PRODUCTION OF CAUSTIC POTASH FROM COCOA POD HUSK

BY

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ABSTRACT

The production of caustic potash from cocoa pod husk (c.p.h.) and the percentage yield from a constant weidht input was investigated. Batch method was used and the results obtained indicated that the percentage yield of caustic potash increases with increase in ashing time.

At the ashing time of three hours the average caustic potash yield was found to be between (2.6 - 5.2)%. It was also observed that the purity of caustic potash obtained increases with ashing time at constant temperature of 300°C.

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DEDICATION

TO MY BELOVED PARENTS AND SISTER,

MR. JEREMIAH AKANDE ONI (ABLE FATHER),

MRS. ESTHER ANIKE ONI (LOVING MOTHER),

AND MRS. SARAH OGUNWOLE (U.S.A)

(CONCERNED SISTER).

CERTIFICATION

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

15/8/9.

PROJECT SUPERVISOR

15/8/91

HEAD, CHEMICAL ENGINEERING DEPARTMENT

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CHAPTER ONE

INTRODUCTION

Caustic potash is one of the most important raw materials which found its uses in many chemical industries. It can be produced from a number of sources, which include the mineral deposit, agricultural products, industrial wastes, etc.

In view of the versatility of this product in industries, efforts has been made to produce it from cocoa pod husk (c.p.h.), which has been found to contain high percentage of potassium oxide (K_2O).

Potassium hydroxide (KOH) is used directly in the production of explosives, gun powder, etc. However, its most important uses are in its converted form. For instance, potassium chloride (Kcl) and potassium sulphate (K_2SO_4) are important constituents in the production of fertilizers. It is also used in the manufacture of hard and soft soaps such as shaving soaps, via potassium carbonate (K_2CO_3). As a constituent of oxidizing agents e.g. potassium permanganate ($KMnO_4$) and potassium dichromate ($K_2Cr_2O_7$), it is used in bleaching, tanning and dyeing processes.

Potassium Nitrate (KNO_3) also called saltpeter is used in the manufacture of local gun powder. Also mixture of potassium Nitrate (KNO_3) and Potassium chlorate ($KClO_3$) are oxidizing agents used in the manufacture of matches. Potassium bitartrate ($KHC_4H_4O_6$), called cream of tartar is also used in baking powders.

Commercial exploitation of cocoa pods husk could be made in the production fedding stuff for animals since the amounts of protein and fibre in the pod bear a close resemblance to those for grass hay.

It has also been found that one third of the pod consists of cellulose which might be separated and made into paper.

Production of caustic potash (KOH) from (c.p.h.) is however preferable for commercial utilization of (c.p.h.) since the problem of using them as an animal feed is difficult to solve, as of rapid deterioration, also analysis of (c.p.h.) has shown that it contains about (0.20-0.21)% of theobromine, which is known to be cumulatively toxic to animals.

Again it has been found that paper produced form (c.p.h.) are of low quality with parchment-like product and bad appearance. Moreso, it was soft and quiet inflexible because of high protein contents and low fibre contents (mainly in the form of fibroid pulp), with a few of them having a length of 0.3-0.5mm instead of the least 0.9mm length for paper making.

Basically three important methods are available for producing caustic potash (KOH) from (c.p.h.). These are batch, semi-bath and continous methods. But due to time and equipment limitation, the batch and semi-batch methods are used in this study.

The objective of the present study therefore, is to produce caustic potash (KOH) from cocoa pod husk (c.p.h.), which for many years has remained to a large extent an agricultural waste with resultant worldwide disposal problem.

After the caustic potash (KOH) have been obtained, the chemical properties will be determined in order to establish its purity. Also some of the important parameters that are necessary for the design of a full scale plant for the production of caustic potash (KOH) from (c.p.h.) will also be investigated.

CHAPTER TWO

2.0. LITERATURE SURVEY

2.1. COCOA TREE AS A CASH CROP AND ITS PODS

Cocoa tree (Theobroma cacao) is one of the most important economic crops of West Africa. It is planted generally for beverages production. e.g. Bournvita, Overtine, Choco-milo, etc. It is a native of tropical America and Africa and is cultivated extensively in tropical America and Africa, West Indies, Brazil, Ghana, Nigeria and Kenya. The World supply comes mainly from Brazil, Kenya, Ghana and Nigeria.

On an average basis, each cocoa tree is capable of bearing fifty (50) pods, measuring (15-22 x 7-10)cm each, and having about thirty (30) good seeds, yielding over 1Kg of cured cocoa. Pods are cut or broken open to extract the seeds which are fermented, dried, roasted, and powdered for beverages production.

In addition to its use as drink, cocoa powder together with certain ingredients such as sugar, spices and sometimes milk is used in making sweet chocolate. Also with the presence of some percentage of protein and fat contents, cocoa is a source of food.

The main product of the crop is the "cocoa beans or seeds". The seeds are about the size of almonds and have a thin skin or shell which averages to about 12% of their weight. The cocoa bean seeds contain theobromine and caffeine 1% or less, protein (15%), starch (15%) and fatty oil (30-50)%. (1)

According to F.R. Irvine (2) cocoa thrives best in deep, well drained neutral or slightly acidic soil, preferably heavy loamy or light clay soil which throughout the year contains enough air and water. Cocoa is sensitive to strong winds and can only be grown near the coast when protected by wind-breaks.

It thrives in a warm humid climate and so must be protected from hot dry winds. It dislikes long drought and prefers a rainfall of (1600-2200)mm annually. They could be grown together with bananas to provide shelters with an average shade temperature of 27^oC (80^oF), with daily and seasonal variation less than 8^oC (15^oF). The farm should be cool with damp air circulating beneath the shelter plant canopy.

