 83.76 | 82.19 | 83.43 |

Using 500ml of 2% $H_2SO_4$

**TABLE 6.5** EXPERIMENT SIX

PARTICLE SIZE RANGE: 0.250mm - 0.500mm

| REACTION TIME | 3 HOURS | | 6 HOURS | |
|---|---|---|---|---|
| RUN | A | B | A | B |
| Mass of sample used(g) | 50 | 50 | 50 | 50 |
| Mass of oxalic acid obtained | 2.3100 | 1.9870 | 1.9870 | 2.0170 |
| Percentage yield of O.acid obt (%) | 4.6200 | 3.9740 | 3.9740 | 4.0340 |
| Conc.of inpure O.acid obt(g/dm ) | 0.5922 | 0.5973 | 0.5931 | 0.5909 |
| conc. | 0.7056 | 0.7056 | 0.7056 | 0.7056 |
| Percentage purity of impure O. acid (%) | 83.93 | 84.65 | 84.06 | 83.74 |

**TABLE 6.6** **EXPERIMENT SEVEN**

**Using** 750ml of 2% $H_2SO_4$

PARTICLE SIZE RANGE: 0.250mm - 0.500mm

| REACTION TIME | 3 HOURS | | 6 HOURS | |
|---|---|---|---|---|
| RUN | A | B | A | B |
| Mass of sample used (g) | 50 | 50 | 50 | 50 |
| Mass of O.acid obtained(g) | 2.6240 | 2.8530 | 2.5630 | 2.4850 |
| Percentage yield of O.acid obtained (%) | 5.248 | 5.706 | 5.126 | 4.970 |
| conc.of impure O.acid obtained (g/dm ). | 0.5861 | 0.5851 | 0.5900 | 0.5970 |
| Conc.of pure acid(g/gm ) | 0.7056 | 0.7056 | 0.7056 | 0.7056 |
| Percentage purity | 83.06 | 83.14 | 83.62 | 84.61 |

## 5. 2 TABLE OF AVERAGE VALUES:

TABLE 6.7: Using 250ml of 2% $H_2SO_4$

| Sample particle size (mm) | Average weight of oxalic acid obt(g) | | Average percentage yield (%) | | Average percentage. purity (%) | |
|---|---|---|---|---|---|---|
| REACTION TIME | 3 | 6 | 3 | 6 | 3 | 6 |
| 0.250-0.500 | .1.476 | 1.246 | 2.951 | 2.491 | 83.77 | 80.79 |
| 0.500-0.710 | 1.137 | 1.397 | 2.794 | 2.273 | 78.97 | 79.87 |
| 0.710-0.850 | 0.948 | 0.855 | 1.895 | 1.710 | 83.39 | 82.27 |
| 0.850-1.000 | 0.590 | 0.599 | 1.180 | 1.197 | 82.61 | 82.14 |
| 1.000-1.400 | 0.546 | 0.397 | 1.093 | 0.793 | 83.73 | 82.81 |

Using 750ml of 2% $H_2SO_4$

| | | | | | | |
|---|---|---|---|---|---|---|
| 0.250-0.500 | 2.739 | 2.524 | 5.477 | 5.048 | 83.10 | 84.12 |

Using 500ml of 2% $H_2SO_4$

| | | | | | | |
|---|---|---|---|---|---|---|
| 0.250-0.500 | 2.149 | 2.002 | 4.297 | 4.004 | 84.29 | 83.90 |

