IMPROVEMENT OF THE PERFORMANCE OF AN EXISTING FATLIQUOR PILOT PLANT

A THESIS

Presented to

The Postgraduate School
Ahmadu Bello University, Zaria

UGWU, Johnson Obunwa

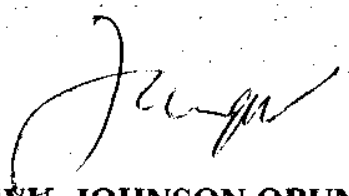
In Partial Fulfillment of the Requirements for the Award of the Degree of Master of Science in Chemical Engineering

Department of Chemical Engineering
Ahmadu Bello University,
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AUGUST, 2000

DECLARATION

I hereby declare that this project has been composed and written by me, and that it is a record of my research work. It has not been previously presented in any of my applications for a higher degree. All literature cited have been duly acknowledged by means of references.



UGWU, JOHNSON OBUNWA

CERTIFICATION

This thesis entitled "IMPROVEMENT OF THE PERFORMANCE OF AN EXISTING FATLIQUOR PILOT PLANT by UGWU, JOHNSON OBUNWA, meets the requirement of the regulations governing the award of the degree of MASTER OF SCIENCE IN CHEMICAL ENGINEERING of AHMADU BELLO UNIVERSITY, and is approved for its contribution to knowledge and literary presentation.

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DEDICATION

To the entire, CHIEF UGWU EJINMA OKO, Ochiri Ozua I, of Umuogbo-Agu family for their monumental role in shaping my life.

ACKNOWLEDGEMENT

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ABSTRACT

Experimental and theoretical investigations were conducted on an existing sulphation pilot plant facility for production of leather lubricant, sulphated oil popularly known as fatliquor. This was carefully and critically done to evaluate the performance of the plant in respect of attaining the required process conditions. These include maintaining the sulphation reaction temperature of 25°C to reduce side reactions to enhance optimum yield of the desired product. In addition, the study evaluated the possibility of using the plant for as much smaller feed as possible, per batch of production, to reduce cost of experimentation on the plant.

The test-runs revealed some design Lapses, such as the reaction temperature rising up to 41"C in the three test runs that were done. This may indict the cooling system that was earlier on, observed to have placed serious restriction on wide variation of process input required of a research project. A redesign of the cooling system was done as part of the investigations to confirm the observed under-functioning. The redesign

established that the total surface area for the heat exchanger of the cooling system was insufficient to perform the required duty. This may not only have been responsible for the design defects but had also placed much restriction on the process feed variation.

The details of the modified design done and being recommended for implementation include, maximum plant capacity of 216kg of oil and a total surface area of 5.74m^2 as compared to initial total surface area of 0.844m^2 and later modified to 2.614m^2. These differences are much and could be the main source of inefficient cooling in the former two designs. The modified design with the largest total surface area is expected to have a better cooling performance and as well reduce the restriction on the variation of the feed materials per batch to make the plant more functional, and less expensive. A provision of 2m tube length was made for inlet and outlet extensions of the coiled tube heat exchanger. The cylinderical section of the reactor vessel required 28 coils of 0.79m uniform diameter and the conical section contained 4 coils of varying diameter.

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LIST OF SYMBOL

SYMBOL	DEFINITION	UNIT
A_s	Surface area	m^2
ϕ_V	Volumetric flow rate	m^3/s
d_c	diameter of tube coil	**m**
D	diameter	**m**
OD	outside diameter	**m**
ID	inside diameter	**m**
C_p	Specific heat capacity	J/kg°C
λ	Thermal conductivity	W/m°C
ρ	Density	kg/m^3
μ	Dynamic viscosity	Ns/m^2
U	Overall heat transfer coefficient	-
ν	Kinematic viscosity	m^2/s
L	Length of tube	**m**
n	Number of tube coil	-
M	Mass	**kg**
ΔH_{rxn}	Heat of reaction	J
Q	Quantity of heat	J
ΔT_{LM}	Log mean temperature different	°C
T	Temperature	°C

$a = \frac{\lambda}{\rho C_p}$	Thermal diffusivity	m^2/S
σ	Surface tension or energy	N/m
φ_H	Heat flux	J/m^2S
α	Heat transfer coefficient	$W/m^{2o}C$
Nu	Nusselt number	-
Pr	Prandtl number	-

Subscripts and Superscripts

i	-	refers to inside
o	-	refers to outside or oil
f°	-	refers to standard state property.
m	-	refers to mixture
s	-	sulphuric acid
w	-	water property
c	-	tube coil

CHAPTER ONE

1.0 INTRODUCTION

An oil can be made soluble in water by reacting it with sulphuric acid to form a sulphated oil, a form of fatliqour. The reaction is necessary to break the interfacial tensions to make oil miscible in water. Fatliquoring is a part of leather chemical finishing process activity. Sulphation in this context is a process of rendering an oil water soluble for stable emulsion with the various chemicals used resulting in a fat which will possess a negative or anionic charge when emulsified. If sulphated oil is used as emulsifier, the emulsion will be anionic and will react accordingly.

It is necessary to introduce lubricants (oils) into leather in the course of its manufacture because most of the fatty matters which in conjunction with water ensure the suppleness of the hide or skin are lost during processing. The processed leather will be brittle and horny without lubricant, making the leather to be less useful. The extent of fatliquoring depends on the type of leather desired,

whether for upholstery or case upper shoe. A special fatliquor with excellent emulsifying capacity and water repelling effects does not contain either ingredients supporting the growth of bacteria and fungi or fatty substances that decay, and they also do not cause fat spews - the application gives the leather excellent mechanical and physical properties like water repellant effect. Natural fats and oils, fatty alcohol, mineral oils, and fatty acids are among commonly used lubricants.

The local demand for fatliquor by tanneries is high with greater percentage if not all is yet to be met by importation. The present practice of total dependence on imported fatliquor is not favourable to our economy as a lot of money in terms of foreign exchange is lost. The current economic recession in the country does not encourage importation, thus there is a dire need for the development of a local substitute for imported fatliquor since the raw materials are locally available in large quantities.

From the above development, the need for a functional pilot plant to process fatliquor arose as part of steps taken to

commercialize viable research results and to promote indigenous engineering efforts in chemical process technology development. This contribution drew attention from some recent laboratory work and modification efforts to make an existing pilot plant for fatliquor production facility more functional.

However, the immediate motivation for this work was that the importance of the product not withstanding, a lot of money, and research efforts have been committed to fatliqour pilot plant project, but in spite of all these modifications, on test running the pilot plant with up to four production batches, the desired quantity and reaction temperature for a better production were not met.

Results of laboratory work on fatliqour development and the need to produce leather lubricants, fatliquor (sulphated oil) on a semi commercial scale formed the basis for this study.

There was a pilot fatliquor plant of 500 litres of fatliquor per batch. The problem with the plant was that the design capacity of the cooling system of the plant placed much restriction on the quantity of the raw materials to be processed. The raw material

requirement of the available pilot plant facility is 500 litres (about two drums) of groundnut oil and up to 100 litres of concentrated sulphuric acid per batch of production. This meant that a test run in the plant costed about N55,000.00 presently on raw materials alone, excluding labour, electricity and equipment depreciation cost. This placed a lot of constraints on research at pilot plant scale considering that several runs were required for research purposes, and these runs may involve huge losses of materials and other process inputs, because the concentrated acid addition if not properly controlled and standard established through test runs, might burn the oil and give products that are not useful. This has a lot of cost implication. Also the temperature of reaction is critical in the production, and with the present facility if small amount of feeds required for research purposes is used to save costs, the cooling system is not extended enough to cool this amounts of raw materials to the desired temperature of sulphation reaction. Therefore to maintain temperature at desired level and reduce process inputs (process variable) as much as possible for research

purposes, the need to modify the plant design arose. These insights and the need for better production strategies at pilot plant scale was the basis of this work. It was against this background that this project, "IMPROVEMENT OF AN EXISTING FATLIQUOR PILOT PLANT" as done.

The hypothesis here was that the design modification of the cooling facility which involved the extension of the surface area of the heat exchanger of the cooling system, would reduce the cost of raw material requirements of the test runs to a manageable size that would make research work more feasible and allow for long range of variation of through-put.

This project has become very important to justify the huge investment on the laboratory research and on the existing pilot plant facility so far.

In 1993 alone, the demand for fatliquor of different classes by local tanneries could be put at ten million naira (Burton and Robertshaw (1940)). The gap in demand is filled by importation. The money spent on the importation of this chemical into the

country yearly, based on demand estimates runs into billions of Naira.

The abundant sources of natural fatty and mineral oils that form the bulk of raw materials for fatliquor production in the country, makes the production feasible. Research efforts put into the identification of needed raw materials for the production of fatliquors of comparable quality to the imported products in the Country have yielded good results. This shows that the use of locally available natural oils and fats for fatliquor production gives a good quality product.

From the above background, the project was generally aimed at commercializing viable research results with a view to promoting indigenous engineering efforts in chemical process industrial development in the country. The modified plant could be used to try production of fatliquor from other oils like castor oil, rubber seed oil and benniseed oil apart from groundnut oil. The specific objectives of this work could be summarized as the following:

(i) The modification, if completed will make further research with the plant more cost effective in terms of reduction of process input variables (raw material requirement);

(ii) Provision of wide range of variation of process input required of a research project, thereby making the plant more functional;

(iii) Towards perfecting the production technology at pilot plant and eventually into commercial large scale production in the country to reduce importation;

(iv) Towards maintaining the reaction temperature between 25°C to 30°C required to produce good quality fatliquor with excellent emulsifying capacity (stable emulsion), water repellant effects and other good properties like not allowing fungal growth as expected of a good fatliquor.

CHAPTER TWO

2.0 SURVEY OF LITERATURE

The purpose of this is to carry out a pilot plant design modification for production of sulphated oil fatliquor. The need to improve upon an existing design to enhance performance was part of the motivation for this study. To achieve the above aims a general literature survey of subjects relevant and pertaining to this investigation were carried out as follows:

For many centuries, the leather lubricants were incorporated into the leather by simply smearing wet leather with mixtures of grease and oil (O'Flaherty et al (1978)). The use of oil in water emulsions (O/W), now called fatliquors, originated during the twentieth century (Wilson (1941)). The early fatliquor was prepared by emulsifying the oil in water through the use of common surface active agents such as soap, and/or the use of protective colloids such as egg albumen, casein or natural gum. Recently, the more modern

surface active agents have come into wide use beginning with sulphated fatty oils and later sulphonated hydrocarbons.

Presently two classes of fatliquors predominated throughout the leather industry namely;

1. the mayonnaise-type fatliquor which is an oil in water emulsion where the emulsifier is primarily soap or soap and protective colloid;
2. sulphated oils of various types. In addition, however, some of the newer synthetic emulsifier of the cationic, nonionic and anionic types are being used for specific purposes. The processing conditions both before the fatliquoring operation and after must be taken into account in selecting the proper fatliquor for a particular job.

2.1 Types of Oils Used in Fatliquoring

The types of oils used include animal fats and oils, vegetable oils, marine oils, mellon and degauss, oil substitutes, synthetic materials, mineral oils and sulphonate.

Oils vary in their melting and freezing points, interfacial tension against water, polar characteristics, viscosity, etc. The cause of these differences is the chemical structure of the various fats and oils. Glycerine is the common structure of all fatty oils, the differences among them exist in the chemical nature of the fatty acids present in the fat. These determine its physical characteristics which are important from the standpoint of fatliquoring and, in addition, determine the manner in which the oil will react with sulphuric acid to form sulphated oils.

The product formed after sulphation determines the characteristics of the emulsion which will be formed from the sulphated oil which, in turn, plays an important part in the deposition of the oil in the leather, which of course affects the characteristics of the leather produced.

2.2 Synthetic Lipids

Deslkan (1974) in his work, "Application of Petroleum sulphonate" reported that leather processing lubricants like

sulphated animal and fish oils may be improved by the use of petroleum sulphonate; some fatliquors now composed of sulphated animal oil, petroleum sulphonate, some coupling agents and mineral oils combined with water can provide leathers with good amounts of lubrication from this combination of softening oils. Germans manufactured synthetic fatty substances using coal, gas and petroleum fractions as starting materials. Research on these materials produced very interesting results on leather, however, it was concluded that the natural fats, oils and waxes were preferred, unless unusual conditions like war hindered the import of the natural materials.

The primary function of lubricant is to prevent adhesion of leather fibre and to influence the degree of fibre cohesion which takes place during the drying of wet leather, fatliquors also give a certain degree of "body" to leather (Kedlaya (1978)). Additional studies on replacement of fats have been reported by Gutensohn (1953) and Pense (1956) who investigated the chemistry of chlorinated Fischer Tropsch hydrocarbons and their properties as

fatliquoring agents; the latter products have the desirable property of resisting bacterial attack and spew.

2.3 Production Alternative Methods

For many centuries, the leather lubricants were incorporated into leather by simply smearing wet leather with mixture of greases and oils. The use of oil in water emulsion, now called fatliquors, originated during the twentieth century (Wilson (1941)).

The early fatliquors were prepared by emulsifying the oil in water through the use of common surface active agents such as soap, and/or the use of protective colloids such as egg albumen, casein, or natural gums. Recently, the more modern surface active agents have come into wide spread use beginning with sulphated fatty oils and later sulphonated hydrocarbons.

The processing conditions both before the fatliquoring operation and after must be taken into account in selecting the proper fatliquor for a particular job. Any generalization on the subject of fatliquor must be used only as a guide to the

understanding of the subject, because any change of specific conditions will give different result. All conditions must be known before an optimum fatliquoring system can be determined for specific formulation for a particular type of leather.

A sulphated product may be formed by esterification of sulphuric acid to an ethylenic linkage. This makes possible the production of a wide variety of chemical compounds falling into the class of sulphated products. In fatliquoring, sulphated oils are by far the most important group within the anionic class from stand point of commercial use. Other compounds within the class which are used are sulphated fatty acids, fatty alcohols, amides and sulphated esters of various types.

Another method of fatliquoring of oils is the sulphonation. A sulphonate and a sulphate are characterized by the following formulas:

$R - SO_3Na$, $R - O - SO_3Na$

(sulphonate), (sulphate)

A carbon to sulphur bond in case of sulphonate distinguishes it from sulphate, a carbon to oxygen to sulphur bond. The process of producing sulphonated oil is sulphonation or sulphitation to produce fatliquor.

Becher (1957) discussed the theory and manufacturing of soap-type emulsions.