Cocoa plant is believed not to be free from serious pests and diseases. Mirids are the most harmful insect pest of cocoa in Nigeria, accounting for about 25% loss of total yield every year. The other is black pod disease. These diseases are capable of infecting the crop at any stage of its development. They are currently controlled by application of organochlorine insecticides such as Gammalin 20, Lindex 20, and Kokatine. In some acute cases of infection, the infected plants are cut off from cocoa farm plantation and burnt to ashes to prevent the spread of the diseases. (3).

2.2. BOTANICAL CONSIDERATION OF COCOA PLANT

By botanical consideration of cocoa plant we mean growing of the plant (cocoa) with other plants for scientific study to determine its structure, and other conditions necessary for its normal growth. From study by Sachs in 1860 and Knop 1865 (1) via water culture experiment to determine which elements are essential for plant normal growth and in which form they are best taken up, it has been shown that potassium is abundantly present in the growing regions of the plant, the flowering parts, in the fruits/pods and seeding parts because of the role it plays in the plant body.

Potassium has been found to be an essential constitutent of the plant protoplasm and is closely connected with its important activities. It is known to act as a catalyst in the synthesis of carbohydrates and proteins. Starch grains are not formed in the absence of potassium. It also helps the growth of the plant and enables it to produce healthy flowers, seeds and fruits.

In the absence of potassium, the growth of the plant is terminated, the stem becomes slender, the leaves lose their colour and gradually wither away. From the important role played by potassium and its compounds in the plant life, it is no surprise why cocoa pod husk (c.p.h.) is very rich in potash.

2.3. STRUCTURAL COMPOSITION OF (C.P.H.)

There are three varieties of cocoa tree which is common to all areas where it is been propagated, these are F_3 Amazon variety, $T_{9/15}$ variety, and Amelonado cocoa variety. From the structural studies of Amelonado variety, it has been shown that cocoa pod husk of each Cultivar is separable into three parts; the "outer" pericarp; the "middle" pericarp, and the "inner" pericarp. Most of crude fibre is located in the middle pericarp. The outer pericarp of the three varieties constitutes over one-half of the whole husk, (i.e. about (44-63)% of the pod husks, on either a fresh or dry weight basis.

On fresh weight basis, the middle pericarp is the smallest component in F_3 amazon variety, whereas in Amelonado and $T_{9/15}$ varieties, the inner pericarp is the smallest. On dry weight basis however, the inner pericarp is the smallest component in the three cultivars. The thickness of both the middle pericarp and the inner pericarp is uniform throughout in diameter in all the three varieties, but the shape and thickness vary in the outer pericarp. (See Table 2.1 overleaf).

TABLE 2.1

Percentage content of the three regions of the pericarp in fresh and dry pod husks of Amazon, $T_{9/15}$ and Amelonado Cocoa.

Sample			Outer pericarp (mm)	Middle pericarp (mm)	Inner pericarp (mm)
AMAZON	Fresh	Mean	54.6	20.0	25.4
		Range	52.6-57.9	19.6-21.0	21.1-27.8
	Dry	Mean	51.0	31.75	17.25
		Range	50.2-53.6	30.5-32.6	14.1-19.3
$T_{9/15}$	Fresh	Mean	63.2	20.10	16.7
		Range	61.8-64.2	19.6-21.5	14.3-18.6
	Dry	Mean	59.30	28.0	12.70
		Range	58.7-62.1	26.4-29.5	8.4-14.9
AMELONADO	Fresh	Mean	54	23.4	22.6
		Range	52.5-62.1	22.9-24.5	21.2-24.6
	Dry	Mean	43.8	43.1	13.1
		Range	42.6-45.4	41.5-44.8	9.8-15.1

The microscopic observation of the transverse sections of the pod husk showed that it is highly differentiated. The outer pericarp stretches from the epidermis to the vascular cambium, it is maily parenchymatous cell containing large number of starch grains and containing small percentage of polyphenols.

The middle pericarp contains mainly lignified Xylem cells made up of sclerenchymatous cells. The inner pericarp consist mainly of parenchymatous cells with thin cell walls, containing a few starch grains, and this is the region of active mineral house of the plant body.

The minerals are generally evenly distributed throughout the plant body, since they constitute the active enzymes for the proper functioning of protoplasm which is present in all parts of the plant body, and as such source of ash (i.e. inorganic constituents) of plant body.(4)

2.4. CHEMICAL COMPOSITION OF COCOA PLANT

The various elements that have entered into the composition of cocoa plant body can be determined by chemical analysis. For this purpose a representative sample of the plant (i.e. samples representing all parts of the plant body) are taken and dried at temperature range of $(90-110)^{o}C$.

Some of the water that the plant contains is driven off, and the proportion of water to total weight of the plant is determined. Generally, cocoa plant are found to contain high percentage of water being 50% in woody parts; 75% in soft parts; and (85-95)% in succulent parts, for example the growing bud and the pods. When the plant is "charred" we get charcoal. The main bulk of this charcoal is carbon.

When the dried plant is carefully burnt over a flame at a temperature of about $600^{o}C$, the organic compounds such as the proteins, carbohydrates, fats, oils, etc. which are contained in the cocoa plant, which constitutes over 90% of the dry matter of the plant are converted into carbondioxide (CO_2), water vapour (H_2O), sulphur oxide (SO); ammonia (NH_3) or free Nitrogen (N_2) and escape as such into the air. The residue left over after the above treatment consists only of inorganic compound which are in-combustible and is known as "ash". Potash (Pot, ash) an oxide of the element potassium (K) was derived from the method used to produce potassium carbonate (K_2CO_3) by evaporating a solution of wood ashes in an iron-pot hence the name potash.(1,5)

The total chemical analyses of the cocoa plant body (including the combustible materials and the ash) shows that there are various elements present in it in easily detectable and measurable quantities. The following 13 elements are constant in all parts:-

Potassium (K), Calcium (C_a), Magnessium (M_g), Iron (Fe) and Sodium (N_a), among the metals; and Carbon (C), Hydrogen (H_2), Oxygen (O_2), Nitrogen (N), Sulphur (S), Phosphorus (P), Chlorine (Cl), and Silicon (Si), among the non-metals.