Figure 1·3

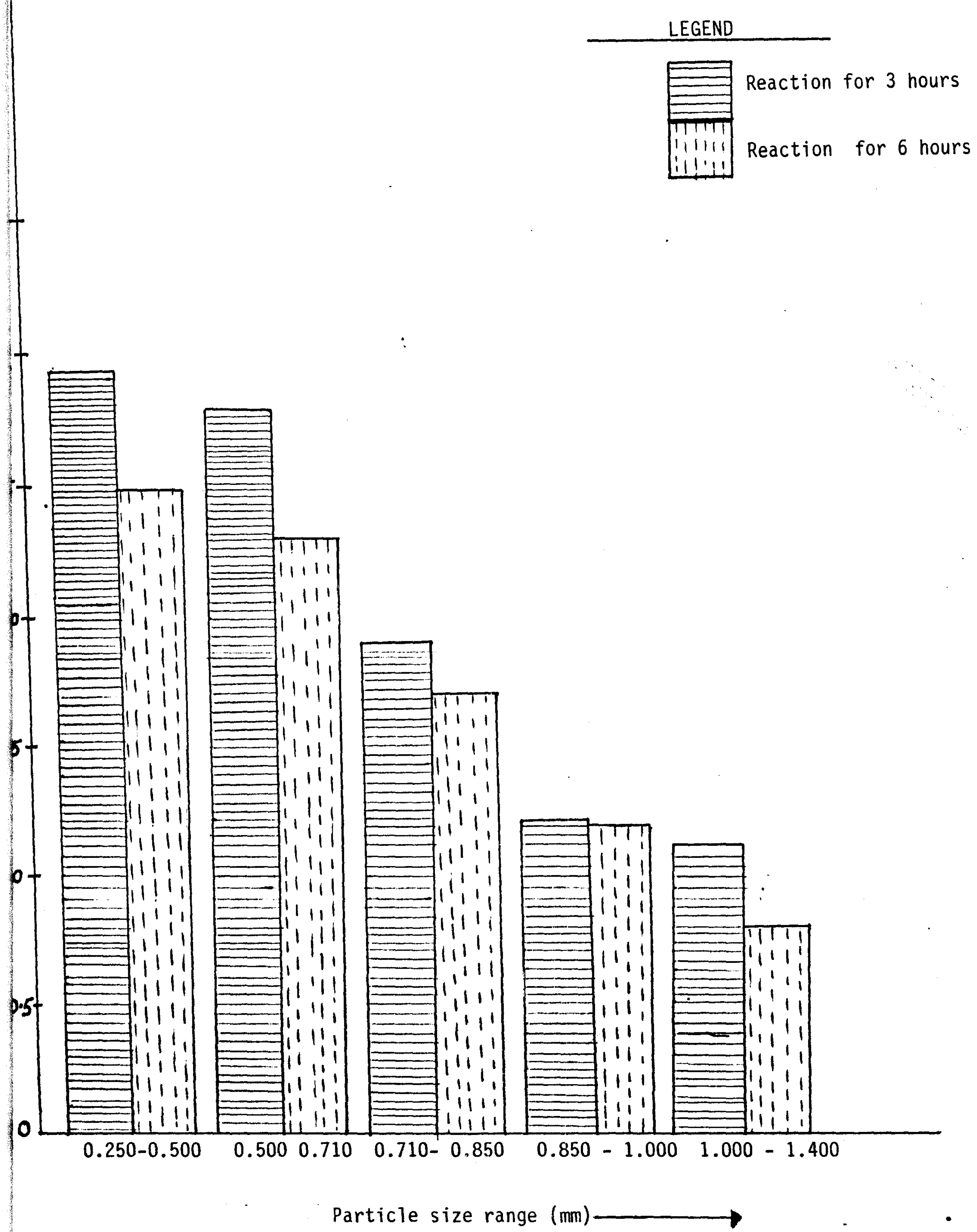


**Fig.1·3:** Bar chart for oxalic acid average yield (%) versus particle size range (mm) using 250ml 2% SO (tetra oxo sulphate (vi) acid)