2.4 **Process Description**

It is extremely important to keep in mind that the conditions and methods in sulphating an oil determine the characteristics of the end product. Starting with the same basic oil, it is possible to produce a wide variety of fatliquors by varying these conditions. Therefore, great care should be taken to avoid categorically classifying all sulphated cod oils or all sulphated neatsfoot oil as being one and the same thing. Bumcke (1927) after evaluating the properties of four sulphated oil from the standpoint if their respective analyses and their effect on leather concluded that an oil cannot be evaluated on one factor alone such as the combined sulphate or total fatty matter. Instead it is the combination of all the

various constituents into a workable whole that determines the value of the particular oil in fatliquoring.

In general, sulphated oils are produced by treating the oil with a given amount of sulphuric acid under controlled conditions of temperature, time and amount of agitation, followed by washing the acid and oil mixture with a salt solution to remove the excess acid. The acid salt solution is allowed to settle and is drained off. The sulphated oil is then generally neutralized with sodium, potassium or ammonium hydroxide until the desired pH is reached. Following this, the moisture content, total alkalinity, etc are adjusted to the desired levels. There are many variations on this process, all of which have distinct effect on the end product.

Meo and Folsom (1953) have summarized the reactions taking place during sulphation as follows:

1. The preliminary reaction when sulphuric acid react with a fatty oil takes place on the hydroxyl group or groups if present, and then may be followed by reaction at the double bond and both reactions result in the formation of ester type

linkages.

2. Due to the presence of water in the reagents as well as water formed as a by product of esterification or self condensation, a variety of compounds results, all of which are intermixed with the product.

3. The investigations of Stiasny and Riess (1926), Burton and Robertshaw (1940), Hart (1937), and Koppenhoeffer(1939) indicated that a sulphated oil is composed primarily of neutral fatty matter, consisting of unreacted glyceride, unsaponifiables possibly diglycerides formed by reaction, fatty acids which possess strong surface active properties.

Analysis of sulphated oils. For routine analysis of sulphated oils, there are in existence in most countries of the world, methods for analysis published by the respective leather chemist associations. The first attempt of analysis was on separate component of the sulphated oil. In recent years, the method of separation of sulphated oil into ;

1. The true fatty oil sulphates.

2. Free fatty acids, and

3. The neutral fatty oil and analyzing is being used.

O'Flaherty (1978) on review of literature on fatliquor concluded as follows; the leather literature is almost completely devoid of sound quantitative scientific information regarding the emulsifiers as they apply to fatliquoring. It is evident from a review of the literature on fatliquoring that many voids remain in our knowledge of the technology of fatliquoring, and also that a great deal of commendable work have been done by a great many competent people but the subject of fatliquoring still offers a challenging field for further research. These conclusions are valid up to date.

2.5 **The Cooling System;**

The two components of the cooling system are the coiled tube heat exchanger and the mixer. Survey of literature to effect modification of these components continued as follows:-

2.6 Coiled Tube Heat Exchanger Design Principles

Power generation, chemical industry and other technologies increasingly employ coiled tube heat exchanger. This has lead to wider research on heat transfer and hydraulic drag from different arrays of tube in a cross flow of gas or a viscous fluid. Tube coils afford one of the cheapest means of obtaining heat-transfer surface (Kern (1950)).

Although there are abundant literatures in Perry(1988) on heat transfer data, design principles and good approaches to equipment design, there still remains a not-well-understood problem which introduces error into every design. This is fouling from the fluids being processed. Fouling is the deposition of unwanted material on a heat transfer surface. Problems associated with fouling include financial loss due to increased energy consumption, high cost of maintenance, capital loss for an otherwise oversized equipment, decrease in throughput and increased pressure drop.

The traditional method of heat exchanger design procedure is usually to "throw in a factor" to account for fouling.

Due to the uncertainty in the values of fouling factors used, equipment over sizing as much as over 400% have been reported (Abanum (1996)). However, rational design procedure for heat exchanger exposed to fouling should allow not only the forecasting of how the fouling deposit will build up, but also the time and the extent to which it will happen, as well as the changing operating variables like temperature and velocity of fluid. To give this study a practical relevance, some of the data used were obtained from our the existing pilot plant.

Beek and Muttzal(1975) gave the average heat transfer coefficient for reactor vessels with coils and other correlations as follows:

$$u\rangle = \frac{(\alpha)d_c}{\lambda} = 0.17R_e^{0.67}\ P_r^{0.37}\left(\frac{d}{D}\right)^{0.1}\left(\frac{d_c}{D}\right)^{0.5} \quad \text{.....(2.1}$$

For $0.25<\frac{d}{D}<0.58$; $0.03<\frac{dc}{D}<0.15$ and $400<Re<2x10^5$.

The standard vessel configuration and that of the reactor vessels are shown in Figures 2.5 and 4.5.

$$\alpha_0 = \frac{N_{um}\lambda_m}{D} \quad \text{.....(2.2)}$$

$$\frac{1}{U} = \frac{1}{\alpha_i} + \frac{1}{\alpha_0} + \textit{Fouling factor} \quad \text{.....(2.3)}$$

Other correlations available were applied as shown in Appendix B as heat exchanger design.

2.7 Principles of Mixing and Separation Unit Operations

Quillen(1954) defined mixing as that which involves "intermingling of two or more dissimilar portions of a material resulting in attainment of a desired level of uniformity, either physical or chemical, in the product". The technology of mixing covers a very wide range of application and the knowledge of it is extremely useful in process operations (mixing of paints, mixed fertilizers, many pharmaceutical products), (Bello(1993)). Mixing and stirring are two conceptions which partly overlap. Mixing is the uniform distribution to a certain scale of inhomogeneities over a certain space. Mixing can be attained by stirring. The object of

stirring can also be to promote heat or mass transfers or to make suspension or emulsion (Beek and Muttzal(1975)). Mixing is usually accomplished with agitator rotating in a confined space. The agitator generates high streams of liquid which in turn entrain stagnant or slower moving regions of liquid resulting in uniform mixing by momentum transfer.

Convention, dispersion and diffusion are the three transport mechanisms that occur during mixing. Viscosity plays a large part in liquid mixing. As the viscosity of the fluid is increased, the mixing process becomes more difficult, since frictional drag retards the high velocity stream and confines them to the immediate vicinity of the rotating agitator.

The selection of a particular mixer will depend among other things, on

i. whether the process is batch or continuous.

ii. the liquid viscosity.

iii. the vessel volume or the desired residence time.

iv. whether the flow is laminar or turbulent.

Various types of impellers are shown in Figure 2.5. Mixing is also carried out to promote intimate contact between materials and\or to provide materials of new properties not necessarily present in the ingredients.

The importance of physical properties that may influence mixing are, for fluids, viscosity, density, relative density and miscibility. Solids; fitness, density, relative density, shape, stickiness and wettability.

If power curve is available for a particular system geometry, it can be used to calculate the power consumed by an agitator at various rotational speeds, liquid viscosities and densities. (Find (NR_E) -▸ power number, ϕ -▸ calculate power consumed). Additional power is required to overcome electrical and mechanical losses which occur in all mixing systems. Since mixing is an operation depending on fluid motion, it is logical to evaluate mixing in terms of fluid mechanic parameters. It has been shown by

Rushton etal (1950) that various conditions of fluid motion can be completely described by the power number, the Reynolds, and the Froude number, namely:

$N_P = \frac{P\rho}{4(N^3D^5)}$, power number } dimensionless groups for mixing

$N_{Re} = \frac{D^2N\rho}{\mu}$, Reynolds number } dimensionless groups for mixing

$N_{fr} = \frac{DN^2}{g}$, Froude number } dimensionless groups for mixing

for geometrically similar systems it is possible to relate power, gravity, size speed of impeller, density and viscosity of fluid by means of the equation,

$$N_P = K (N_{Re})^m (N_{fr})^n$$

where values of K,m&n are available for variety of mixing situation and are listed by Rushton et al (1950).

N_P = K for fully developed turbulence (high Reynolds number, no swirl, no surface vortex & baffles are in place).

and power is given by $N_P = \frac{K.\ \rho.N^3D^5}{g}$

Power is the product of flow and head, developed, at the impeller ($\phi_v \Delta h$) which is proportional to the cube of speed and fifth power of size; therefore $\phi_v \alpha ND^3$ and $\Delta h \alpha N^2D^2$.

Details from other literatures consulted are shown in the appendices.

2.8 Important Reactions in Fatliquor Production

Some of the important reactions that oils/fats and other resulting products undergo on addition of concentrated sulphuric acid to fatty oils.

1. Hydrolysis

   C_3H_5 (00CR)$_3$ +3HOH → $C_3H_5(OH)_3$ +3HOOCR, accelerated by high temperature and pressure and catalysed by acid.

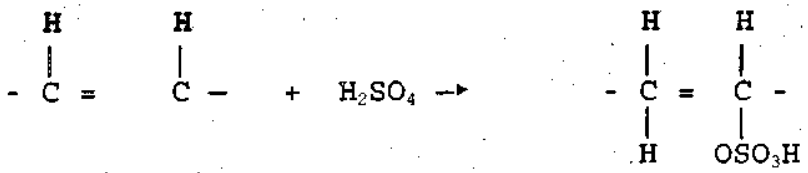
2. Saponification

   C_3H_5)OOCR)$_3$ + NaOH → C_3H_5 (OH)$_3$ + 3 NaOOCR

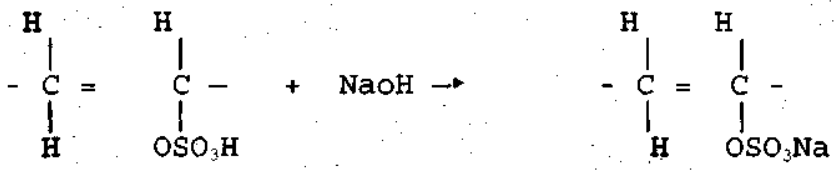
3. Sulphation:

   Unsaturated acid (radicals) readily react at double bond with strong sulphuric acid to introduce either the - OSO_2 OH

group or, at elevated temperatures - SO_2OH group.

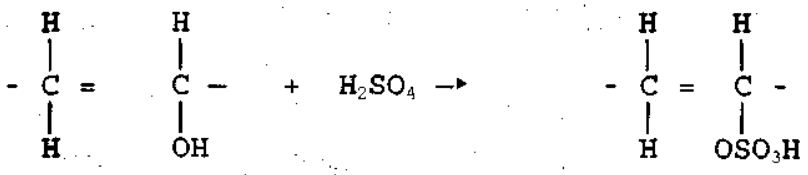


Neutralisation with NaOH



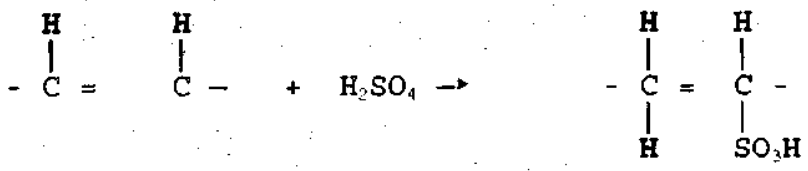
Sulphated (tri) glyceride.

Hydroxyl group and other reactive side group of fatty acid radical component are readily sulphated,



e.g castor oil.

Sulphonation: at elevated temperature, the carbon-sulphur bond is formed instead of the ester bond between carbon and oxygen of sulphated group at the α - position of the fatty acid

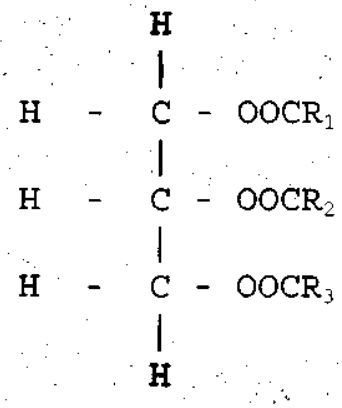


4. Polymerisation:

Oil rich in conjugated fatty acid radicals like tung oil polymerises very fast. Linoleic acid rich oils polymerises but only very slowly.

Theory of Fatliquor:

The chemistry of oils or fats are synonymous with the chemistry of the fatty acid radicals that constitute the triply cerides since the difference between various fatty component acid radical of the triply cerides e.g. of molecular structure of Triglycerides with R_1, R_2 and R_3 representing the hydrocarbon radicals (with or without side groups such as OH) of the fatty acid components of the oils/fats.



A typical sulphated oil or fat is a mixture of many substances, among are neutral oil or fat, free fatty acids, sulphated oil or fat and fatty acids, combined fatty acids, polymers etc. The fatliquoring quality of any sulphated oil or fat that results from sulphation process depends to a very large extent on the products and by products of such process. Sulphatability of a given oil increases with increased proportion of H_2SO_4 (conc) up to a certain level and falls thereafter, increase also with time duration up to a level, and but varies from oil to oil and sulphating conditions (Olawale (1993)).

If temperature goes beyond 25°C, there will be a lot of side rxns resulting in many by products like $S0_2$, H_2S and others there by decreasing fatliquor yield and quality.