Besides there are other trace elements like Boron (B), Manganese (Mn), Zink (Z), Copper (Cu), etc. The average chemical composition of cocoa plant body Carbon 45%, Oxygen 42.0%, Hydrogen 6.5%, Nigrogen 1.5%, and ash 5.0%. (1)

2.5. CHEMICAL COMPOSITION OF COCOA POD HUSK (C.P.H.)

The chemical composition of the husk of all the three varieties showed a similar pattern in the distribution of substances among the component parts. The highest concentration of crude fibre was found in the middle pericarp, followed by the outer and the inner pericarps. About 75% of total crude fibre of Amelonado variety is located in the middle pericarp whereas for Amazon and $T_{9/15}$ varieties, the values are 55% and 44% respectively.

The middle pericarp also gave the lowest values for the other substances. The outer pericarp and the inner pericarp generally gave a better nutrient balance than the whole husk, since both regions contains less crude fibre and higher levels of crude protein, ash, ether extract and Nitrogen free extract.(4)

Apart from the chemical composition given above, the most comprehensive and complete analysis of cocoa pod husk (c.p.h.) appears to be that published by Dittmar(6), which gives the maximum and minimum figures obtained from pods from five different sources. These are as shown in Tables 2.2 and 2.3.

Table 2.2

Composition of Cocoa pods (DITTMAR)[6]

Composition	Percent
Crude Protein	5.69 - 9.69
Fatty Substances	0.03 - 0.15
Glucose	1.16 - 3.92
Sucrose	0.02 - 0.18
Pectin	5.30 - 7.08
Nigrogen Free Extract	44.21 - 51.27
Crude Fibre	33.19 - 39.45
Theobromine	0.20 - 0.21
*Ash	8.83 - 10.18

Table 2.3

Composition of the Ash of Cocoa pods

Composition	Percent
C_aO	0.22 - 0.59
M_gO	0.40 - 0.52
*K_2O	2.85 - 5.97
P_2O_5	0.30 - 0.49
SiO_2	0.06 - 0.14
PH	6.10 - 7.00

2.6. Possible Commercial Utilization of (C.H.P.)

From the complete analysis of cocoa pod husk (c.p.h.) as published by Dittmar(6) in Tables 2.2 and 2.3 above, it can be seen that they contain substances of nutritional value and is also very rich in many mineral components which suggest their possible uses and exploitations in the following areas:-

1. As Sources of Polysaccharides:

From Table 2.2 Dittmar showed that cocoa pod husks contained crude protein, Pectin, Nitrogen-free extract, crude fibre and ash. A hot water-soluble polysaccharide extracted from cocoa pod shows 2% yield. And was shown to be composed mainly of L-rhamnose, L-arabinose, D-galactose, D-Maunose, together with small amount of glucose, xylose and an unidentified pentose. These are very important source of food as sugar.(6)

2. As Feedingstuff for animals in Tropical Dairy Rations:-

Latin American dairymen have long been searching for a low-priced palatable ingredient that they can substitute for corn in dairy rations. Corn is scarce in tropics of Latin America and besides it, is the basic diet for human beings. As a result it is usually very expensive feedingstuff for animals, hence the need to find alternative method for feeding the animals.

One of the recent and promising possibilities which come to the attention of these farmers is a meal made from fresh cocoa pods from which the pulp and the beans have been removed.

In Almirante, Panama, where the meal got early test in dairy rations, it was made by putting the pods into Gordon dryers, keeping them there for 42 hours at 180°F to get the moisture down to approximately 4%, and then pulverizing them in a hammer mill into product that had about the consistency of coarse corn meal.

The pod meal had the advantage of being in plentiful supply where cocoa is widely grown.[6]

3. As sourve of Fertilizer:

Considering the nitrogen and phosphorous contents of cocoa pod husk they could be placed in the same category as manure for farm animal. The potash content however is particularly high, (see Table2.3), the use of pods, therefore as fertilizer from cocoa pod husk would be very appropriate.[6]

4. As a source of Paper Manufacture:

The cellulose contents of cocoa pod husk amount to about one third of the pod. It has been suggested that it might be separated and made to high quality paper. This possibility was explored in Germany in 1939.[6]

5. Furfural Production:

The possible use of the pentosans (hemicelluloses) in the pod as a source of furfural has also been investigated. Furfural is an important chemical which finds many uses in the oil refining and plastic industries and as solvent. An analysis of cocoa pods husk made some years ago at the colonial products Laboratory (now the Tropical products Institutes) London, shows a potential yield of furfural of 9%.[6]

6. Pectin extraction:

According to Dittmar (Table2.2) cocoa pod contain 5.3 to 7.08% of pectin. This is quite high, even in comparison with the established commercial sources of pectin, such as orange pulp (3.5 - 5.5)%, Lemon pulp (2.5 - 4.0)% and apple pomace (1.5 - 2.5)%. Pectin is used commercially as food sources.[6]

7. Source of caustic potash:

Considering Table2.3, the ash content of cocoa pod husk shows a high percentage of potassium oxide (K_2O) which can be used to produce caustic potash commercially.[6]

2.7. CAUSTIC POTASH PRODUCTION FROM COCOA POD HUSK (C.P.H.)

Definition:

By production of caustic potash from cocoa pod husk we mean the recovery of (KOH) from mineral constituents which has found its way into the cocoa pod husk from the mother rock (soil) via absorption, adsorption, diffusion and osmotic activities of the plant life.

Recovery generally implies to reclaim or bring back to original form, hence chemical recovery of this nature is a phenomenon whereby chemicals which have undergone changes either chemically or physically are regenerated into their usual form using a number of articulated and well controlled processes.

Three general methods are available for the production of (KOH) from cocoa pod husk. These are batch, continuous and semi-batch methods.