FIGURE 1·4

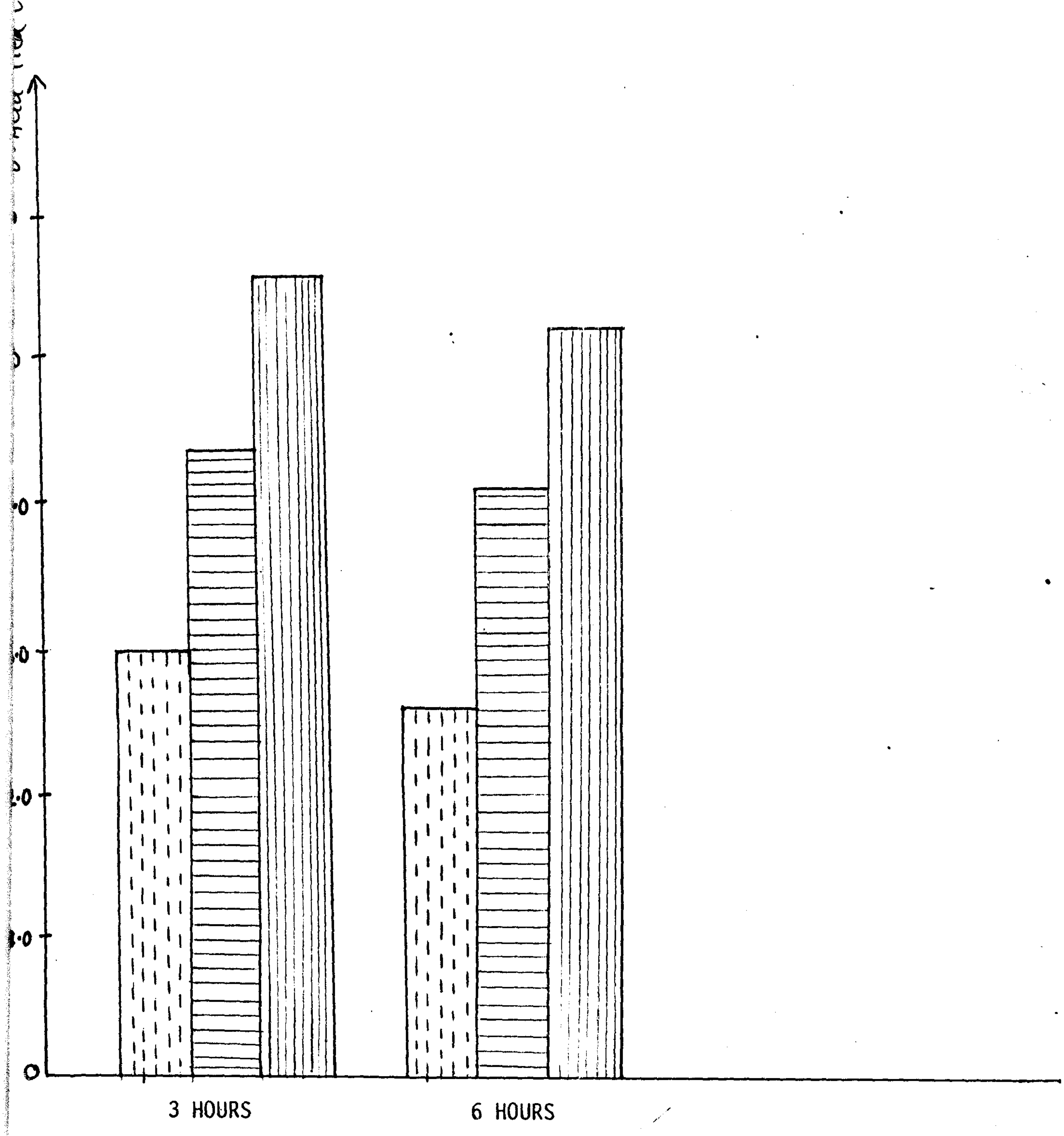


**Fig 1·4:** Bar chart for average percentage yield versus time using results of particle size range 0.250 - 0.500 mm.

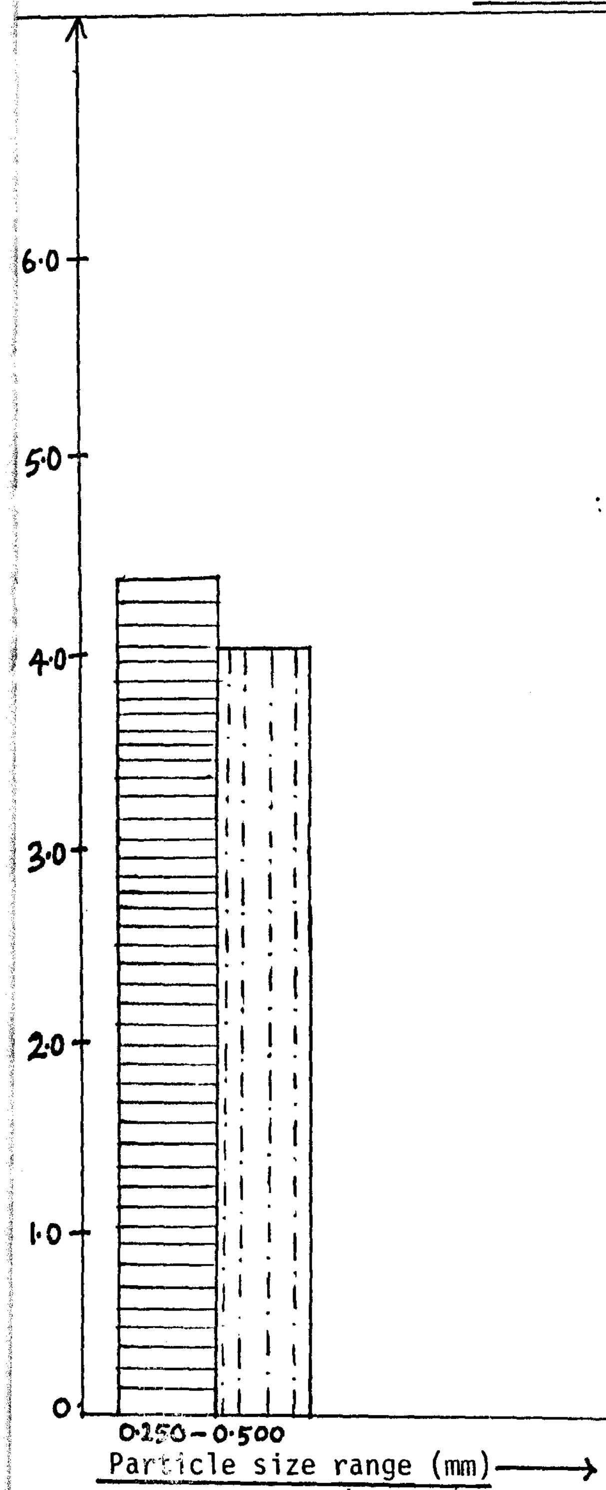
LEGEND

⁞ Using 250 ml 2% $H_2SO_4$

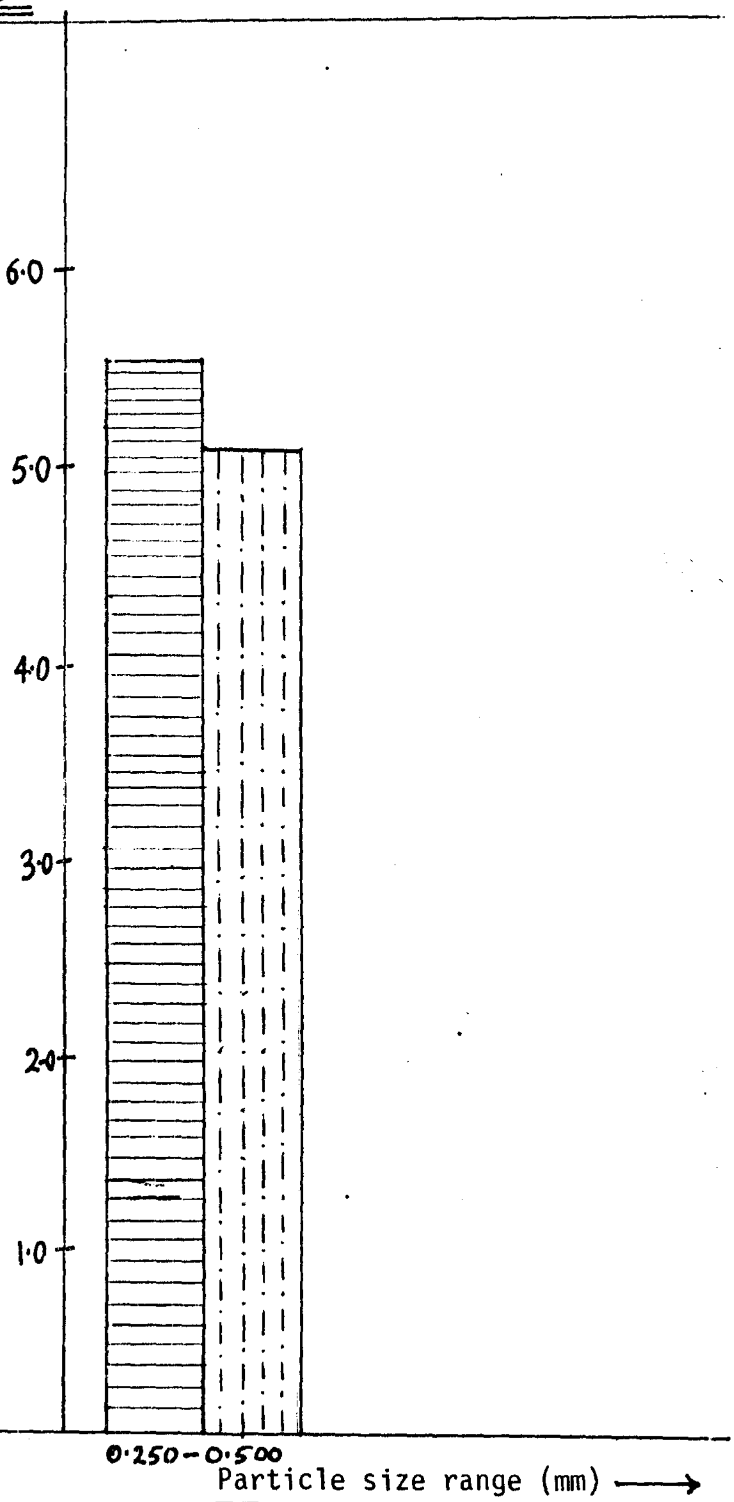
☰ Using 500 ml 2% $H_2SO_4$

▥ Using 750 ml 2% $H_2SO_4$

FIGURE 1·5



ig 1·5: Bar chart of average yield Versus particle size range using 500ml of 2% $H_2 SO_4$



Bar chart of average yield versus particle size range using 750 ml of 2 % $H_2SO_4$

LEGEND

▤ REACTION TIME OF 3 HOURS

▥ REACTION TIME OF 6 HOURS

# CHAPTER SIX

## DISCUSSION OF RESULT:AND CONCLUSION:

### 6.0 DISCUSSION OF RESULT:

The average amount of oxalic acid produced for each run is as given in table 6.7 The table shows that for increase in particle size of sample there was a decrease in the amount of oxalic acid produced. This may be due to the larger interfacial area of reaction provided by the smaller particle size sample as against the smaller interfacial area of reaction provided by the larger particle size sample. From table 6.7 with 250ml of solvent and reaction time of 3 hours, 0.250 - 0.500mm particle size sample yielded 1.476g of oxalic acid while 0.500 - 0.710mm, 0.710-0.850mm, 0.850-1.00mm and 1.000-1.400mm particle size samples yield 1.397g, 0.948g 0.590g and 0.546g respectively. The same pattern of result was obtained for reaction time of 6 hours. Fig 1.3 indicated that oxalic acid yield for the reaction time of 3 hours was more than that for the reaction time of 6 hours. This relation is not enough to establish a general relationship between oxalic acid yield and reaction time. The reason for the lower oxalic acid yield for the reaction time. The reason for the lower oxalic acid yield for the reaction time of 6 hours may be due to more evaporation of the reacting solvent (2%$H_2SO_4$) than when the reaction time was 3 hours.

Fig 1.4 showed an increasein oxalic acid yield with an increase in the volume of solvent for a given sample. Table 6.7 indicate that for particle size range 0.250 - 0.500mm and reaction time of 3 hours, when 250ml of 2% $H_2SO_4$was used 1.476g of oxalic acid was produced. Whenthe volume of the solvent ($H_2SO_4$) was increased to 500ml and 750ml, then quantity of oxalic acid produced was 2.149g and 2.739g respectively. this showed an increase by about 50% of oxalic acid obtained for each 250ml increase in volume of 2% $H_2SO_4$ with reaction time of 3 hours. Similar trend of result was obtained for the reaction time of 6 hours.