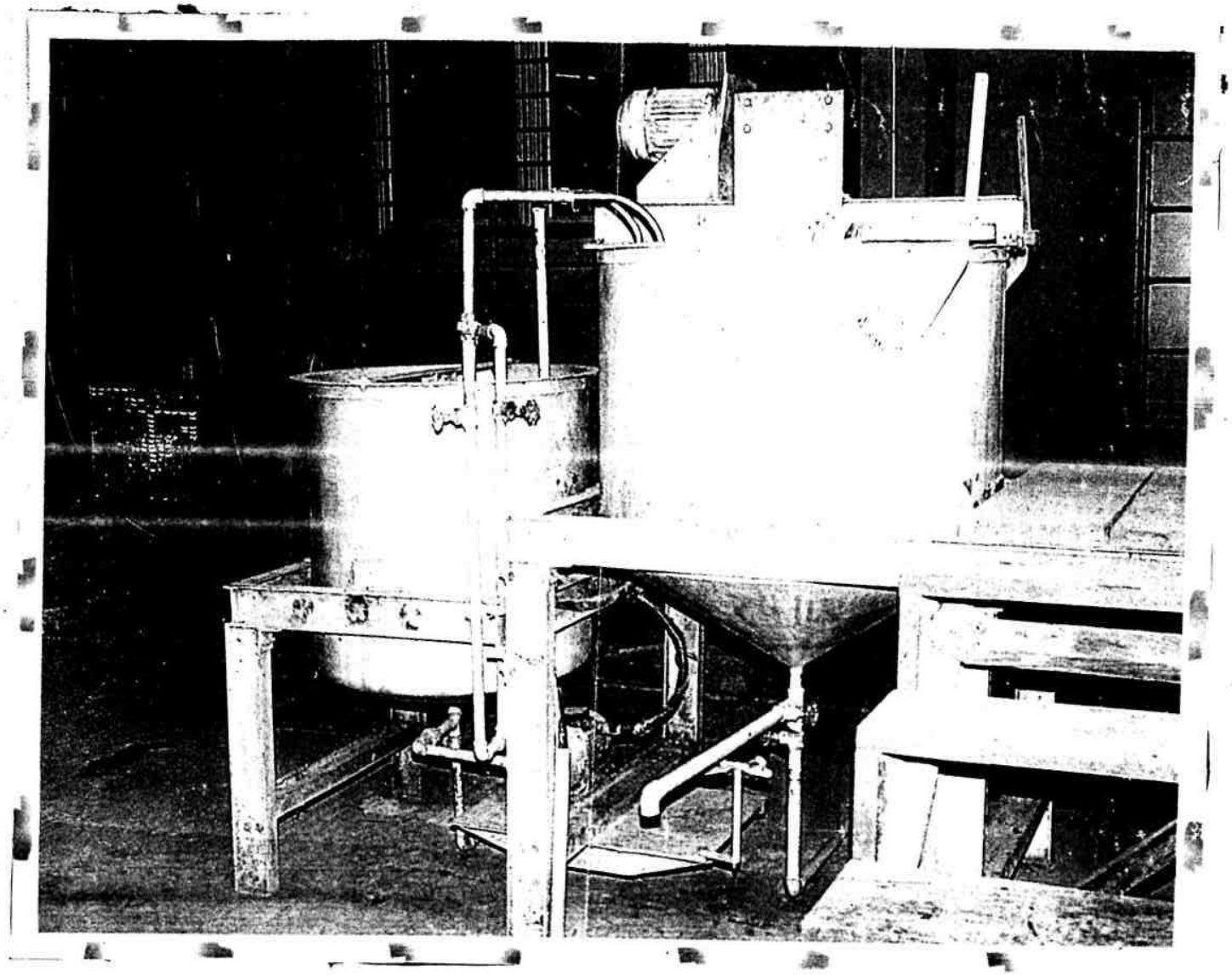
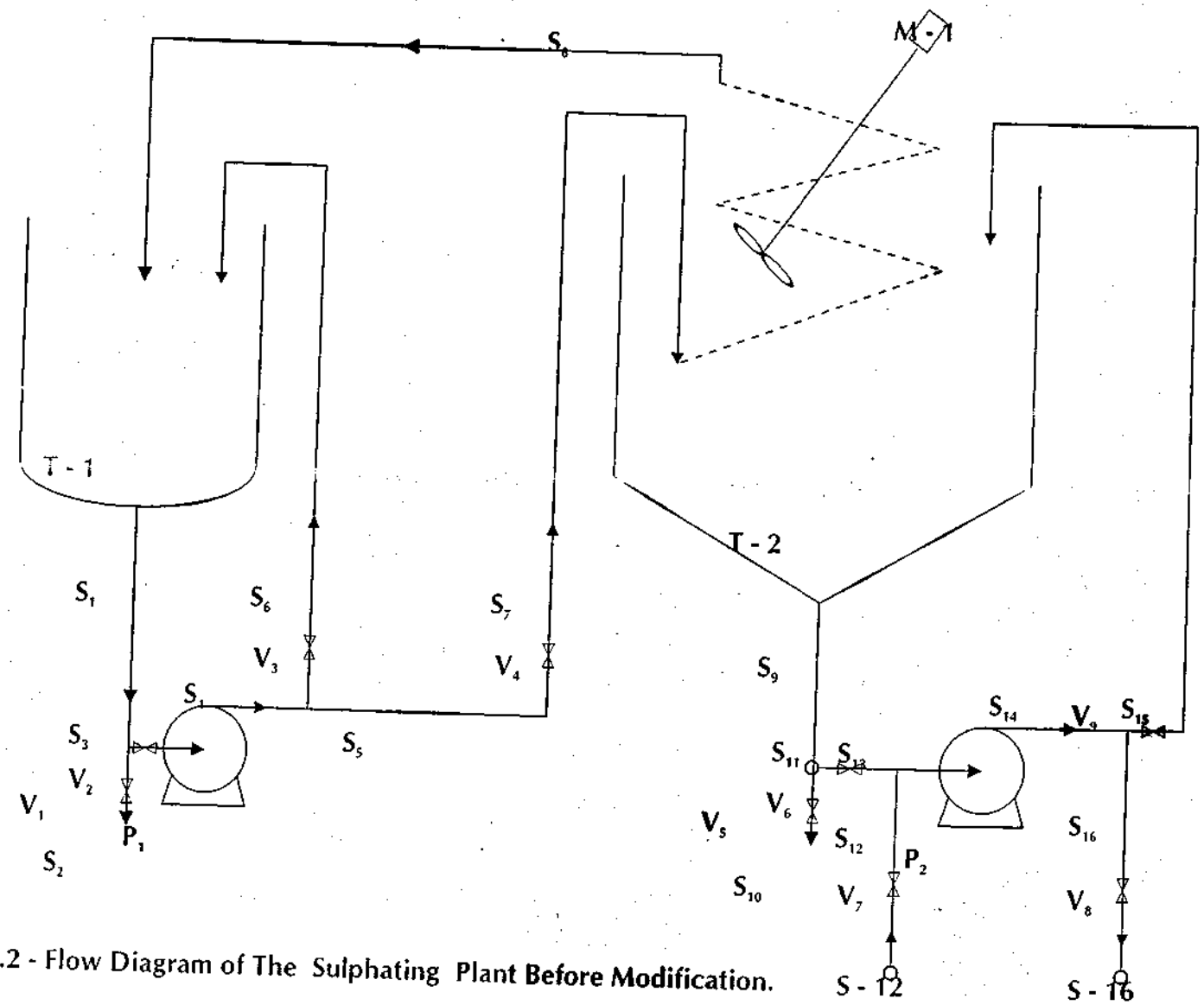


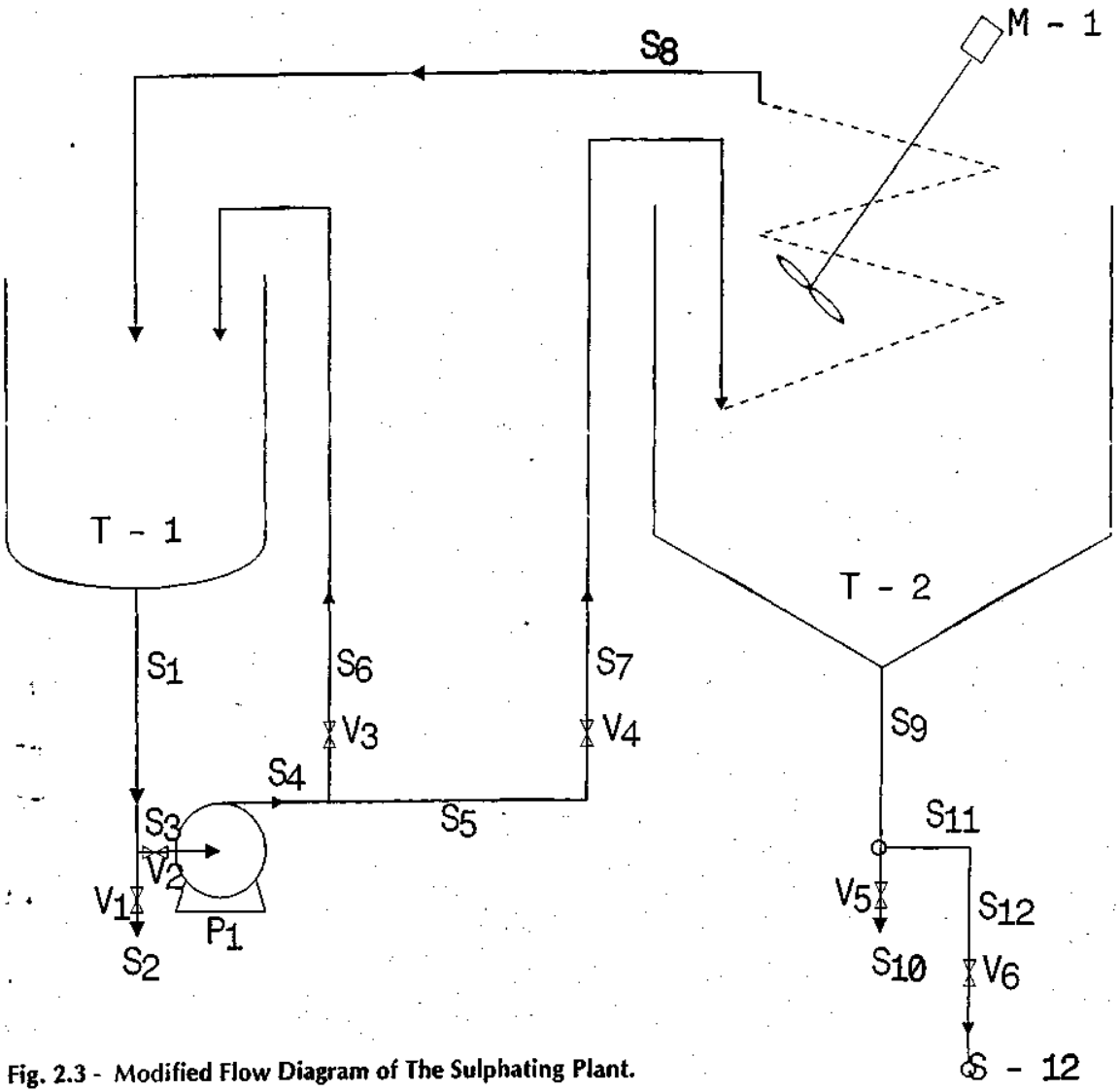
Fig. 2.1 Fatliquor pilot Plant, (Sulphating Plant)



KEY

SYMBOL	DESCRIPTION
T -1	Cooling/Heating Fluid Tank, 320 Litres Max.
T - 2	Sulphation Reactor, 570 Litres Max.
S_1 - S_{16}	Pipe Network, 3/4 Inch. Nominal Size
V_1 - V_9	Valves, 3/4 Inch.
M - 1	Motorized Stirrer, 2HP//220/380 Volts.
P_1 - P_2	Pump Drives, 1HP//220/380 Volts. 3.75/2.17A.
S - 12	Feed/Wash Liquor Inet
S - 16	Product Outlet

Fig. 2.2 - Flow Diagram of The Sulphating Plant **Before Modification.**



KEY

SYMBOL	DESCRIPTION
T -1	Cooling/Heating Fluid Tank, 320 Litres Max.
T - 2	Sulphation Reactor, 570 Litres Max.
S_1 - S_{12}	Pipe Network, 3/4 Inch. Nominal Size
V_1 - V_6	Valves, 3/4 Inch.
M - 1	Motorized Stirrer, 2HP//220/380 Volts. 6.7/3.9A.
P_1	Pumps Drives, 1HP//220/380 Volts. 3.75/2.17A.
S - 12	Product Outlet

Fig. 2.3 - Modified Flow Diagram of The Sulphating Plant.

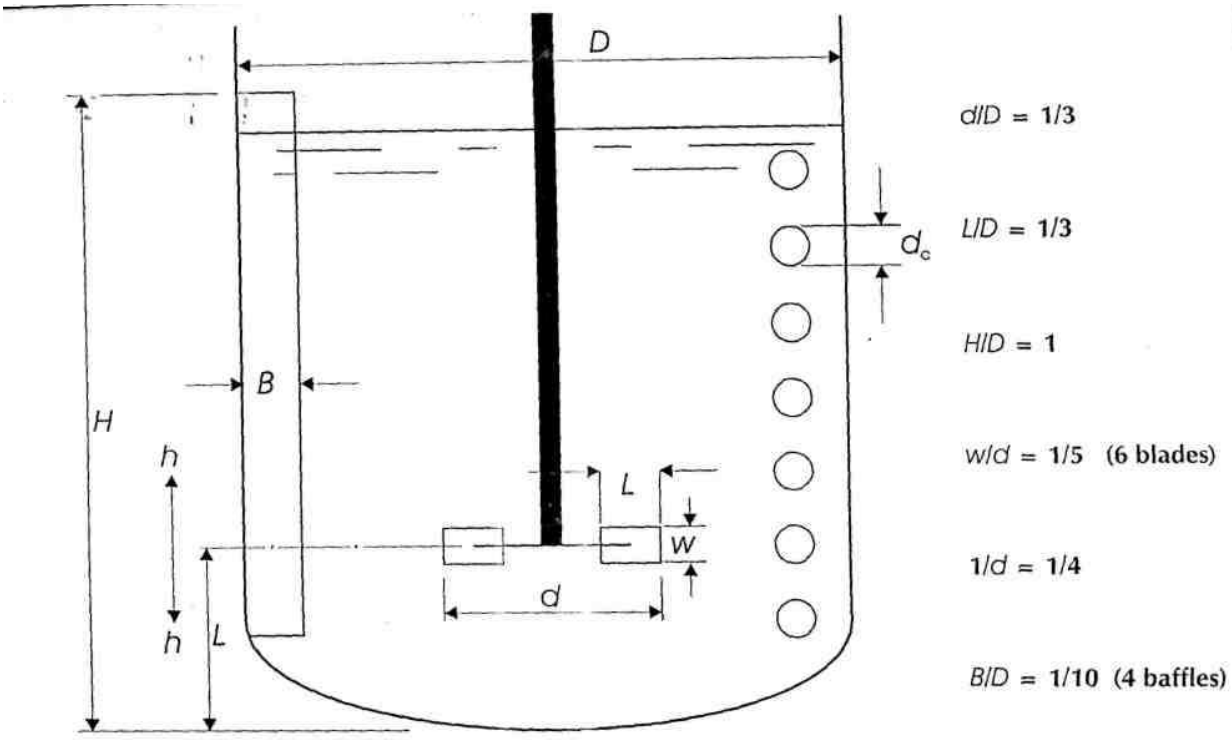


Fig. 2.4 - Standard Configuration of Stirred Vessel, Beek and Muttzall (1975).

