DEVELOPMENT OF A SUITABLE PROCESSING TECHNIQUE FOR KAOLIN CLAY FOR INDUSTRIAL APPLICATION

BY

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DEDICATION

To mum and 'gina

CERTIFICATION

We certify that this work was carried out by MR. AJAKA Oyedele Ebenezer in the Metallugical & Materials Engineering Department of the Federal University of Technology. Akure; and to the best of our knowledge, this work has not been submitted elsewhere for the award of a degree.

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Chapter Four: Results and Discussion

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ABSTRACT

Production of kaolin clay of very high purity, brightness and whiteness was undertaken in this study for the purpose of developing an economic processing technique for working any local kaolin clay deposit.

Clay materials were collected from locations such as Akure, Ifon and Ijero. The chemical analysis of Akure clay was carried out using the wet-leach technique while that of Ifon was obtained from a previous work by Delta Steel Company, Aladja and the various compositions showed that iron was responsible for colouration. Ijero clay was not analysed.

Classification and jigging technique were employed at the primary processing stage to separate the coarse impurities from the clay; and an oxide reductive bleaching technique was used in a secondary processing to remove colour. A greyish-white product was obtained from the initial red samples. The purity of this final product was determined by measuring the iron content with an atomic absorption spectrophotometer.

Results showed that an integration of a multi-stage classification process and the oxide reductive bleaching process gave a good product; while jigging produces an unsatisfactory result. The results further indicated that the reductive bleaching process can be employed to remove the colour induced by iron from any kaolin clay and that the whiteness and purity of the final product can meet most industrial specifications.

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CHAPTER ONE

INTRODUCTION

Kaolin (or china clay as it is commonly known) has become an important mineral to man since its uses were discovered. It has a wide range of application and its use therefore cuts across many industries. Among its many areas of application are in paper making (as fillers and coating grades), in ceramic wares production, paints making, and in the pharmaceutical industry. Kaolin also finds application as fillers in plastics manufacturing, cosmetics, as discolourant in oil and grease production, and as cracking catalyst in petroleum refining.

Pure kaolin is a refractory material and is therefore useful in foundry practice and other high temperature applications. Also, pure kaolin has good electrical insulating property. It is therefore used in the manufacture of electrical socket insulators.Kaolin is also entensively used in building and furniture making. Up to date, the mineral is said to be the major source of aluminium oxide used in fiber glass production. In view of the diverse applications of the industrial mineral, kaolin, its importance in the development of a strong industrial base in any nation cannot be overemphasized.

The various industrial applications of kaolin require varying degree of purity and brightness below which this industrial mineral may not be useful to those industries. As example, it is said that over 80% of kaolin used in paper making are used for paper coating [Forbus et al, 1993, veglio et al, 1993]. It is therefore important that a high degree of whiteness and brightness are attained in any kaolin clay processed

for these applications. Thus the need to produce high purity kaolin in Nigeria is very great.

In Nigeria, kaolin clay is found in abundance and in large deposits of workable qualities as shown in Figure 1.1 and Table 1.1. It is interesting to note that inspite of the abundance of this mineral in the country, most of the user industries still import processed kaolin (especially those requiring high purity and brightness as in paper coating, food processing and other special applications). The reason for this surge in importation is that there is no suitable processing technique in the country to meet the specifications of these industries. The effect of this on the industries and on the national economy alike cannot be over emphasized.

This study is therefore an effort towards the development of a suitable and economic processing technique that can be employed in the recovery of the local kaolin clay deposits to produce high purity kaolin that will meet the standards and specifications of the various users.

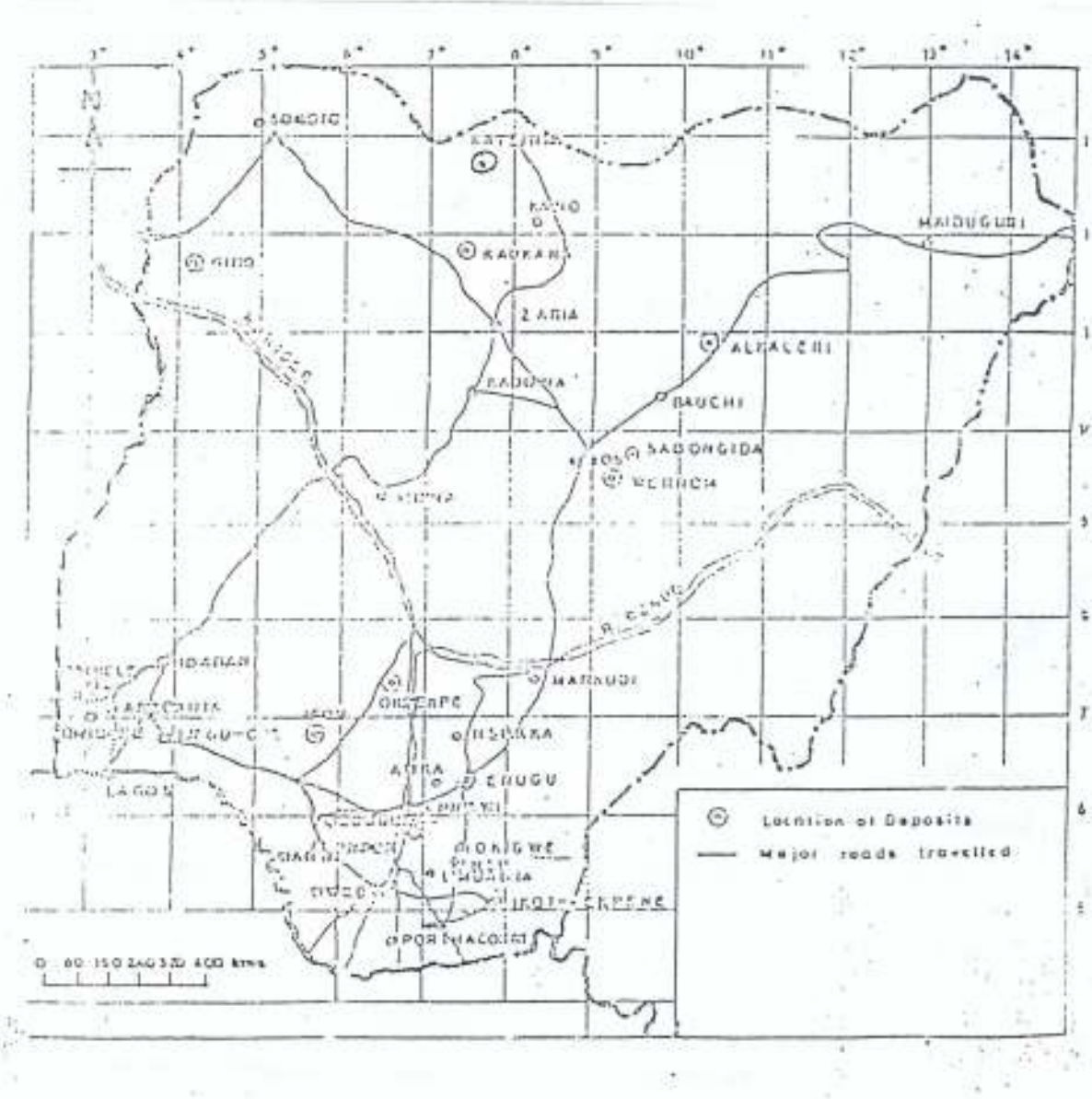


FIGURE 1.1 : MAP OF NIGERIA SHOWING THE LOCATIONS OF SOME IMPORTANT CLAY DEPOSITS.
(Delta Steel, Aladja : Physical and Chemical Properties of some Nigerian clays)

Table 1.1 IMPORTANT CLAY DEPOSITS IN NIGERIA

(Ministry of Mines and Power:- Inventory of Nigeria Minerals and Miners 1990).

S/NO.	STATE	LOCATION	TYPE	RESERVE (TONNES)
1.	Anambra/Enugu	Ozubulu Ihiala & Nnewi L.G.A.	Kaolinitic	4,200,000
2.	Anambra /Enugu	Enugu	Fireclay	50,000.000
3.	Katsina	Kankara	Kaolinitic	3,400,000
4.	Kaduna	Mararaba - Rido	Brickclay	5,524,000
5.	Ogun	Onibode, Lishabi Bamajo Miroko	Kaolinitic	2,800,000
6.	Plateau	Jos and Ropp	Kaolinitic Brickclay	Very Large
7.	Borno	Maiduguri, Biu	Kaolinitic	Large
8.	Edo /Delta	Ukwunze	Brickclay	To be fully Investigated
9.	Imo /Abia	Bende, Awomama, Nsu, Orlu & Ohaozara L.G.A.	Brickclay	" "
10.	"	Umuahia	"	" "
11.	Taraba / Adamawa	Garkidda	Kaolinitic	Not fully assessed
12.	Kano	Dawakin Tofa Minjibir Tsanayawa	Kaolinitic	Large
13.	Sokoto / Kebbi	Illo kaoje	Kaolinitic	Very Large
14.	Ondo	Ifon / Omiafara	Kaolinitic	40,000,000

Table 1.2 CHEMICAL COMPOSITION OF SOME NIGERIAN CLAY SAMPLES

(Delta Steel Aladja - Physical and Chemical Properties of some Nigerian Clays).

SAMPLE	Al_2O_3	SIO_2	Fe_2O_3	CaO + MgO	L.O.I
NSU	30.22	50.6	1.90	1.08	10.54
UKPOR	33.20	48.0	1.20	0.73	12.10
OZUBULU	19.31	58.30	1.55	1.25	14.16
ENUGU	22.71	55.0	2.32	1.95	16.35
ONIBODE	39.30	42.30	trace	1.05	14.2
ORUN	34.55	50.50	2.05	1.10	15.50
OSHIELE	28.30	53.40	1.35	0.88	15.0
WERROM I	36.12	45.16	2.30	0.85	14.00
WERROM II	38.14	41.92	-	0.30	14.40
ALKALERI	25.43	54.50	1.05	1.0	15.73
KANKARA	38.67	44.50	-	1.3	16.70
GIRO	38.72	41.26	2.10	1.48	14.00
SABON GIDA	25.88	25.32	13.10	3.20	18.36
IFON	36.80	47.90	0.67	1.11	12.32
OKPEKPE	24.30	53.20	1.45	1.30	16.86

CHAPTER TWO

LITERATURE REVIEW

2-1 Kaolin: A Review of its Characteristics And Properties

Kaolin belongs to a large group of fine-grained minerals called "clay". There are different types of clay but kaolin is just one of a number of hydrous aluminium silicate minerals. Other types of clay commonly encountered include bentonite, the fullers earth variety, ball clay, and fire clay.

Geologically kaolin has been found to occur in four forms namely: kaolinite, nacrite, dickite and halloysite. Among these, kaolinite and halloysite are said to be of most common occurrence with halloysite occuring in two hydrated forms usually represented as hylloysite $2H_2O$, ($Al_2O_3SiO_2$. $2H_2O$), and halloysite. $4H_2O$, ($Al_2O_3SiO_2 4H_2O$) (Marshall, 1982).

In the industry, kaolin is classified as light kaolin, light natural kaolin and heavy kaolin. Most industrial classifications are usually based on grain size; hence in the paper making industry which is the largest user of kaolin, grading is done numerically according to grain size for coating grades, and premium grades and so on. For example, we have numbers 1,2,3. coating grades according to the fineness of the grains (Bowman et al 1993).

Like most natural minerals kaolin occurs in most of its deposits with a number of impurities which must be removed to certain extent in order to meet the specifications of the various users. Depending on the type of deposit, impurities usually associated with kaolin clay include silica (SiO_2), mica [(K,Na,Ca) $(Mg, Fe, Li, Al)_{23}(Al, Si)_4 O_{10} (OH,F)_2$], feldspars [(MAl $(Al,Si)_3O_8$- M: K, Na, Ca,Ba, Rb,Fe,Sr)] and some amount of ferrogenous minerals such

as oxides of iron (Fe_2O_3), rutile (TiO_2), siderite ($FeCO_3$). (Veglio et al, 1993, Heinskanen, 1993, and Ryan, 1978). Few of these impurities are normally associated with a particular deposit and their amount varies with the type of deposit (Heinskanen 1993).

Bowman et al, (1993) and Heinskanen, (1993) showed that kaolin clay has some of the smallest grain size ever known, with particle size sometimes below 12 micrometers and that kaolin is naturally resistant to most acid and alkalis.

2.2 KAOLIN PROCESSING TECHNIQUES

Each of the impurities mentioned above has different effects on the quality of the kaolin clay. Naturally, pure kaolin is white or greyish-white, but the presence of such impurities as oxides of iron and titanium usually impact colours which range from red to light pink (Heinskanen, 1993, Veglio et al, 1993). Other impurities like silica, mica and other coarse components are only responsible for the gritting of the as-mined clay (Bowman et al, 1993, Heinkanen, 1993). Thus kaolin processing involves the removal of both the gritting components and the colour.

Some of these impurities can be separated by physical methods such as gravity separation technique for example jigging, while others will require chemical processes.Thus complete kaolin clay purification is usually accomplished in a two-stage process, each requiring different techniques:

These are:

Primary processing techniques and

Secondary processing techniques

Primary processing techniques are those processing methods that are applied for the removal of those impurities that can be easily separated from the clay usually

the gritting components. The definition of primary processing technique also includes any method that is first applied to the as-mined clay. Secondary processing techniques on the other hand are those methods employed for the removal of any impurity that can not be separated by the primary method. Usually secondary recovery techniques are applied to remove colour and improve brightness.

It should be mentioned here that, for some industrial specifications, only the primary processing methods are applied to attain the required level of purity. Depending on the nature and amount of impurities present in a particular kaolin clay deposit, various methods have been employed for both primary and secondary processing stages. Generally, some of the methods are the common mineral processing techniques which include classification, floatation, electrosmosis, magnetic separation, jigging, chemical reductive bleaching , and microbiological processes and are well described in literatures (Kelley and Spottistwood 1982; Wills, 1982, Heinskanen, 1993, Forbus et al, 1993, Bowman et al, 1993). Some of these techniques can serve both as primary and secondary processing techniques while some especially classification and jigging can serve only as primary processing methods. Thus a combination of some of these methods is usually employed to attain the highest degree of purity in kaolin clay Processing.

2-2-1 PROCESSING BY CLASSIFICATION

Classification is a process of separating ore particles into two or more fractions on the basis of size or specific gravity difference. This difference enables different settling rates to be achieved by individual particles in a viscous fluid such as

which classification takes place are called classifiers (Heinskanen, 1993). Classification can generally be divided into two types depending on whether the fluid used is a gas or a liquid. Thus we have wet classification and dry classification.

Principle of Classification

The principle of classification as a mineral processing technique is well established and documented in literatures, (Heinskanen, 1993, Kelley and Spottistwood 1982, Wills 1982).Generally, the principle enables one to determine the settling velocity of a particle in a classifying column or what is otherwise known as classifiers. Two applicable laws are used in establishing the terminal settling velocity of a particle in a sorting or classifying column. These are Stoke's law for viscous flow and Newton's law for turbulent flow.

Based on Stoke's law the terminal velocity of a spherical particle in a classifier can be expressed as:

$$v = \frac{gd^2(s - f)}{18\mu} \qquad (1)$$

On the other hand, the terminal settling velocity of a spherical particle according to Newton can be expressed as:

$$v = \frac{[(3gd(s - f)]^{1/2}}{f} \qquad (2)$$

Where:

v = terminal settling velocity,

g = acceleration due to gravity

d = diameter of particle

s = density of particle

f = density of sorting or classifying fluid (air or water)

μ = viscosity of the sorting fluid

Thus equations (1) and (2) are strongly dependent on particle size and density, and the density of the fluid medium. The difference in particle sizes and densities between kaolin and most of its gritting impurities makes the principle of classification most effective in primary kaolin processing. Stoke's law is applicable in elutriators or gravitational classifiers since settling in these classifiers is solely due to viscous resistance; but Newton's law will apply effectively in centrifugal classifiers (centrifuges and cyclones) where turbulence is expected.

Wet Classification

Wet classification is the technique most commonly employed in the primary stage of kaolin clay recovery. Classification is usually employed for degritting, that is, removal of coarse particles.In wet classification, water is the classifying fluid commonly used. This may be done in either gravitational classifier, or centrifugal classifier or a system of integration of both gravitational and centrifugal classifiers.

The only material usually necessary at this stage is the as-mined clay itself. If the clay is mined by the hydraulic system (in which a powerful jet of water is used to disintegrate the clay rock in-situ), the resulting slurry is then diverted to reservoirs from where it is fed into classifiers (Bowman et al, 1993), Heinskanen, 1993). However, if the clay is mined in lumps and transported to the processing site, the as- mined clay is first crushed and then slurried with water using a powerful plunger.

The slurry is then pumped into a system of classifiers for degritting where impurities such as silica, mica and other heavy particles are removed.

Wet Classification in Elutriators

Elutriators are generally gravitational classifiers. Their usual mode of operation is such that clean water is made to rise slowly from the base of the classifier at a constant velocity. The water either overflows the circumference of the sorting column or is discharged with some special mechanisms. The feed introduced is separated into two fractions the sink and the overflow. In the case of kaolin classification, the sink consists of the coarse particles while the overflowing water carries the degritted clay particles up and to a slurry tank.

Usually, most of the gravitational classifiers also have a mechanical device that helps to collect the coarse impurities at a point in the classifier where they are finally discharged.

Wet Classification in Centrifugal Classifiers

Centrifugal classifiers are generally of two major types

(a) Mechanical classifiers -centrifuges

(b) Non-mechanical classifiers -hydrocyclones.

In hydrocyclones, the feed is usually introduced under pressure to achieve separation while in mechanical classifiers or centrifuges, a high speed (in-built) impeller provides the centrifugal force.

These classifiers especially the cyclones are the most efficient equipment for kaolin clay recovery. They serve many purposes. They are used for degritting, for cutting into different size fractions or grades and there are special dewatering cy-

clones and centrifuges (Heinskanen, 1993). Feed particles separation in centrifugal classifiers is very complex, so many factors have been neglected here in order to provide a brief explanation of the behaviour of particles in these classifiers.

Usually, centrifugal classifiers introduce a centrifugal or radial force in addition to the gravitational and drag forces on a feed particle which causes the particles to move in a spiral path in the classifier. Heinskanen, (1993) showed that the feed entering a cyclone starts to move downward in a spiral path but as all the materials cannot leave the classifier via the apex opening, some of the material must reverse their direction and flow back in an upward spiral. Thus two spirals are developed: one moving upwards and the other downward and both rotates in the same direction. Heinskanen, (1993) further showed that the downward spiral is at the classifier wall and contains the coarser particles which move down and escape through the apex opening while the upward spiral is innermost and contains the fine particles which finally escape through the vortex finder. In mechanical classifier, the fine particles move up and escape around the circumference of the inner cylinder of the classifier while the coarse particles move downward around the conical wall and escape through the apex opening.(Figure 2.1).