It will be observed from table 6.7 that there was no clear relationship between percentage purity of oxalic acid obtained and sample particle size and also between percentage purity and volume of solvent.

From correlation and regression analysis (appendix 2) it was observed that the correlation coefficient for the relations was approximately + 1. This implied a strong linear relation between the analysed variables. That shows that an increase in volume of solvent results in increase in quantity of oxalic acid obtained for the reaction time of 3 hours and 6 hours. (as shown on fig. 1.4 appendix 2).

## 6.1 CONCLUSION:

From the general analysis of the results obtained, it can be concluded that oxalic acid obtained is dependent used. The smaller the particle size of sample the higher the oxalic acid yield and the more volume of 2% $H_2SO_4$ used the more oxalic acid produced. For 250ml of 2% $H_2SO_4$ and reaction time of 3 hours, the oxalic acid obtained for the smallest particle size of 0.250-0.500mm was 2.95% while the largest particle size range of 1000 - 1400mm gave a yield of 1.093% oxalic acid. Similar pattern of result was obtained for reaction time of 6 hours.

With 500ml of 2% $H_2SO_4$and reaction time of 3 hours, the oxalic acid yield for 0.250-0.500mm was 4.297% while when, the volume of solvent was increased to 750ml the oxalic acid yield was 5.477% similar trend of result was obtained with reaction time of 6 hours.
Generally, the results indicated that oxalic yield for a reaction time of 3 hours was higher than for the reaction time of 6 hours. The percentage purity of oxalic acid obtained was found to be between 78-84%. No clear relationship between percentage purity

of oxalic acid obtained and particle size of sample or volume of solvent was established.

The correlation and regression analysis of result indicated a strong positive linear relationship between quantity of oxalic acid produced and volume of solvent used. The correlation coefficient (r) was found to be approximately + 1.

## 6.2; RECOMMENDATIONS:

(1) Equipment/materials for the experiment should be made readily available as inavailabily may cause in accuracy of results of the experiment.

(2) Investigation of the rate of crystallization should be carried out to determine the uptimum temperature for crystalliuzation.

(3) Work on the purification of oxalic acid obtained from the bark of pucalyptus camaldulensis should be carried out.

(4) Comparison of the purity of oxalic acid obtained from the bark of eucalyptus camaldulensis and that obtained from other sources should be carried out.

(5) Relationship between temperature of agitation and oxalic acid yield should be investigated.

(6) Investigation on the effect of the quantity of sample of eucalyptus camaldulensis bark and the percentage yield should be carried out.

(7) More work on the effect of solvent concentration to precentage yield of eucalyptus camadulensis should be done.

(8) More work on the reaction time should be carried out in order to determine a clear relationship between the reaction time and oxalic acid yield from the bark of eucalyptus camaldulensis.

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# APPENDIX ONE.

Determination of percenatge purity and percentage yield of oxalic acid.

Data available for oxalic acid.

molecular weight = 126.00

(a) Sample calculation for percentage yield using result of experiment 1 (table 6.0) for size 250 - 500 m.

the mass of sample used = 50g

mass of oxalic acid obtained are;-

for 3 hours - 1.5441g and 1.410g

for 6 hours - 0.956g and 1.535g

To calculate the percentage yield we use the expression;

$$\text{percentage yield} = \frac{\text{mass of oxalic acid obtained}}{\text{mass of sample used}} \times 100\%$$

;. percentage yield when mass of oxalic acid obtained is 1.541g is

$$= \frac{1.541}{50} \times 100\% = 3.082\%$$

When mass of oxalic acid obtained is 1.410g the percentage yield is

$$= \frac{1.410}{50} \times 100\% = 2.82\%$$

The results above are for agitation periodof 3 hours. The average percentage yield for the above is

$$= \frac{3.082 + 2.820}{2} = 2.951\%$$

The same procedureis followed for agitation periodof 6 hours and for other experiments and results tabulated as shown on the results analysissection.

(b) Determination of percentage purify from experiment 1.

(i) the equation for the neutralization reaction of oxalic acid sodium hydroxide solution of 0.01 M concentrate is represented by the following equation:-

$NaOH + H_2C\ O_4 \text{ -- } Na\ HC_2O_4 + H_2O$

(ii) To prepare solution of oxalic acid: 0.16g each of pure and impure oxalic acid were dissolved in 100ml of distilled water.

(iii) Titration is carried out using the prepared solution Todetermine the molar of the pure oxalic acid we use the expression.

$\frac{MA\ VA}{MBVB} = \frac{B}{A}$ where

Va = volume of acid used = 10.00ml

Ma = molarity of acid ?