Batch Processes:

These are those processes in which a certain amount of the initial substances is charged into an apparatus, processed for a given period of time and when the processing is completed the products are discharged. The operation may then be repeated several times.[7]

Advantages:

(i) Batch processes has the advantage that it can be carried out on small scale (i.e. low apparatus) capacity. Under such conditions batch processes show better economic parameters.

(ii) Batch equipment from an abandoned process can be procured at a low transfer cost to the current project.

(iii) When continuous process equipment has not been satisfactorily developed the batch-process equipment has been satisfactorily demonstrated, batch processes can provide short-range solution, when emergency deadlines must be met.

(iv) When yields and quality of products can not be achieved by continuous processing because of such parameters as very low reaction rates and long residence times in the processing equipment, batch process can be used.

Continuous Processes

These are those processes in which the raw materials are introduced into the apparatus and processed, and the products are removed simultaneously and continuously.

Advantages:

(i) No irrational waste of time in stopping the apparatus for charging in the raw materials and discharging the products out, since some products proceeds continuously for days, months even years. Therefore as compared with batch processes, continuous processes are most economical in most cases.

(ii) Continuous processes make it possible to obtain a high yield and uniform product quality.

(iii) Continuous processes are easier to mechanize or automate, and they ensure that process conditions are continuous and constant.[8]

Semi-batch processes:

These are the intermediate between the batch and continuous processes. They are usually used at the pilot stage of process design. It is usually aimed at concentrating the product to the desired concentration.

Advantage:

They are used when the batch and continuous processes proved unsuccessful.[8]

2.8. COMMERCIAL EXPLOITATION OF COCOA PODS FOR THE PRODUCTION OF (KOH) IN NIGERIA.

The world production of cocoa beans now exceeds one million tonnes a year, and the figure is steadily on the increase. From this quantity of dried beans it can be calculated that the total weight of pods from which they were harvested was of the order of nine and a half million tonnes.

Better still, from this world figures the annual production of cocoa beans from Nigeria is about 300,000 tonnes, which means about 2,000,000 tonnes of dried cocoa pods are thrown away annually.

Nigeria is among those other tropical countries which need to develop all her available resources. Any profitable use(s) of cocoa pod therefore, would be of great value to the economy of those countries concerned.[9]

CHAPTER THREE

3.0. EXPERIMENTAL WORK

3.1. LABORATORY METHODS

In the laboratory, there are three kinds of caustic potash (KOH) extraction methods. These extraction methods are the batch method, semi-batch method and the continuous extraction method.

3.1.1. THE BATCH METHOD

In this method the "ash" to be extracted is weighed and poured into a beaker and distilled water of specific volume is also added and boiled for specified time. The solution that resulted is then centrifuged and filtered using filter paper.

The filterate obtained is then evaporated to dryness to obtain the caustic potash (KOH).

3.1.2. THE SEMI-BATCH METHOD

This method is similar to the batch method described above. The difference is that the filterate obtained from the first raffinate of the batch-experiment is used to dilute the fresh "ash" to be extracted in the second batch. This is repeated for (3 - 5) more batches depending on the intended concentration of caustic potash to be produced, and the final filterate from the last batch heated to dryness to obtain the (KOH). It has been shown that this method produces high concentration of caustic potash.