The largest particle of the feed fraction that escapes as the overflow is called the cut size. Particles larger than this cut size will escape through the apex opening. Heinskanen, (1993) said that the cut size depends on many factors among which are: cyclone diameter, votex finder diameter, feed inlet diameter, apex diameter, free height and angle of cone, feed rate, pulp density and viscosity and feed pressure. Thus the kaolin clay recovery process that requires cutting the final product into different size

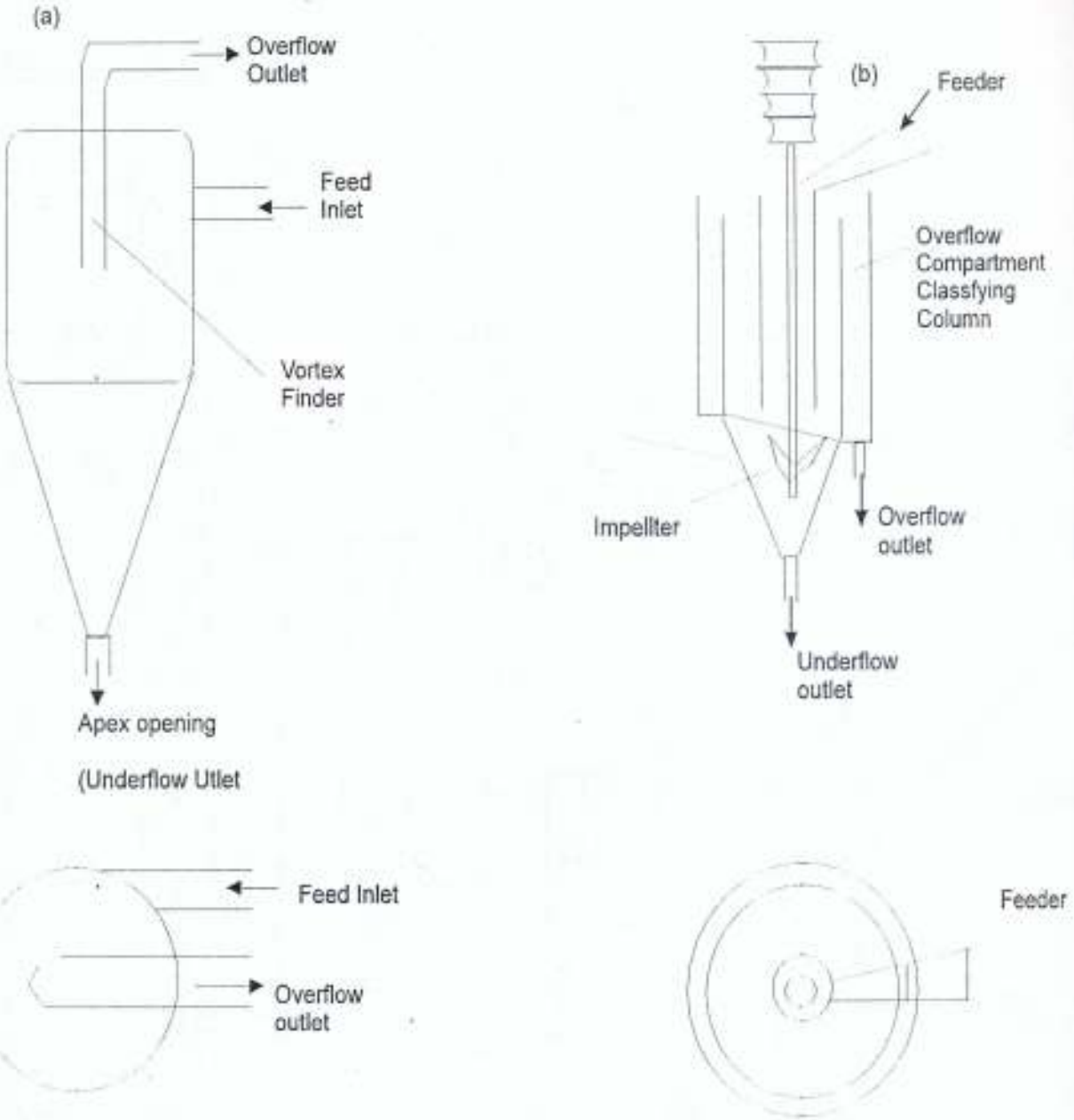
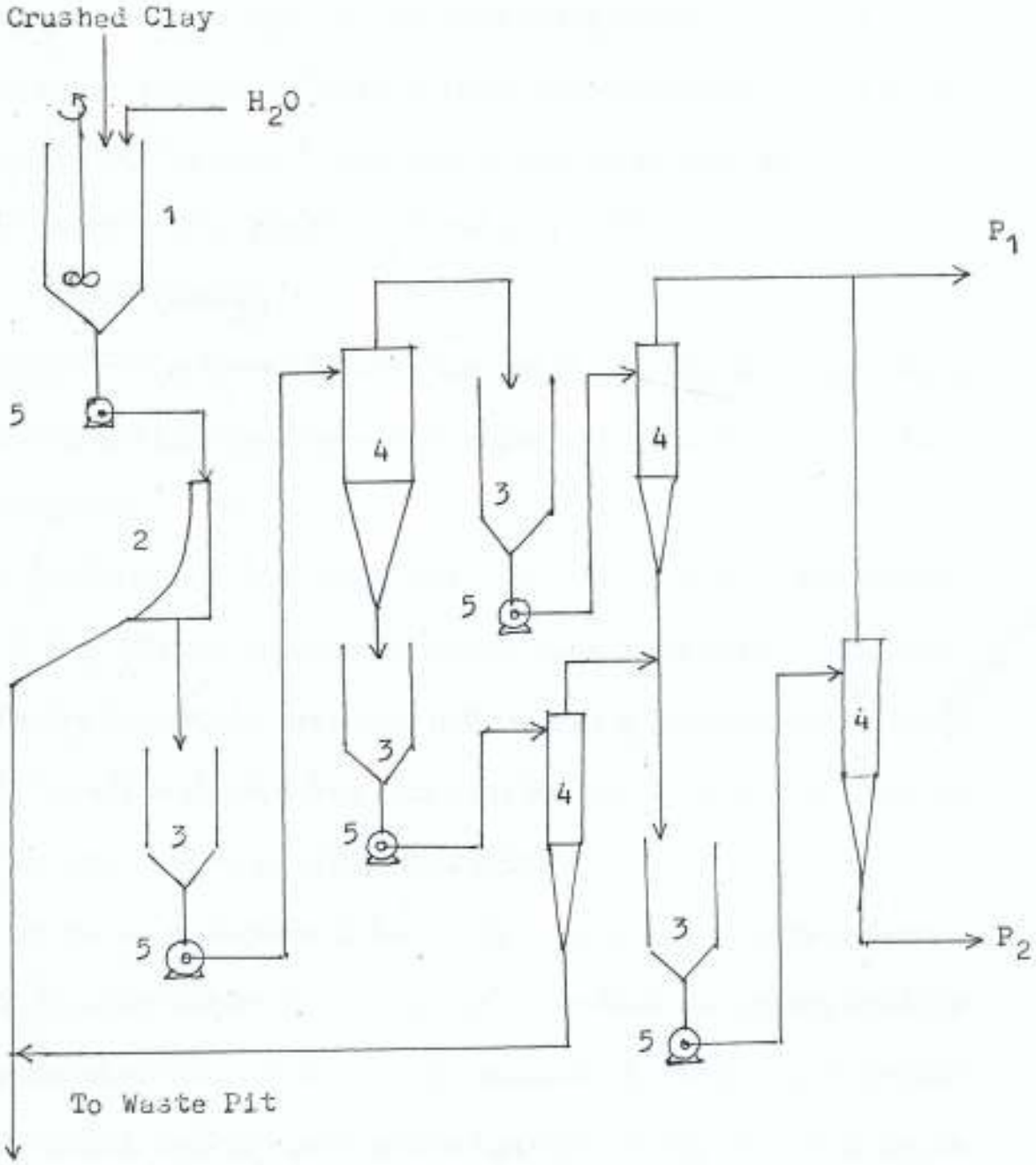


Fig. 2.1 Types of Centrifugal Classifiers
(a) Hydrocyclone (b) Centrifuge



1. Plunger
2. Spiral or any gravitational classifier
3. Slurry tanks
4. Hydrocyclones
5. Pumps
6. P_1 & P_2 - Clay Products.

Fig. 2.2 : A multi-stage Classification process producing two grade products. (Heinskanen, 1993)

fractions usually consists of many cyclones of varying diameter, integrated in a mult-stage classification process such that the overflow form one classifier serves as feed for the next Figure 2.2 is a simple flowsheet for multi-stage classification of kaolin employing a variety of these classifiers. (Heinskanen, 1993).

Dry Classification

The significant difference between wet and dry classification is the sorting fluid used. While in wet classification water is the fluid commonly used, dry classification employs air.

Dry classification is more adaptable to clay processing than other minerals because it is more effective in separation of fines from coarse particles. The equipment used in dry classification are similar to those used in wet classification except that they are usually in closed systems because of the dust involved. Thus there are dry classifiers which employ air as their fluid medium.

If a secondary purification of kaolin clay is necessary in order to remove colour after dry classification, the only applicable secondary processing technique is dry magnetic separation. Because of this limitation, dry processing is normally applied to kaolin clay with very good as-mined quality or to clay whose only impurities are coarse silica grains. It may also be employed in processing clays whose specification for purity can be met by this technique alone without a secondary purification.

Feed Preparation for Dry Classification

The feed material is the as-mined clay. The principle involved in dry classification requires that the kaolin clay be fed dry into the classifier and then separated into two size fractions. Thus in preparing a feed for dry classification, the as-mined clay is first crushed, dried and then ground lightly before feeding into the classifier. No serious grinding is required. This is to maintain a wide size difference between the clay particles and those of the impurities -- a property that is very essential for an effective classification.

The crushed clay is usually dried in a chamber maintained at about 120°C. As the clay looses its water content, it tends to stick together (forming a cake) so that light grinding is required to liberate the components thoroughly. In some systems, drying and grinding are carried out simultaneously in the same equipment.

Separation In Dry Classifiers.

Most dry classifiers are elutraitors or gravitational classifiers. Generally, most of the available designs contain a sorting column in which air is blown up from the base. The blower is made in such a way that the speed is adjustable so that the velocity of the rising air can be varied. The feed is usually introduced dry. The manner in which the feed is introduced depends on the design. In some systems, the feed is blown up simultaneously with the air, while in others the feed is introduced independently in a counter direction to the rising air in the column.

(Figure 2.3) shows a sketch of dry classifier.

The dry feed introduced into the sorting column is separated into two size fractions. The dust and the coarse particles. As the feed is introduced or blown up from the base of the classifier, the larger particles attain terminal settling velocities that are greater than the velocity of the rising air and fall back to the bottom of the column. The lighter and finer particles are carried in the rising air current through a screen to a product bin.

In most designs, the screen is made of rotating vanes whose aperture varies with the speed of rotation. As the speed is increased, the aperture between vanes (or blades) decreases. This screen performs two major functions. It prevents any particle that has passed through it from returning because it is also a blower, and secondly it allows only fine particle to pass through. Dry classification usually follow a sequence shown in (Figure 2.4)

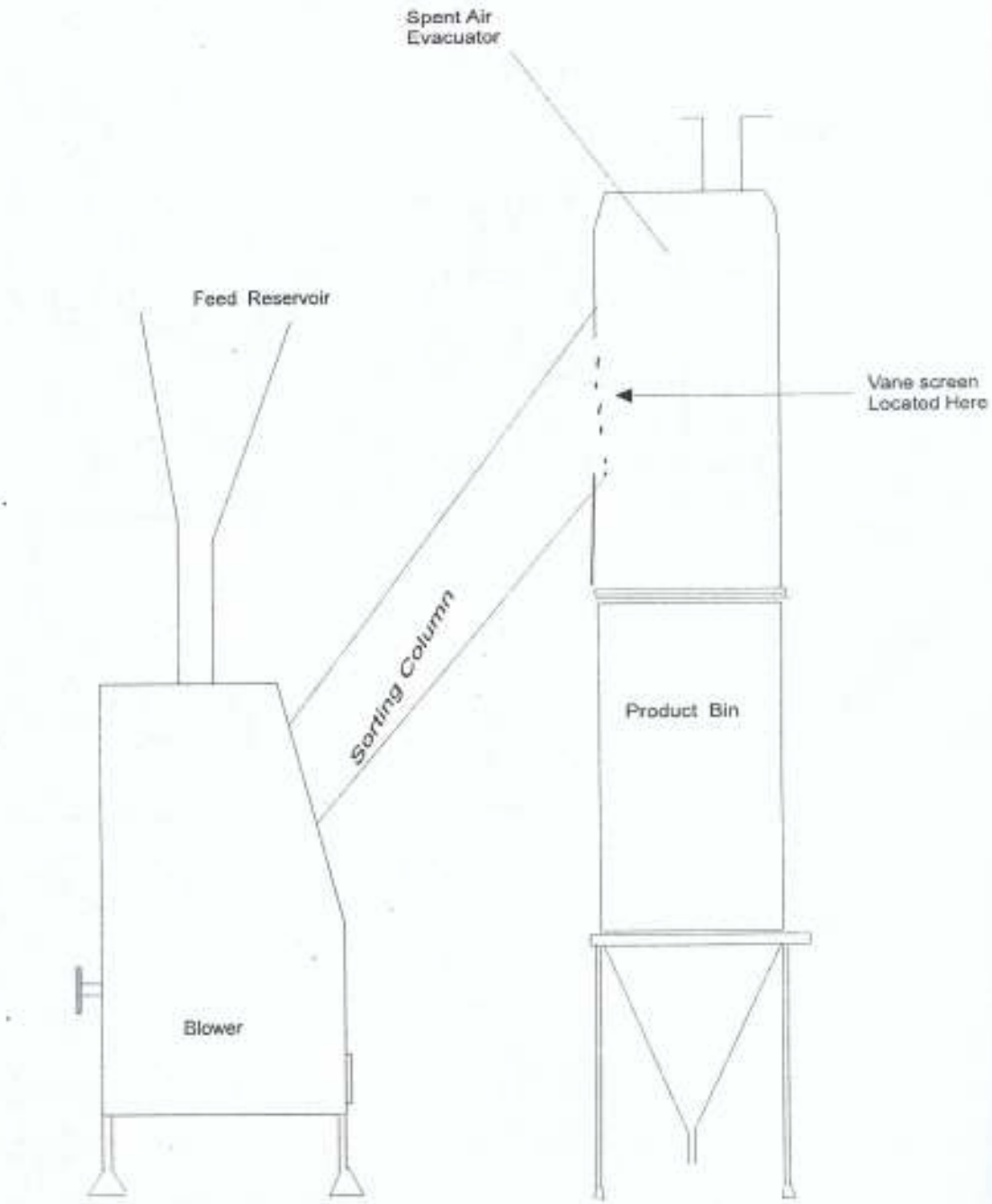
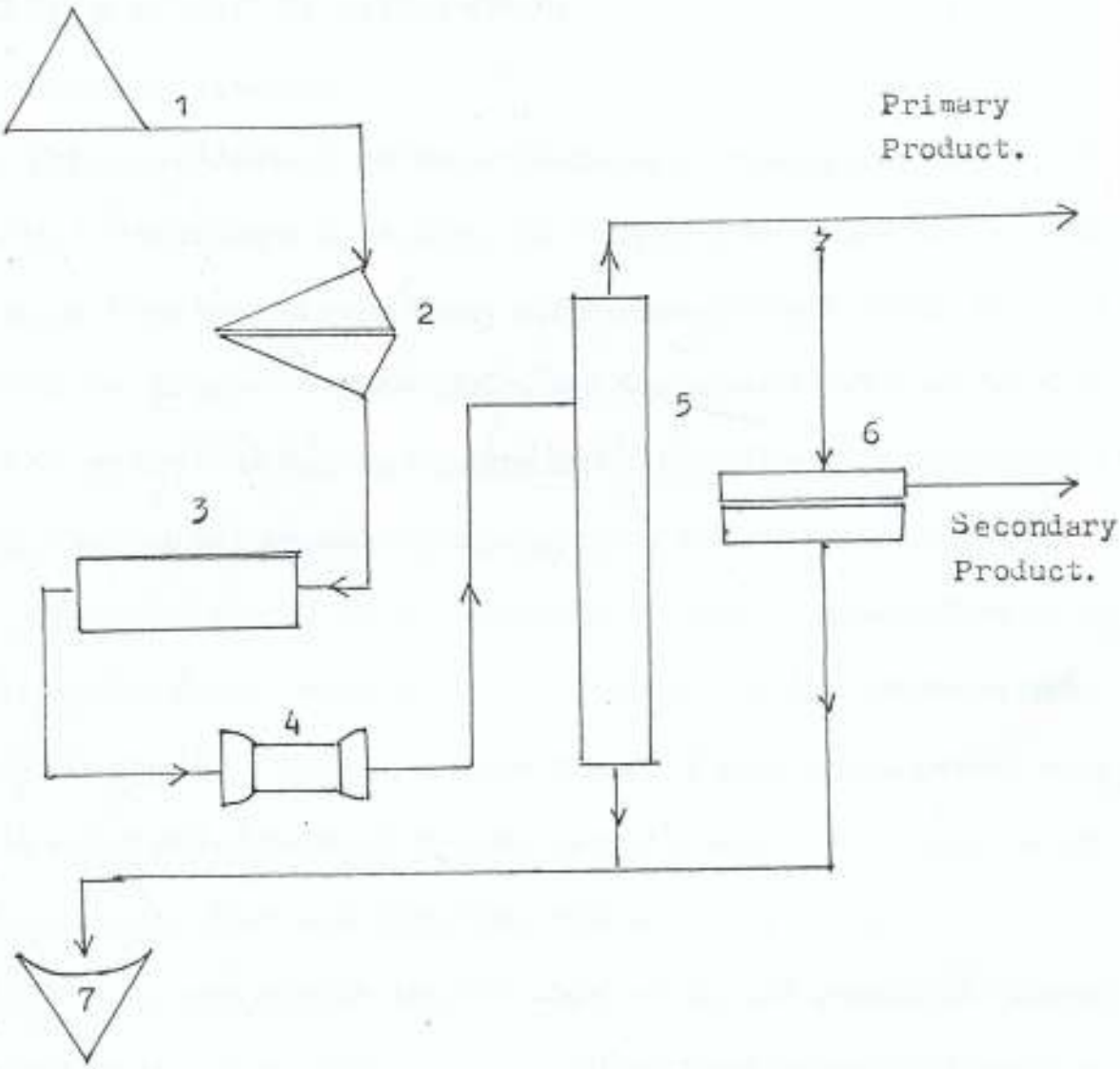


Fig. 2.3; A rough sketch of a dry classifier (courtesy Kavitex Industries Ltd.)



1. Clay pile
2. Crusher
3. Oven (possibly tunnel)
4. Mill
5. Classifier
6. Magnetic Separator
7. Waste Pit

Fig. 2.4 : A flowsheet for dry classification of Kaolin clay with magnetic Separator.

2.2.2 PROCESSING BY FLOATATION

Review of Principles

The most widespread and modern technique in mineral processing is froth floatation. This technique is commonly used because it can be applied to any mineral ore no matter how complex (Kelley and Spottistwood Wills, 1982). Floatation technique was developed in 1906 (Kelley and Spottistwood, 1982) and has since then been applied to all minerals including kaolin clays. However, the technique is usually employed as a secondary processing option after classification.

Floatation is simply a selective separation of mineral components from an ore containing other minerals in the same pulp by floating the desired component preferentially. Its principle is based on selective adhesion of some mineral particles to air and the simultaneous adhesion of the other particles to water when conditioned with necessary reagents in the same pulp (Wills, 1982;).

Naturally, most minerals adhere to water and thus will always sink. But the hydrocarbons and some other organic compounds have the peculiar property of adhering to air and thus, always float in water. This property of differential adhesion is utilized in developing various floatation reagents which are used in making selective separation. Floatation reagents are usually grouped as promoters or collectors, activators, depressors and conditioners based on their functions.

Materials Preparation And Method in Kaolin Clay Floatation.

All clays are generally treated alike in floatation. In order to reduce cost and increase production rate floatation should be employed as a secondary processing technique in kaolin clay recovery (usually after classification) since it is only applied to remove colour through the removal of oxides of iron (Forbus et al, 1993) Veglio et al, 1993). Thus before floatation is carried out, the as-mined clay is first classified and the product of classification is then subjected to floatation technique.

Floatation can be done in either of two ways depending on which one is economical or which reagents are available. First, the impurities (iron oxide) can be floated and the clay left in the pulp.Secondly, the clay itself may be floated leaving the impurities behind.

Materials preparation will therefore have to take these alternatives into consideration. The first option is better when there is only one component present as impurity. This is because the proportion of the impurities is always far less than the quantity of clay in the charge, and a particular charge will only be treated once. However, if there are many components present as impurities, employing the first alternative will require that a particular clay charge be treated as many times as the number of impurities present, since only one component of the charge may be floated at a time. In such a situation, the second alternative is better, that is, floating the clay and leaving impurities behind.

The method employed in kaolin clay floatation is the same with other minerals. As in all floatation processes, a high density pulp, containing about 75% kaolin is prepared from the classified clay. A depressant is added, depending on which

component is to be floated. If the iron oxide (that is impurity) is to be floated then the clay is depressed by the addition of sodium silicate. A collector for the iron oxide is then added after which a frother is also added and the pH of the pulp adjusted as necessary by the addition of suitable conditioners. Usually iron oxides are floated in an acid circuit with pH range between 3 and 5. The pulp is finally diluted to about 30% solid with the addition of clean water and then aerated to float the required component.

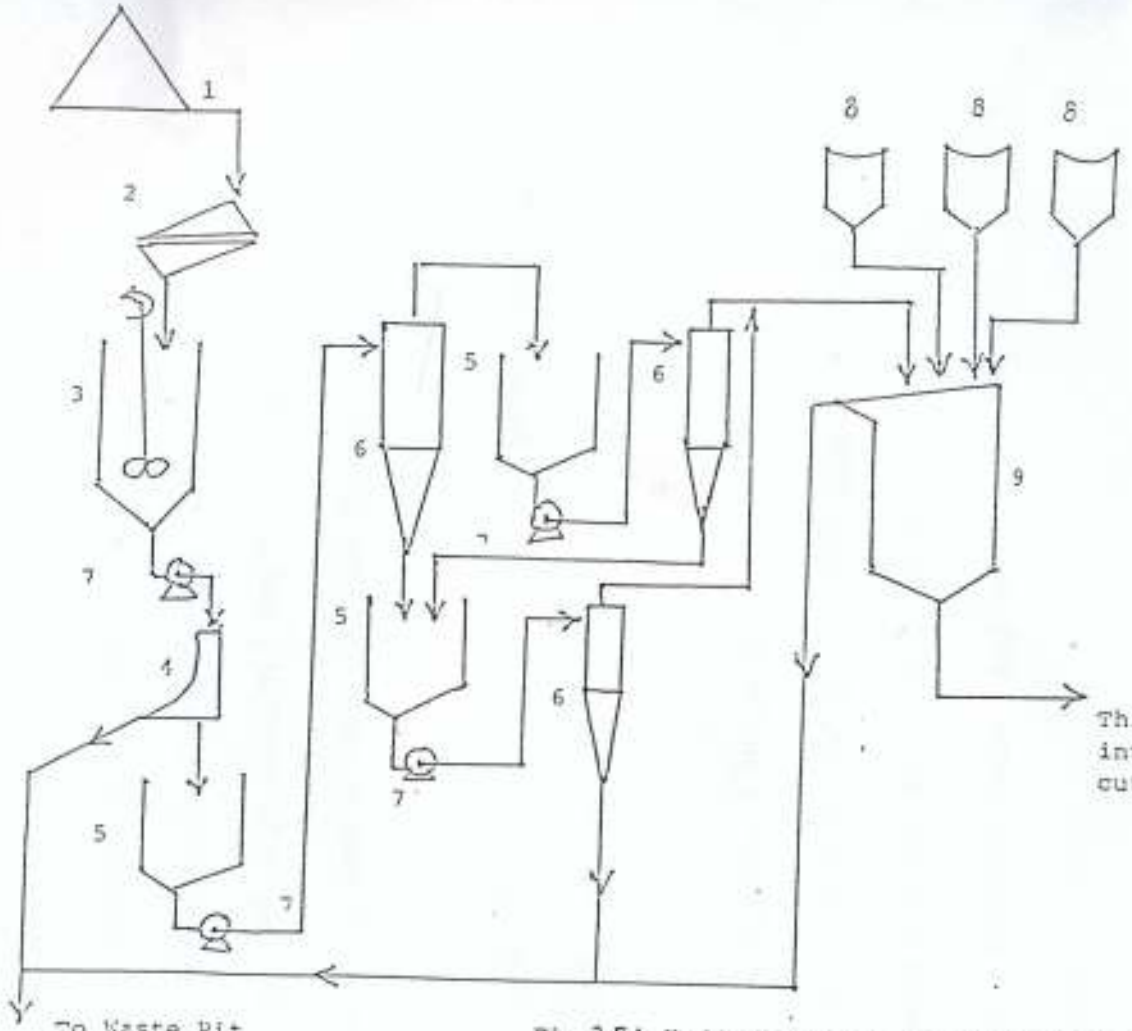
The appropriate reagents for floating any mineral and their dosage can be obtained from standard floatation handbooks. Clays are also usually floated in an acidic circuit at about pH of 3.

