PRODUCTION OF GUN POWDER USING MADAR AS A SOURCE OF CHARCOAL

BY

NDAGANA LADAN (89/1143)

DEPARTMENT OF CHEMICAL ENGINEERING SCHOOL OF ENGINEERING AND ENGINEERING TECHNOLOGY MINNA.

A PROJECT SUBMITED TO THE DEPARTMENT OF CHEMICAL ENGINEERING SCHOOL OF ENGINEERING AND ENGINEERING TECHNOLOGY, FEDERAL UNIVERSITY OF TECHNOLOGY, MINNA, NIGERIA.

IN PARTIAL FULFILMENT OF REQUIREMENT FOR THE AWARD OF DEGREE IN CHEMICL ENGINEERING B.ENG. (Hons).

DECEMBER, 1998.

i.

CERTIFICATION.

This report has been read and approve as meeting the requirement of the lepartment of chemical engineering in the school of engineering and engineering Fechnology Federal University of technology Minna for the award of bachelons of engineering BENG (Hons) Degree in chemical engineering.

Mr. Akiwale Akinbode.

Dr. J.O Odigure Msc ph.D MNSE Head of Department

External Examiner

Date

Date

Dean School of engineering and engineering technology

8.8

DEDICATION.

I dedicate this work to my father late Mohammed Ladan and my mother Mrs. Fatima Ladan Shabakaji.

ACKNOWLEDGEMENT.

My sincere thanks goes to almight allah, who has made it posible for me to conclude this work. I wish to express my appreciation to my brothers Moha'd Gana and Moh'd F. Ladan whose financial and moral support has sustained me throughout my stay in the University. I wish to thank my Supervisor Mr. Akiwale Akinbode for his effort to the success of this project.

I appreciate my gratitude to my class mate Mallam Ismail Isah Rikoto whose endrmous contribution to the success of this work can not be over looked, and to my brother Ibrahim Hassan who has encourage me through out my course. Finally I am grateful to my class mates in chemical engineering department.

TABLE OF CONTENTS

	(i)
Little page	(ii)
Certification Dedication	(iv)
Abstract Table of content	PAGE
	••••
.200 The objection of study	•••••• -
.300 Method and scope .400 The relevance of study	
HAPTER TWO	4
	· · · · · · · · · · · · · · · · · · ·
.100 History of Gum powder	5-6
.200 Black powder and its raw material classification	6
.200 Black powder and its raw material classifier .300 The raw material	
.310 Potassim nitrate	9
.320 Sulphur	9-10
.330 Charcoal the criding on the c	composition of
.331 Effect of carbonization temperation and diffamily	
charcoal hlack powder	12-16
.400 The buring properties of the black powder	17
.500 The milling of the ingredients	17
1 1 1 not nowder	18
	DACE
CHAPTER THREE	119
	···· +
1.000 The experimetal	••••••±۶ 10
3.100 The equipment for the experiment 3.200 The raw materials	····· 19
8.200 The raw materials 8.210 Carbonization of the wood	
8.300 Carbonization of the wood 8.300 The experimetal procedual	
too Tooting	· · · · · · · · · · · · · · · · · · ·
3.400 Testing	PAGE
CHAPTER FOUR. 4.000 The Results	
4.000The Results	
4.100 Calculations	
4.200Tables of results	23
4.300 Graph of rate against particle size	ter and the second s Second second

CHAPTER FIVE

CHAPI	DR TIVL	
5.000	Design project	
5.100	Introduction	••••• 24
5.200	Flow sheet of the production process	••••• 25
5.300	Materrial balance	••••• 26-27
5.400	Power requirement by Crushes & ball mill	••••••28
5.500	Detail design of Ball mill	•••••29
5.510	Determination of material requirement	••••• 29
5.520	Choice of Grinding mill	29-33
5.530	Consisty and power requirement	••••• 33
5.600	Material for construction	
5.700	Conting of aquinment	
5.800	Safety consideration	
5.000		
CHAP'	FER SIX.	PAGE
6.000	Discusion of Results	36-37
		PAGE
CHAP	TER SEVEN	17102
7.000	CONCLUSION, RECOMMENDATION, NUMENCLATURE	
7.100	densition and recommedation	······ 38 39
7.200	Numeclature	••••••
7.300		40
1.000	Appendix	• • • • • • • • • • •

ABSTRACT.

This project focused on the production of gun powder using Madar tree as a local source of raw material, other material used in the mixture are pottasium nitrate and sulphur.