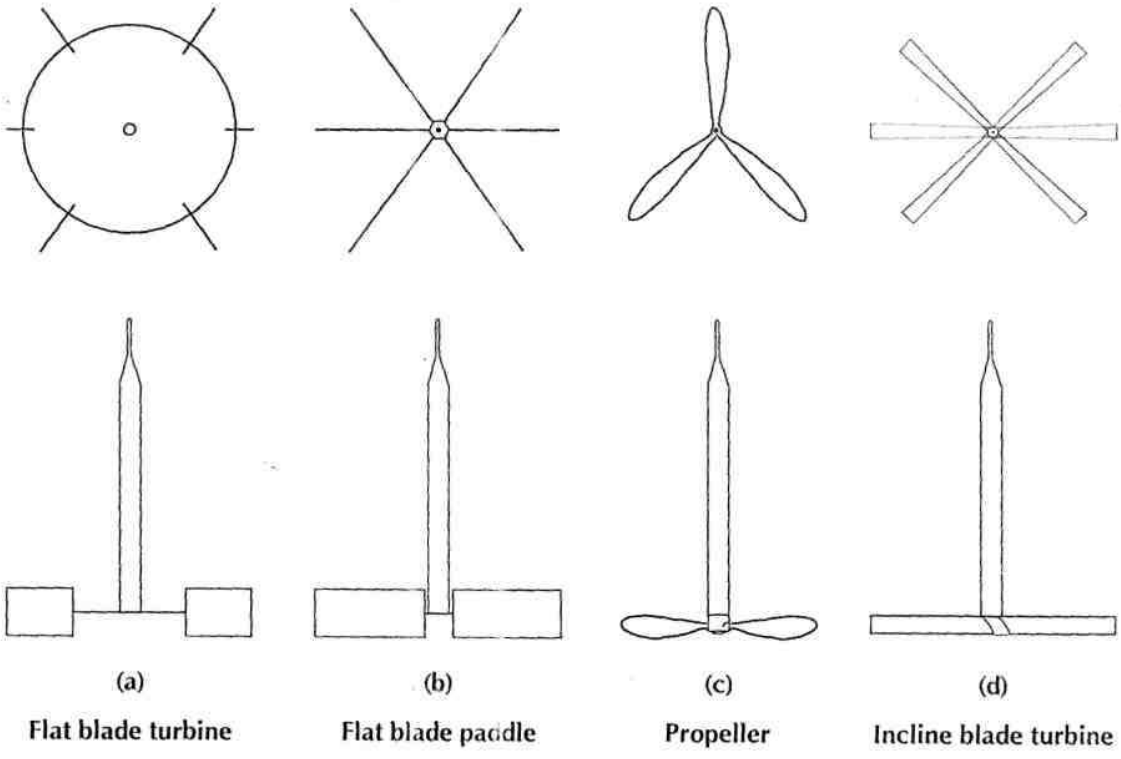


Fig. 2.5 - Various types of Impellers, Beek and Muttzall (1975).

CHAPTER THREE

3.0 EXPERIMENTAL PROCEDURE

From the problem definition, various methods of solution were suggested and examined. However the various limitations of some of these methods do not make them feasible. The scope of this work may not go beyond the chosen method of solution contained in this report. Four sets of experiments were carried out for this work as follows;

3.1 Experiment I: Laboratory determination of temperature profile of sulphation reaction

A set of experiments were carried out as follows:

The first approach was to determine the temperature profile of sulphation process at laboratory scale. This was done with the aim of getting the peak temperature of sulphation and using the peak temperature data to design coiled tube heat exchanger required for the cooling system. A measured value of groundnut oil was put in

a beaker, and a measured value of concentrated sulphuric acid was titrated into the beaker, and stirred. The addition of acid was not continuous. It was at the rate of 0.25mls to 0.3mls at intervals of 10 minutes. Each addition was stirred for 5 minutes before recording the temperature and the next addition, but the actual process of sulphation required continuous addition and stirring of the mixture, therefore this method may not give the correct data for the design.

In the second trial the temperature records were taken when the laboratory air conditioner was on and off. As well as in the third trial the temperature changes were noted at the same interval with the first and second and later at intervals of 2, 5 and 8 minutes. Still there was no continuous condition as the addition had to be stopped for stirring and noting of temperature changes on each addition. In the fourth trial of this first set of experiments, the temperature was continuously rising even above the required sulphation reaction temperature, meaning that there could have been other side reactions.

3.2 Experiment II: Determination of temperature profile of sulphation reaction at pilot plants for test run one.

This experiment was eventually adopted for use in this design because the existing pilot plant facility reflecting the actual sulphation process was used. The data got from this experiment were used for the design under the following assumptions;

i. That the heat released by the concentrated sulfuric acid `Q' during sulphation of the oil was the only heat of reaction, ΔH_{rxn}.

ii. Negligible heat losses to the environment.

3.2.1 Materials;

(i) Thirty (30) litres plastic aspirator bottle (graduated)

(ii) Three (3) mercury in bulb thermometer

(iii) Plastic buckets

(iv) Five plastic, 25 litres jerry cans

(v) 180kg of refined groundnut oil

(vi) 174 litres of sodium chloride solution(25%w/v)

(vii) 39 litres of sodium hydroxide solution (20%w/v)

(viii) 54kg of concentrated sulphuric acid 30%w/w based on oil.

(ix) Fat liquor pilot plant, Figure 3.1

3.2.2 **Procedure;**

Various instruments and equipment were used in the experiment but only the main experimental set up for the run are shown in Figure 3.0. Other tools used are be described in this procedure. 180kg of refined groundnut oil was measured into the sulphation reactor vessel at a room temperature of 23^{0}C. The first modified coiled tube heat exchanger was used to cool the oil to a temperature of 15^{0}C before the start of sulphation. The cooling water temperature in spite of the addition of ice blocks rose from 10^{0}C to 12^{0}C at start of the sulphation. The oil was continuously stirred with a motorized stirrer on addition of concentrated sulphuric acid at a feed rate of 0.25 litres per minute for 2 hours. The progress of reaction temperature is shown onTtable 3.1 and Figure 3.1. A maximum temperature of 41^{0}C was recorded, but later with the addition of more ice block to the cooling system, it brought down

the temperature to 32.5^{0}C which was closer to the required temperature of sulphation, 25^{0}C. On completion of the required addition of acid, stirring continued for more than 30 minutes for washing and salting out preparations.

3.2.3 **Washing and salting out;**

The resulting acid and oil mixture was washed with 25%w/v brine solution to remove the excess acid (sulphate ions). The product mixture was stirred for more than 15 minutes to allow for proper mixing and was allowed to settle over night or usually for 3 hours. To monitor the layer separation, some quantity of the reaction mixture was collected in a test tube for observation since the reactor vessel was not transparent. The aqueous layer was discarded by draining of the settled acid salt solution.

3.2.4 **Neutralization;**

The top layer, the sulphated oil was neutralized using 20%w/v sodium hydroxide solution to a pH of 6-7, and at which the emulsion of the reaction product was fairly stable in water. It took an hour to discharge a total product which weighed approximately 270kg.

3.3 Experiment III: Determination of temperature profile of sulphation for test run two.

The experiment II described above was repeated for 220kg of oil and the required weight of acid. The temperature change records are shown in Tables 3.2 and Figure 3.2.

3.4 Experiment IV: Determination of temperature profile of sulphation for test run three.

The procedure for experiment II was repeated except that there were changes in the quantity of raw materials used as follows;

3.4.1 Materials;

(i) 15 tins of 12kg each of refined groundnut oil=180kg

(ii) concentrated sulfuric acid=54kg

(iii) 36kg of NaCl in 242 litres of water (20%w/v)

(iv) 43 litres of 25%w/v sodium hydroxide solution.

Acid flow rates for sulphation;

For the first 60 minutes acid flow rate was 1.5l/5 minutes ≈ 0.3l/min

For the second 60 minutes was 1.0l/5 minutes ≈ 0.2l/min.

The temperature changes and volume of acid used for the process

are shown in Table 3.3 and also Figure 3.3 shows the temperature profile for the reaction.

Room temperature was 31^{0}C.