Floatation Equipment

There are various floatation equipment available for use in mineral processing and any of these can be adapted to kaolin floatation. Generally, all floatation equipment are made up of some essential features and there are no exception to kaolin beneficiation by floatation.

These features include:

1. A cell or tank whose volume depends on the designed production capacity.
2. An agitator or stirrer which helps to mix and condition the pulp properly. In most cases involving other minerals, all floatation reagents are added at the milling stage. In this case, kaolin floatation is different because it is necessary to first classify the clay before floatation.



1. Clay Pile
2. Crusher
3 Plunger
4 Spiral Classifier
5. Slurry tanks
6. Hydrocyclones
7 Pumps
8 Flotation Reagents meters
9 Flotation Cell

This product can be introduced into a classification circuit to cut into different size grades.

Fig. 2.5: Kaolin Processing with flotation for Secondary recovery. (Heinskanen, 1993, flotation added)

3. A source of aeration which introduces air into the pulp.

All other provisions different from these essential features are ancillary.

Figure 2.5 is a simple flowsheet for kaolin processing incorporating floatation technique for secondary purification (Heinskanen, 1993).

2.2.3 THE ELECTROSMOSIS PROCESSING TECHNIQUE

This is another wet processing technique that has been applied to kaolin processing. The principle of operation is similar to that of electrolysis.

In this technique, the as-mined clay is first crushed and then slurried with water to give a well dispersed pulp of about 40% solid. The slurry is then passed through sand throughs where silica and other coarse impurities are removed and the kaolin slip flows into channels where larger kaolin particles settle. The overflow from the channels contains fine-grained kaolin and this is fed into the electrosmosis machine where the final separation takes place simultaneously with dewatering. The large grained kaolin slip is also passed into the electrosmosis machine for the removal of the colouring impurities.

Purification in The Electromosis Machine.

The machine itself consists essentially of a semi-circular trough containing a half cylinder cathode grid and a centrally rotating drum which serves as the anode. The machine is fed with a direct current (dc) of about 170 amperes at 100volts in a sodium silicate electrolyte solution with the clay slip. As the current flows, The

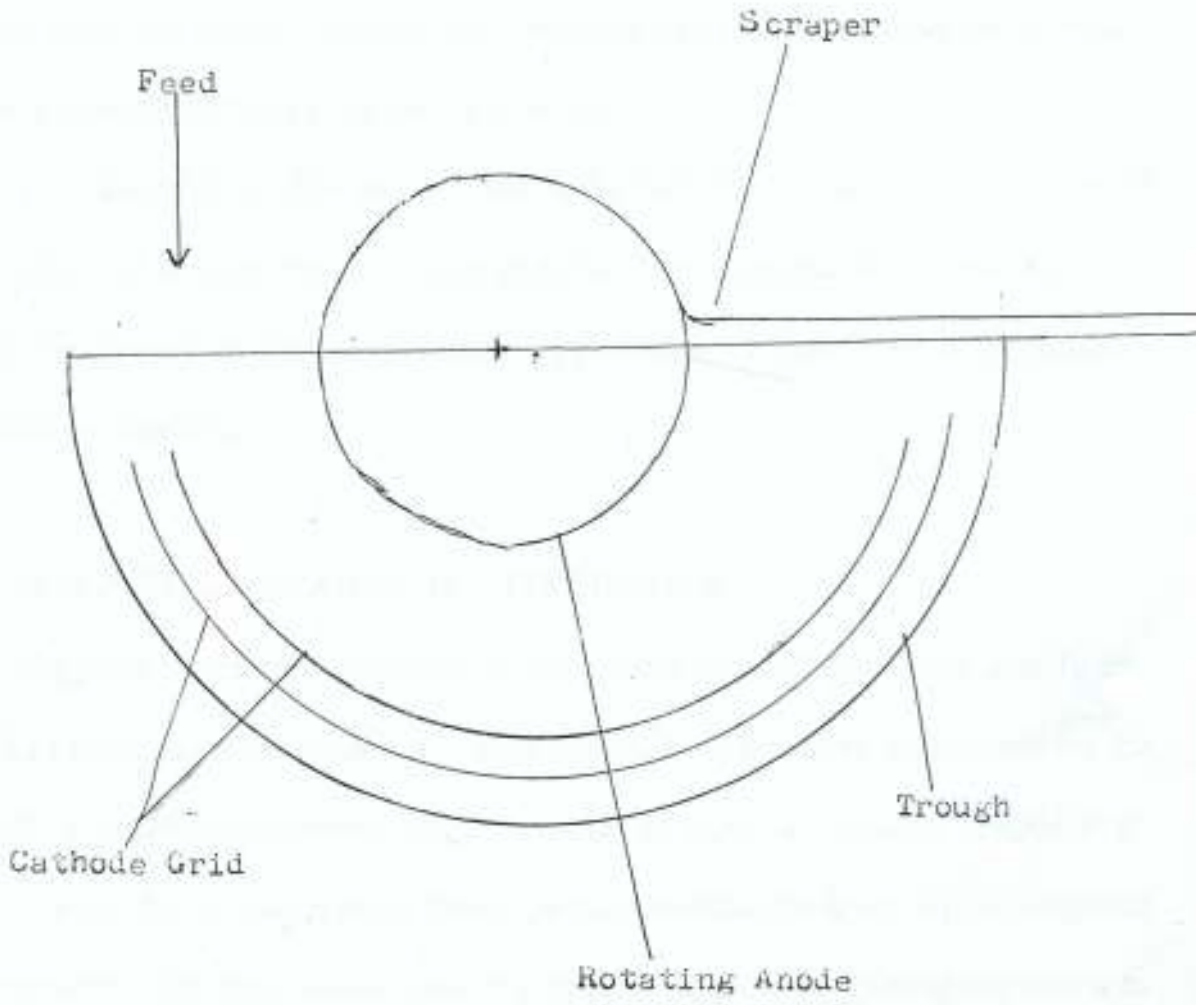


Fig. 2.6 : A simplified diagram of an Electrosmosis Machine.

electro-positive impurities such as iron, titanium oxide and others are collected at the cathode while the electro-negative clay attached itself to the rotating anode which carries it to scrapers where the clay is removed.

The advantage of this process lies in the fact that it produces two grades of kaolin clay and dewatering in a single process. The technique is however costly to employ because of the high consumption of electricity. Figure 2.6 is a simpliified elctrosmosis machine.

2.2.4 MAGNETIC SEPARATION TECHNIQUE

Magnetic separation technique is one of the physical processes usually employed in kaolin clay beneficiation. This technique is however useful only for the removal of magnetic impurities such as oxides of iron and titanium (Veglio et al, 1993). Hence the technique alone cannot be used to attain the level of purity required for most industrial applications since it will not remove such gritting impurities as silica, mica, and other non-magnetics. Thus, it is usually employed as a secondary processing technique after primary degritting by classification.

The process can be carried out wet or dry. It is the only technique yet known by which kaolin clay processed or degritted by dry classification can be discoloured. Figure 2.4 shows a kaolin dry purification process incorporating a magnetic separator.

Principle of Magnetic Separation

As with magnetic separation for other minerals, a dry kaolin feed or a slurry is passed through a magnetic field where the magnetic components are removed and the clay with any retained non-magnetic impurities are carried away from the magnetic field to a product bin.

Usually a high intensity magnetic separator is used. This technique is applicable in kaolin processing only when colouring due to the presence of oxides of iron and titanuim is involved because kaolin clay itself is non-magnetic. Any of the numerous magnetic separator, used in mineral processing can be adapted to kaolin purification. The continuous wet magnetic separators are especially very useful in this case. This is because removal of magnetic components and settling of non-magnetic gritting particles can take place simultaneously in the separator while the clay flows out. But for efficient separation, it is necessary that the clay is first degritted by classification since magnetic separation will not remove the non-magnetic and diamagnetic components. However, if the clay was degritted by dry classification, a continuous dry magnetic separator can be used. The advantage of this method is that it can be applied as the secondary processing technique after a primary wet or dry classification. Also a single equipment may be used to degrit and remove colour. Magnetic purification can be used to attain a high production rate since all features of the separators are usually designed for continous operation; and in case the separator uses permanent magnets, the additional cost of electricity due to the

use of electromagnets is removed and the technique can thus be one of the cheapest in kaolin clay recovery.

2.2.5 JIGGING TECHNIQUE IN KAOLIN PROCESSING

A Review of Principles

Jigging is a gravity separation techique that separates mineral particles on the basis of either specific gravity or size difference accomplished in a bed fluidized by a pulsating current of water (Wills, 1982). Although jigging is applicable in kaolin processing, but with the availability of efficient classifiers, jigging is hardly considered when clay processing is intended.

Jigging involves using a sieve of appropriate size to which the mineral is fed. Depending on the design of the jig, either the jig sieve is moved in a stagnant water or the water pulsates while the jig sieve remains stationary. Most modern jigs employ stationary sieve in a flowing and pulsating stream of water. There are also various equipment with vibrating, gyrating and reciprocating sieves. Jigging can only be employed for degritting in kaolin clay processing. Thus it is applicable only as a primary processing technique.

Feed Preparation And Method

Feeding may be done wet or dry. If wet feeding is intended, then the kaolin clay is first crushed and slurried to give a high density pulp of about 75% solid. The dense slurry is fed directly into the jig where it is diluted by the flowing and pulsating

water. If however the clay is to be fed dried, then a light grinding is necessary after crushing. The dry clay is fed to the jig where it is slurried and dispersed by the pulsating water. No special method is required because jigs are available for this purpose except an improvisation is intended. Since kaolin clay has particle sizes between 10 micrometers and 50 micrometers (Heinskanen, 1993, Bouiman et al, 1993), it is necessary that the jig sieve be around 52 micrometers. Jigging in kaolin processing should be done in a flowing and pulsating stream of water rather than stagnant pulsating stream. This is to allow the fine clay particles discharged from the jig sieve to flow away with the water current; otherwise they will move back into the jigging area in the upward stroke, if the water is stagnant.

However, production rate in jigging is far lower than in classification, and there is no possibility of cutting the clay slip into different size grades as in classification with centrifugal classifier. Thus jigging will not be economical for commercial kaolin clay recovery.

2.2.6 **OXIDE REDUCTIVE BLEACHING TECHNIQUES**

These techniques are generally applied in the secondary processing of kaolin clay in most industries. They are usually wet processing techniques and are essentially employed to remove colour from the clay and improve its brightness.

The aim of most of these techniques is the reduction of the iron oxide to a soluble form that can subsequently be removed from the kaolin pulp by filtration. The removal of the coarse gritting impurities such as silica and mica, has never

e any significant problem in kaolin clay processing. These impurities can be oved by simple differential settling-a principle employed in most classifica- processes. The most difficult processing problem in kaolin recovery is the oval of colours. These colours have been established to be due to the pres- e of iron and titanium (forbus et al, 1993, Veglio et al, 1993). Hence the re- val of iron is the most troublesome process in kaolin clay processing. Forbus l (1993) and Veglio et al (1993) showed that iron is usually present in the form ematite (Fe_2O_3) and sometimes as jarosite ($AFe_3(SO_4)2\ (OH)_6$ [A: K^+, Na^+, It is further shown that in most cases when iron is responsible for buration in kaolin, it is present in the form of oxide with the oxidation state of hat is, as ferric ion (Fe^{3+}) which is insoluble in water and thus difficult to ove. However ferrous ion (Fe^{2+}) is soluble in water and usually gives a green our (Kreshokov and Yaroslavtsv 1979). Thus the aim of most of the reductive cesses is to reduce the insoluble ferric ion (Fe^{3+}) to the soluble ferrous form $^{2+}$) which is subsequently removed with the process water.

Most of the useful methods of bleaching are chemical processes. hough such physical processes as magnetic separation and floatation tech- ue, have also been used to remove colour from kaolin clay but their products not as satisfactory as those of the reductive bleaching processes. Some of reductive processes include: reductive bleaching with zinc hydrosulphite, h sodium hydrosulphite, sodium silicate, chlorine, iron powder and sulphur xide gas and a

number of organic substances. These processes are well described in literatures (Forbus et al, 1993, Veglio et, al 1993, Crudev et al, 1978 and Brin and Eremin , 1969). Most of these processes reduce the ferric ion (Fe^{3+}) to Fe^{2+}) such that the following equation is satisfied.

$$Fe^{3+} + xA + \ldots\ldots \longrightarrow Fe^{2+} + yB + \ldots\ldots\ldots\ldots \quad (3)$$

Some of these methods are briefly discussed here.

Reductive Bleaching With Zinc Hydrosulphite.

Forbus et al, (1993) showed that this technique was the major bleaching method for kaolin before the 1970s. The reductive subtance is the sulphite ion in the zinc hydrosulphite. The process involves the production of zinc hydrosulphite on site. This is done by the addition of zinc dust to sulphurous acid. The acid itself is prepared by passing sulphur dioxide gas into water. The zinc hydrosulphite so produced was then added to an unbleached classified kaolin slurry in an acid medium to solubilize the ferric iron and produce an iron-free clay. The process is said to have been abandoned in the 1970s due to the environmental concerns about zinc in waste water.

Reductive Bleaching With Iron Powder And Sulphur Dioxide Gas.

(In-situ Bleaching).

This process was developed and patented by Engehard in the 1970s after the unresolved problem with the removal of iron hydroxide by-product form his former process involving the use of iron hydrosulphate (Forbus et al, 1993). The process

described as an in-situ process because all the materials were added directly to e unbleached slurry at the site. The materials used in this technique are the nbleached (but already classified) clay, iron powder, sulphur dioxide gas, alum nd sulphuric acid. The technique involves preparing an unbleached slurry of bout 30% solid from the classified clay. The iron powder is mixed with a small mount of slurry (already bleached if possible) and added to the slurry to be eated. Alum is also added and the pH adjusted to betweeen 2.5 and 3.0 with e addition of sulphuric acid (Forbus et al, 1993). The sulphur dioxide gas is nally passed into the pulp for about an hour while stirring.

Forbus et al, (1993) showed that the reaction taking place is the formation f iron hydrosulphite in the slurry as shown by the following equation.

$$Fe + 2SO_2 \longrightarrow FeS_2O_4 \qquad (4)$$

But if iron is present in the form of hematite (or any oxidation state of 3) en, the hematite is ionized in the acid medium and reduction can be also take ace according to the following equations:

$$Fe_2O_3 + 6H^+ \longrightarrow 2Fe^{3+} + 3H_2O \qquad (5)$$

$$H_2O\,(1) + SO_2\,(g) \longrightarrow H_2SO_3\,(aq) \qquad (6)$$

$$H_2SO_3 \longrightarrow 2H^+ + SO^{2-}{}_3\,(aq) \qquad (7)$$

$$2Fe^{3+}\ 3H_2O + SO_2^- \longrightarrow 2Fe^{2+} + H_2SO_4 + 2H_2O \qquad (8)$$

Effects of the Various Factors

The particle size of the iron powder was found to affect both the ightness of the final bleach and the reaction time. While maximum brightness otential of 86.8

was reached in 16 hours with a 1500 micrometers iron powder, a 44 micrometer powder reached the same brightness potential in less than an hour of the reaction (Forbus et al, 1993).

The optimum quantity of iron required is said to vary with the type of kaolin clay being processed; but generally, it is said that between 0.2kg/ton and 0.5kg/ton are sufficient for any type of kaolin clay. Excess of iron is said to suppress brightness and that the suppression can be overcome with the passing of more sulphur dioxide gas.

Sulphur dioxide gas is the source of the sulphite ion which is the main reducing agent in this process. Its optimum level is said to vary between 4.5kg/ton and 6kg/ton. sulphur dioxide gas is also said to cause a rapid pH decrease. The major function of the acid is for pH regulation and as flocculant. It also enhances the ionization of the iron oxide in solution. The most effective pH range for this process is between 2.5 and 3.0 (Veglio et al, 1993).

The function of alum is to flocculate the clay and thus increase production rate through reduction of solid loss. It also helps to reduce the level of sulphur dioxide gas used. About 2 to 3 kg/ton of alum are said to be sufficient for optimum performance. It is also said to be necessary for brightness. The in-situ bleaching technique can easily be installed as part of any kaolin clay processing system, since it does not require the construction of a separate and elaborate chemical plant to produce the reducing agents. Figure 2.7 shows a kaolin recovery flowsheet incorporating the in--situ process. (Veglio et al, 1993).

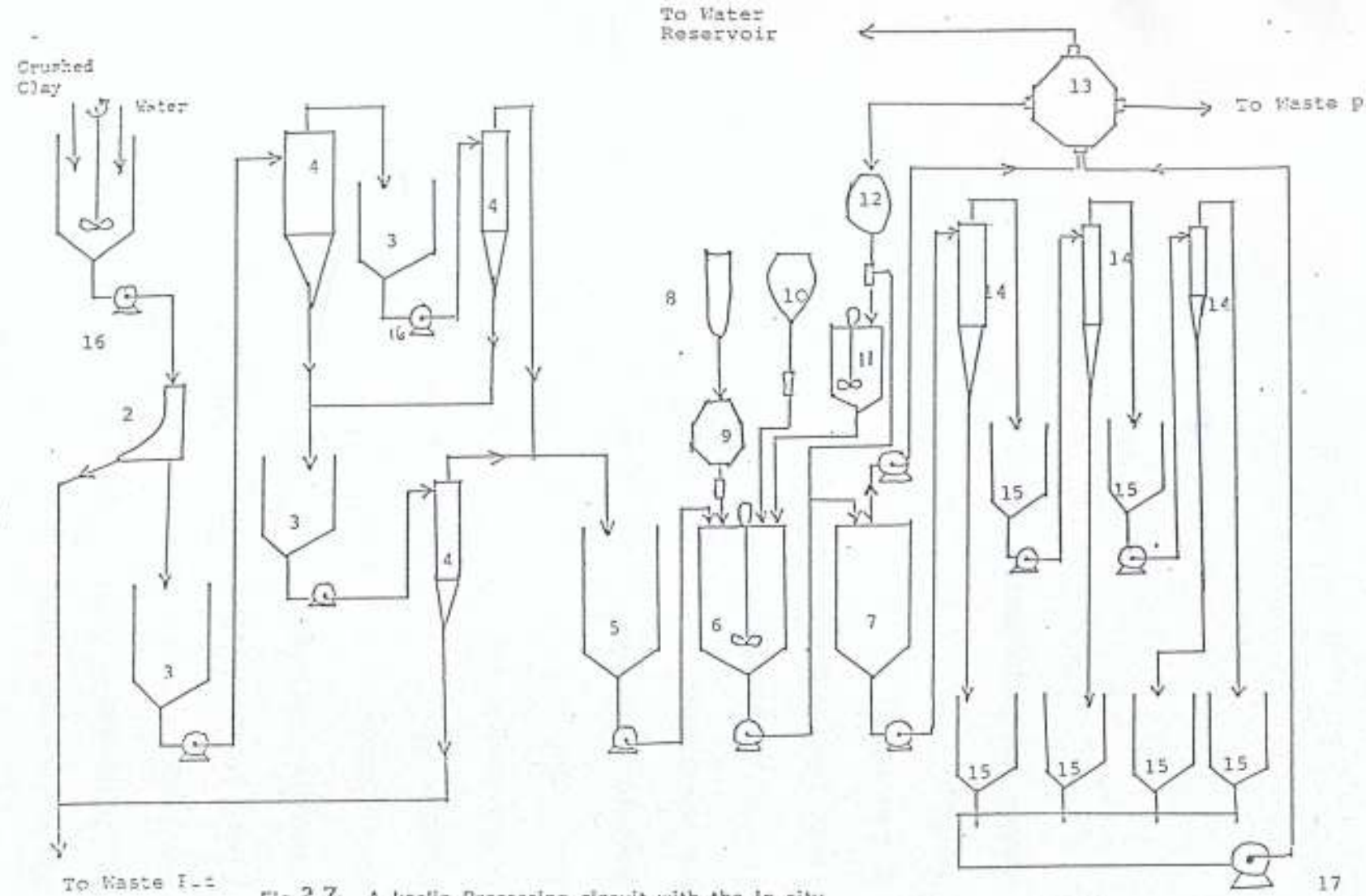


Fig.2.7: A kaolin Processing circuit with the In-situ Reductive Bleaching process. (Heinskanen, 1993 Forbus et al, 1993)

Components of Figure 2.7

1. Slurring tank with plunger
2. Spiral Classifier
3. Slurry tanks
4. Primary Processing Hydrocyclones
5. Unbleached Slurry tank
6. Bleaching tank
7. Washing tank
8. Sulphur dioxide Gas
9. Vapourizer
10. Sulphuric acid meter
11/12 Iron power meter
13. Material recovery unit
14. Grades processing cyclones
15. Product tanks
16. Pumps.
17. General Dewatering pump.

2.2.6 REDUCTIVE BLEACHING WITH ORGANIC SUBSTANCES

All the reductive bleaching processes earlier mentioned employ in organic substances as reductants. However organic materials have also been used in some kaolin clay processing techniques and the number of organic materials that can be used are many. Veglio et al (1993) listed such materials as molasses, hydrolyzed cellulose and other agro- industrial wastes.

A similar process was undertaken in this study and described fully in Chapter three.