Mb = " " base = 0.01M

Vb = volume of base used = 5.60ml

Na = mole ratioof acid = 1 ? from equation

Nb = " " base = 1 of reaction

$\frac{B}{A} = 1$ ;. MAVA = MBVB

$===MA = \frac{MB\ VB}{VA} = \frac{5.6 \times 0.01}{10.0} = 0.0056\ M$

Concentration of the pure acid.

== Molarity of acid x molecular weight

== 0.0056 x 126.0

==0.7056g 1dm

for the impure oxalic acid (one obtained)

MA= ? ; MB = 0.01M, Va = 10.00ml; VB = 4.70ml

;. MAVA = MB VB

$MA + \frac{MBVB}{VA} = \frac{0.01 \times 4.70ml}{10.0} = 0.0047M$

Concentration of impure acid

= molarity of impure acid x mol.weight

$= 0.0047 \times 126.0 = 0.592g/dm^3$

From the two concentrations above,

the percentage purity of the impure oxalic acid

$$= \frac{\text{concentration of impure oxalic acid}}{\text{Centration of pure oxalic acid}} \times 100\%$$

$$= \frac{0.5920}{0.7056} \times 100\% = 83.93\%$$

The same procedure is used to obtain percentage purities of other oxalic acids obtained from the other experiments.

## APPENDIX TWO

## OXALIC ACID YIELD VERSUS VOLUME OF ANALYSIS FOR REACTION TIME OF THREE (3) HOURS.

| $n$ | x | y | $x^2$ | xy |
|---|---|---|---|---|
| 1. | 250 | 1.476 | 64500 | 369.00 |
| 2 | 500 | 2.149 | 25000 | 1074.50 |
| 3 | 750 | 2.739 | 562500 | 2054.25 |
| | 1500 | 6.364 | 875000 | 3497.75 |

$$\bar{X} = \frac{\Sigma x}{n} = \frac{1500}{3} = 500$$

$$\bar{y} = \frac{\Sigma Y}{n} = \frac{6.364}{3} = 2.1213$$

In making a prediction of y from x i.e. oxalic acid yield from quantity (volume) of solvent. We first calculate the slope of the regression line (b) and the valve of y when x is zero i.e. 'a'.

the regression equation is $\hat{y} = bx + a$ where

$\hat{y}$ = predicted valve of y

a = valve from which y is predicted.

$$b = \frac{\Sigma xy - \Sigma x \Sigma y / n}{\Sigma x^2 - (\Sigma x)^2 / n}$$ where $n$ = number of variable = 3

from the table above,

$$b = \frac{3479.75 - 1500 \times 6.364/3}{875000 - (1500)^2/3} = \frac{3497.75 - 3182}{875000 - 750000}$$

$$= \frac{315 . 75}{125000}$$

$$\therefore b = 0.002526$$

To calculate 'a' we use the expression $a = \bar{y} - b\bar{x}$

where $\bar{y} = 2.1213$, $\bar{x} = 500$ and $b = 0.002526$

$\therefore a = 2.1213 - 0.002526\ (500)$

$= 2.1213 - 1.263 = 0.8583$

Hence the regression equation becomes:

$$y = 0.002526\ x + 0.8583$$

We then substitute in the value of x into the equation to obtain the table for the estimated point of dependence y

| $x$ | $y$ | $\bar{y}$ | $\hat{y}$ | $\hat{y}-\bar{y}$ | $(\hat{y}-\bar{y})^2$ | $y-\bar{y}$ | $(y-\bar{y})^2$ |
|---|---|---|---|---|---|---|---|
| 250 | 1.476 | 2.1213 | 1.4898 | -0.6315 | 0.39879 | 0.6453 | 0.41641 |
| 500 | 2.149 | 2.1213 | 2.1213 | 0 | 0 | 0.0277 | 0.00077 |
| 750 | 2.739 | 2.1213 | 2.7528 | 0.6315 | 0.39879 | 0.6177 | 0.38155 |
| $\Sigma$ 1500 | | | | | 0.79758 | | 0.79873 |

from the table above, the coefficienct of determination is

$$r^2 = \frac{\Sigma(\hat{y}-\bar{y})^2}{\Sigma(y-\bar{y})^2} = \frac{0.79758}{0.79873} = 0.99856$$

$\therefore$ the correlation coefficient (r) is

$$r = + \sqrt{0.99856} = \pm\ 0.99928$$

$$r \simeq \pm\ 1.0$$

Since the regression coefficient is positive then it implies that $r \simeq 1.0$ which shows that the correlation of the yield versus volume of solvent is a linear one.