SKETCH OF SEMI-BATCH METHOD

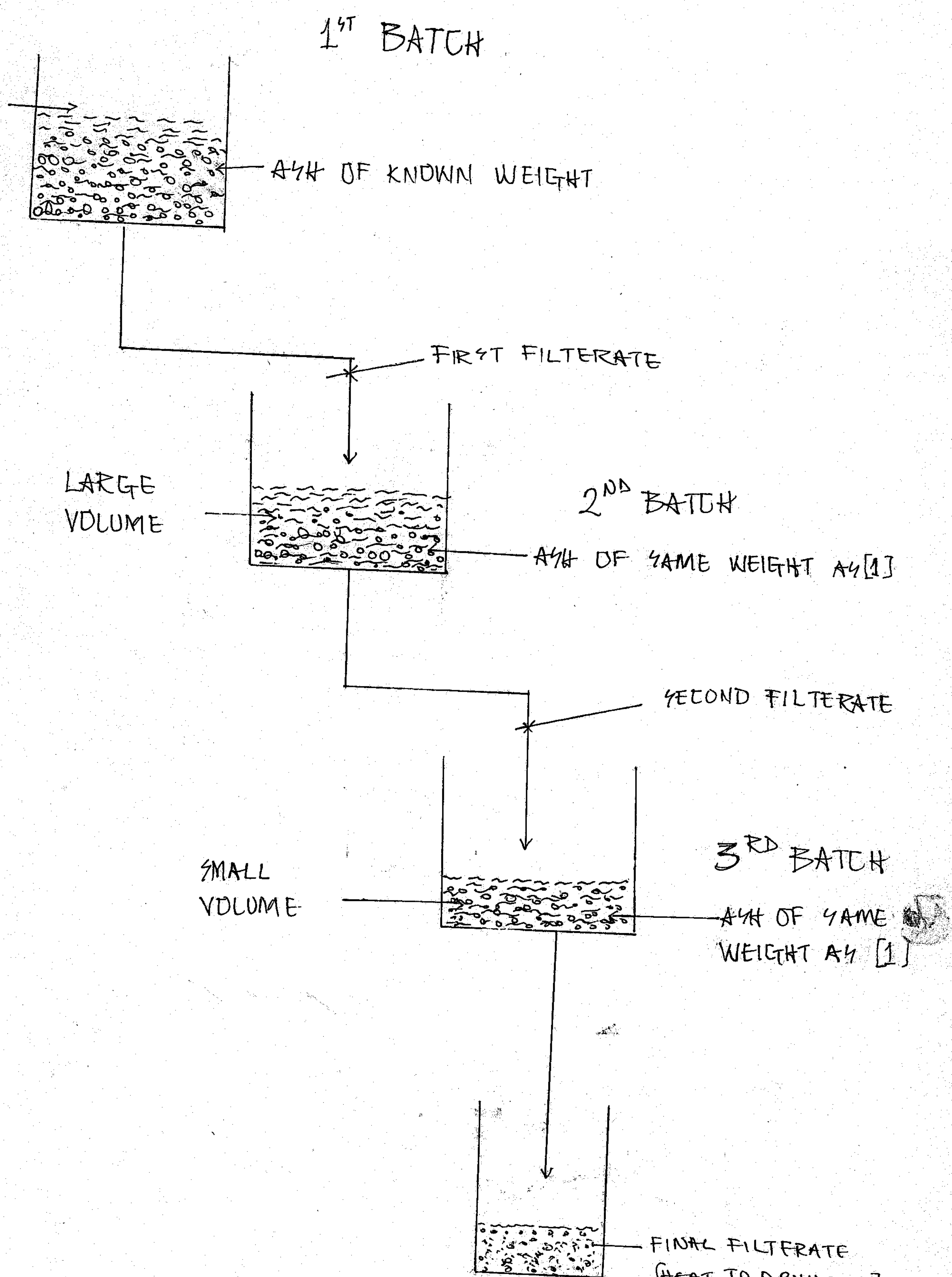

FIG. 3.1

3.1.2.1. THE CONTINUOUS METHOD

Continuous method is usually used in industries for mass production processes. This method has not been fully developed for laboratory experimentation. It is expected here that the raw material "ash" is introduced into the apparatus, processed and the product removed simultaneously and continuously.

3.2. LEACHING OF CAUSTIC POTASH FROM (C.P.H.) USING BATCH METHOD

Definition:

Leaching is the extraction of soluble component which may be solid or liquid from an insoluble solid by means of a solvent.[13 & 14]

The leaching process would generally produce two output streams:

(i) solution of solute and solvent (extract)

(ii) mixture of inert solutes, solvent and insoluble solid. (rafinnate).

Before the extraction of caustic potash the following processes were carried out on the wet-weight of (c.p.h.).

1. DRYING

Drying is a physical separation process whose objective is the removal of liquid phase from a solid phase by means of thermal energy.[10] The wet-weight of cocoa pod of 25.3Kg was dried to constant weight of 3.8Kg which is 15% of the wet-weight.

The drying was done in the sun for 26 days to reduce the moisture content of the pods and prepared the pods for ashing process. Proper drying enhances ashing. The average ambient temperature at this period was 32^{o}C.

Two samples of (c.p.h.) X_1 and X_2 were chosen and their drying rate behaviour monitored, their drying rate data is shown in Table 4.1. The drying curves were also plotted for the chosen pods as shown in Fig. 4.1.

2. CRUSHING

In crushing the weighed sample of dried (c.p.h.) was crushed (i.e. breaking into smaller pieces) with motar to reduce their total area and make them easily placeable in the crucible ready for ashing.

3. WEIGHING

The weight of the samples of (c.p.h.) to be processed were taken using an electronic weighing balance.

4. ASHING

Ashing implies burning of combustible material at control temperature in limited supply of oxygen. The dried (c.p.h.) samples were ashed in oven at constant temperature for one, two and three hours respectively, and caustic potash (KOH) was extracted from different samples of "ash" obtained.

3.2.1. EXPERIMENTAL PROCEDURE

1. 50gm of ashed (c.p.h.) was weighed and poured into a 2000ml beaker and 1 litre distilled water added.
2. The resulting solution was boiled using hot-plate with magnetic stirer at 100°C with maximum stiring for 3 hours.
3. The resulting mixture was centrifuged using (Shermond) centrifuge machine, and the supernatant solution filtered using filter papers.
4. The residue obtained from filteration was added to solid residue left in the beaker after centrifuging.
5. The filterate from (4) was collected in a weighed beaker labelled E_1.
6. Another 1 litre of distilled water was added to the beaker containing the residue and (2) carried out again.
7. While (6) was going on the filterate from (5) was boiled off until a constant weight of the beaker was obtained. E_1
8. Steps (3) - (5) was carried out on mixture obtained in (6) labelling the filterate beaker E_2.
9. Steps (2) - (8) was repeated for 1 more time with 1 litre distilled water. The filterate beakers were labelled E_1, E_2

and E_3 respectively.

10. Solutions of known concentration was prepared from the residues in beakers E_1 - E_3. The solutions were then titrated against 0.1 MHcl to determine the caustic-potash (KOH) strength, using pH -metre to determine the neutral points since the colour of solution is dark-brown and acid-base, indicator titration can not be used.

11. The mass of (KOH) residues obtained in each beaker E_1 - E_3 after evaporation to dryness were weighed. The experiment was repeated for different "ash" samples.

CHAPTER FOUR

4.0. RESULTS, DISCUSSION AND CALCULATION

4.1. Drying Results

The drying rate data is as given in Table 4.1. below:

TABLE 4.1. DRYING RATE DATA OF (C.P.H.)

Time in (Days)	Weight of sample pods in (gm)	
	Pod X_1	Pod X_2
1	212.03	167.16
2	Not weighed	Not weighed
3	Not weighed	Not weighed
4	85.83	67.86
5	66.22	51.86
6	58.67	43.57
7	56.10	40.84
8	55.22	40.03
9	Not weighed	Not weighed
10	Not weighed	Not weighed
11	54.32	39.21
12	54.00	39.00
13	53.95	38.94
14	53.85	38.83
15	Not weighed	Not weighed
16	Not weighed	Not weighed
17	Not weighed	Not weighed
18	53.62	38.63
19	53.60	38.63
20	53.55	38.62
21	53.35	38.62
22	53.28	38.42
23	Not weighed	Not weighed
24	Not weighed	Not weighed
25	53.28	38.42
26	53.28	38.42

4.2. LEACHING RESULTS

In this experiment, the duration of ashing was varied for one, two and three hours respectively, while other variables like temperature of ashing, mass of the sample were kept constant at 300°C and 100gm respectively.