2.2.7 MICROBIOLOGICAL PROCESSING TECHNIQUES

Microbiological processes are some of the recently developed techniques for removing colour from kaolin clay. These processes involve the use of heterotrophic bacteria and yeast as reductive substances (Toro et al, 1992; Veglio et al, 1993, and Grudev et al, 1978). The mechanism of reduction may be similar to that of the use of organic materials or to the use of algaes in removing some heavy ions from waste water.

However there are said to be some unresolved problems about the industrial application of these techniques; so they are not commonly applied in the industries today (Veglio et al, 1993).

2.3 DEWATERING IN KAOLIN PROCESSING.

Dewatering is the process of removing water from the final kaolin slurry. Most industrial kaolin recovery plants employ wet processing techniques though there are

recovery process from the primary stage through the secondary stage. Although some end users purchase the processed clay in slurry form (Bowman et al, 1993), most users require the mineral dry, hence majority of the kaolin processing plants bagged their products dry. In most processing plants, the final slurry may contain only about 40% solid and 60% water. Dewatering is therefore an important process in kaolin clay recovery (Bowman et al, 1993)

2.3.1 Methods of Dewatering.

Dewatering can be carried out in a number of ways. Such methods include filtration, decantation and evaporation. Among these, filtration is almost exclusively used in kaolin clay processing. Decantation is not used because it is slow, crude and results in the loss of much solids. Evaporation is not applicable because of the large volume of water usually involved and the possibility of leaving some salts and metal ions in the final products since only water and the volatile components will evaporate during heating. Hence filtration is the only applicable method. Filtration helps to remove all soluble salts and metal ions from products .

With proper treatments of the clay slurry, filtration can be sued to achieve a high production rate although it also results in loss of some solids especially when filtering ultra-fine clay particles. Thus filtration is usually the rate controlling step in most kaolin clay processing plant (Bowman et al, 1993). Total dewatering in kaolin recovery is not usually achieved by the filtration process alone since the product must be bagged dry (with minimum moisture content) Hence "drying is usually employed after filtration in some plants.

2-3-2 Filter Aids in Kaoling Dewatering

Kaolin clay is said to have some of the smallest particle sizes in the mineral kingdom (Bowman et al, 1993; Heinskanen, 1993 and Pearl 1967). Though this small particle size makes kaolin separation form most of its coarse impurities easier and faster; the same factor (small size particle) results in loss of recovery especially during dewatering since the particles can easily pass through small openings and filter cloths in the filtering machine (Bowman et al, 1993).

To alleviate this production limiting problem, various filter aids are often used. These aids are agents usually added to the clay slurry to enhance filtration performance and thus increase production. The primary functions of these agents are: flocculation, coagulation, thickening and combination of these.

Until recently, alum was the main filter aid commonly used. Acids (especially sulphuric acid) have also been used. The function of both alum and the acid or a combination of them as filter aid is principally flocculation, which keeps the particles from passing through filter cloth. Various chemicals have also been developed (Bowman et al, 1993). These filter aids are said to be capable of increasing production rate by between 32% and 55% through increased filtration performance (Bowman et al, 1993). The primary mode of functionality of the Nalcofiltermax products is coagulation mechanism

Some floatation reagents are said to have also served efficiently as filter aid in kaolin dewatering. Examples are Aeromine 3035 and 3037 and the Aeropfloes, all

produced by the American Cyanamid Company, USA.

2-3-3 Dewatering Equipment

Many dewatering equipment are available in mineral processing but only filtration and drying equipment are usually employed in kaolin recovery

Filter machines commonly used in kaolin dewatering are continuous vacuum filters, pressure filters or filter presses. In most cases too, the centrifuges or cyclones used in the clay recovery are also used in dewatering. Any of the drying equipment that can be adapted to the particular plant may be used but the temperature must not be more than 120°C.

CHAPTER THREE

EXPERIMENTAL MATERIALS, EQUIPMENT AND METHODS

-1 MATERIALS

Kaolin clay recovery in this study was carried out in two sequential stages, amely the primary processing stage and the secondary processing stage;

-1-1 Material For Primary Processing

The only material used in this processing stage is the as-mined kaolin clay. lay materials were collected from three locations: Akure, Ifon and Ijero. The on clay is more-coarse to the touch as a result of the large size of its silica ontent. The Akure clay is more unctuous to the touch because its silica and her coarse impurities are of smaller sizes than those of Ifon and Ijero.

Akure clay is light-red as mined but becomes red when slurried. Ifon clay yellowish-white or white with some yellowish inclusions but turns pink when urried, and Ijero clay which appears pink as mined becomes light red in slurry. he chemical analysis of Akure clay was done using the wet-leach technique vhich involves leaching the different chemical components from a solution of e ore using appropriate reagents) while that of Ifon was obtained from a previ- us study carried out by the Delta steel Company Aladja.

1.2 Materials For Secondary Processing

The materials used in this stage of the recovery are:

) The classified or degritted kaolin clay from the primary processing stage.

) Common edible cassava starch, purchased wet and prepared as described under materials preparation

(3) Sulphuric acid; Commercially available technical grade was used in this study Hydrochloric acid was also used in this process and this is also commercially available in different grades.

3.1.3 Materials Preparation

For the classification process, the clay was only crushed in a laboratory pulverizer to small lumps for easy slurrying, but for jigging, the crushed clay was ground lightly in a ball mill because it was intended for dry feeding. The classified clay for the secondary processing stage was dried in the oven at a temperature of about 115°C. The cake so obtained was ground in the mortar to powder form for easy weighing and slurrying

The cassava starch used as reducing agent in the secondary processing was also dried in the oven and then crushed to powder in the mortal. 300 grams were hydrolysed in an autoclave at 121°C for 90 minutes with sulphuric acid. The resulting solution was allowed to cool and excess calcium carbonate powder was added to consume the remaining acid. The filtrate was then decolourised with activated carbon, evaporated at atmospheric pressure and then cooled to collect the crystal. At the completion of the reaction excess acid was removed by reaction with calcium carbonate, the solution was then evaporated or heated to dryness. The remaining cassava starch was sieved to remove any lumps. This non-hydrolysed starch was used directly as required.

3.2 EQUIPMENT AND METHODS

Two experimental techniques namely classification and jigging were used at

ductive bleaching process was employed. For the classification process, a Denver 8" centrifugal classifier (a centrifuge with four speeds) was used. The essential features of the machine are shown in Figure 3.1 and the operating principle involves the use of centrifugal force produced by an impeller to separate a feed into two size fractions - an overflow and an underflow. The overflow consists mainly of clay and some fine impurities. The underflow contains the heavy particles with some amount of clay.

The important parts of the classifier are the classifying column in which classification takes place. The column consists of two parts the upper cylindrical part and the conical base which carries the apex opening for the underflow outlet. There is also the overflow compartment which is also cylindrical but made a little taller than the classifying column the overflow compartment slopes round the circumference of the classifier with a drain hole located at its lowest point, through which the overflow materials are collected. The impeller is located inside the conical base of the classifying column with only little clearance from the wall.

As the feed is introduced into the classifying column, the coarse materials are thrown to the wall where they slip down and escape through the apex opening of the conical base. The clay and some fine impurities however whirl up and overflow the circumference of the classifying column into the overflow compartment where they are drained out.

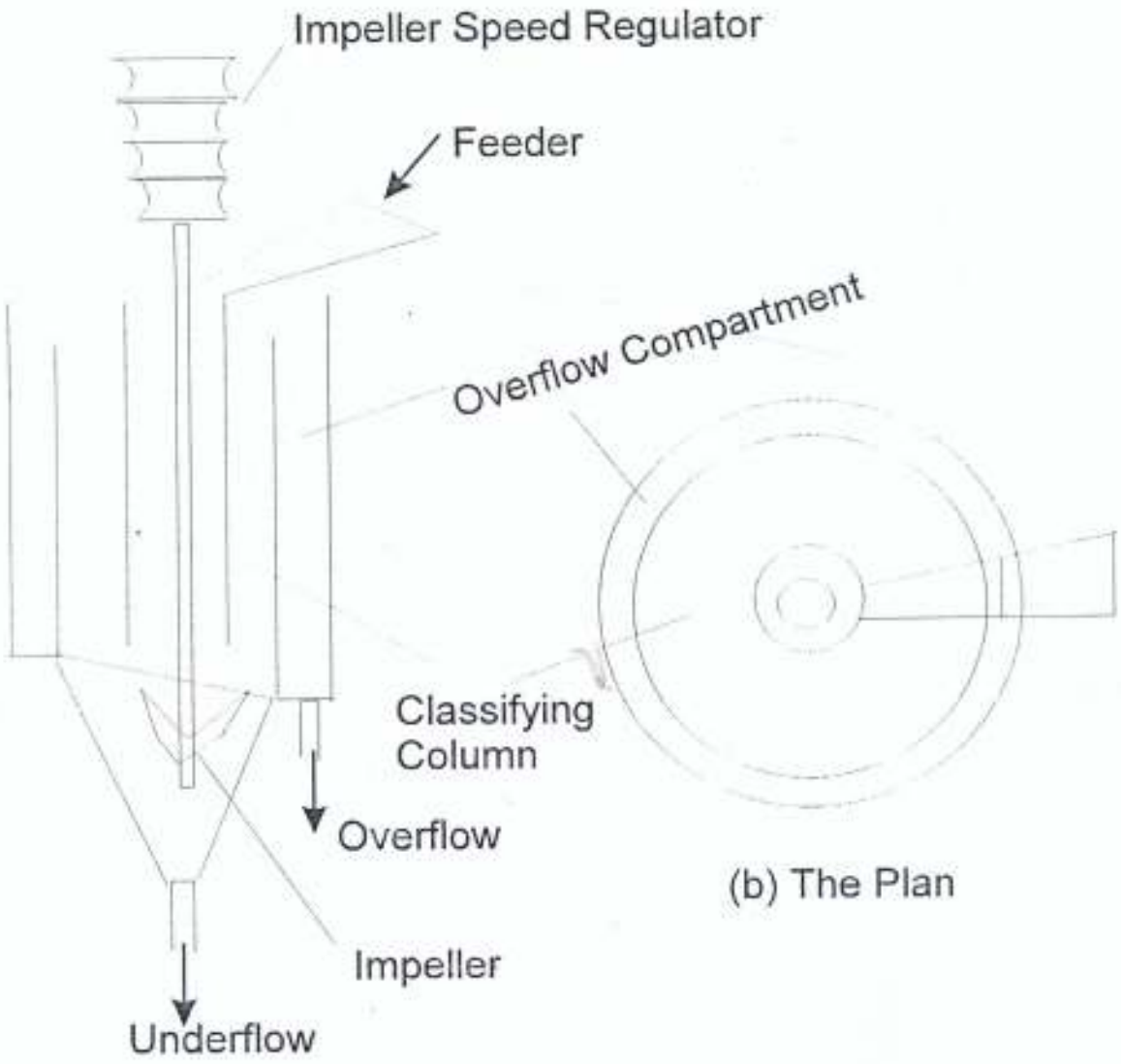


Fig. 3-1; Functional feactures of the Denver Centrifugal Classifier.

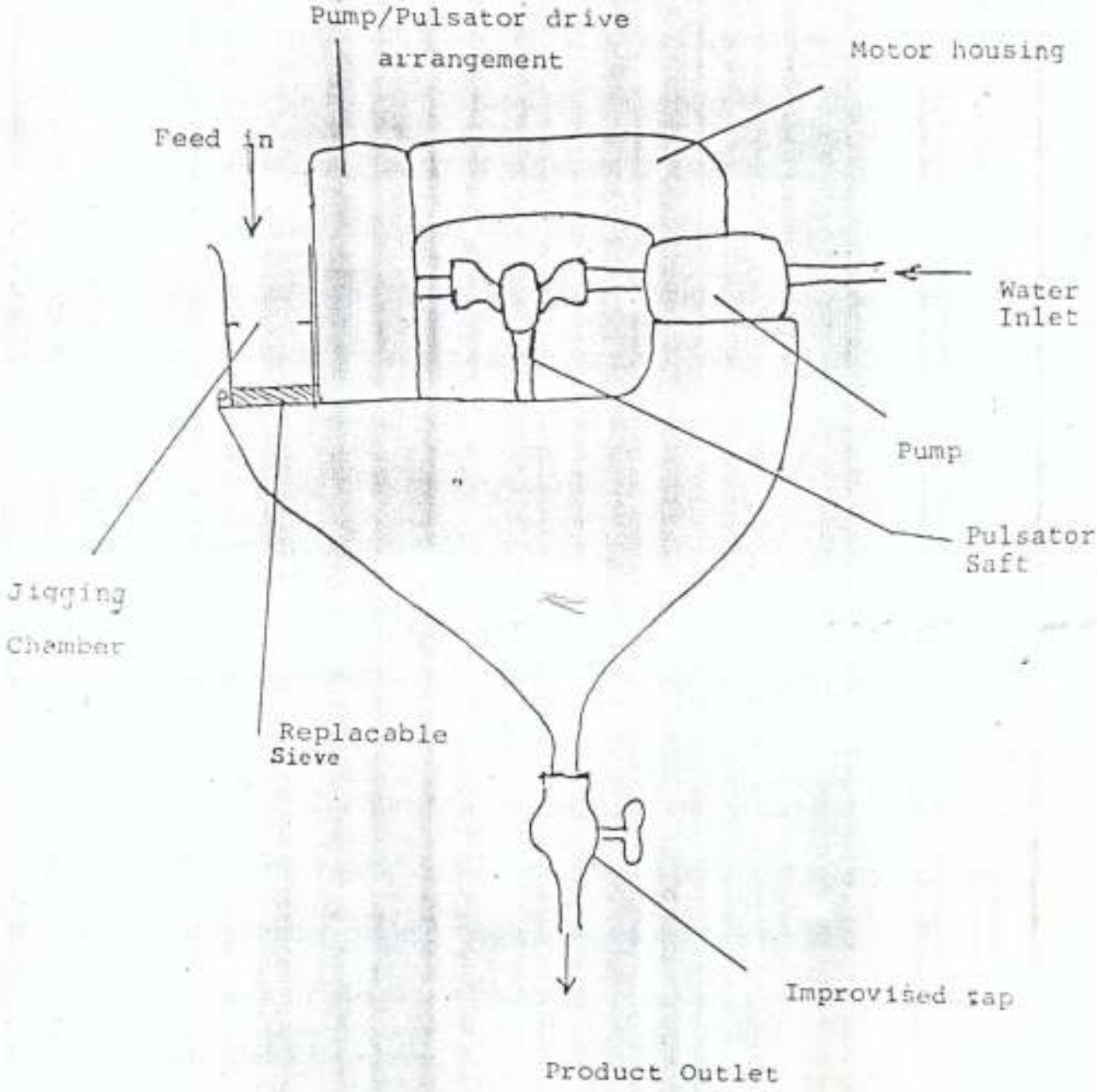


Fig. 3 • 2: Functional Parts of the Denver Mineral jig.

Jigging was done with a Denver simplex mineral jig. The functional part of the machine (shown in Figure 3.2) consists of jigging chamber containing a sieve holder which can be fitted with a sieve of the desired size and the pump which draws water into the jig for use of the pulsator. The pulsator is driven by an electric motor and it causes the water to pulsate or move up and down thereby separating the fines and discharging them through the sieve. There is also the underflow compartment with an outlet for the collection of water and any discharged material. Crushing was done in a laboratory pulverizer while grinding was carried out with a ball-mill both located at the mineral processing laboratory of the Federal University of Technology Akure.

The oxide reductive bleaching process employed in the secondary processing stage of this study was undertaken in a 250 ml beaker at atmospheric pressure. All pH measurements were done at 30°C with a Jenway Model 3015 digital pH meter. Temperature was measured throughout with a mercury-in glass thermometer with 100°C maximum point and -10°C minimum point. Heating was carried out with a Clifton model MSH-L electric hot plate with magnetic stirrer and temperature regulator. Sample weighing was done with a Metler AE 160 digital balance which weighs to four places of decimal.

3.2.1 Experimental Procedure

By Classification

One kilogram of the crushed clay was slurried with water using a multi-speed plunger unit1 there were no lumps of clay in the slurry. The slurry was then diluted to about 20% solid and fed into the classifier from a side reservoir which was agitated continually by stirring. The classifier speed was varied for

ach kilogram of clay processed and the completion time and coarse recovery in the nderflow in each case were measured and recorded.The nature of the overflow and nderflow materials were also observed with regards to relative volume, amount of lay and impurities. The results of measurements and observations made for aver-ge of three runs during the classification process are shown on Table 4.1

By Jigging

During jigging, the lightly ground clay was used. A sieve of size 53 micrometers was fitted into the sieve holder and measured quantities of the clay were placed n the jigging chamber. The outlet of the machine was improvised with a tap which vas closed at the beginning of each jigging operation. When the pulsating water had aturated the feed in the jigging chamber, the tap was opened to collect the underlow. Each jigging was continued until clear water started to come out from the jig.

The completion time for jigging each charged quantity of clay was measured nd the coarse materials retained an the screen were collected, dried and weighed. The quantity of clay charged, the observed completion time and amount of retained articles are shown in Table 4.2

By Reductive Bleaching Process

For each experimental run carried out in this process, 20 grams of degritted lay were slurried with 150ml acid solution in a 250ml beaker at different pH values, anging between 0.00 and 1.00 (Veglio et al, 1993 used pH as low as 0.20 in a imilar process.), varying addition of hydrolysed and non-hydrolysed starch, and the nixtures were

heated at different temperatures while stirring.

The Measure of Response

The measure of response in this process is the extent of iron removal by solubilization and this may be done in either of two ways.

(1) By direct measurement of the concentration of the Ferric and Ferrous ions in solution at different stages of reaction, or

(2) By measuring the brightness of the sample as the reaction progresses, that is by observing colour changes during reaction

In this study, the later method was adopted and colour changes were observed during the reaction process and time for reaction to reach "completion " determined. The colour of the sample being treated was observed to change from tis initial red, through milky to light-grey and then finally black. Samples were washed at these different stages. The samples from the black pulp remained black or dark, the milky colour sample gave a dull white product, while the light-grey sample produced a bright-greyish-white powder which is considered the best among the three products.

The iron content of the unbleached sample as well as those of the different process stages were determined with an atomic absorption spectrophotometer. The results obtained are given in Table 4.6

The light-grey colour stage of the process was then chosen as the **"visible completion"**stage and the time for the pulp to reach this completion stage is the **measure of response** in this process. But preliminary runs showed that this visible

completion state is suppressed at some pH , temperature and starch addition, so that the pulp changed almost directly from red to a dark-grey colour. Hence for the purpose of optimization, all reactions in this study were continued until the pulp turned dark.

The process was first run with all three clay samples at pH 0.25 and 0.6g of hydrolysed and non-hydrolysed starch in 150ml acid solution containing 20g of clay. The purpose of this was to determine whether the technique is clay-specific or not . The response measured is the time taken for the pulp to change colour from red to the visible completion stage. The results are shown in Table 4.3 The responses of the three clay samples showed that this technique is not clay-specific. It can therefore be applied in the recovery of any kaolin clay coloured or contaminated by iron. In view of the large amount of data that will be required for optimization and the large volume of acid that will be used, all three clay samples could not be used separately in the rest of the experimental runs. Hence Akure clay which has the poorest quality among the three was chosen for the rest of the study.

The effects of the main factors such as pH, temperature and the quantity of starch (hydrolysed or non-hydrolysed) on this bleaching process were determined. This is to identify the most significant factors or variables and their optimum values on the process. The results obtained are given in Tables 4.8,4.9, and 4.10 The effect of stirring was investigated separately by varying the stirring rate and measuring the completion time. The result obtained from this is shown in Table 4.7

4.3 ACID RECOVERY AND RECYCLING

The assumed equation for reduction in this process shows that the final process fluid still contains the acid component in solution as indicated by the presence of the hydrogen (H^+) in the product of reduction

$$24Fe^{3+} + C_6H_{12}O_6 + 6H_2O \longrightarrow 24Fe^{2+} + 6CO_2 + 24H^+ \qquad (9)$$

It is therefore possible to recover the acid used from the final process fluid. As a matter of fact, the equation indicates that the final process fluid will be more acidic than the initial pulp since the water in the pulp is decomposed to produced more acid components. The process of acid recovery has been suggested for further studies

However, the filterate from the bleach with the acid can be recycled without actually recovering the acid alone if only the solubilised materials especially the ferrous ion Fe^{2+} - can be removed from the filterate. Removal of soluble metals and metal ions can be done by passing the filterate from the bleach through an ion exchange column or use, an exchange resin. The resulting fluid then contains an ion from the exchange column or resin (which should be unreactive in the bleaching process) The resulting fluid can then be recycled to fresh bleaching tank and the pH adjusted as necessary.

Also since the ferrous ion in the filterate is soluble and will not interfere with the bleaching process (since itself is a product of the process), the filterate can be diluted with water, the pH adjusted as desired and recycled for use in fresh bleaching

3.4 CUTTING THE BLEACH INTO GRADES

In commercial kaolin clay processing cutting the final bleach into size grades is usually done with the aid of various small diameter centrifugal classifiers (especially the hydrocyclones). These classifiers can also be used in the laboratory if they are available.

In this study however, the final kaolin bleach obtained from the secondary process was successfully cut into different size grades using "timed settling" In this process, the kaolin slip was cut into size grades by allowing the particles to settle naturally in water after being uniformly dispersed. The slurry was then cut at different time intervals. Naturally, the larger particles settle first.

A slurry containing about 40% kaolin was prepared from the bleached product. Five 500ml beakers were used for the cutting with a separating funnel. The slip was well dispersed by shaking and then allowed to settle for five minutes after which the funnel was tapped into one of the beakers. More water was added to the slip in the funnel, dispersed, allowed to settle again before tapping. The procedure was repeated for the remaining beakers at time intervals of 15 minutes, 25 minutes and 35 minutes, and the remaining slip was washed into the last beaker. All products were allowed to settle overnight before careful dewatering and drying. The textures of the products showed that they were of different size grades.