## OXALIC ACID YIELD VERSUS VOLUME OF SOLVENT ANALYSIS FOR REACTION OF SIX (6) HOURS:

| | x | y | $x^2$ | xy |
|---|---|---|---|---|
| 1 | 250 | 1.246 | 62500 | 311.50 |
| 2 | 500 | 2.002 | 62500 | 311.50 |
| 3 | 750 | 2.524 | 562500 | 1893.00 |
| Σ | 1500 | 5.772 | 875000 | 3205.50 |

$$\hat{x} = \frac{\sum x}{n} = \frac{1500}{3} = 500$$

$$\hat{y} = \quad = \frac{5.772}{3} = 1.924$$

From the regression equation $\hat{y} = bx + a$

$$b = \frac{\sum xy - \sum x \sum y/n}{\sum x^2 - (\sum x)^2/n} \quad \text{where } n = 3$$

$$= \frac{3205.50 - 1500 \times 5.772/3}{875000 - (1500)^2/3} \qquad = \frac{3205.5 - 2886}{875000 - 75000}$$

$$= \frac{319.5}{125000} = 0.002556$$

$\therefore$ b = 0.002556

we calculate 'a' from

$a = \bar{y} - b\bar{x}$ where $\bar{y} = 1.924$ and $\bar{x} = 500$

$\therefore$ a = 1.924 − 0.002556 (500)

= 1.924 − 1.278 = 0.646

This implies that the regression equation becomes

$\hat{y} = 0.002556\,x + 0.646$

| $x$ | $y$ | $\bar{y}$ | $\hat{y}$ | $\hat{y}-\bar{y}$ | $(\hat{y}-\bar{y})^2$ | $y-\bar{y}$ | $(y-\bar{y})^2$ |
|---|---|---|---|---|---|---|---|
| 250 | 1.246 | 1.924 | 1.285 | 0.639 | 0.40832 | -0.678 | 0.45968 |
| 500 | 2.002 | 1.924 | 1.924 | 0 | 0. | 0.078 | 0.00608 |
| 750 | 2.524 | 1.924 | 2.563 | 0.639 | 0.40832 | 0.600 | 0.36000 |
| 1500 | 5.772 | | | | 0.81664 | | 0.82576 |

From the table above, the coefficient of determination

$$r = \frac{\Sigma(\hat{y}-\bar{y})^2}{\Sigma(y-\bar{y})^2} = \frac{0.81664}{0.82576} = 0.988956$$

$\therefore$ the correlation coefficient r is

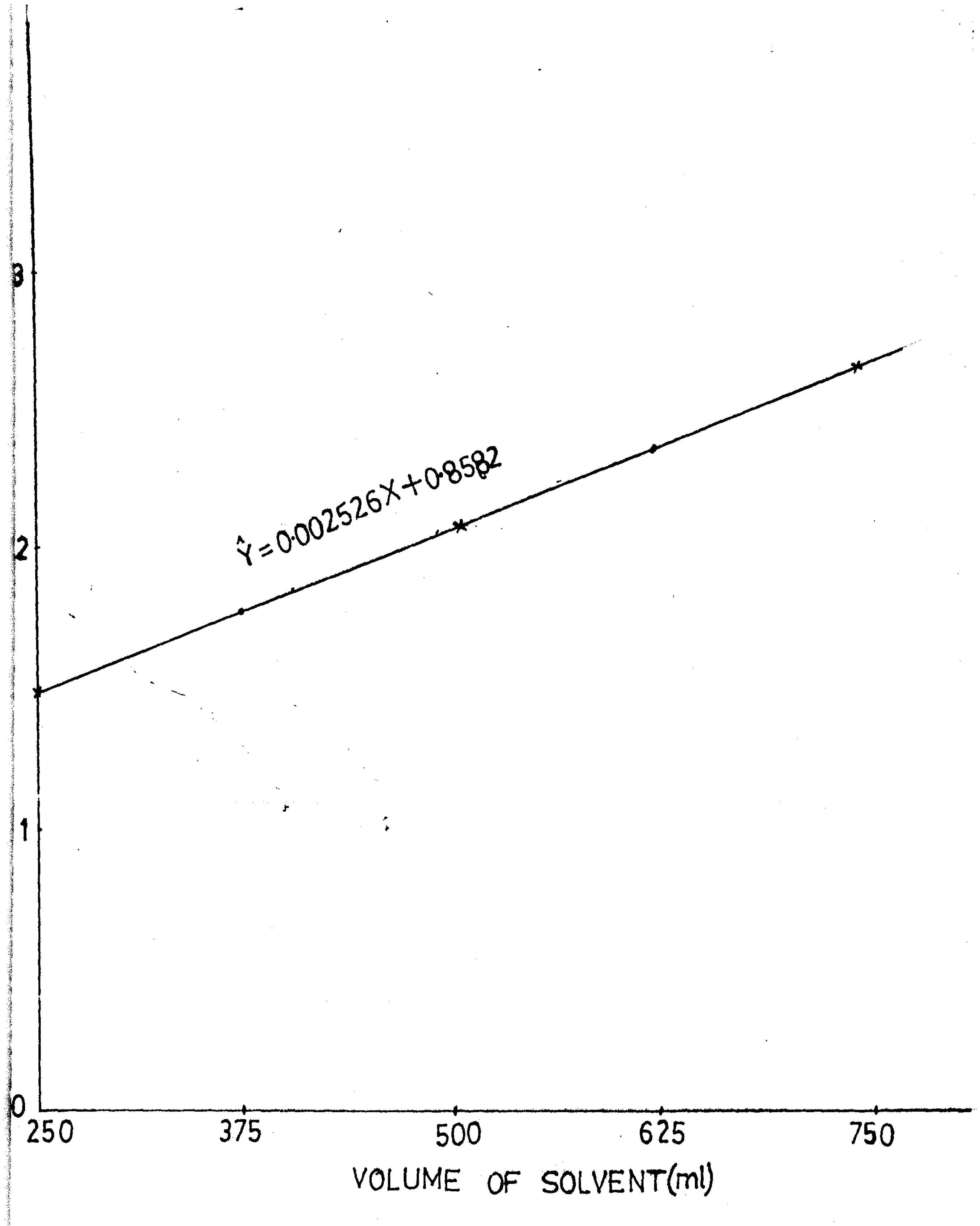
$$r = + \sqrt{0.988956} = \pm 0.994463$$