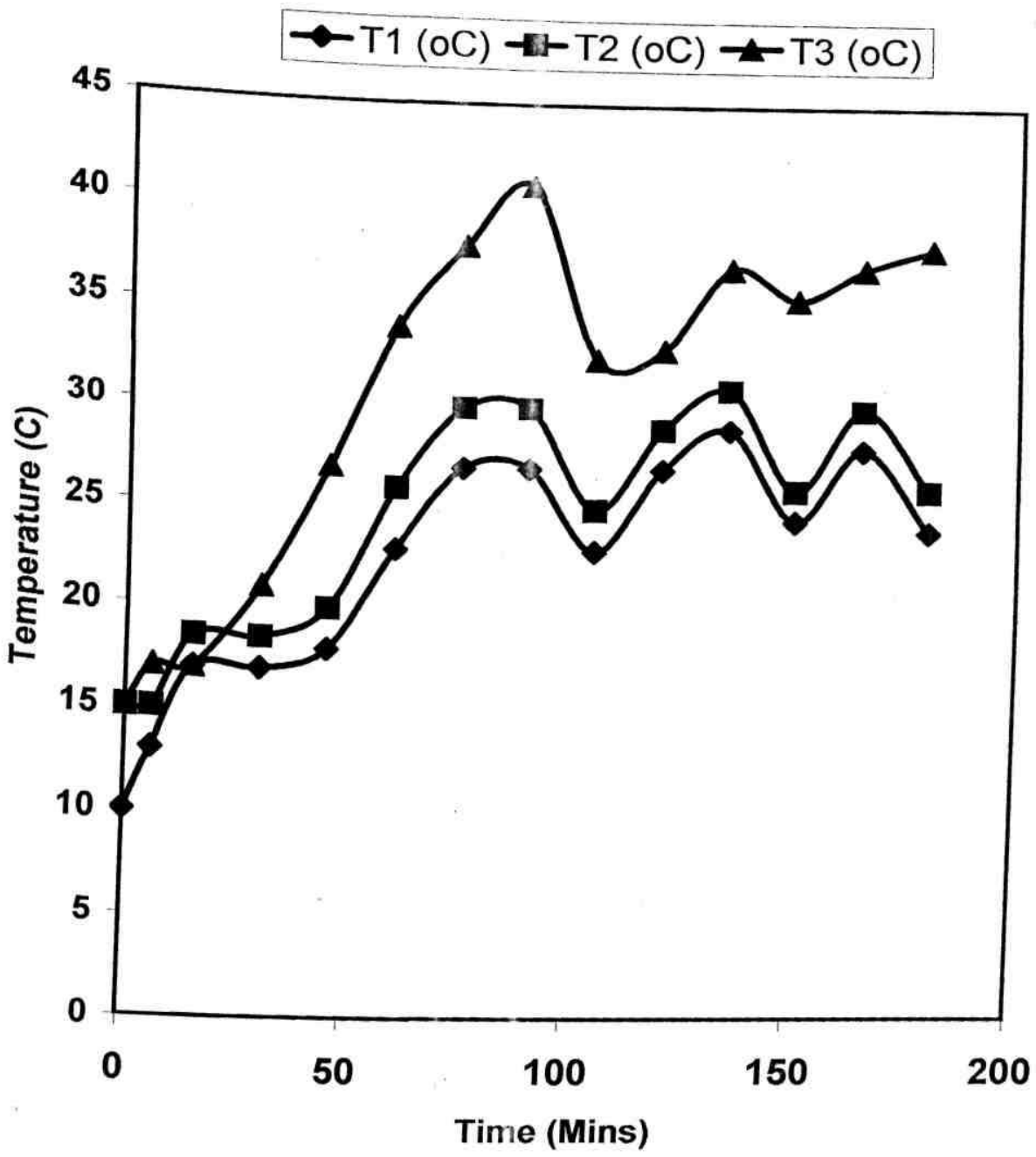


Fig 3.1 Temperature Profile of Sulphation of Groundnut Oil For Test Run I

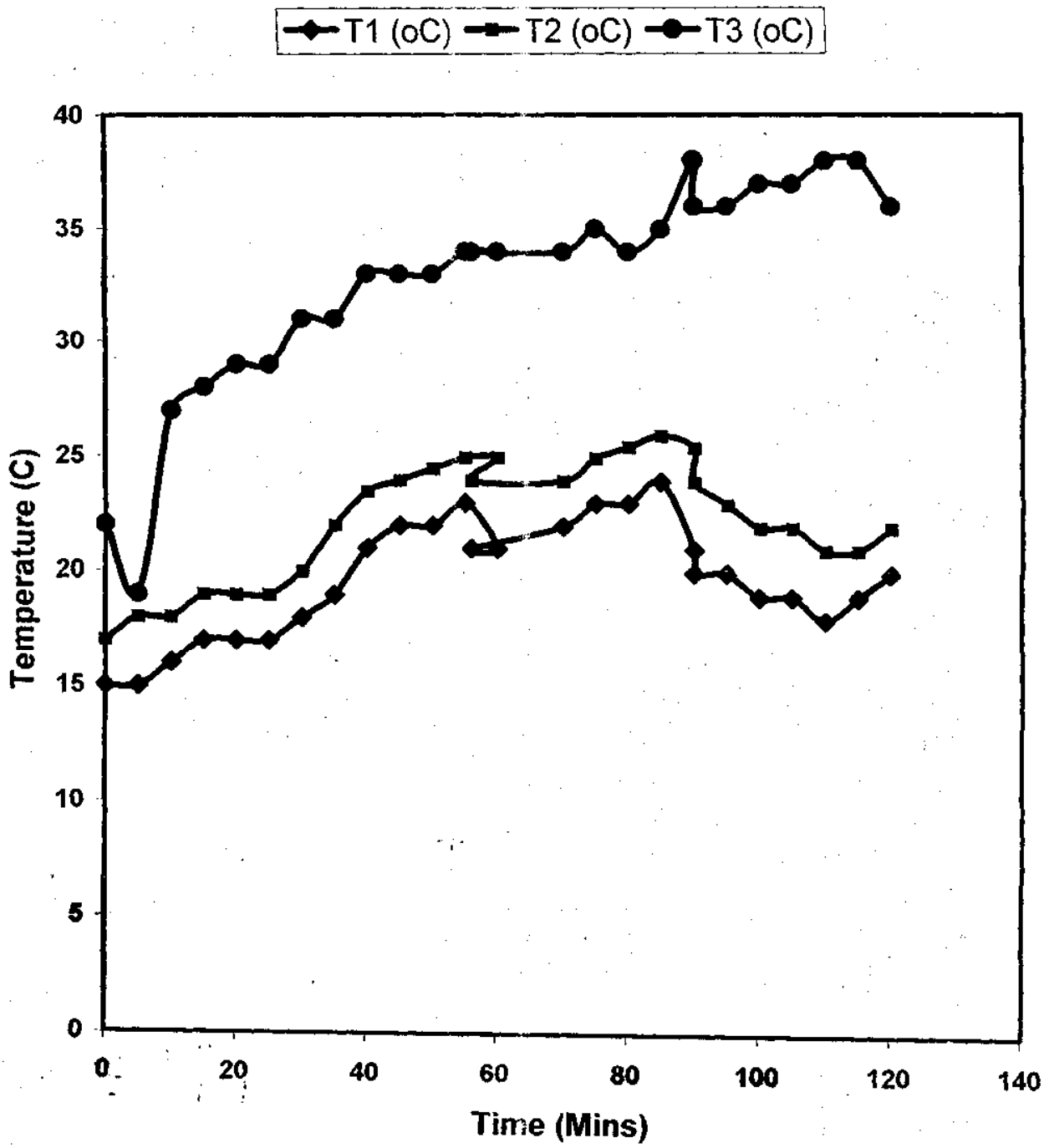


Fig 3.2 Temperature Profile of Sulphation of Groundnut Oil for Test Run II

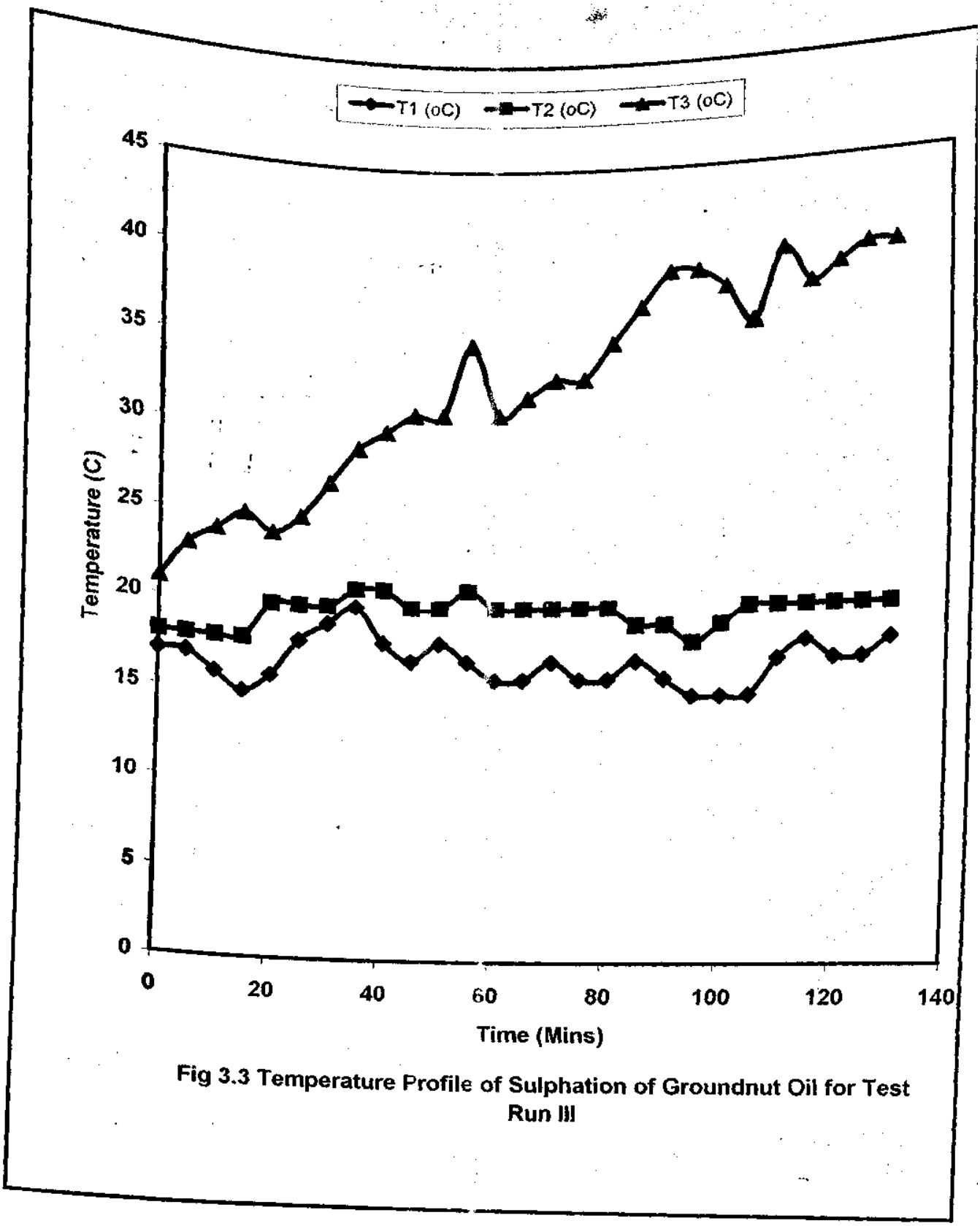


Fig 3.3 Temperature Profile of Sulphation of Groundnut Oil for Test Run III

CHAPTER FOUR

4.0 DESIGN MODIFICATION

4.1 Modification of the Cooling System Design

Experimental method and model construction for parameter estimation was used for this modification. The first approach was conversion of the coiled tube heat exchanger diameter of 0.055m to a smaller diameter of 0.025m to get more number of coils that would extend to larger surface of the reactor system. This was to enable the system to allow for smaller feeds as much as possible for a batch for research purposes to make it cost effective. A smaller diameter, 0.025m stainless pipe was recoiled and the total surface was extended.

On test running, it was not easy to maintain a reaction temperature of 25^{0}C as required by the process. The reaction temperature rose up to 41^{0}C in the three production batches carried out. Hence the motivation for this study was to redesign the plant

cooling system to bring the reaction temperature down to 25^{0}C or closer to it while observing other process conditions, and as well to reduce other process input required per batch to make research work more feasible in the plant. The importance of fatliquor production in the country and the huge investment of NARICT to this project also mobilized for this work.

To achieve the above, a more consistent and scientific redesign approach of the cooling system involving coiled tube heat exchanger design was adopted as the focus of this study.

4.2 Coiled Tube Heat Exchanger Re-design

Energy balance model in conjunction with experimental method of solution was employed. The experiments were as earlier described.

This involved the determination of the heat exchanger duty and a suitable heat exchanger surface area that would maintain the reaction temperature for the sulphation of groundnut oil for fatliquor production at 25^0 C. The coiled tube heat exchanger was to remove

heat of reaction of concentrated sulphuric acid and oil at reaction temperature of 25^0C. For lack of data on heat of reaction of the reactants and product complexes, the determination of the heat of reaction from the standard heat of formation or standard heat of combustion data was not possible; rather expensive and hectic experimental process and model construction for parameter estimation were used.

4.3 **Process Modelling.**

For lack of data on the heat of reaction for the process, the modelling of the heat transfer process for the sulphation of groundnut oil using concentrated sulphuric acid with some experimental input were used in the estimation of the process design parameters.

Assumptions;

(i) That the heat released by the concentrated sulphuric acid `Q' during sulphation of the oil was only the heat of reaction of oil and acid ΔH_{rxn}.

(ii) Negligible heat losses to the environment.

4.3.1 Heat exchanger design data and duty;

The duty of the exchanger was to ensure that the reaction temperature for sulphation of groundnut oil for fatliquor production did not go beyond 25^{0}C. The heat exchanger was to remove the heat of reaction of concentrated sulfuric acid with oil at a reaction temperature above 25^{0}C. The inlet and outlet temperatures for the cooling water are shown in Tables 3.1,3.2 & 3.3. The fluid properties, densities, viscosities, thermal conductivities and specific heats are shown in Appendix A.

4.4 Design Principles for Determination of Heat of Sulphation Reaction;

For an exothermic reaction, the energy required to hold the products of the reaction together is less than that required to hold reactants together and the surplus energy is released, the reverse is true of an endothermic reaction, (Himmelblau (1989)). The energy change that appears directly as a result of a reaction is termed, heat

of reaction, ΔH_{rxn}. To take account of possible energy changes caused by the reaction, in the energy balance, in the enthalpy of each individual constituent an additional quantity termed the standard heat (really enthalpy) of formation $\Delta H(^{\circ}F)$ was incorporated. Heat of reaction (ΔH_{rxn}) can be calculated from:

(i) standard heat of formation data and

(ii) standard heat of combustion data, but for lack of these and complex nature of the reaction mixture and products in the batch reactor, the modelling using available experimental data was employed in this work.

4.4.1 **Heat balance;**

The heat balance method was applied to evaluate ΔH_{rxn} for the above process as follows.

With respect to the assumptions above, the heat balance taken at maximum attainable temperature during sulphation, maximum heat duty for the exchanger, for the cooling water and the reaction mixture for the batch process, the heat generated by the reaction mixture during sulphation, ΔH_{rxn} is equal to heat taken up by the

reaction mixture and the cooling water . The reaction mixture includes, sulphated oils, by products and unreacted materials for the batch.

$$\therefore \Delta H_{exn} = \Phi = MmCP_m (DT)_m + M_w CP_w \Delta T_w \ldots\ldots\ldots\ldots 4.1$$

$$but \quad \Phi_H = \frac{\Phi}{t} = \frac{M_m}{t} CP_m \Delta T_m + \frac{M_w}{t} CP_w \Delta T_w \ldots\ldots\ldots 4.2$$

$$\Phi_H = \Phi_m CP_m \Delta T_m + \Phi_w CP_w \Delta T_w \ldots\ldots\ldots 4.3$$

The heat exchanger design equation is

$$\Phi_H = \bigcup A \Delta T_{LM} \ldots\ldots\ldots 4.4$$

The above equations were used in the evaluation of heat exchanger design parameters. The detailed calculations are shown in Appendix B.

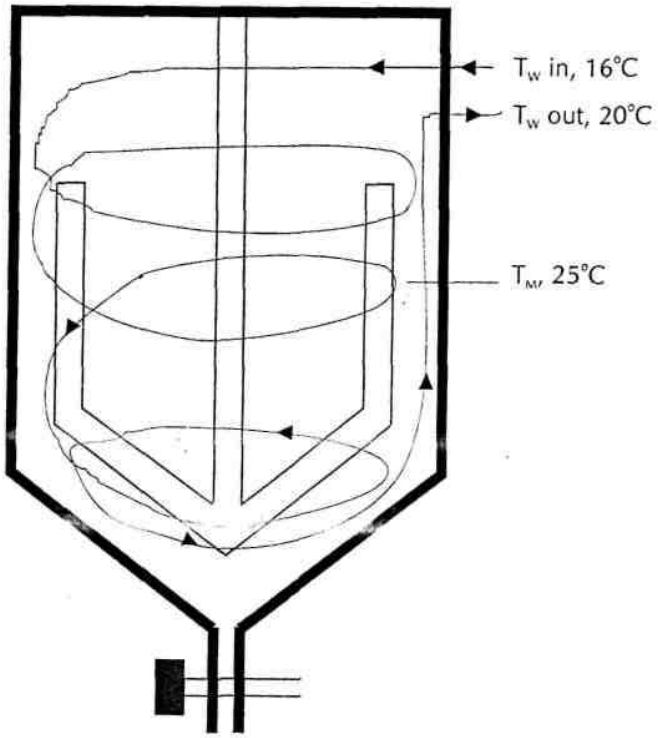


Fig. 4.1 - Coiled Tube Heat Exchanger,
Sketch of Heat Flow in the Reactor Vessel.

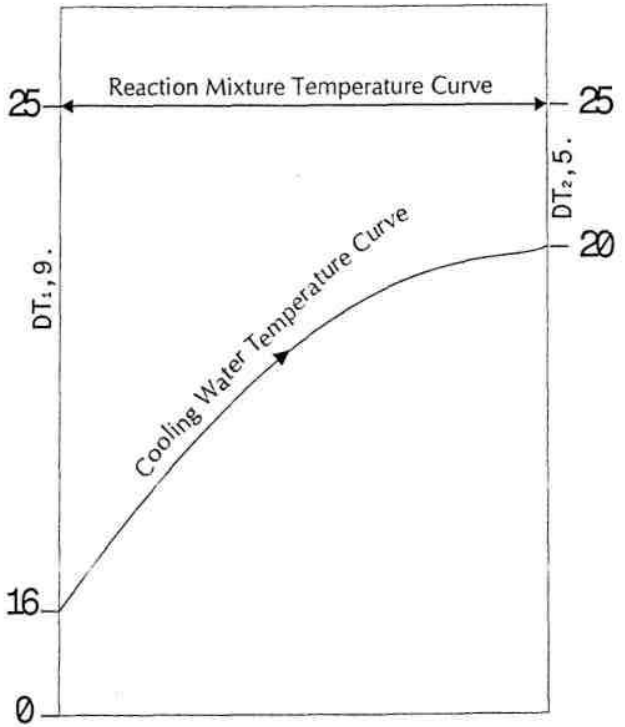


Fig. 4.2 - Graphical Sketch of Heat Flow in the
Reactor Vessel in Degrees Centigrade (°C).

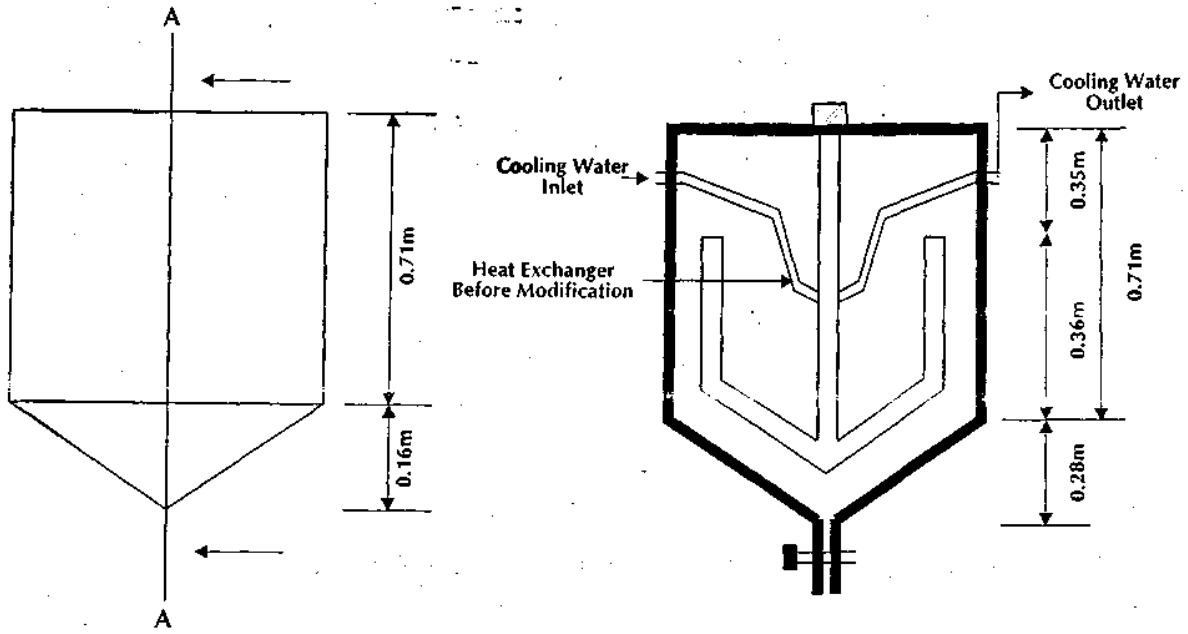


Fig. 4.3 - Side View and Cross Section of Reactor, with Heat Exchanger before modification.