CHAPTER FOUR

RESULTS AND DISCUSSION

4.1 RESULTS

The results of all studies described in chapter three are shown in Tables 4.1 to 4.11

Tables 4.1 **Chemical Composition of Kaolin Clay Materials Used**

Clay Samples	As Mined Colour	Colour in Slurry	% Al_2O_3	% SiO_2	% Fe_2O_3	% CaO + MgO	% K_2O	% LOI
Akure	Light-red	red	39.96	44.07	1.45	1.02	0.25	14.25
Ifon	Yellowish-White	Pink	36.8	47.0	0.67	1.11	-	12.32
Ijero	Pink	light red	Not available					

Table 4.2 **Recovery Pattern by Classification.**

Speed of Classifier	Recovery Pattern		Completion Time (Min)
	Underflow	Overflow	
1st	72.7% coarse with limited amount clay	Much clay with some coarse particles	11
2nd	77.06% Coarse with some clay	Much clay with coarse particles	12
3rd	78.46% coarse with increased amount of clay	Much clay with some coarse particles	17
4th	80.34% coarse particles with fairly high amount of clay	Reduced quantity of clay with some fine coarse particles	19

The underflow materials were washed and the coarse recovery dried, weighed, and expressed as a ratio of the course content of the feed to obtain the percent coarse recovery in this table.

Table 4.3 **Jigging Result**

Quantity jigged (g)	Jigging Time (min)	Amount of Retained particles (g)	Quantity. of clay recovered	% Recovery
50	3	18.62	31.39	62.76
150	6	5638	93.62	62.41
250	8	98.42	151.58	60.63
350	14	125.37	224.63	64.18
500	32	186.42	313.68	62.72

Table 4.4: **Completion time for runs with all three clay samples.**

Sample	pH	Temp ^{0}C	Starch (g/L)	Stirring (rpm)	Completion time (min)
Akure	0.50	90	3	300	48
Ifon	0.50	90	3	300	36
Ijero	0.50	90	3	300	45

Table 4.5: **Direct determination of pH at instant colour change**

Runs	pH	Temp ^{o}C	Hydrolysed starch g/L
1st	0.04	81	3
2nd	0.07	88	3

Table 4.6 Iron Content At Different Process Stage

Process Stage	Iron content (ppm)	% of unbleached	Iron Solubilized (ppm)	% of Unbleached
Unbleached sample	2430	-	-	-
Milky colour stage	857	35.26	1573	64.74
Light-Grey Colour stage	269	11.06	2161	88.94
Dark colour stage	226,	9.28	2204	90.72

Columns 3 and 5 of Table 4.6 are obtained by expressing the iron content at different stages of the bleaching reaction as percentage of the iron content in the unbleached but classified clay.

Table 4.7 Effect of stirring Rate on the Bleaching Process

Stirring Speed (rpm)	Completion Time (Min)	Temp (^{0}C)	pH	Starch (hydrolysed) (g/L)
0	35	90	0.40	3
200	20	90	0.40	3
400	22	90	0.40	3
600	33	90	0.40	3

Table 4.8: **Effect of pH on reaction time** 54

pH	Amount of Starch g/l	Completion time (min)	
		Hydrolysed Starch	Non Hydrolysed Starch
1.00	8	37	72
0.75	8	22	52
0.40	8	7	29
0.00	8	Turns dark before 95°C	2
1.00	6	43	81
0.75	6	28	63
0.40	6	14	33
0.00	6	-	4
1.00	4	51	98
0.75	4	35	76
0.40	4	16	43
0.00	4	-	9
1.00	2	76	134
0.75	2	54	120
0.40	2	27	65
0.00	2	Time not significant	14
1.00	0	52	Time for change
0.75	0	?	from red to a
0.40	0	11	light colour
0.00	0	2	

Constant factors: Temperature = 95°C
Stirring rate = 300rpm.

Table 4.9: **Effect of starch quantity on reaction time.**

Amount of Starch (g/l)	*Temp °C*	*Completion Time (min)*	
		Hydrolysed Starch	*Non Hydrolysed Starch.*
8	95	18	40
6	95	25	45
4	95	31	53
2	95	49	78
0	95	Infinite	Infinite
8	90	23	47
6	90	29	53
4	90	36	62
2	90	55	86
0	90	Infinite	
8	80	48	98
6	80	53	106
4	80	60	114
2	80	76	133
0	80	-	-
8	70	117	165
6	70	121	170
4	70	130	176
2	70	148	191
0	70	-	-

Constant factors: pH = 0.50

Stirring rate = 300 rpm

Table 4.10: **Effect of temperature on reaction time.**

Temp °C	pH	Complition Time (mim)	
		Hydrolysed Starch	Non-Hydrolysed Starch
95	1.00	60	107
90	1.00	69	116
80	1.00	98	154
70	1.00	185	247
95	0.75	44	83
90	0.75	53	91
80	0.75	85	135
70	0.75	160	221
95	0.60	34	67
90	0.60	42	74
80	0.60	66	122
70	0.60	139	201
95	0.40	19	52
90	0.40	21	57
80	0.40	50	95
70	0.40	121	172

Constant factors: Starch quantity = 3g/L
Stirring rate = 300rpm.

Table 4.11: **Result of a preliminary run on effect of temperature.**

Temp °C	Time (min)	Amount of Starch g/L	pH	Stirring Rate (rpm)
90	57	4	0.75	300
80	89	4	0.75	300
70	162	4	0.75	300

4.2 DISCUSSION

4.2.1 Classification

Result and observations made during the classification process in this study show that classification is one of the best methods for kaolin or clay recovery in general.

The classifier used has four speeds (Table 4.2). The first speed is the highest and it gives about the best result. Although the actual amount of clay in both the overflow and the underflow could not be measured; it was observed that as the speed of the classifier was decreased, the amount of clay that reported with the underflow also increased. It was also observed that the time taken by the overflow materials to reach the top of the classifying column would have been reduced if the diameter of the classifying column was smaller; and in that case too, the light , finer, clay particles would have reached the top of the column first and then get discharged, thus allowing the possibility of producing grades of smaller grain sizes with high degree of purity. Thus it can be concluded that, among the factors which affect the grade and purity of the products of a centrifugal classifier are: the size of the classifier and the speed of the impeller or feed pressure in the case of hydrocyclones. It therefore becomes obvious that the smaller the classifiers diameter, the finer the grade produced, and the faster the speed, the higher the quantity of clay recovered though with the possibility of decreasing purity since some fine grained impurities will also inescapably report with the overflow.

Also observed from the result of the classification process is that as the speed of the classifier decreases the total volume of materials reporting to the underflow increases while that reporting to the overflow decreases. Finally; the result also indicate that a single classifier cannot completely separate the clay and coarse impurities. Thus for total degirtting and efficient recovery of clay by classification process a system of integration of classifiers is necessary. For the removal of the very large particle size impurities, a gravitational classifier and a large diameter centrifugal classifier may be used at the earlier stages of a multi-stage classification circuit. This can then be followed by series of small diameter classifiers depending on the number of grades to be produced. Figure 5.1 (a) is a suggested multi-stage classification process.

4.2.2 **Jigging**

The result of jigging experiments are given in Table 4.3. It was observed that the increase in jigging time with increasing feed were small at lower feed quantities; but as the quantity jigged increased beyond some critical value, the time increased sharply. The results in Table 4.3 are represented in Figure 4.1 and it shows that above 250g feed quantity, the time increased sharply. This increase in time indicates that the production rate with jigging will be reduced as the feed quantities are increased. The reason responsible for this increase in jigging time is thought to be due to the increasing amount of retained particles on the jig sieve. This retained particles temporarily blind the screen especially during the downward stroke thus reducing

the rate of discharge of the fine clay particles through the screen. One other problem observed with clay jigging is the degree of purity of the final product. Although a screen or sieve of smaller aperture than the one used in this study may be employed, but the fact is that fine grained impurities such as mica, feldspars which can have grain sizes as small as 20 micrometers (Henskanen, 1993) will also be discharged through the screen since unlike classification, the differences in the densities of the feed components are very effective in this type of jigging. Hence, large amount of impurities of grain size less than the sieve apperture that is , 53 micrometer, were contained in the final products.

It will also be difficult to produce clay of different grades with jigging because as screen of smaller apperture are used, jigging becomes difficult and production rate drastically reduced. Hence jigging should be of little consideration when commercial clay processing is intended.

4.2.3 Reductive Bleaching

This processing stage of the study is about the most important because; first its aim is to remove colour which is the most troublesome contaminant of the kaolin materials. This is carried out by the removal of iron which is responsible for the colouration. Thus, the efficiency of iron removal by this process will determine its effectiveness. It has been shown that when iron is responsible for colouration, it is present as ferric ion (Fe^{3+}) since ferrous ion (Fe^{2+}) is soluble in most aqeous solution. The process of iron removal therefore involves the reduction of the ferric

ion to ferrous from by gain of an electron. The reducing agent - which is starch in this particular study-provides the radical which produces the excess electron. The equation for the reaction according to Veglio et al, (1993) is believed to be:

$$Fe_2O_3 + 6H^+ \longrightarrow 2Fe^{3+} + 3H_2O \quad (10)$$

$$24Fe^{3+} + C_6H_{12}O_6 + 6H_2O \longrightarrow 24Fe^{2+} + 6CO_2 + 24H^+ \quad (11)$$

Secondly, the technique is a chemical process, unlike the physical processes of classification and jigging employed in the primary stage and so series of unseen interactions are to be expected between variables which can be controlled to achieve optimum performance of the process.

The results and observations made during the experimental runs indicate that though all the factors investigated are necessary for an effective iron removal from the kaolin clay but some factors or variables are most significant

The most significant variables are found to be pH, temperature and time. As a matter of fact, time is so significant that reaction to total completion was almost impossible as it takes several hours to burn the carbon produced by the decomposition of starch. Another reason thought to be responsible for the difficulty in carrying the reaction to total completion is that as starch is broken down, producing carbon oxygen and hydrogen, water is simultaneously ionized to make more oxygen available for the oxidation of the carbon to carbon dioxide; when all water in pulp is been used up, the oxygen produced by the decomposition of starch alone are not

pulp darker as more starch is decomposed without complete oxidation

It is possible to integrate this process as part of a commercial kaolin clay recovery as suggested in chapter five and shown on the suggested flowsheet for a complete kaolin clay recovery process - Figure 5.1. The effects of the various variable and their interactions in this bleaching process are discussed in the following sections

Effect of Temperature

The effect of temperature on the bleaching characteristics was undertaken to determine the trend of the bleaching reaction. This was carried out by increasing the temperature and holding other factors constant. The result shows that as the temperature increases the reaction rate increases. This is most expected as predicted by the general reaction rate rules.

The result of preliminary investigation in Table 4.11 shows that no reaction may take place at 60°C or that reaction rate at this temperature and below may be very slow. Similarly at 100°C and above, the liquid content of the pulp may evaporate. Hence in this study, temperature range was limited to 70°C and 95°C

Figures 4.3 to 4.6 show the plots of time-temperature parameters obtained by keeping constant, the pH quantity of starch and stirring rate. The figures were obtained form Tables 4.9 and 4.10. From the figures, it can be seen that no reasonable reaction will take place below 70°C. Thus, temperature is a significant variable in this process.

Effect of pH

The effect of pH on the bleaching reaction was investigated by varying pH at constant stirring rate, temperature and quantity of starch; and determining the time to completion. Tables 4.8 and 4.10 show how the pH variation with other factors or variables was carried out, and the results of these variation are plotted in Figures 4-11 and 4.12 for both hydrolysed and non hydrolysed starch.

Result of preliminary investigation shows that no reaction will take place at high pH values especially those above 1.00. This is responsible for the choice of the pH range used in this process. As can be seen form the plots in Figures 4.11 and 4-12 faster reactions were obtained at low pH usually in the rage 0.5 and 0.75. At very low pH values especially those below 0.4, the visible completion stage for the reaction tends to be suppressed and the sample changed almost directly to dark-grey or black at some temperatures. This suppression is observed to be more pronounced at high starch addition. It is therefore thought that acid addition that results in pH below 0.40 may not be necessary if the reaction must be interrupted at the light-grey visible completion stage. The plots of pH time parameters showed that the time required to carry the reaction to the dark-grey colour stage or reaction time generally, decreases somehow linearly as pH decreases, and it appears that there is a pH value at which no time will be required between the addition of the acid and the completion of reaction. This pH value is represented by the intercept marked K on the pH axis in Figure 4.11, and as the plots showed, this point varies with the temperature and the quantity of starch added.

During the preliminary runs, it was observed that as the acid is being added to the pulp, a pH value was reached at which the pulp changed instantly without heating or stirring from red to dark-grey or black. The pH at this point of instant colour change was roughly determined by adding acid continuously to pulp preparation of the composition shown in Table 4.5 without heating or stirring until the colour changed instantly to dark-grey. The values recorded in Table 4.5 are obtained from three trials. This pH value is expected to be related to the intercept K in Figure 4•11.

The addition of acid to the pulp generates much heat so that the final temperature at which the colour change occurred was also measured. The average value for pH and temperature are pH = 0.05 and temperature 85°C.

Effect of Time.

Time in this process is dependent on the other variables - pH, temperature, quantity of starch added, and stirring rate. As can be seen from the results, as pH decreases at constant temperature, starch and stirring rate, the time to reach completion also decreases and at high quantity of starch the pulp will turn dark within a very short time.It was also observed that at high pH values, high temperature may be used to achieve completion, but if pH is low, lower temperature may be used to attain completion in about the same length of time. See Figures 4.11 to 4.16

The dark colour developed by the pulp during the reaction is as a result of the decomposition of starch molecules and it takes almost infinite time to burn off all the carbon. Thus, it is almost impossible to carry the reaction to total completion. Hence, time is a very significant factor in this process. It is the measure of response

Effect of Stirring Rate.

The effect of stirring speed on the bleaching reaction was investigated seperately by varying stirring rate and determing the completion time while the other factors or variables were held constant. The results obtained are shown in Table 4.7. The result can help to explain the interaction of stirring with the other variables and therefore its effect on the whole bleaching process. When there was no stirring, the time taken to reach completion was highest as shown on Table 4.7. Also at higher stirring speeds, more time was taken to attain completion, but at some intermediate stirring rates, shorter time was required. As can be seen from the table, completion time falls from maximum at zero stirring rate to a minimum at between 200rpm and 400rpm and then rises towards maximum at higher stirring rate. (Figure 4.2).

This behaviour shows that stirring was neccessary possibly to bring about adequate contact between the reacting species and thus increase the reaction rate. At high stirring rate however, the rate of pulp agitation became so high that little conctact time was allowed between reacting species, thus leading to reduction in the rate of reaction which is responsible for the longer reaction time at high stirring rates. Also since the process was carried out at atmospheric pressure, the high stirring rate makes the pulp loose heat to the surrounding by convection. Hence to achieve faster reaction at high stirring rate, the rate of heating must be increased.

Effect of Starch

The effect of starch was investigated by varying the quantity of starch added to pulp while holding other variables constant. Tables4.8,and 4.9 show how the

hydrolysed and non hydrolysed starch were used in the course of experimentation. The process of reduction is represented by the following equation

$$24\ Fe^{3+} + C_6H_{12}O \longrightarrow 24Fe^{2+} + 6CO_2 + 24H^+ \qquad (11)$$

This equation indicates that the starch component responsible for reduction in this process is glucose $C_6H_{12}O_6$ which is obtained from starch hydrolysis described under material preparation. It can be seen from the experimental procedure that if the clay were absent from the pulp, then the run will somehow be a process of hydrolysis. Thus, starch is also used directly in some runs without previous hydrolysis. The non-hydrolysed starch is first hydrolysed in the course of reaction before reduction of ferric ion can take place. Hence, the runs with non-hydrolysed starch take longer time than those with hydrolysed starch. This is the only obvious advantage of hydrolysed starch over the non-hydrolysed.

Figures 4.7 to 4.10 are starch quantity -time parameters plotted from Tables 4.8 and 4.9. As can be seen from the figures, as the quantity of starch is reduced, the completion time increases. This behaviour suggests that the process of starch decomposition in the pulp is gradual and it depends on the quantity available in the pulp

With high quantity of starch in the pulp, the visible completion stage was suppressed and the pulp quickly turned dark; but as the amount of starch added was gradually reduced, the visible completion stage becomes clearly defined so that maximum brightness was achieved. Observation shows that the quantity of starch that gives the best results lies between 4g/L and 6g/L as most runs with 2g/l of starch did not give satisfactory result apart from being too slow to attain completion.

It was also observed that though low pH and high temperature lead to increased rate of reaction, the quantity of starch in pulp must be sufficient to cause total reduction and 4g/L is observed to be sufficient in this process.

From a comparison of the results of non-hydrolysed starch with hydrolysed starch, it can be seen that a combination of non-hydrolysed starch quantity, temperature and pH can be chosen that will give good result in about the same length of time as hydrolysed starch; thus saving the need for previous hydrolysis. This can be seen from the interpolation of the result for both hydrolysed and non-hydrolysed starch as shown by Figures 4.11 to 4.16. For example , the intersection or closeness of the lines for 8g/L of non-hydrolysed starch and 2g/L of hydrolysed starch in Figure 4.11 shows that about the same reaction time will be required for completion using these two quantities of starch at the same temperature, pH and stirring rate. The same trend can be seen in Figure 4.12, 4.15 where temperatures of 80°C and 95°C can be used to complete reaction in about the same length of time for hydrolysed and non-hydrolysed starch respectively at the same pH, and starch quantity, while stirring steadily. The same trend is observed in Figures 4.13, 4.14,4.15 and 4.15. This trend therefore shows that starch hydrolysis may not be necessary if correct combination of factors is carried out.

It was necessary to dissolve the non-hydrolysed starch in small quantity of water before adding to the acid solution or pulp and then adjust the pH as desired. This is because non-hydrolysed starch added directly to the pulp tend to form small

lumps which were later burnt as lumps of floating carbon on the pulp. The effects of this is that the quantity of starch available for actual reduction was less than the quantity added.

It can also be seen from the equation for reduction that the water in the pulp is used up in the process to produce more acid components, sufficient quantity of water must therefore be used to obtain good result. This can be done by increasing the liquid to solid ratio.