TABLE 4.2.

EXPERIMENT ONE (Ashing at 300°C for 1 hour)

Exp. Runs	Amount of impure (KOH) obtained in (gm)	% purity of (KOH) obtained	Boiling Temperature (°C)	pH-meter Temperature (°C)	pH-Values
$E_{1,1}$	6.20	09.89	99.5	30	9.02
$E_{1,2}$	2.82	14.83	99.0	30	9.78
$E_{1,3}$	1.70	15.54	99.0	30	9.85

The colour of caustic potash (KOH) obtained is dark-brown.

TABLE 4.3.

EXPERIMENT TWO (Ashing at 300°C for 2 hours)

Exp. Runs	Amount of impure (KOH) obtained in (gm)	% purity of (KOH) obtained	Boiling Temperature (°C)	pH-meter Temperature (°C)	pH-Values
$E_{2,1}$	6.76	24.72	99.0	29	10.08
$E_{2,2}$	3.20	21.19	99.0	29	10.00
$E_{2,3}$	1.27	19.07	99.0	30	9.80

The colour of caustic potash (KOH) obtained is light-brown.

TABLE 4.4.

EXPERIMENT THREE (Ashing at 300^{o}C for 3 hours)

Exp. Runs	Amount of impure (KOH) obtained in (gm)	% purity of (KOH) obtained	Boiling Temperature (^{o}C)	pH-meter Temperature (^{o}C)	pH-Values
$E_{3,1}$	6.20	27.50	99.0	30	10.73
$E_{3,2}$	2.51	25.80	99.0	29	10.60
$E_{3,3}$	1.02	24.90	99.0	29	10.45

The colour of caustic potash (KOH) obtained is whitish-brown.

N.B. (i) Sample calculation for percentage purity and other calculations are shown in the Appendix.

(ii) Weight of treated "ash" is the same for all the experiment (50g).

(iii) Preparation of known solution of the obtained (KOH) is the same for the three experiments.

TABLE 4.5.

ANALYSIS OF RESULTS

Exp. No.	Exp. Runs	Amount of Impure (KOH) obtained in (gm)	% Purity of (KOH)	Amount of Pure (KOH) obrained in (gm)	% yield of (KOH) for each Expt.	Efficiency of Extraction (%)
1	1	6.20	09.89	0.61318	2.6	43.3
	2	2.82	14.83	0.418206		
	3	1.70	15.54	0.26418		
2	1	6.26	24.72	1.547472	4.51	75.2
	2	2.20	21.19	0.466125		
	3	1.27	19.07	0.242173		
3	1	6.20	27.50	1.70500	5.21	86.8
	2	2.51	25.80	0.647580		
	3	1.02	24.90	0.25398		

NOTE: (i) About 6% of (c.p.h.) "ash" contained (KOH) in terms of K_2O.

(ii) Sample calculation of efficiency of extraction is shown in Appendix.

TABLE 4.6.

EFFICIENCY DATA

Time of Ashing in (Hrs)	% yield of (KOH)
1	2.6
2	4.51
3	5.21

4.3. DISCUSSION OF RESULTS

In Table 4.2. (i.e. result of ashing at 300^{o}C for 1 hour) it can be observed that the pH and the strength of the obtained (KOH) increases with extract obtained from subsequent raffinates of the experiment. Their pH is 9.02, 9.78 and 9.85 respectively. The explanation for this observation is that the period of ashing (one hour) in this case, is not long enough to completely burn-off all the organic matter of (c.p.h.). Thus, there are some (KOH) associated with the unburnt organic matter not yet dissolvable in the solution. Hence there is no equilibrium between the solvent (distilled water) and solute (K_2O).

But with subsequent raffinates treatment such (KOH) tend to dissolve more in the solution and are extracted which lead to increase in pH and strength of (KOH).

From Tables 4.3. and 4.4. respectively, it can be observed that the pH and strength of the obtained (KOH) decreases with each raffinate of the experiment. This is due to exhaustion of the available (KOH) in the ash and the blockage of the microstructural pores of extraction due to "ash impurities" since the ash becomes finer with increase period of ashing. The efficiency of this method is high compare with the value obtained by Dittmar in Table 2.3. (See Table 4.5.).

Also from Table 4.6. it could be deduced that increase in time of ashing resulted in increase in percentage yield of (KOH) obtained for each experiment. See Fig 4.5. The percentage yield is 2.6, 4.5 and 5.2% respectively.

The reason for this observation is that as period of ashing increases, there is more complete burning off of organic matter in the (c.p.h.) making the KOH in the c.p.h. more readily available for dissolution and their consequent extraction which lead to increase in percentage yield.

From Table 4.5. also the efficiency of extraction increases with increase in period of ashing. The reason for this observation is associated with more (KOH) extraction as the period of ashing increases.

4.4. CONCLUSION

The production of caustic potash (KOH) from cocoa pod husk (c.p.h.) had been investigated and it can be concluded that for higher yield of (KOH) to be obtained from a given dried (c.p.h.) there is need to "ash" for long period of time as shown in this project.

The maximum time of ashing was three hours at 300^{o}C, while the minimum was one hour. The highest percentage of (KOH) extracted was 5.2% for the maximum period of ashing, while the minimum percentage of (KOH) extracted was 2.6% for the minimum time of ashing.

The figure obtained for three hours of ashing may not be the maximum permissible (KOH) that can be extracted, but the yield need to be balanced with other parameters in an optimization exercise.

4.5. RECOMMENDATION

1. It is necessary to study other parameters like variation in the temperature of ashing and the mass of sample. The knowledge of this will help to compare the percentage yield and efficiency of (KOH) extracted with this project.
2. Further work should be carried out by increasing the period of ashing further and determine its effects on quality of (KOH) obtained.
3. More work should be done to determine the physical and chemical properties of (KOH) obtained from (c.p.h.). This would enable comparison to be made between (KOH) obtained from (c.p.h.) and those obtained from other sources.
4. Lastly, further work is recommended on the effect of washings on yield and purity of the extracted (KOH).