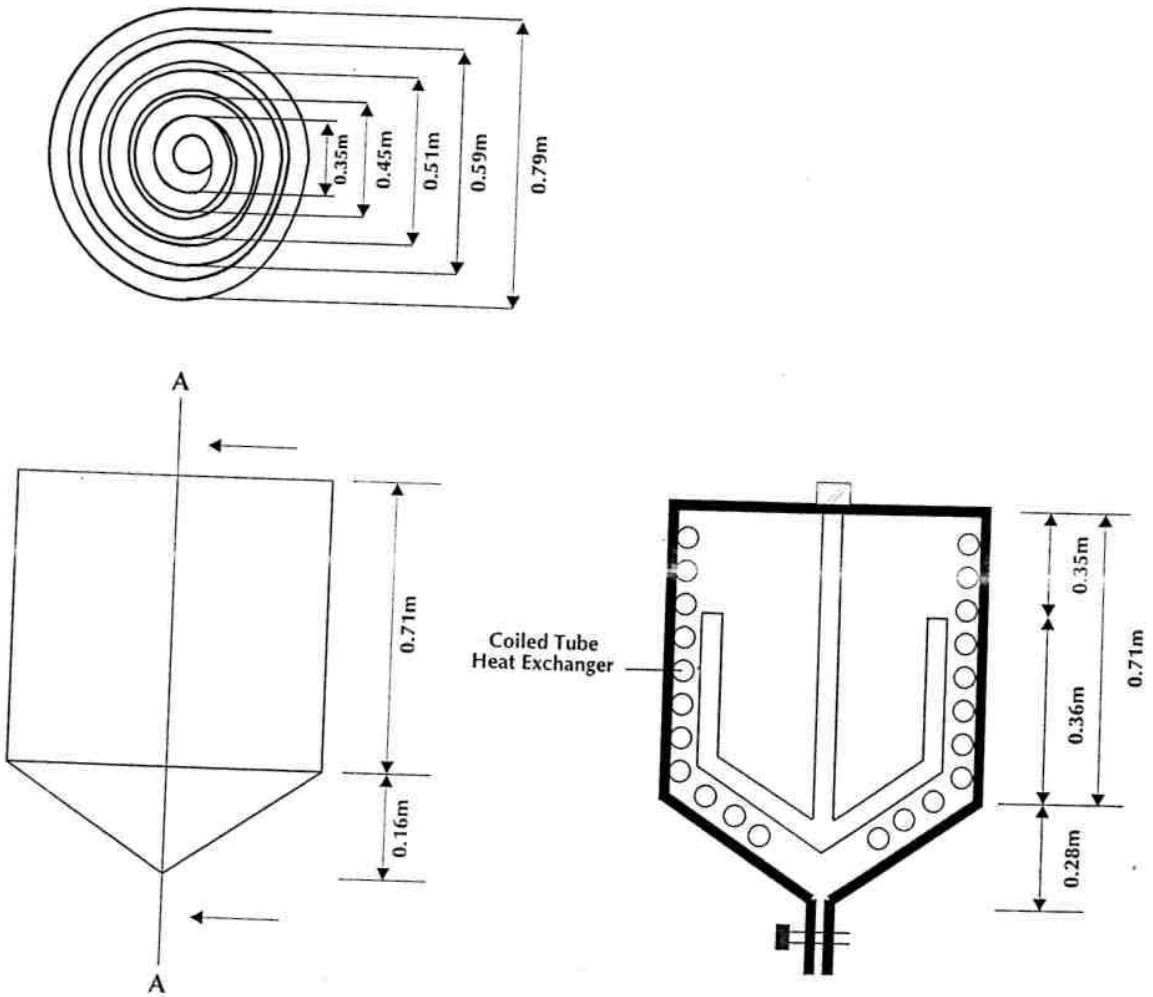
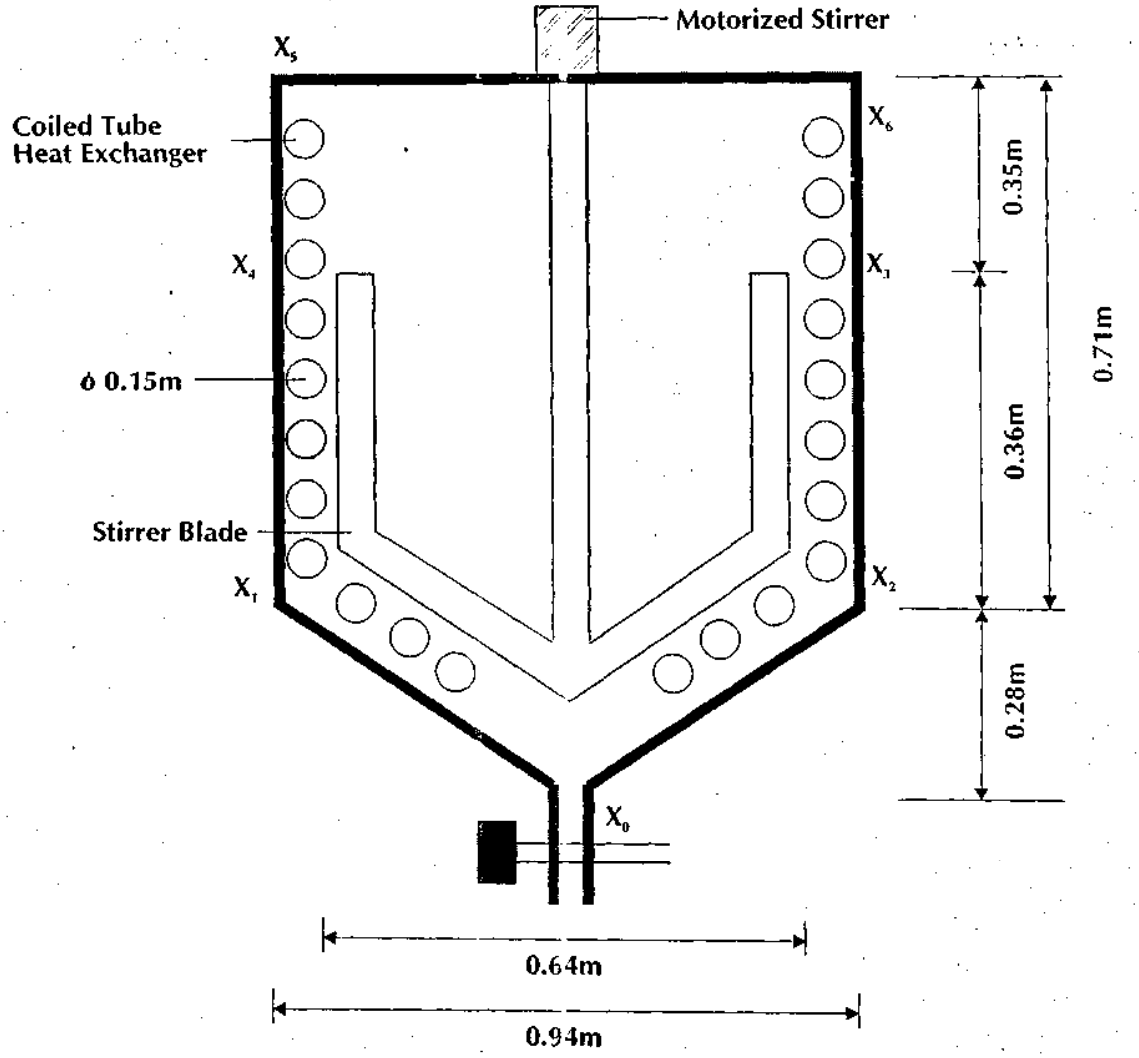


Fig. 4.4 - Plan,Side View and Cross Section of Reactor, with Modified Coiled Tube Heat Exchanger.



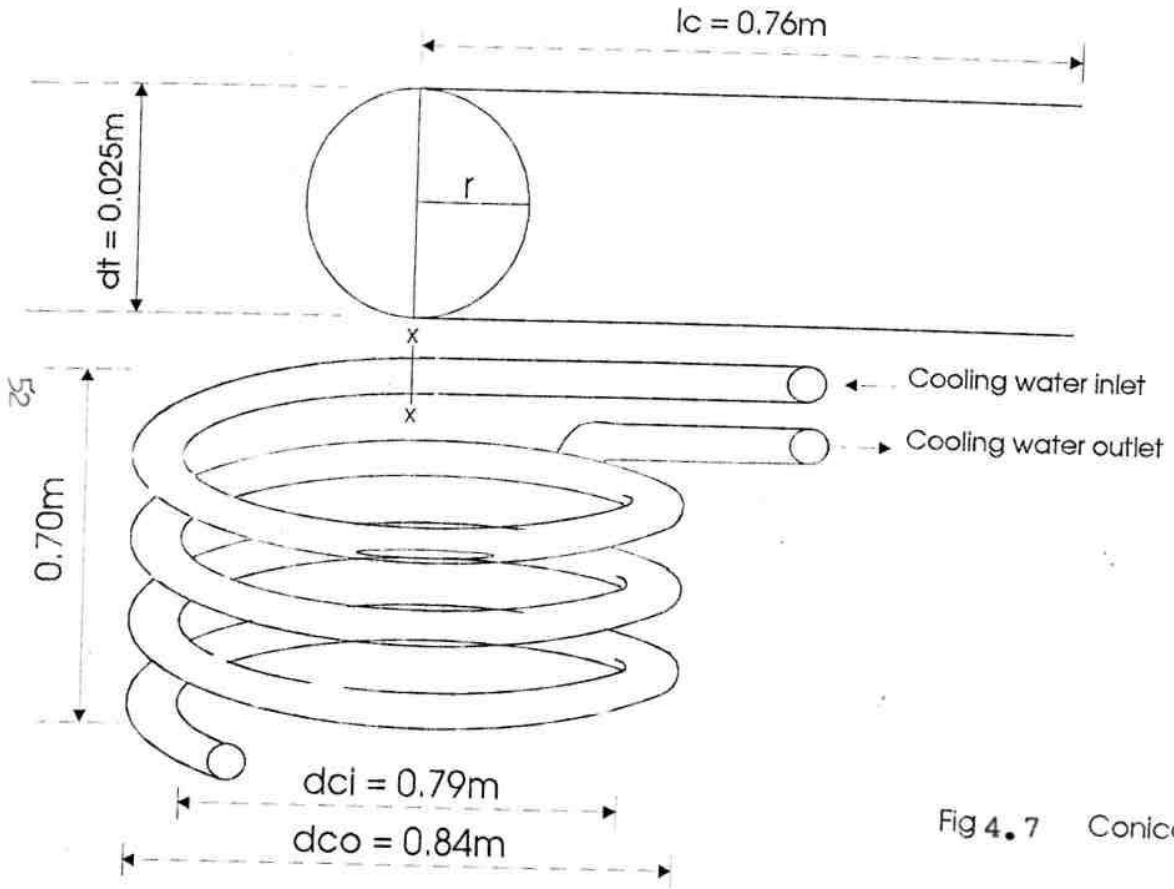
VOLUME MEASURES ABOVE X_0

$\overline{X_1X_2}$ = 65 Litres

$\overline{X_3X_4}$ = 315 Litres

$\overline{X_5X_6}$ = 560 Litres

Fig. 4.5 - Reactor Internals and Dimensions, Cross Sectional View (AA).



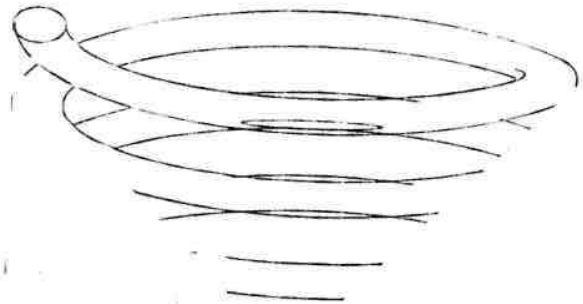


Fig 4.7 Conical Section of the Coiled Tube Heat Exchanger

FIG. 4.6 Dimensions of the Cylindrical Section of the Modified Coiled Tube Heat Exchanger for the Reactor

CHAPTER FIVE

5.0 RESULTS AND DISCUSSION

5.1 Temperature Profiles of the Trial Runs

Changes in temperature for the sulphation process for the three trial runs are shown in Tables 3.1, 3.3, and 3.5 and Figures 3.1, 3.2 and 3.3. These changes in temperature, resulting in rise of temperature of reaction up to 41°C, well above process requirement of 25°C in spite of all efforts of modification was the motivation for this study.

The temperature recorded in Tables 3.1 and 3.2, show that in spite of the addition of ice block to bring down the reaction temperature, it was still rising, this may indicate that the total surface area of the coiled tube heat exchanger was not enough for the expected duty. It may also suggest that the mixture agitation was not sufficient, because there were instances where the temperature of the reaction mixture were different at the top and the

bottoms. The first configuration of the heat exchanger to cover about $^1/_3$ of the reactor vessel volume as in Figure 4.3 was the reason why the heat exchanger was changed to coiled tube heat exchanger. The three test runs show that the coiled tube is yet to bring about the required process temperature of 25°C. The temperature records of the test runs show that sometimes when ice blocks were added the temperature of the cooling fluid became low, yet it did not bring about the desired cooling effect in the reaction mixture. The stirrer and the reaction vessel configuration could be part of the problem of not having the required cooling effect. However the inadequate surface area for cooling appears to be the major problem.

Figures 3.1, 3.2 and 3.3 show graphically the temperature profile results of the three test runs. The three curves for each of the figure are temperature against time, T_1, for inlet temperature profile of the coolant, T_2, the outlet temperature profile of the coolant and T_3, the reaction mixture temperature profile curve which is the real temperature of the sulphation of groundnut oil process for

production of sulphated oil. In the three figures, that of the sulphation is on top of the other two curves. Under the experimental conditions employed in, this curve explained the reaction process better. The curve agreed with the theoretical representation of the temperature profile for sulphation of groundnut oil. Though the curve had several peaks, the first peaks are the highest and represents the maximum reaction temperature. At this peak it is assumed that this is the point of maximum heat input of the exothermic reaction to the reaction system. At this point, the design was based on the principle that if the temperature could be maintained at 25°C, that at any other point in time of the reaction, the temperature would not exceed 25°C. Theoretically, the peak temperature of 25°C would give the optimum yield of sulphated oil, where all the sulphated radicals were taken up by oil. Whatever radicals that were left may result in side reactions that impaired the desired sulphation reaction. Temperature above 25°C for the sulphation of groundnut oil may have promoted undesired reactions and products.