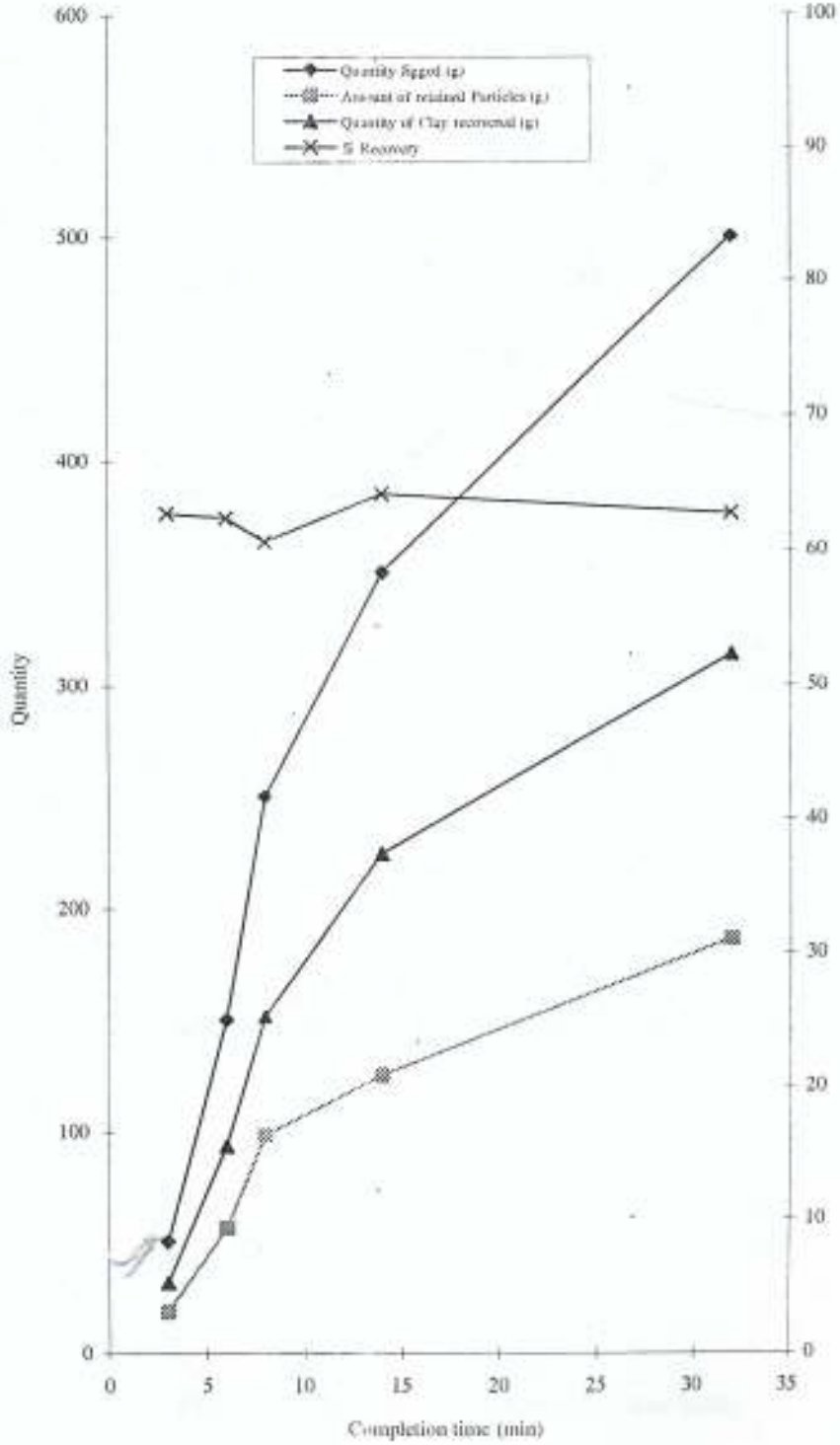


Fig 4.1: Jigging Result

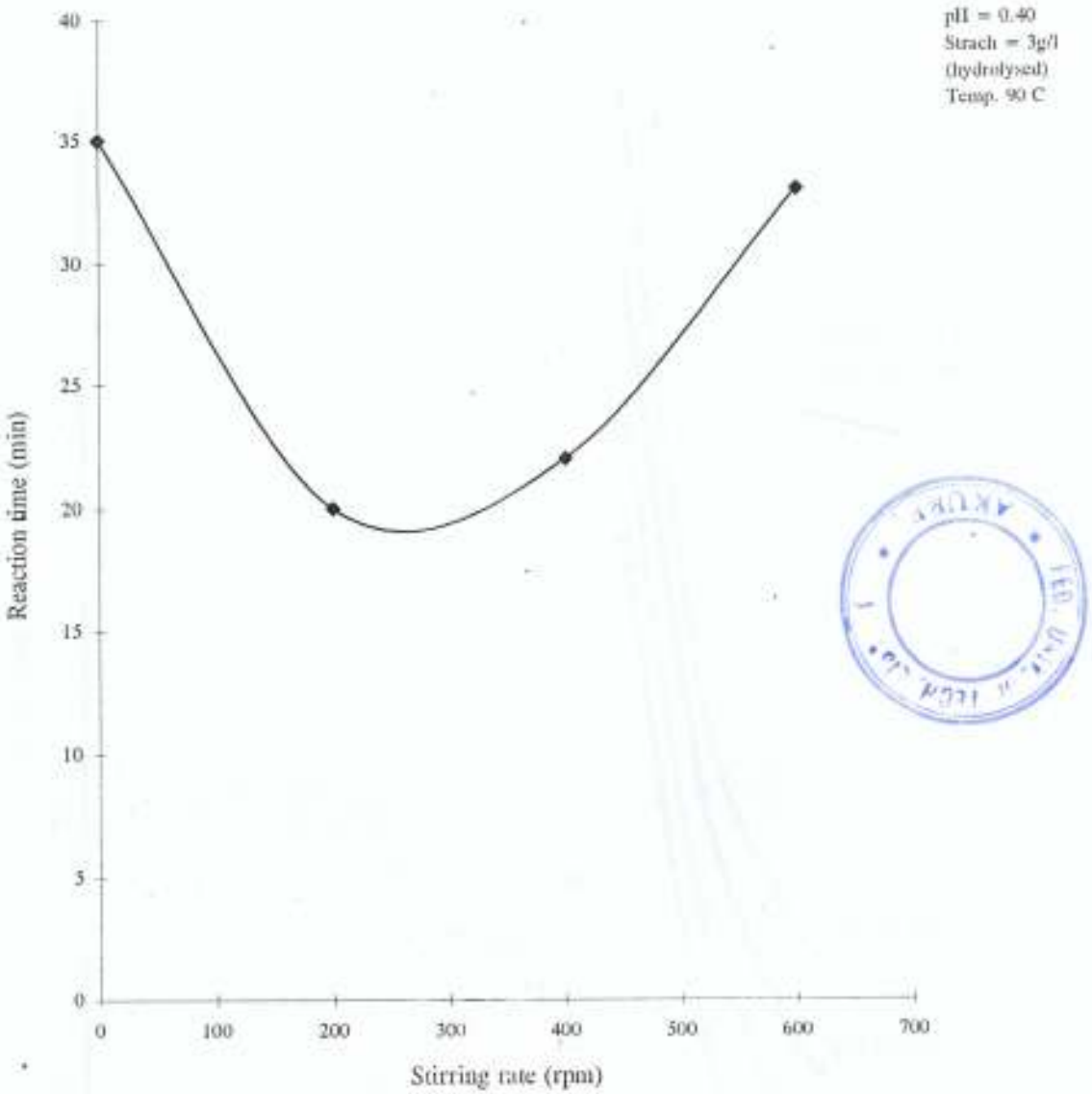


Fig 4.2: Effect of stirrring on reaction time

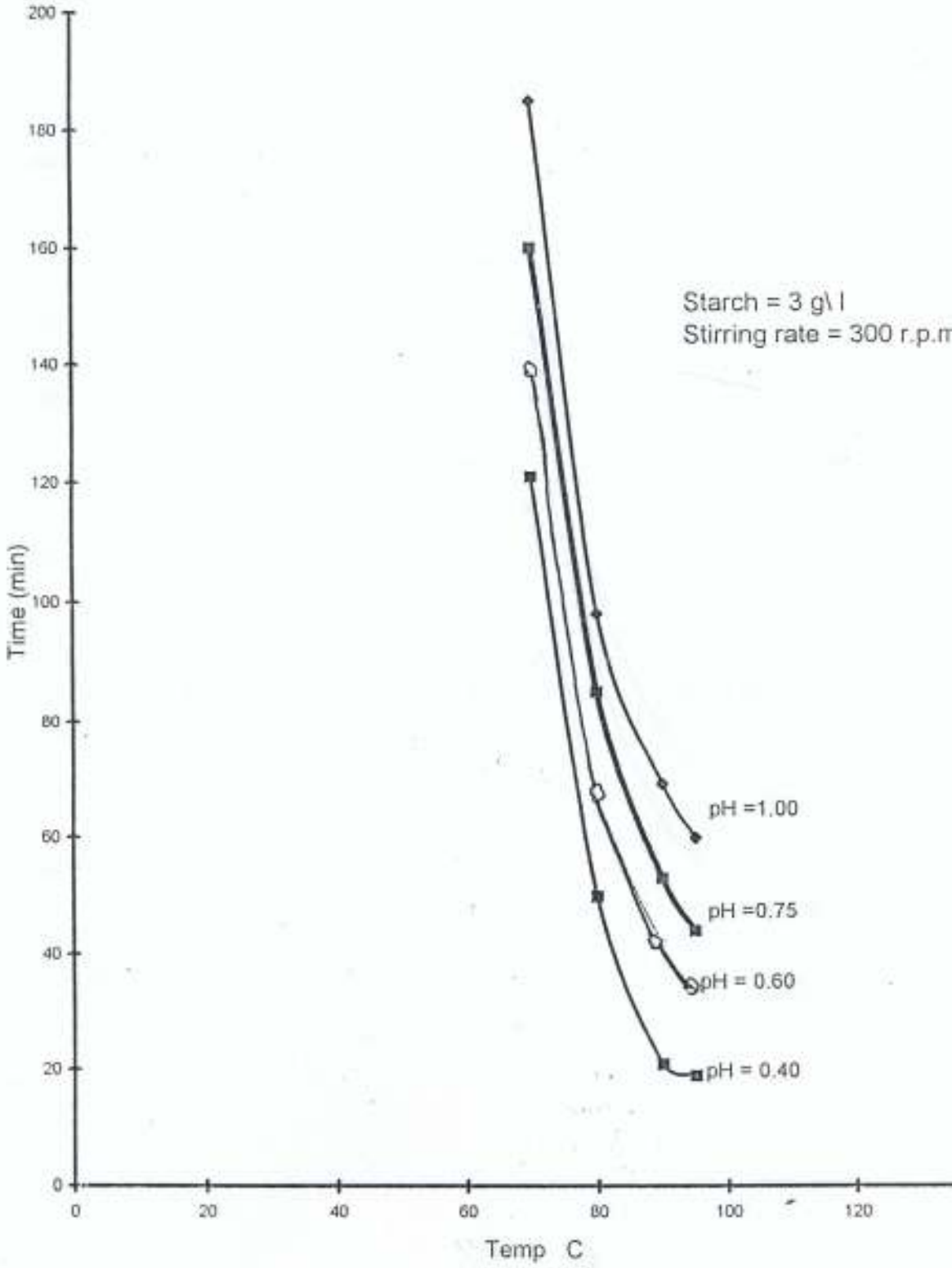


Fig. 4 -3: Reaction time from temperature variation at different pH for hydrolysed starch

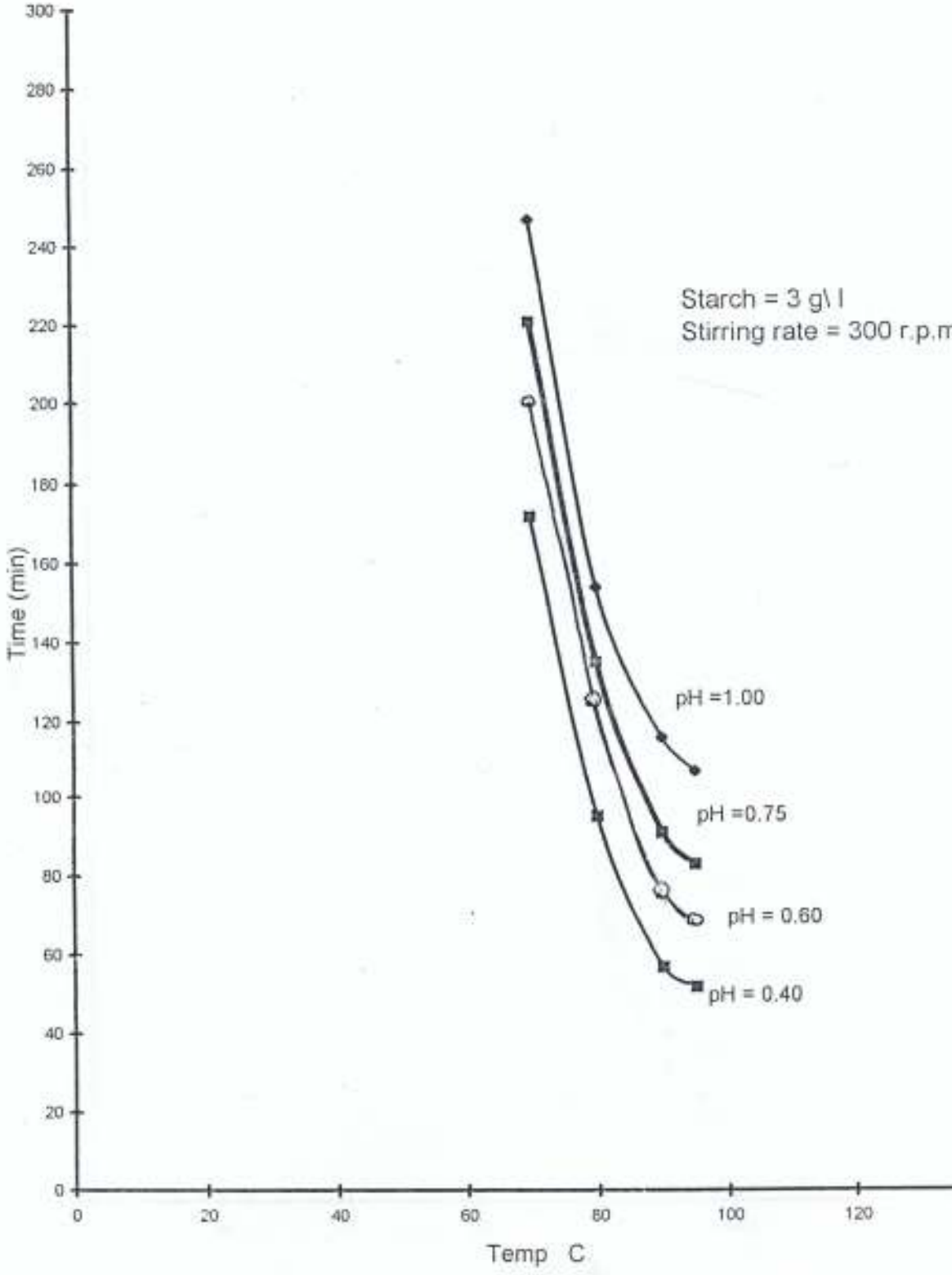


Fig. 4 -4: Reaction time from temperature variation at different pH for non-hydrolysed starch

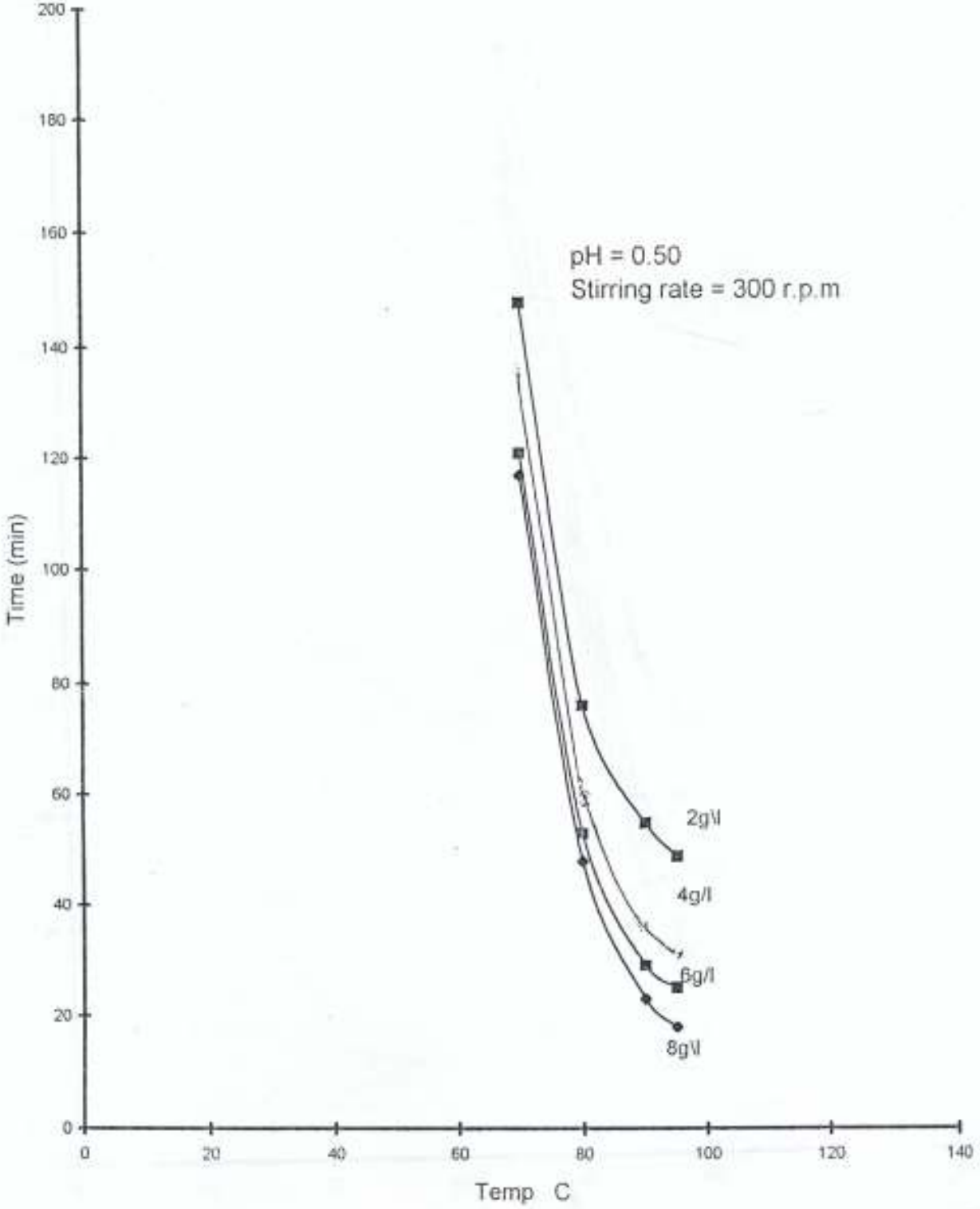


Fig. 4 -5: Reaction time from temperature variation at different quantity of starch for hydrolysed starch

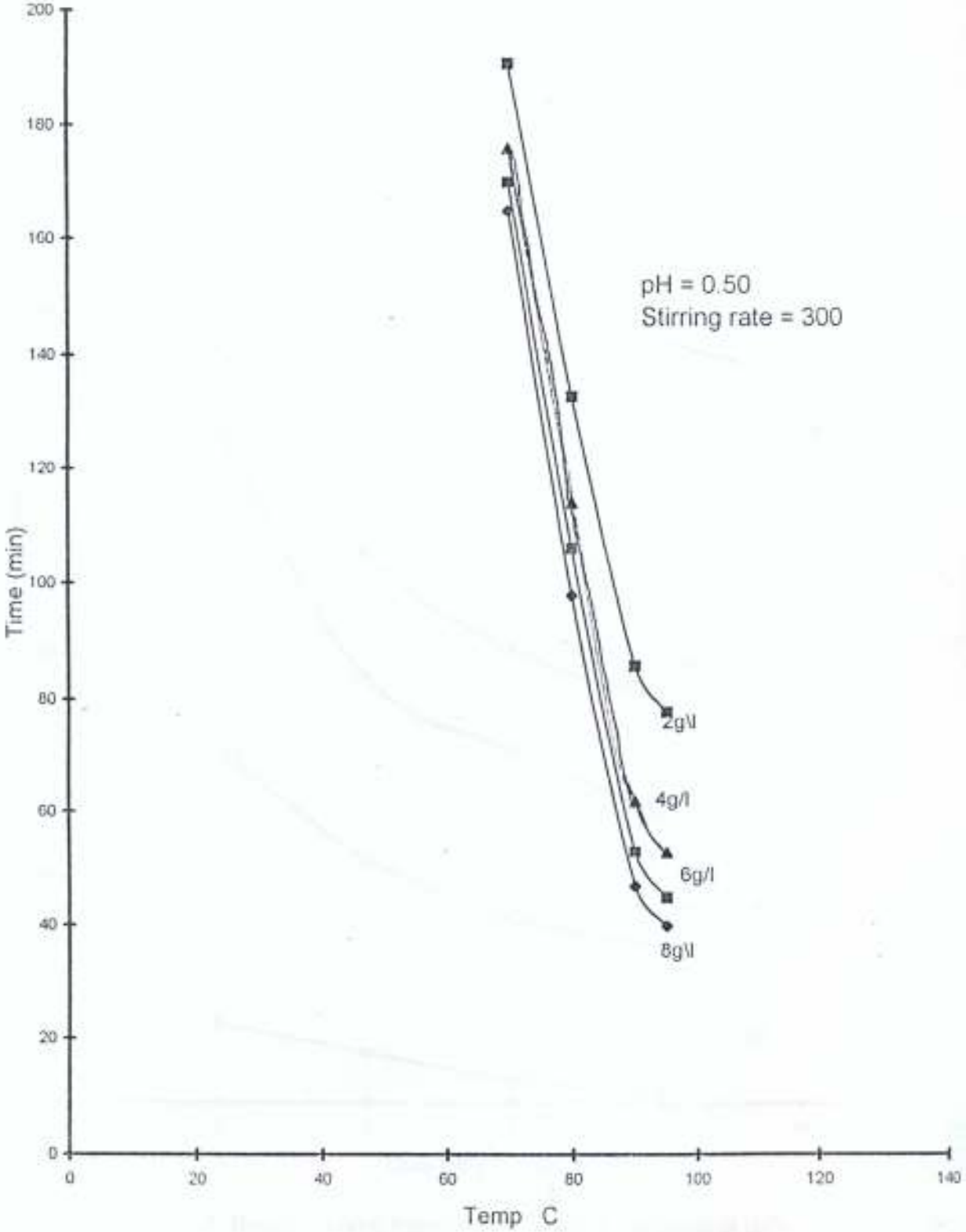


Fig. 4 -6: Reaction time from temperature variation at different quantity of starch for non hydrolysed starch

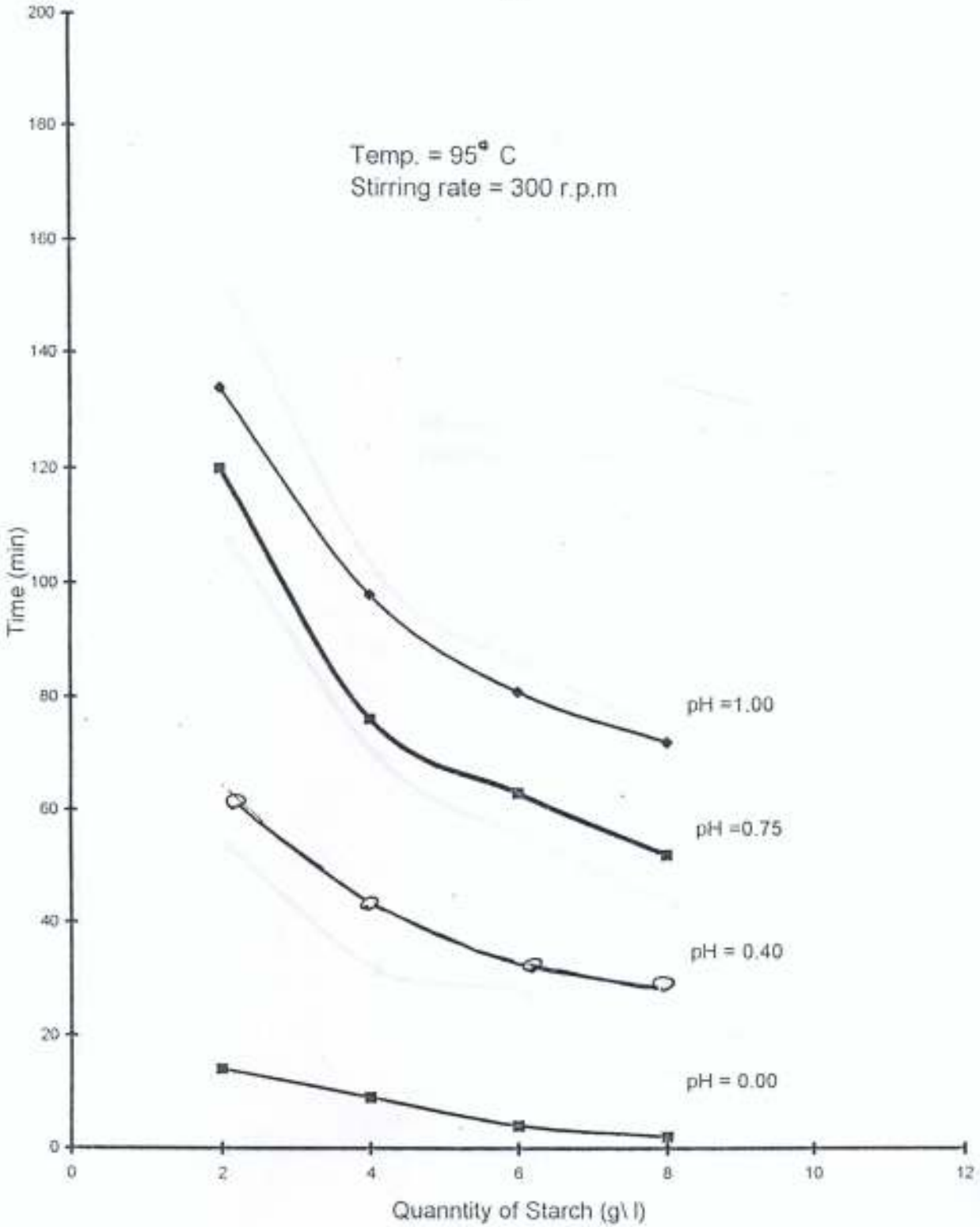


Fig. 4 -7: Reaction time from starch quantity variation at different pH for non-hydrolysed starch