$$r \simeq \pm 1$$

Since regression coefficient is positive then

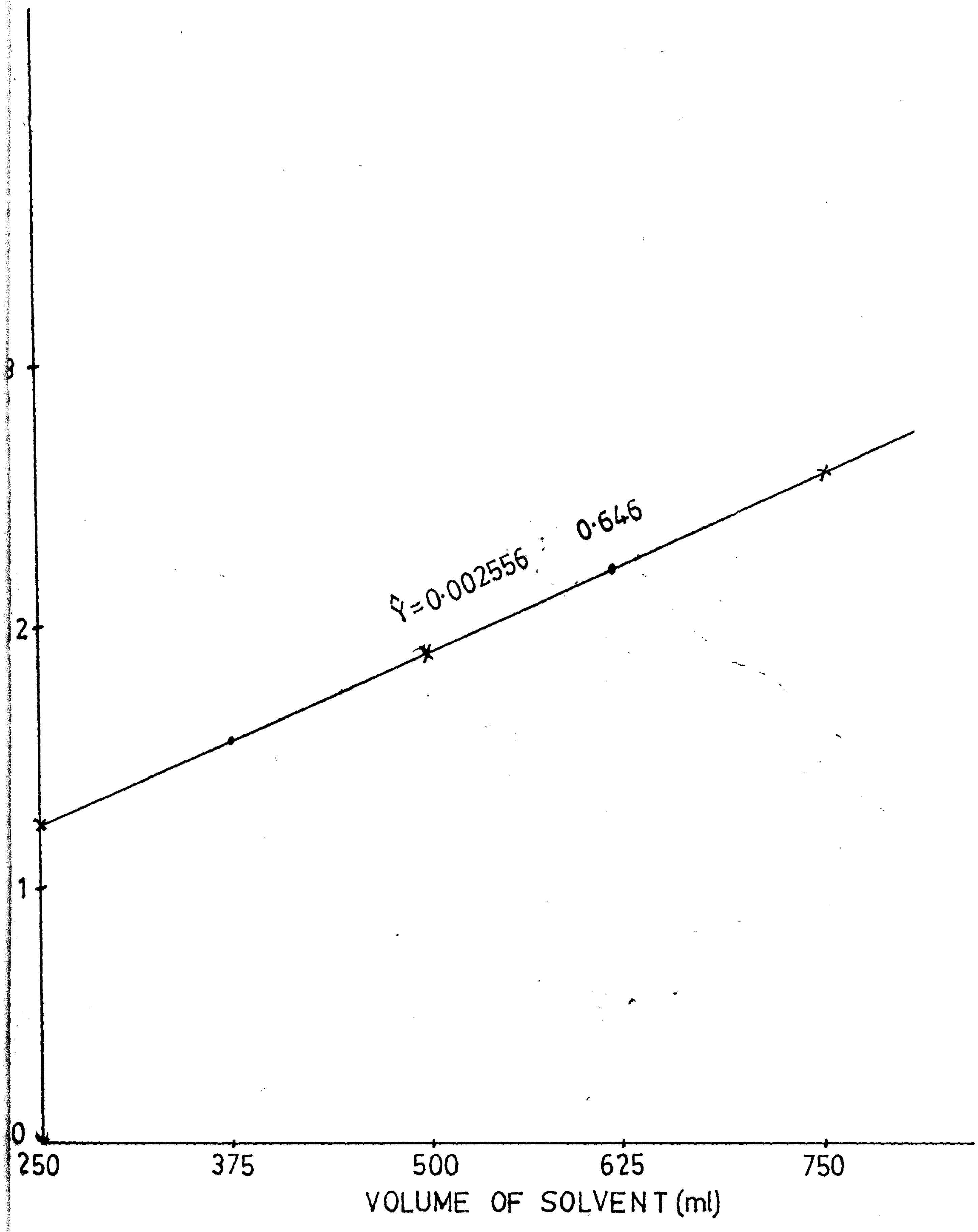
$r = 1$

this indicates that the yield of oxalic acid versus volume of solvent is a linear one and that the relationship is a very strong one.



GRAPH OF OXALIC ACID AVERAGE YIELD VERSUS VOLUME OF SOLVENT(ml) (2%$H_2SO_4$).

FOR REACTION TIME 0 3 HOURS.



GRAPH OF OXALIC ACID AVERAGE YIELD VERSUS VOLUME OF SOLVENT(ml) (2%$H_2SO_4$).

FOR REACTION TIME OF 6 HOURS.