Table 5.1 Comparison of results of design modification recommended, with original and first modified designs

		Original Plant	First Modification	Recommended Modification
(i)	Maximum capacity of groundnut oil per batch	216kg	216kg	216kg
(ii)	Total surface Area of heat exchanger	$0.844m^2$	$2.614m^2$	$5.74m^2$
(iii)	Total tube length	4.22m	35.84m	74.24m
(iv)	Internal Diameter	0.055m	0.025m	0.025m
(v)	Outside Diameter	0.060m	0.030m	0.030m
(vi)	Number of coils at cylindrical section of 0.79 uniform coil diameter	Not coiled	14	28
(vii)	Number of coils at conical section of very Diameter (Fig.4.1) and (Fig.4.6)	Nil	Nil	4
(viii)	Shape of reactor vessel	Cylindrical/ Conical	Same	Same
(ix)	Stirrer blade	Not standard	Same	Same

5.2 Modified Design Dimensions and Configuration

The modified design to process 216kg of oil maximum capacity puts the total surface area required to carry out the defined duty at 5.74m^2. The total length of tube (stainless) of 0.025m internal diameter required was 74.24m. A provision of a tube length of 2m was also required for inlet and outlet extensions. The cylindrical section of the reactor vessel required 28 coils of 0.79m uniform diameters and the conical section of the reactor vessel should contain 4 coils of the stainless tube with varying diameter of the coiled tube as in Figure 4.1, other details of the dimensions and the configuration for the modified coiled tube heat exchanger that forms part of the cooling system are shown in Figures 4.6 and 4.1. The provision of the base coil at the conical section of the reactor vessel is very important as to allow wide variation of feed requirement for a batch process for research purposes.

5.3 Comparison of Modified Design with the Former and Standard Components;

(1) Heat exchanger surface area:

The former includes new heat exchanger and the old heat exchanger design being compared with the modified design under study. The total surface area for

heat transfer in the old design is 0.844m^2 and the new is 2.614m^2 and 5.74m^2 for the modified design. The difference for the three is much and could be the main source of inefficient cooling in the former two. The modified with the largest surface area is expected to have a better cooling performance.

(2) **Heat exchanger configuration:**

The heat exchanger arrangement for the old design in Figure 4.3, does not allow for larger surface area for cooling and it places a constraint on a batch feed variation. The new and the modified design are coiled tube arrangement which allows for larger surface area for cooling in the pilot plants system. However, the modified may be able to cool the system more efficiently and effectively because of larger surface area.

(3) **Shape of reactor vessel:**

The shape of reactor vessel is the same with the three design and vessel dimensions compared favourably with the standard and vessel dimensions of figure impellers but closer to propeller type of impeller in Figure 2.5. The type of blade for the three designs are shown in Figure 4.5. There were no baffles for the three designs. The implication of this is that the power consumption is likely to be higher. However, the stirring degree for the former designs were good as shown

during the test running experiments. This is because vortex was not much even though the flow was laminar. Also the product of the test run showed lack of proper agitation. Proper agitation could be achieved if baffles are introduced to the reactor vessel and as well titrate the washing and the Neutralization liquids into the system as against the usual pouring of measured quantity at intervals.

(5) **Feed requirement constraint:**

The two former design have a constraint for feed, as can be seen in Figure 4.3, that any feed below the heat exchanger facility may not be processed, as that will amount to no cooling, since the reaction mixture may be burnt as a result of the exothermic reaction. This does not allow for variable feed for research purposes as a lot of material could be burnt in trial run that requires large material. It makes the trial runs not to be cost effective in terms of process inputs. The second design does not have the conical section of the coiled tube heat exchanger, therefore the feed cannot be varied as much as in the modified design.

5.4 Production Process

The pilot plant production process for the old plant (1st design) can be illustrated with Figure 2.2 and Figure 2.3 for the second and the modified design.

In general, sulphated oils are produced by treating oil with a given amount of sulphuric acid under controlled conditions of temperature, time, and amount of agitation. There are many variations on this basic process all of which have a distinct effect on the product, (O'Flaherty and Roddy (1978)). The results of the trial runs production process could be discussed as follows:

(1) **Sulphation Process**

The sulphation process for the two test runs for the plant were carried out as described in the experimental. Inspite of all the cooling efforts of additional ice blocks to bring about the desired temperature of reaction, maximum reaction temperatures of 41°C and 39°C were recorded for the first, second and third runs respectively. The sulphation was completed after 2 hours in each case.

(2) **Salting out and washing**

The salting out and washing was done, 30 minutes after the acid addition, with 20% w/v of NaOH, sodium hydroxide solution with constant cooling and agitation. The washing lasted for 30 minutes in each case. The agitation and cooling were put off and the reaction mixture was left for an hour to settle. A sample was collected in a small beaker to monitor the complete layer separation time since the reaction vessel is not transparent. The time interval between

washing and the beginning of neutralization is to allow for complete layer separation; but should not be left for longer periods before Neutralization, because there is possibility of desulphation.

(3) **Neutralization**

Before neutralization, the bottom layer that contained water and salt used in washing were decanted. The temperature profile of this stage of reaction showed exothermic reaction. In each of the test runs, the decant liquid showed a green colouration suggesting possibility of contamination from pipe lines, since the colour was supposed to be just like the salt water used for washing. pH of 6.0 and 6.5 in each case show complete Neutralization.

(4) **Products**

The products of the first test run was better because the emulsion was stable and the product lubricant did not thicken after discharge as was the case in second trial. In both cases the stored products had to be stirred again to ensure proper agitation. However, it was noted that agitation improved, showing some turbulence because of the increased volume of reaction mixture that made the impeller blade to be fully immersed and the edges of the impaler helped to tear the

liquid apart. The emulsions were stable but the colours were changing showing deviation from a standard fatliquor. The improper mixing may have resulted from not titrating the wash liquid and the Neutralization reactant into the reaction mixture. The product output in each case fall short of the theoretical expectation. This may be as a result of the undesired reaction and product from the effect of temperature above the desired reaction temperature.

5.5 Design Consideration for the Pilot Plant Design Modification.

The test run experimental investigation to determine what component of the plant was to be modified narrowed the faulting components to the cooling system. However, there might be other approaches to the problem but the focus and scope of this work was on the cooling system.

The cooling system is made up of the:

i. The cooling fluid Tank;

ii. The cooling water pump;

iii. Motorized stirrer;

iv. Coiled tube heat exchanger (could be used for cooling and heating but designed for cooling).

The evaluation of these components identified the coiled tube heat

exchanger as the one requiring major design modification and slight adjustment of the cooling water pump and the stirrer blades. Also identified to improve the cooling and the production process were some operational changes, addition of wash liquid and Neutralization materials precisely, as will be highlighted in the recommendations for remedies.

5.6 Design Modification of the Coiled Tube Heat Exchanger

The duty of the heat exchanger is to fix the reaction temperature for groundnut oil sulphation process using concentrated sulphuric acid for fatliquor (sulphated oil lubricant) at 25°C. The heat exchanger is to remove the heat of reaction of concentrated sulphuric acid and oil at a reaction temperatures above 25°C (ΔHrxn at 2°C). For an exothermic reaction like this sulphation process, the energy required to hold the products of the reaction together is less than that required to hold the reactants together and the surplus energy is released, the reverse is true of an endothermic reaction, Himmelblau (1989). The energy change that appears directly as a result of a reaction is termed heat of reaction, ΔH_{rxn}. To take account of possible energy changes caused by a reaction, in energy balance, we incorporate in the enthalpy of each individual constituent an additional quantity termed the standard heat (really enthalpy) of formation, $\Delta H°f$.

Heat of reaction (ΔH_{rxn}) can be calculated from:

i. Standard heat of formation data;

ii. Standard heat of combustion data for simple substances.

The complex nature of sulphated oil lubricant mixture (fatliquor), several undesired side reactions and products for the sulphation process under discussion and unavailability of experimental data, standard heat of formation and combustion to evaluate heat of sulphation of groundnut oil using conc.H_2SO_4 resulting in a complex mixture and side reactions made the use of above theoretical method for our design impossible.

The leather literature is almost completely devoid of sound quantitative scientific information regarding these materials as they apply to fatliquoring, O'Flaherty and Raddy (1978). Rather process modelling, expensive and hectic experimental method of generating result to estimate design parameters were used in combination. Originally, it was intended to apply the two methods for the design modification and compare results but for lack of standard heat data for complex mixtures for the theoretical method. The coiled tube heat exchanger design modification is shown in Figures 4.6, 4.1 and 4.5. The desired temperature range for this reaction process is 25°C - 30°C and the modification design was done using

25°C to allow for design deficiencies of up to ± 5°C or as a safety factor. The design calculation details are shown in Appendix B.

5.7 Material of Construction and Equipment/Component Selection

The critical factors for consideration for selection of construction material for the coiled tube heat exchanger are availability, cost and corrosion. Availability factor and cost on the high side, not withstanding 3/4" stainless steel was chosen because corrosion is on the high side for this exothermic process. The 3/4" stainless steel pipe thickness small enough as not to hamper the flow of heat from the reaction mixture to the cooling fluid so as not to cause additional temperature gradient was selected.

The use of lead cladding to cover the inside and the outside of the reactor vessel is justified for safety measure against corrosion. The agitator blades were also covered with lead cladding for the same purpose.

1. Reactor vessel component

For this process the reactor vessel with coiled tube heat exchanger is better than the jacket vessel type for effective and efficient cooling.

2. **The agitator (Impeller)**

The impeller is very critical to the design since all he principal steps of the process require agitation. The swirling of the reaction mixture was observed especially when the reactor was not operated at full capacity. This may be due to the fixed speed of the stirrer motor. A motorized stirrer with variable speed may be preferred.

3. **The cooling fluid pump**

This pump also operates at fixed flow rate and this may account for a very high temperature gradient between the coolant and the reaction mixture. If it had a variable head it would have been better to observe the gradient of temperature at varying flow rate. However, the valves were used to make up for this varying flow rate which actually did not give any indication of attaining close range to the desired reaction temperature.

4. Coolant tank

The size of the coolant tank does not seem to constitute any problem to the process especially for the fact that coating of the tank does not allow for energy losses or gain. However the inlet and outlet of the heat exchanger goes to the tank,

but the outlet could be separated from the tank by the use of a rubber hose to make the monitoring of temperature change and the quantity of cooling water used less cumbersome.

5. **Temperature measuring device**

The temperature measurement is presently done by extending mercury in glass thermometer with a rope tied to the top of the thermometer to enable it go down both the coolant tank and the reactor vessel as much as possible. The temperature readings may not be reliable enough. For temperature measurements, a wide range of equipment are applicable, these includes; thermocouples, thermistors, resistance thermometers and radiation pyrometers. The temperature for our system under discussion is below 60°C, therefore thermocouple is suitable.

5.8 Design Modification Constraints

1. Lack of Design information on the existing pilot plant facility

This information would have been a very good basis for the modification. The only information available on the plant is the facility only. There were no, operation manuals, design details, process flow diagram or description on the pilot

plant. In the absence of all these the modification had to start with fundamental design principle that took more time and energy.

As earlier highlighted, the use of theoretical concept of standard heat of formation and combustion was not possible for lack of data for the complex reaction mixture products of the process. Provision of some of these basic information could be the major contribution of this work.

2. Cost of materials

The high cost of materials needed for this work was a major constraint. The cost of each trial run for the preliminary investigation was about N39,800.00 excluding labour, electricity and equipment depreciation costs. The cost covered by that amount are as follows as at 1996;

i. A drum of groundnut oil 220kg at N34,000.00

ii. 54kg of concentrated sulphuric acid at N45/kg

iii. 25kg of sodium chloride (NaCl) at N25/kg

iv. 12kg of sodium hydroxide (NaOH) at N100/kg

v. containers of about N500 per batch of production.

The cost becomes bigger when you consider that in trial runs for research, you may not get good products for sales as to recover your investments. There are

chances of acid burning the whole oils and you get no good products. This limited the trials to three.

Also the cost of procurement of 3.4" stainless pipe on the existing facility and the cost of coiling the pipe was up to N150,000 as at 1996. So to fabricate this modified design an estimated sum of N200,000 would be needed.

Provision of ice blocks for the cooling of the process was a problem as the plants is yet to have its' own ice making machine. There was also the energy problem as light was not steady for the production period, therefore standby generator fuel and the operator should be available each time production is going on.

Finally, the energy balance model used for this work gave good result especially in terms of reducing the raw material requirement for a batch production, since now the coil has to be extended to the conical section. This allows cooling even for small feed materials.

CHAPTER SIX

6.0 CONCLUSIONS

Some years back, following the increasing demand for fatliquor for production of good quality leather in the country, a fatliquor (sulphated oil) pilot plant was acquired by National Research Institute \for Chemical technology (NARICT), Zaria. The plant has been under- utilized for research, as a result of technical problems.

In spite of all earlier modification efforts, the problems are yet to be solved. The increasing demand for the product, time, money and amount of research efforts put into the development of this product was the motivation for this work. As a result, this project was commissioned.

In this study, theoretical and experimental investigations were conducted on the existing and earlier modified fatliquor pilot plant facility with a focus on meeting the required process conditions to reduce the overall cost of experimentation in the plant. To achieve this objective, a consistent technical approach for the design modification was done.

The results obtained from bench scale experiment conducted to generate temperature profile and data for parameter estimation for the design modification was discarded. This was because the process condition varied with the real process insipte of all attempts to make it similar, therefore may not give reliable results, as was indicated from its variation from theoretical stand point of view.

The results of the pilot plant test run experiment conducted for preliminary evaluation of heat transport within the system served as a better data base for the design modification. This is because apart from reflecting the real process, gave the modification a practical relevance, and may give a better solution.

Other conclusions that may be drawn based on the results of the theoretical, experimental and design modification carried out and being recommended for implementation include:

(i) For lack of data on the standard heats of formation and combustion for fatliquor product mixture (not compound), and other side product complexes, this approach could not be used for the modification even if it was for comparison of results with the employed method.

(ii) The laboratory determination of temperature profile curve failed to give peak temperature of sulphation for parameter estimation, because of lack of

adequate control of temperature of sulphation. This was because theoretically you may not attain optimum temperature if the reaction temperature goes beyond 25°C - 30°C, as other side reactions may be favoured rather than the sulphation, and the temperature continues to rise, rather than having a peak at 25°C.

(iii) Theoretically under controlled temperature, the sulphation curve is expected to have a peak at 25°C, since the temperature is supposed to be rising up to 25°C, when all the sulphate ions (SO_4^{2-}) have been all taken up by all the oil radicals present, there will be no reaction, resulting in sloping of the curve from peak temperature of 25°C.

(iv) The observed total surface area of 5.7m^2 for the coiled tube heat exchanger from the design modification study may confirm the inadequate cooling surface area, suggested by the results of the test run experiments. Noting that the original and the first modified designs had total surface areas of 0.844m^2 and 2.614m^2 respectively.

(v) The extension of the coiled tube heat exchanger to cover the conical section may ensure proper control of reaction temperature and may reduce cost of experimentation, because of reduction in terms of process constraints, as

experiment for small feeds (raw materials) can be carried out within the conical section, that had no cooling facility earlier than the modification.