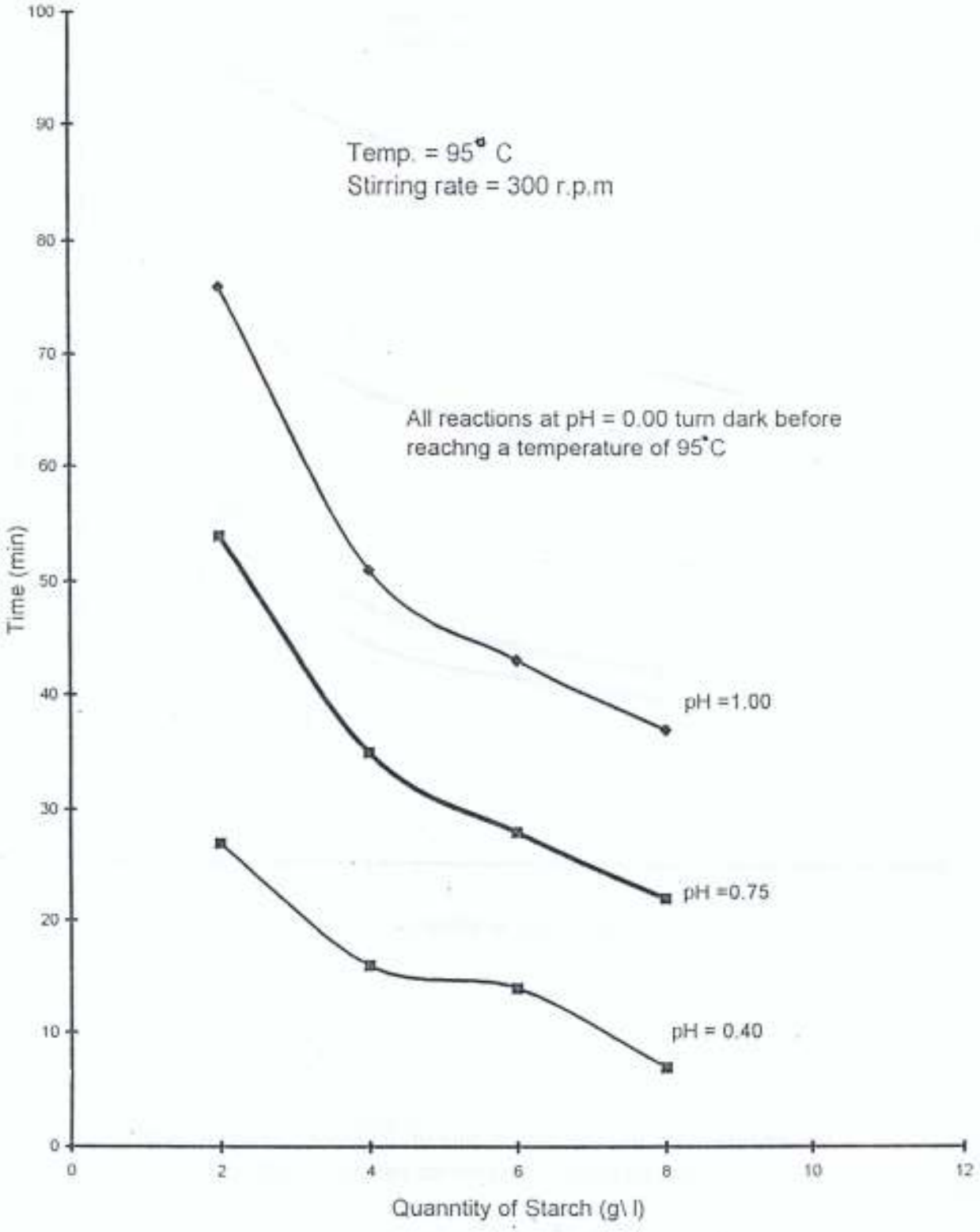


Fig. 4 -8: Reaction time from starch quantity variation at different pH for hydrolysed starch

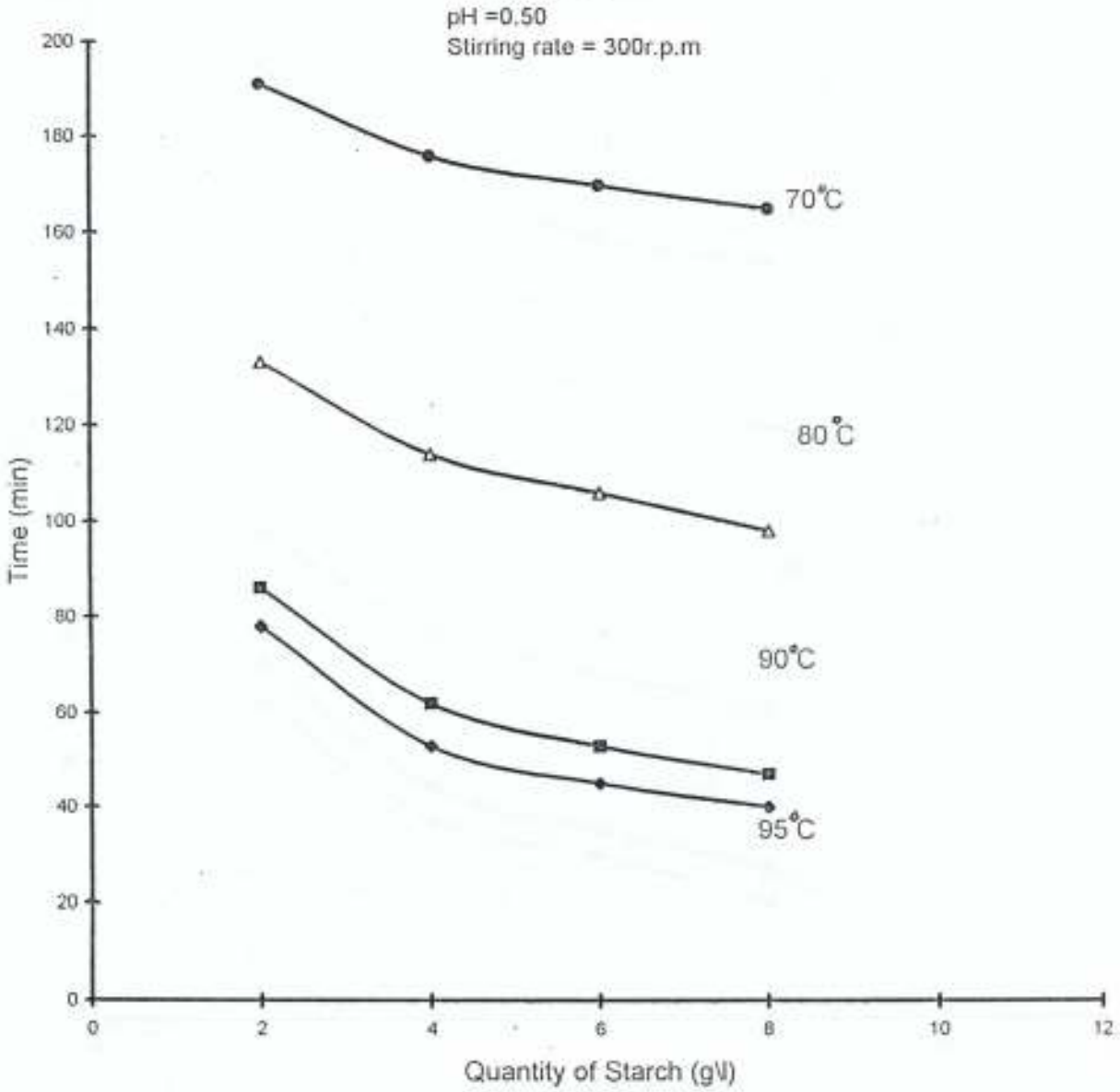


Fig. 4.9: Reaction time from starch quantity variation at different temperatures for non hydrolised starch

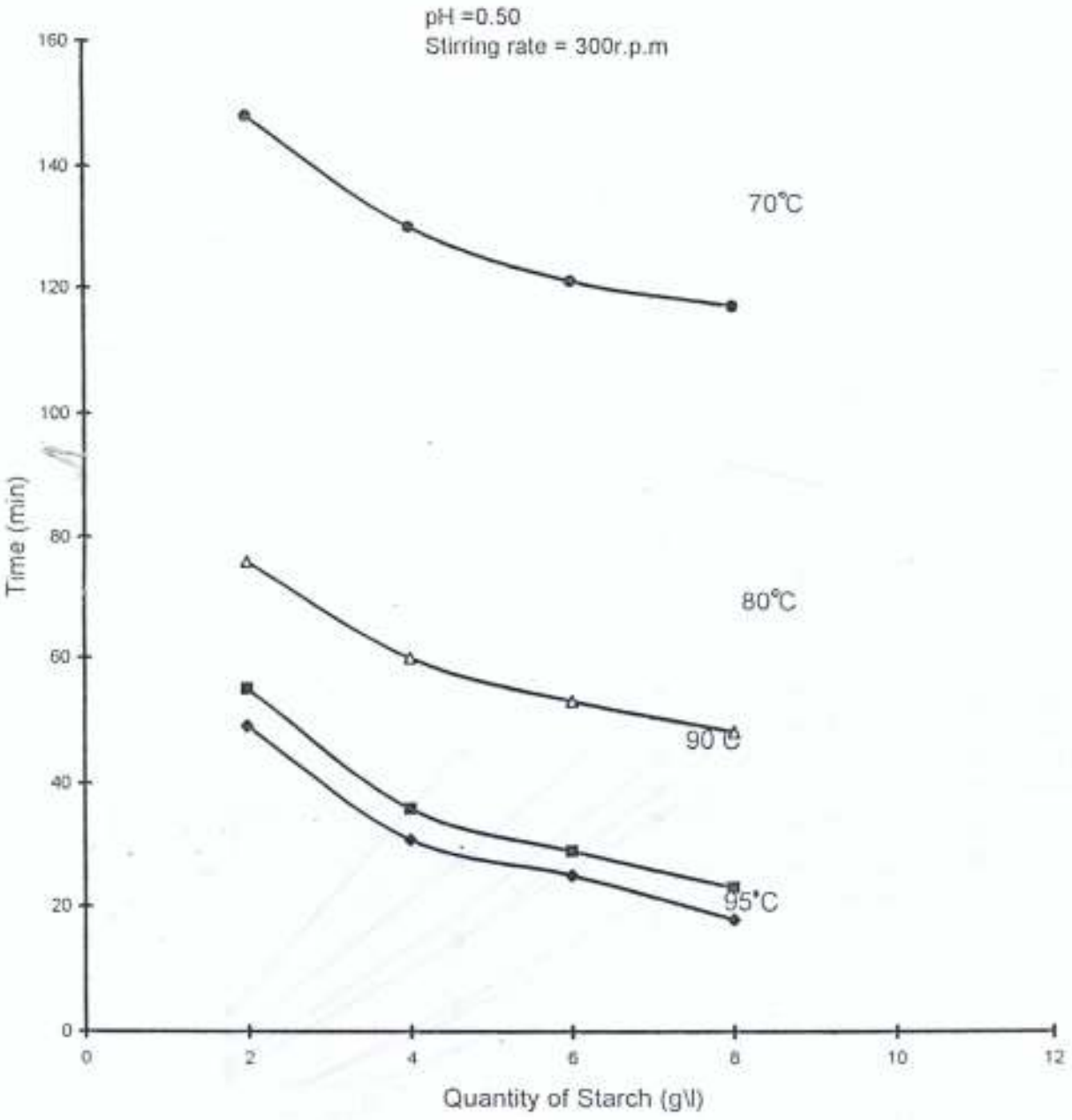


Fig. 4.10: Reaction time from starch quantity variation at different temperatures for hydrolised starch

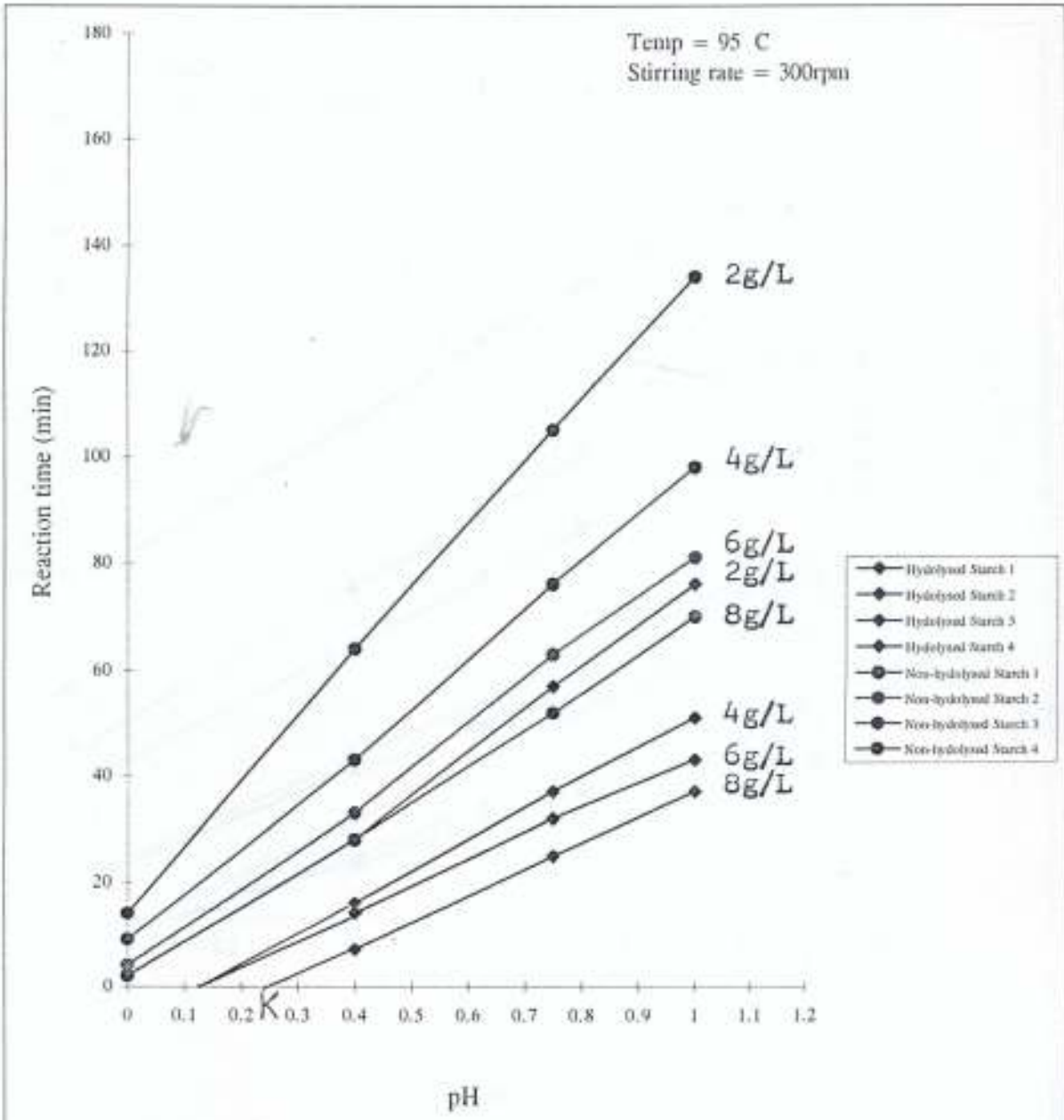


Fig 4.11: Reaction time from pH variation at different starch for hydrolised and non-hydrlysed starch combined

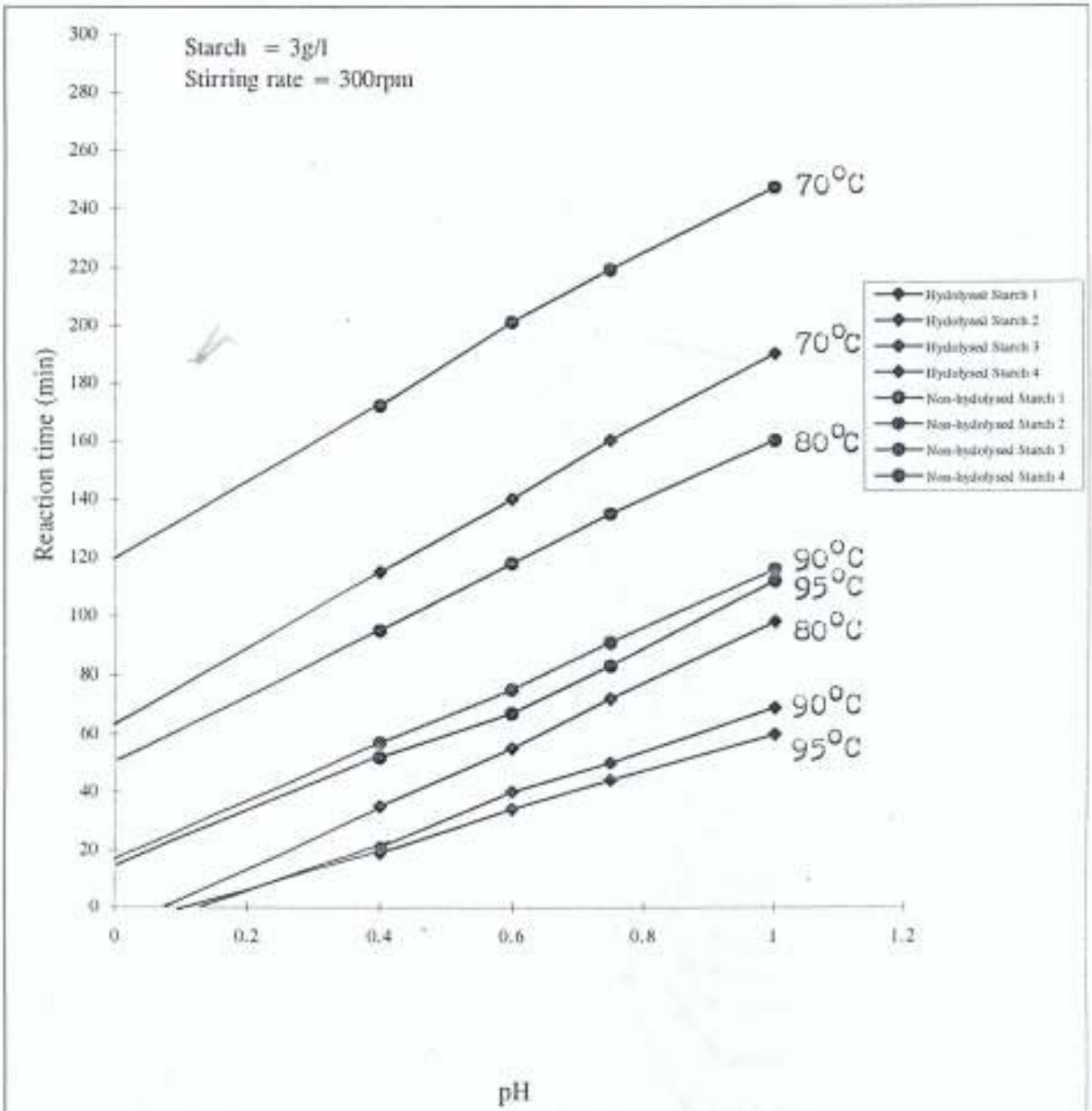


Fig 4.12: Reaction time from pH variation at different temperature for hydrolised and non-hydrolysed starch combined