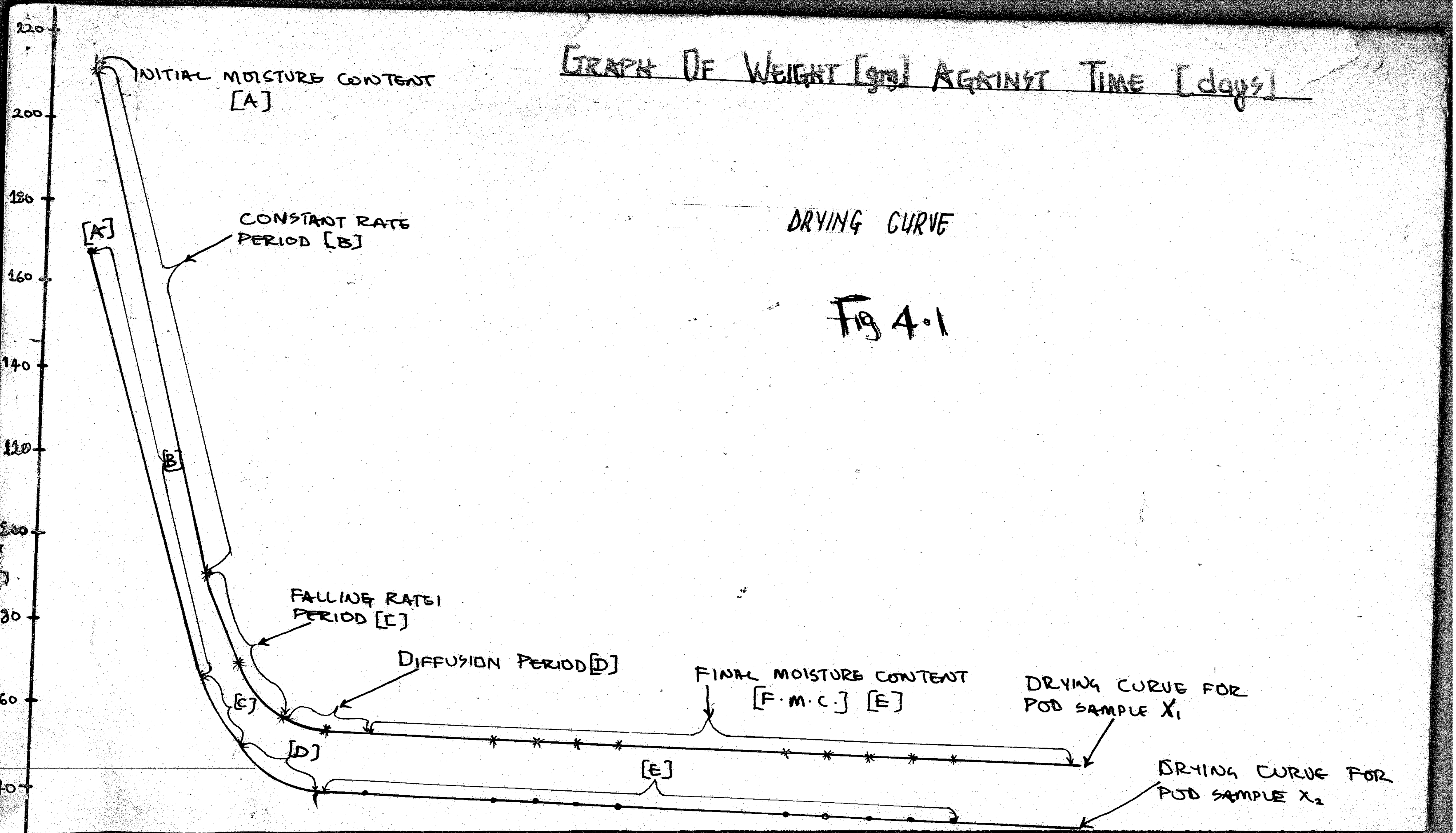
GRAPH OF WEIGHT [gm] AGAINST TIME [days]
DRYING CURVE
INITIAL MOISTURE CONTENT [A]
CONSTANT RATE PERIOD [B]
FALLING RATE PERIOD [C]
DIFFUSION PERIOD [D]
FINAL MOISTURE CONTENT [F.M.C.] [E]
DRYING CURVE FOR POD SAMPLE X1
DRYING CURVE FOR POD SAMPLE X2
[A]
[B]
[C]
[D]
[E]
220
200
180
160
140
120
80
60

Fig 4.1

GRAPH OF % YIELD OF KOH V/S TIME IN HRS.