(vi) Analysis and evaluation of results from this study permitted the development of the design modification which is consistent with experimental observation, and is being recommended for implementation with a view to control sulphation temperature and to reduce cost experimentation.

CHAPTER SEVEN

7.0 RECOMMENDATIONS

Based on the results of this investigations and design, the following recommendations can be made to improve the operation of the pilot plant and as well for future work on fatliquor (sulphated oil lubricant) production.

To rectify the underfunctioning of the pilot plant, the suggested remedies include:

1. **Increase cooling capacity**

The modified design should be constructed and the test running should be done to test the hypothesis that the modification will fix the reaction temperature at 25°C. The reconstruction of the coiled tube heat exchanger will provide additional cooling coil as established by the modified design.

The cooling water pump should be changed to one with a variable speed, so as to enable a selection of optimum cooling water flow rate for the cooling process. Provision of two ice making machines to make sufficient ice blocks to bring down the inlet temperature of the cooling water should be done. The cooling water should not be recycled to the coolant pot.

Alternatively, a refrigeration unit could be added to the cooling system for more efficient cooling.

2. **Improve the mixing efficiency**

This may help to increase the cooling efficiency as well as improve the product quality. This could be done by increasing the stirrer speed, installing baffles and changing stirrer impeller configuration to conform to standard configuration in figure 2.5. The mixing efficiency could also be improved to enhance the process by titrating the washing and Neutralization chemicals into the reactor rather than the usual addition of measured volumes into the reaction mixture

3. **Temperature measurement**

A good temperature measuring device not mercury in glass thermometer should be provided for the reactor, coolant inlet and outlet. This will enable keeping a valid record of temperature for proper analysis and monitoring of the plant operation.

4. **Utility**

Two shower points should be provided to treat acid spill cases.

Stand by generator fuel and operator should always be provided to take care of

power failure, as disruptions of the process affects product quality.

A lot of information on fatliquor mixture are not available. These includes the kinetics of the reaction process, the heat of combustion and formation and heat of reaction. Work on any of the above area is highly recommended as this will surely improve the quality of literature on fatliquor.

Also it has been suggested or documented that petroleum sulphonate addition improves the quality of sulphated oil and could substitute a large proportion of groundnut oil in fatliquor production especially now that groundnut oil is expensive, therefore, this is a good area for further work.

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APPENDICES

APPENDIX A:

Table 1.0: Physical Properties of Some Materials Used for the Design Modification

	Cold Water 18°C	Groundnut Oil 23°C	Concentrated Sulphuric acid H_2SO_4, 23°C
ρ(kg/m^3)	998	940	1.8364
μ(Ns/m^2)	1 x 10^{-3})	40 x 0.001ps	20 X 0.001PS
Cp(J/kg°C)	4.19 x 10^3)	$\frac{A}{\sqrt{d_4^{15}}}+B(T-15)$	1.424
K(W/m°C)	0.60	1.730735	0.21 X 10^{-2}

A = 0.450, B = 0.007, C_p = 1.962.

Source: Perry's Chemical Engineers Handbook, Sixth Edition

APPENDIX B: Design Modification Details

From the general design equation for Heat Exchanger Design,

$\varphi_H = UA\Delta T_{lm}$ ..... (1.1)

$$(1)\ \Delta T_{Lm} = \frac{\Delta T_2 - \Delta T_1}{Ln\left(\frac{\Delta T_2}{\Delta T_1}\right)} = \frac{5 - 9}{ln\left(\frac{5}{9}\right)} = 6.805$$

(2) To calculate U, we apply,

$$\frac{1}{U} = \frac{1}{\alpha_i} + \frac{1}{\alpha_o} + \text{Fouling Factor} \quad \text{..... (1.2)}$$

The average heat transfer coefficient to reactor vessel with coils was given by Beck and

Muttzal (1975) as,

$$\langle Nu \rangle = \frac{\langle \alpha \rangle d_c}{\lambda} = 0.17 Re^{0.67} P_r^{0.37} (d/D)^{0.1} (d_c/D)^{0.5} \quad \text{.....(2)}$$

For $0.25 < d/D < 0.58$; $0.03 < d_c/D < 0.15$ and $400 < R_e < 2 \times 10^5$

but for our vessel configuration, d/D = 64cm/94cm = 0.6 ≈ 0.58,

$d_c/D = 2.5/94 = 0.0266 \approx 0.03$

ρ_m = $0.3(1,836) + 0.7(940) = 1208.8$ Kg/m^3

μ_m = $0.3(0.002) + 0.7(0.004) = 0.0034$ Ns/m^2

R_e = $\frac{nd^2}{\nu}$ (for stirred vessel)

$$\nu = \frac{\rho_m}{\mu_m} = \frac{0.0034}{1208.8} = 2.85 \times 10^{-6}$$

$$R_e = \frac{4.9 \times (6.4)^2 \times 10^{-3}}{2.85 \times 10^{-6}} = 7.04 \times 10^4$$

Therefore, the flow is turbulent and Re. of 7.04×10^4 falls within the range,

$400 < Re < 2 \times 10^5$, therefore equation (2) is applicable.

$$P_r = \frac{C_{Pm}\, L_m}{\lambda_m}$$

C_{pm} = 0.7(1,962 j/kg°C + 0.3(1,424 j/kg°C) = 1,800.6 j/kg°C

λ_m =0.7(0.079 x 1.730735) + 0.3(0.21 x 1.73075) = 0.0957 + 0.1090
= 0.2047 w/m°C

$$P_r = \frac{(1,800.6)\,(0.0034)}{0.2047} = 29.907$$

$P_r^{0.37} = (29.907)^{0.37} = 3.516$

$P_r^{0.67} = (7.04 \times 10^4)^{0.67} = 1769.594$

$$\langle Nu \rangle = \frac{\langle \alpha \rangle d_c}{\lambda} = 0.17(1769.594)(3.516)(0.6)^{0.1}\,(0.0266)^{0.5}$$

$$= 163.918$$

Therefore, the heat transfer coefficient for the stirred reactor vessel with coiled tube,

$$\alpha_o = \frac{N_{um}\lambda_m}{D}$$

$\alpha_0 = \frac{163.918 \times 0.2047}{0.94} = 35.69\ w/m^2oC$

but $1/U = 1/\alpha_i + 1/\alpha_0 +$ fouling factor

Fouling factor for our system is given by Peters and Timmerhaus (1980) to be 1/519.2205

for α_i, we apply correlation for flow in spiral tube,

For the nature of flow in the tube (coiled),

$$R_{ew} = \frac{\rho_w V_d}{\eta_w}$$

$R_{ew} = \frac{1000 \times 0.344 \times 0.025}{1 \times 10^{-3}} = 8.6 \times 10^3 = 8{,}600$

indicating turbulent region.

$$P_{rw} = \frac{CP_w\eta_w}{\lambda_w} = \frac{4.19x10^3 x1x10^{-3}}{0.60} = 6.983$$

$P_{rw}^{1/3} = (6.983)^{1/3} = 1.910$

$P_{rw}^{0.75} = (8600)^{0.75} = 893.046$

$N_{uw} = 0.04\ R_{ew}^{0.75}\ P_{rw}^{1/3}$, for pipe with smooth walls.

$N_{uw} = 0.04\ (893.046)(1.910) = 68.229$

but if a circular pipe is wound to a spiral (diameter 2R), the heat transfer coefficient compared to that of a straight tube is increased by a factor of (1 + 3.5 D/2R) if $10 < R_e/2R/D)^{1/2} < 200$, due to the occurrence of systematic eddies.

Diameter of the tube, D = 0.025m and spiral diameter, 2R = 0.84m

$(2R/D)^{1/2} = (0.84/0.025)^{1/2} = 5.797$

So, $R_e/2R/D)^{1/2} = 8600/5.797 = 1483.641$, falls outside the range $10 < R_e/2R/D)^{1/2} < 200$, therefore the heat transfer coefficient is not increased by (1 + 3.5 D/2R) factor implying that there was no systematic eddies.

$$\alpha_i = \frac{N_{um}\lambda_m}{d} = \frac{68.229x0.60}{0.025} = 1637.496w / m^{2o}C$$

$1/U = 1/\alpha_i + 1/\alpha_0 + 1/519.2205$

$= 1/1637.496 + 1/35.69$

$1/U = 0.0006 + 0.0280 = 0.028619$

$U = 1/0.028619 = 34.942$

(3) ASSUMPTIONS;

(i) That the heat released by concentrated sulphuric acid during sulphation, Q is only the heat of reaction, ΔH_{rxn}.

(ii) Negligible heat losses to the environment, then taking a heat balance at maximum attainable temperature during sulphation, for the cooling

water and reaction mixture for the batch process, the heat generated by the reaction mixture during sulphation, ΔH_{rxn}=heat taken up by the reaction mixture and the cooling water. The reaction mixture include, sulphated oils, by products and unreacted materials for a batch.

therefore $Q = \Delta H_{rxn} = MmC_{pm} (\Delta Tm) + MwC_{pw} (\Delta Tw)$

but $\varphi_H = Q/t = M_m/t\ C_{pm} (\Delta T_m) + M_w/t\ C_{pw} (\Delta T_w)$

$\varphi_H = Q/t = \varphi_m\ C_{pm} (\Delta T_m) + M_w/t\ C_{pw} (\Delta T_w)$

$\varphi_H = \varphi_m\ C_{pm} (\Delta T_m) + M_w/t\ C_{pw} (\Delta T_w)$

$\varphi_H = (234)/10200\ (1800.6)(41\text{-}15) + 300/10200\ (4180)$

$(30\text{-}12)/34.942$

$\varphi_H = (1074.0 + 63.33)J/S = 1137.33J/S$

but $\varphi_H = UA_s\Delta T_{LM}$

$A_s = \varphi_H/U\Delta T_{LM} = 1137.33/34.942 \times 6.805 = 4.78m^2$

SCALE UP

180 Kg of oil requires $4.783m^2$ surface area coiled tube heat exchanger.

Therefore design for 216 kg oil capacity requires

$216/180 \times 4.783m^2/1 \approx 5.74m^2$

$As = 5.74m^2$

$\pi dLcn = A_s$

$n = 5.74/\pi(0.025)(2.56)$ = 28.548 coils

= ≈29 coils

Total length of coils required = 29 x 2.56m

= 74.24m

These coils are to cover the cylindrical and the conical sections of the reaction vessel. The cylindrical section should take 26 coils of length 2.56m at a clearance of 50m from the walls and the conical section to accommodate the remaining 7 coils at varying diameter.

Appendix C1:

Table 3.1: Temperature Profile of the Sulphation Reaction for the First Test Run in thePilot Plant.

Time`t' (Mins.)	Volume of acid used from aspirator at				
	0.251/min. Control	Actual Reading	T_1 (°C)	T_2 (°C)	*T_3 (°C)
0	0.00	0.00	10	15	15
6	1.500	1.44	13	15	17
15	3.75	3.60	17	18.5	17
30	7.50	7.20	17	18.5	21
45	11.25	10.80	18	20	27
60	15.00	14.40	23	26	34
75	18.75	18.00	27	30	38
90	22.50	21.60	27	30	41
105	26.25	25.20	23	25	32.5
120	30.00	28.80	27	29	33
135	33.75	32.40	29	31	37
150	37.50	36.00	24.5	26	35.5
165	41.25	39.60	28	30	37
180	45.00	43.20	24	26	38

T_1 = Inlet temperature of coolant
T_2 = Outlet temperature of coolant
*T_3 = Temperature of reaction mixture.
Room Temperature = 23°C.

Appendix C2:

Table 3.2: Temperature Profile of Neutralization Reaction at 10 Minutes Intervals.

Time (mins)	10	20	30	40	
T_1(°C)		16	22	23	24
T_2(°C)		16	22	23	24
*T_3(°C)	27	34	34	30	

Appendix C3:

Table 3.3: Temperature Profile of Sulphation for the Second Test-Run.

Time 't' (Mins.)	Volume of acid in aspirator at 0.25l./Min				*
	$Control_{(t)}$	$Reading_{(t)}$	T_1 (°C)	T_2 (°C)	T_3 (°C)
0	4.0	4.0	15	17	22
5	5.50	5.5	15	18	19
10	7.00	7.5	16	18	27
15	8.50	9.00	17	19	28
20	10.00	13.0	17	19	29
25	11.50	13.8	17	19	29
30	13.00	15.5	18	20	31
35	14.50	18.00	19	22	31
40	16.00	20.00	21	23.5	33
45	17.50	20.50	22	24	33
50	19.00	21.00	22	24.5	33
55	20.50	22.0	23	25	34
60	22.00	22.5	21^{+}	25	34
56	23.50	23.10	21	24	34
70	25.00	24.00	22	24	34
75	26.50	24.50	23	25	35
80	28.00	25.50	23	25.5	34
85	29.50	26.00	24	26.0	35
90	30.00	31.00^{-}	21^{+}	25.5	38
90	26.50	25.00	20^{+}	24	36
95	28.00	26.00	20	23	36
100	29.50	31.00	19	22	37
105	31.00	34.00	19^{+}	22	37
110			18	21	38
115			19	21	38
120			20	22	36

T_1 = Inlet temperature of coolant
T_2 = Outlet temperature of coolant
T_3 = Temperature of reaction mixture.
+ = Addition of ice block
- = Addition of acid.
Room Temperature = 27°C.

Appendix C4:

Table 3.4: Temperature Profile of Sulphation for the Third Test-Run.

Time 't' (Mins.)	Volume of acid used at 0.3l/min and 0.2l/min				
	Control(ℓ)	Actual(ℓ)	T_1 (°C)	T_2 (°C)	T_3 (°C)
0	1.0	1.0	17	18	21
5	2.5	2.0	17	18	23
10	4.0	3.0	16	18	24
15	5.5	3.5	15	18	25
20	7.0	4.5	16	20	24
25	8.5	6.0	18	20	25
30	10.0	9.10	19	20	27
35	11.5	10.5	20	21	29
40	13.0	11.8	18	21	30
45	14.5	12.8	17	20	31
50	16.0	12.5	18	20	31
55	17.5	14.6	17	21	35
-60	19.0	18.00	16	20	31
65	20.0	19.6	16	20	32
70	21.0	21.2	17	20	33
75	22.0	22.3	16	20	33
80	23.0	24.0	16	20	35
85	24.0	25.4	17	19	37
90	25.0	26.42	16	19	39
95	26.0	26.24	15	18	39
100	27.0	27.0	15	19	38
105	28.0	29.2	17	20	36
110	29.0	30.6	18	20	40
115	30.0	31	17	20	38
125	31.0		17	20	39
130	32.0		18	20	40

T_1 = Inlet temperature of coolant
T_2 = Outlet temperature of coolant
T_3 = Temperature of reaction mixture.