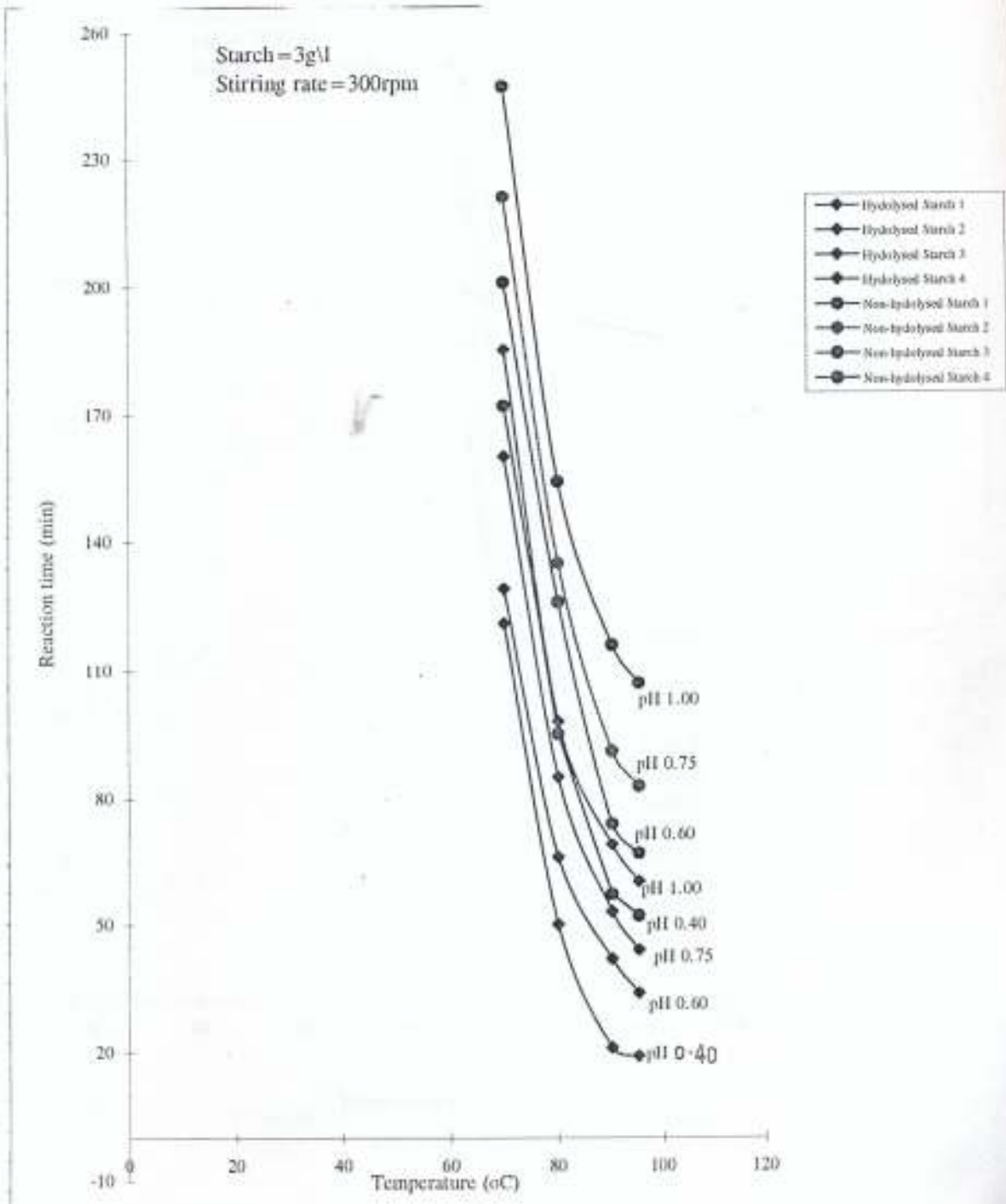


Fig 4.13: Reaction time from temperature variation at different pH for hydrolised and non-hydrlysed starch combined

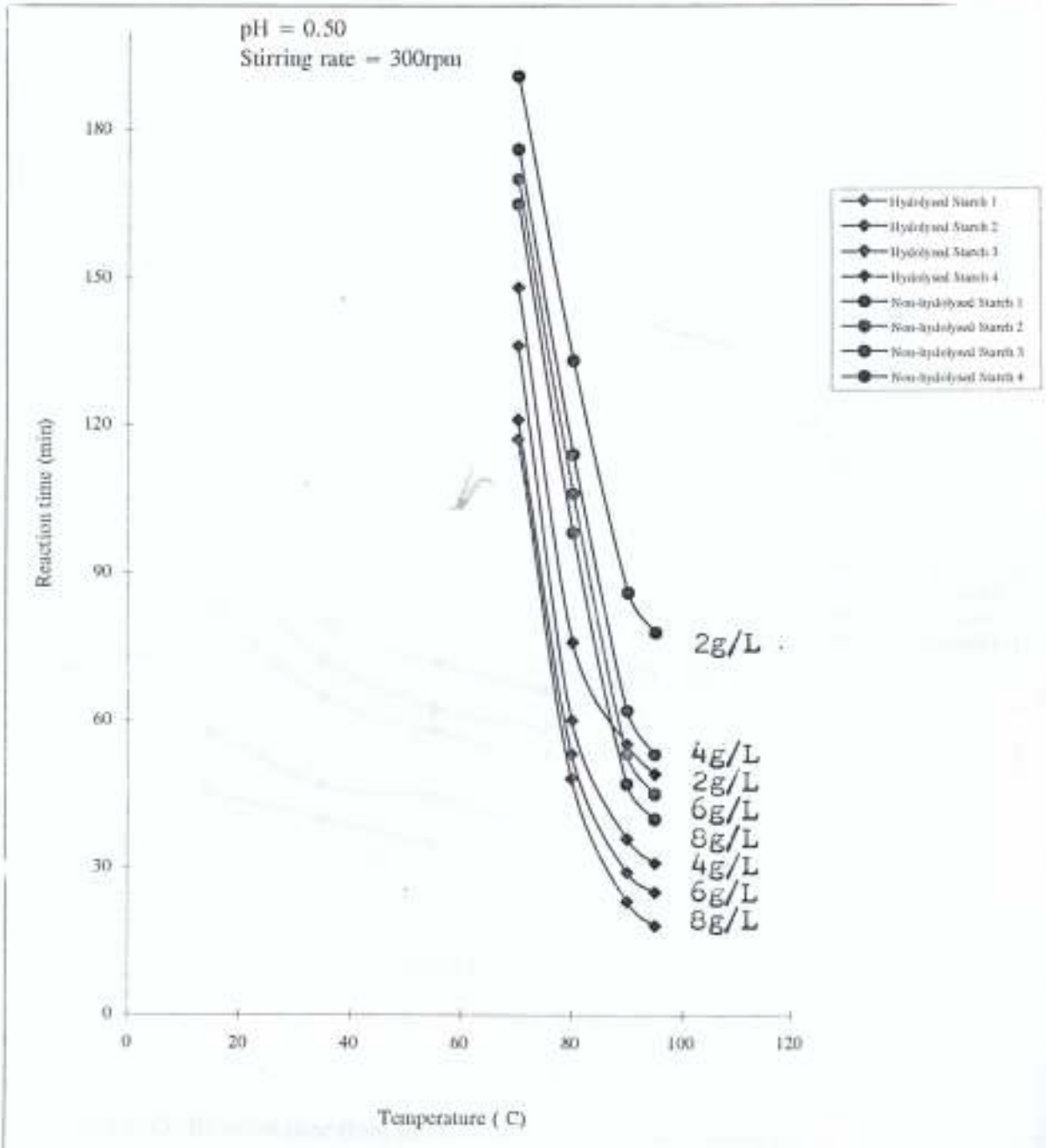


Fig 4.14: Reaction time from temperature variation at different starch quantity for hydrolised and non-hydrlysed starch combined

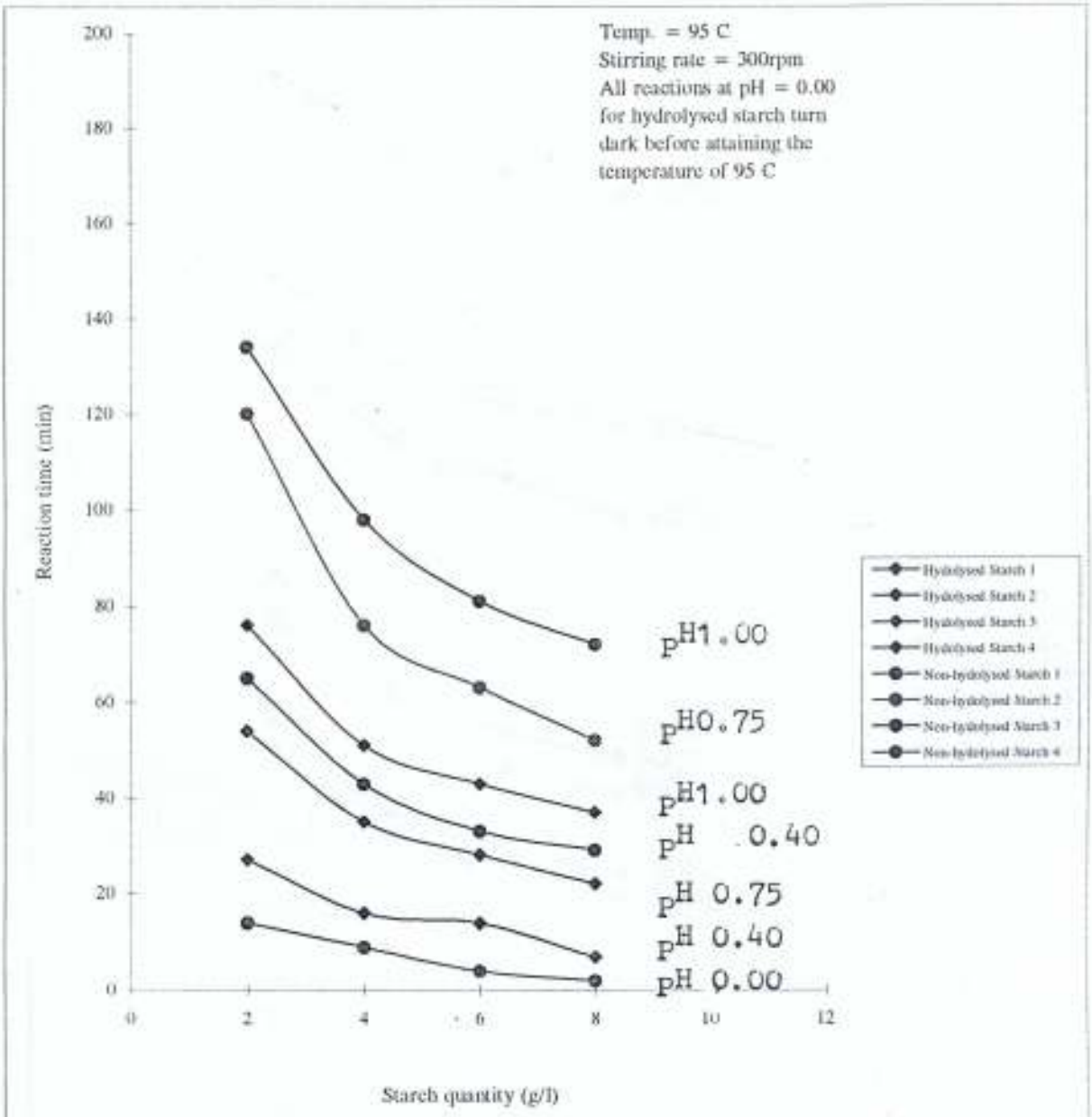


Fig 4.15: Reaction time from starch quantity variation at different pH for hydrolised and non-hydrlysed starch combined

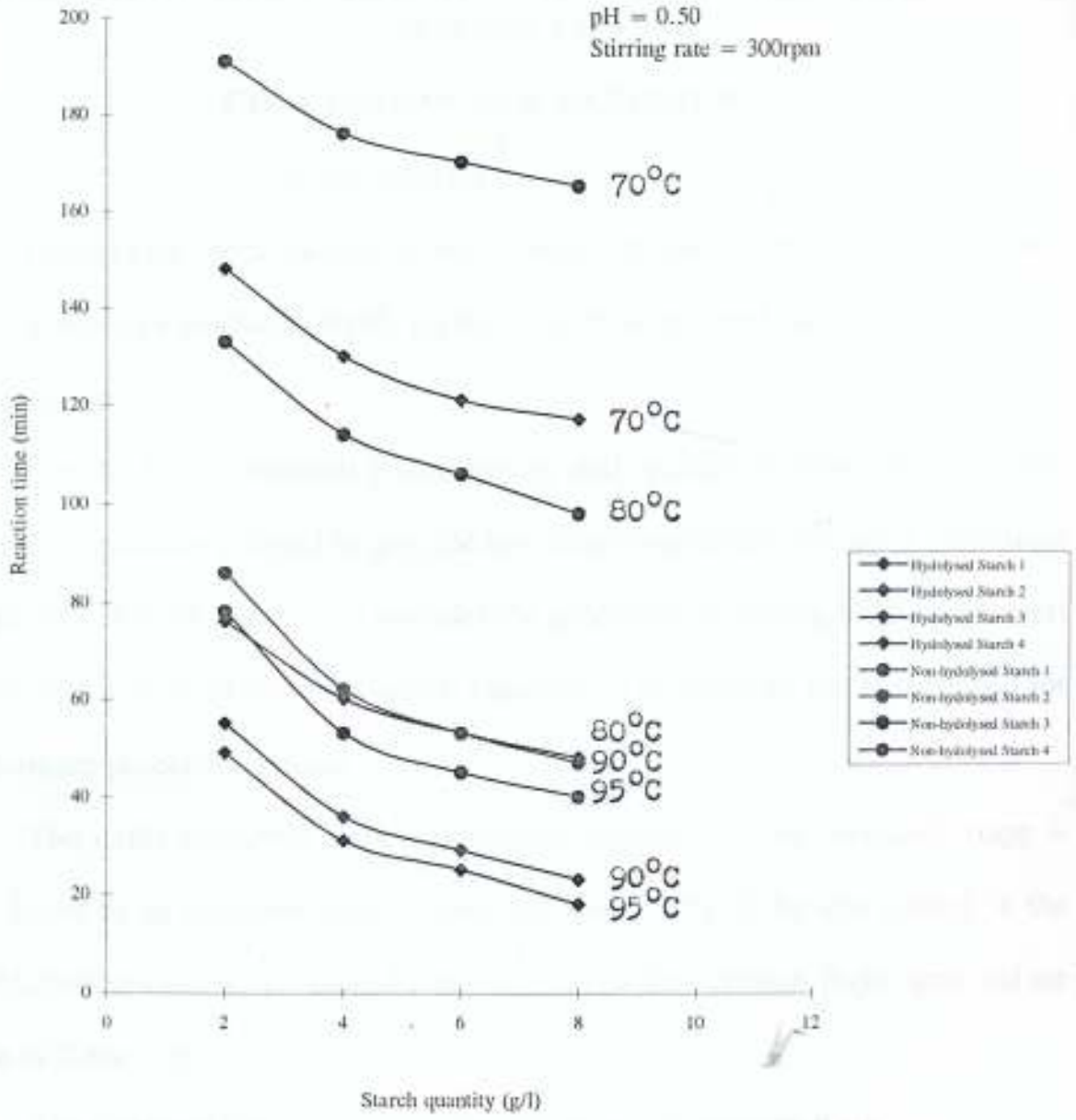


Fig 4.16: Reaction time from starch quantity variation at different temperature for hydrolised and non-hydrlysed starch combined

CHAPTER FIVE

CONCLUSION AND RECOMENDATION

5.1 Conclusion

Kaolin clay from various deposits were successfully processed in a two - stage process to produce a highly purified clay that can meet most industrial specifications.

Among the two methods (classification and jigging) employed at the primary stage, classification is found to give the best result (especially the use of centrifugal classifiers). Classification also provides the possibility of cutting the final products into grades and gives high production capacity. It is therefore the best method for the primary proccessing stage.

The oxide reductive bleaching process employed in the secondary stage is also found to be effective, since it removes about 90% of the iron content in the unbleached sample at the point of interruption of the reaction (light -grey colour stage in Table 4.6)

The values of the various variables that give optimum result are:

For hydrolysed starch:

Solid (clay) content of pulp	=	30-35%
pH Range	=	0.4-0.75
Temperature range	=	90°C-95°C
Quantity of starch	=	4g/L-6g/L
Stirring rate	=	250rpm

For Non-hydrolysed starch:

Solid (clay) content of pulp	=	30-35%
pH Range	=	0.25-0.50
Temperature range	=	90°C-95°C
Quantity of starch	=	4g/L-6g/L
Stirring rate	=	250rpm.

The major advantages of this bleaching process include.

1 The use of only two materials (acid and starch), Starch is a cheap and abundant raw material and the process is not specific about the source of starch. The acid used can be recovered and recycled.

2 No toxic products are produced that can make the process environmentally dangerous as with other bleaching processes with heavy metal ions in final process or waste water.

3 The materials recovery and cost advantages in this process is expected to compensate for the need to provide heating.

Hence this process is expected to be one of the cheapest techniques for kaolin clay processing for any industrial application.

5.2 RECOMMENDATION

Based on the results of findings in this study, the enumerated advantages of some of the processes undertaken and the fact that the reductive bleaching process is neither kaolin clay nor starch-source specific, the processes emphasized under conclusion in this chapter are recommended for industrial recovery of any local kaolin clay deposit

For the removal of coarse and other gritting particles, an integration of gravitational classifiers and various diameter hydrocyclones installed as suggested in Figure 5.1 may be useful. Smaller hydrocyclones are useful in processing for different grades and should be installed after the bleaching process (or any secondary processing technique employed) to cut the final product into required size grades as in Figure 5.1

For the removal of colour in the secondary processing stage, the reductive bleaching process with starch and sulphuric acid is recommended for integration into the recovery circuit shown in Figure 5.1 (b) and the following values of the variables used in order to achieve maximum performance

For hydrolysed starch

Solid content	=	35%
pH	=	0.40
Temperature	=	95°C
Quantity of Starch	=	4g/L

For, non-hydrolysed starch:

Solid content	=	35%
pH	=	0.25
Temperature	=	95°C
Quantity of Starch	=	4g/L
Stirring rate	=	250rpm

For acid recovery in the secondary processing stage of this study, the design of the system should be such that the bleach is filtered before washing as indicated by the links between (11) and (14) in Figure 5.1 (b). The filterate is treated for acid recovery or recycling and the product or the bleach thoroughly washed to remove all solubilized materials

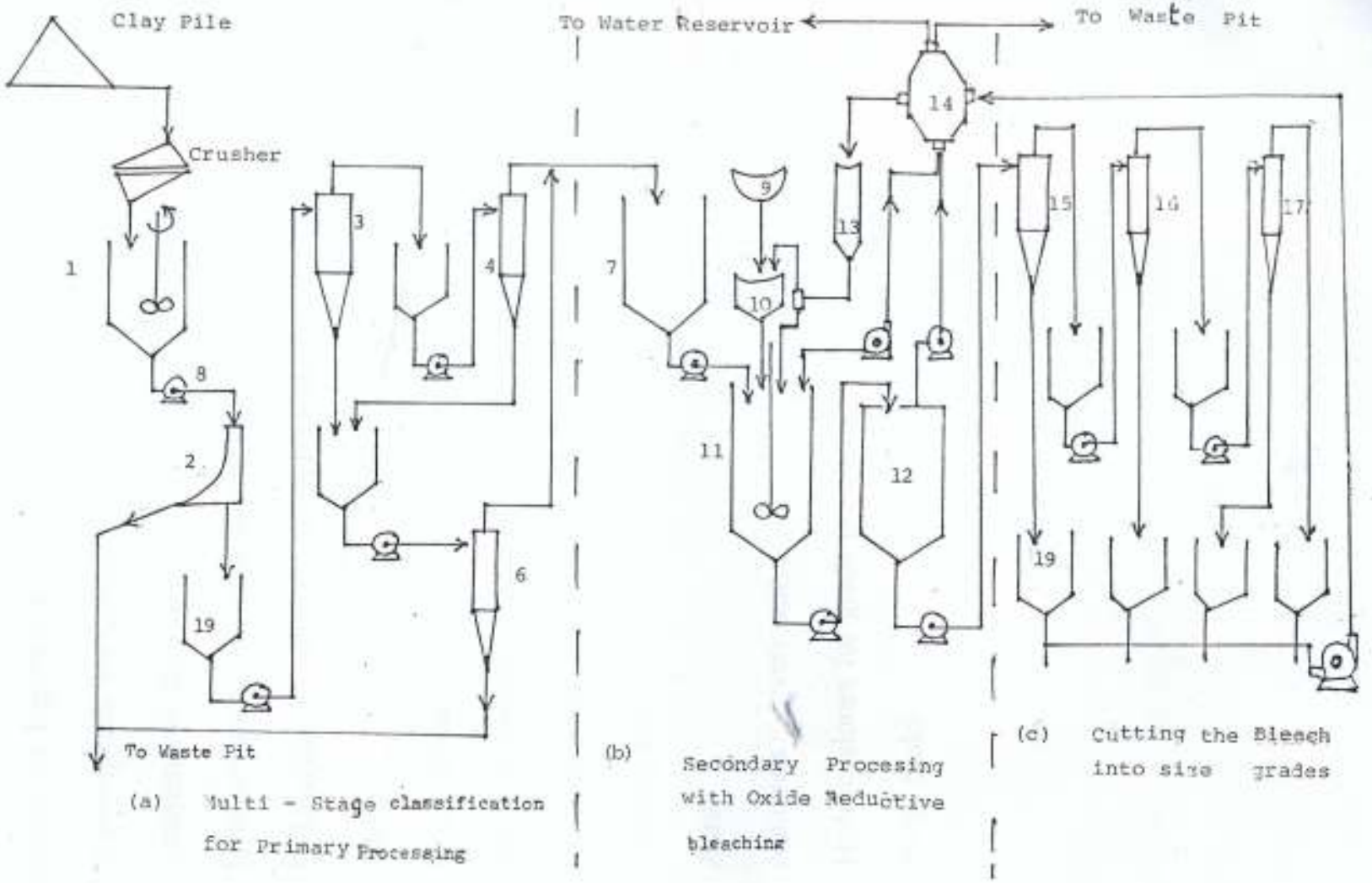


FIG. 51: Flow circuit for a complete Kaolin clay Processing. (Suggested for Complete Kaolin Processing)

Components of Figure 5.1

1	-	Slurrying tank with plunger
2	-	Gravitational classifier
3-6	-	Hydrocyclones of various diameters
7	-	Unbleached classified kaolin, slurry tank
8	-	Pumps
9	-	Starch container/meter
10	-	Starch hydrolyser
11	-	Bleaching tank
12	-	Washing tank
13	-	Acid
14	-	Materials recovery unit
15-18	-	Hydrocyclones for grade processing
19	-	Slurry tanks

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