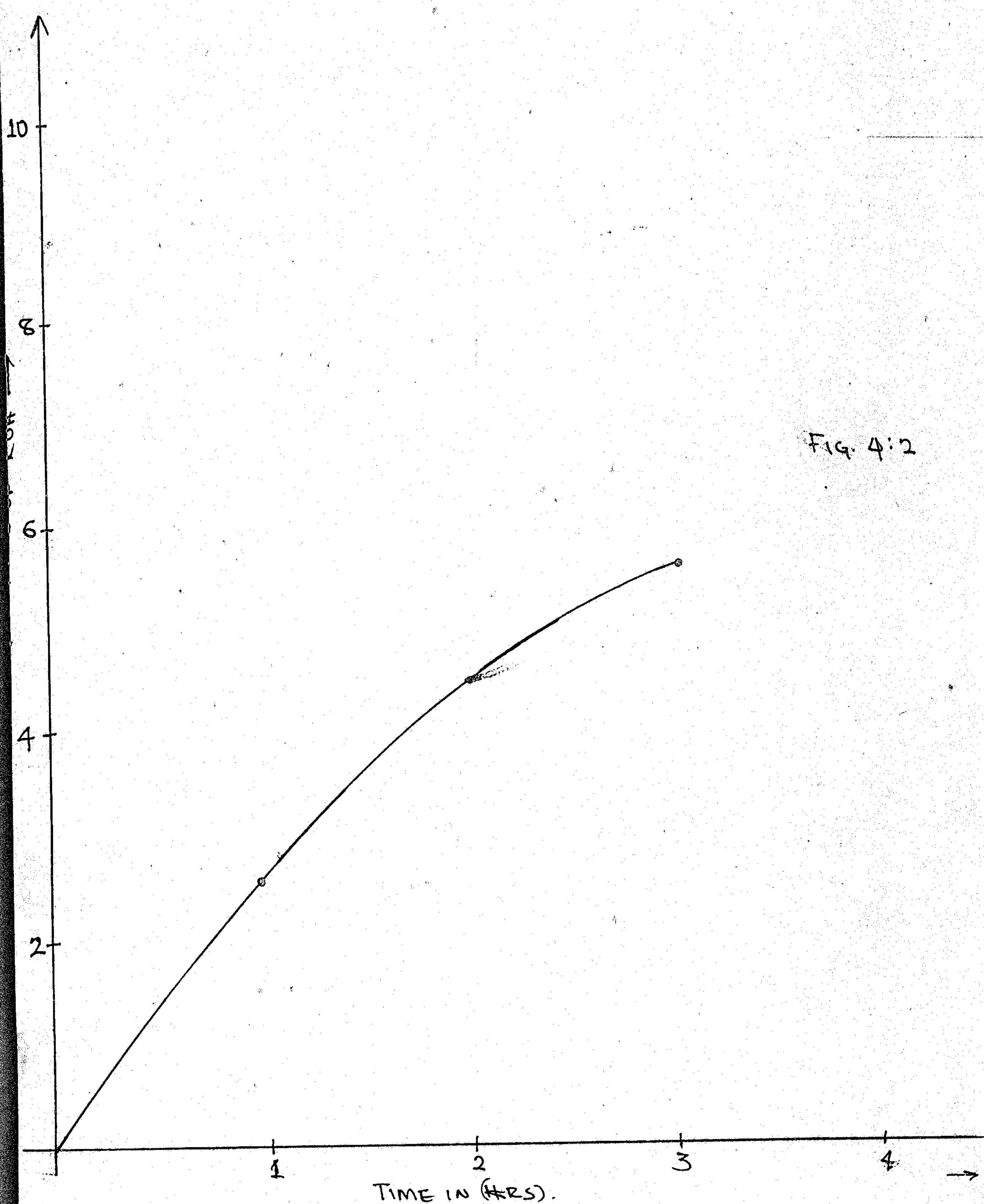

FIG. 4:2

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APPENDIX

PROPERTIES OF CAUSTIC POTASH (KOH)

Molecular Weight	=	56.10
Colour	=	White
Crystal Form	=	deliquescence
Specific gravity	=	2.044
Melting point	=	380^oC
Boiling point	=	1320^oC
Solubility in cold water	=	970^o
Solubility in hot warer	=	178^{100^o}

Sample Calculation for strength determination

(i) Equation of reaction:-

$$KOH + Hcl \longrightarrow Kcl + H_2O$$

(Salt)

Molar Ratio = 1 : 1

(ii) Preparation of known solution of (KOH) obtained:-

0.5gm of obtained (KOH) was taken and dissolved in $250cm^3$ of distilled water.

Concentration of (KOH) prepared = $\frac{0.5}{250}$ = $0.002g/cm^3$

(iii) Calculation

VA = $0.705cm^3$

MA = 0.1M

VB = $20cm^3$

MB = ?

Note

Where

VA = Volume of acid used (Hcl)

MA = Molarity of acid (Hcl)

VB = Volume of base used (KOH)

MB = Molarity of base (KOH)

(iv) Formular $\frac{MAVA}{MBV_B} = \frac{1}{1}$

$MB = \frac{MA\ VA}{V_B} = \frac{0.1 \times 0.705}{20}$

$= 3.525 \times 10^{-3}$ moles

$MB = 3.525 \times 10^{-3}$ moles

Strength/Concentration of (KOH) = MB X Molecular Weight of (KOH)

$= 3.525 \times 10^{-3} \times 56.10$

$= 0.1977525 g/dm^3$

$= 0.0001977525 g/cm^3$

$= 1.977525 \times 10^{-4} g/cm^3$

(v) % Purity $= \frac{0.0001977525}{0.002} \times \frac{100}{1}$

= 09.89%

N.B.

Other values follow the same calculation method.

Sample Calculation of Percentage Yield of (KOH) extracted from experiment One

Table 4.5

The amount of extracted (KOH) for each run in grams is 0.61318, 0.418206, and 0.26418 respectively.

The initial "ash" processed is 50gm

Total (KOH) extracted $= 0.61318 + 0.418206 + 0.26418$

$= 1.295566gm$

% Yield of (KOH) $= \frac{1.295566}{50} \times \frac{100}{1}$

$= 2.6\%$

N.B.

Other % yield calculation follow the same method.

Sample calculation of efficiency of (KOH) extracted

Formular

$$\frac{\text{KOH obtained}}{\text{KOH available (in terms of } K_2O)} \times 100\% = \frac{2.6}{6.0} \times \frac{100\%}{1}$$

$$= 43.3\%$$

BASIC DEFINITIONS IN DRYING

(i) Rate of drying: This is the amount of water or liquid (usually expressed in Kg), removed per square meter of drying area per hour (Kg/m^2hr).

(ii) Constant-rate period: This is the part of the drying process during which the drying rate is constant and is controlled by external factors.

(iii) Falling-rate period: The part of the drying during which the drying rate varies with time.

(iv) External drying factors: The independent variable associated with the conditions and flow of gas-phase.

(v) Internal drying factors: The properties of the solid that influence the transport of heat and mass within the solid-phase.

(vi) Bound Moisture: The amount of water in the solid which exhibits a vapour pressure less than normal for the pure liquid.