The wood from madar tree was carbonised using muffle furnance at a temperature of 500°C. The carbonized charcoal and sulphur were mill separetly. The three component were then mixed together at a proportion of 67.2%, 21.3% and 11.5% for pottasium nitrate sulphur and charcoal respectively, water was added to maintain 1-1.5% moisture content, depending on the quantity to be produce. This helps to in coporate pottasium nitrate in to the mixture, the mixture was mill with mortar and pestle. During of mixture was done on sunlight or natural sun, which finally mill to obtained require fines.

Diameter of particle size ranging 25, 20, 15, 10 and 5mm were Tested for the rate of explosion. After testing it was dicovered that 5mm diameter of particle size gives high rate of explosion, which indicate that fine powder increase the reactivity of solid and best for gun powder.

Vill

CHAPTER ONE

1.0.0 INTRODUCTION

1.1.0 BACK GROUND OF STUDY

Gun powder or black powder as the name implies is black in colour with irritating smell. It was known centuries B.C by replacing oxygen with solid to obtain a better power ratio after notice that, there are small amount of power available for esplosive involves gaseous at any given volume.

Gun powder was originated in china by a franciscan order ROGER BACCON in 13 century. The manufacture of Gun powder stated in 14th century in Britain and by 18th century was introduce to Nigeria. This powder is a mixtures of combustible materials, There are, of three types viz. pottassuim nitrate, Sulphur and charcoal. Hence, it is define as amixture of combustible material readily ingnited even in complete absence of air and burn vary rapidly therefore could simply mean a substance which in its self capable of producing a quantity of gase under high pressure and being able to produce this gases so rapidly under centain condition that the surround are subjected to a strongh dynamic.

The commoly used Gun powder has the composition of 75% by potassium nitrate, 10% by sulphur and 15% by charcoal. The potassimm nitrate and sulphur are local available in the market in their pure state or could be prepare. While charcoal are obtain from woods such as willow, hamp, elders, popular or harzel usually found in the forest or could be alternatively obtain from cassava stem, calabash, and maize Cab (commoly use by local hunter).

The origin of Gun powder has a number of advantages to the developed and developing countries. Amoung its advantages include the use of Gun powder in small proportion for the production of cockers, in military fields in mining centers and by the local nunters. The readily availability of its raw materials make its conomical valuable for used.

The method is limited on size distribution of funishing grain. Further effort may be made to incoporate powder with water proof to avoid shutt failure at low tempreture

1.40

THE RELEVANCE OF STUDY

This project is prepare to produce a Gun powder at cheaper rate to meet the high demand by the local hunter as a result of the incressing in population and they are not get asses to the modern wipound for fighting against wild animals. arms. Table 2.2 show an example of such powder. While for type 2. It is must burn vigorously so as to give an effect as close as posible to detonation. It consist either of gains with a density of 1.8 or compresses cylindical pellets with a density of 1.35-150 and a central peforation [6]. According to Andreyew [5] black powder may buring at a pressure low than atmospheric and dependence of the rate of buring on the pressure may be expressed by the equation U=72p0.24(2(1)

where U is a linear rate of buring in con/min and P the pressure in atmosphere.

The compounent of black powder are clasify in to phosophore and anxoplosives atcording to Whrop and Handrick [(5) The primary source of black powder KNo3 is a phosophores and other additives as auxoplosive. According to these Authours. A phosphores has been define as a group of atom which is capable of forming explosives compound on introduce into hydrocabon. While group which do not then selve produce properties but may influrence others in the same way as that of auxochromic group vary the colour intensity and shade of the dye are called anxoplotives.

2.30

THE RAW MATERIAL.

2.31 POTASSIUM NITRATE.

The primary source of Gun powder is potassium the xonitrate. It is obtaiable from nature deposits in hot counties for example in ceylon, egypt, mexico, India, Iran and some areas of the USSR. It occur there as the result of the microbiological oxidation of organic nitro compounds and of the reaction of the product with the alkaline component of the soil. On being referred by cystalization such saltpetre was used for the manufacture of black powder

Still another method for the preparation of KNo₃ directly from Kcl and nitric acid was used recently. Reaction process at temperature between 55-85^{oc} according to the approximate equation.

 $4Kc1 + 5 HNo_3 - Nad + c1 + H_2o + 4KNo_3 + Hc1$

-(2.6)

Potassium salt petre prepare by any of these method is refimed i.e it is recystallized from water by a method chosen in accordance with the impurities known to exist in the product. Cystalization is facilitated by great diffence in solubility of potassium nitrate at high and low temperature. It is ten times more soluble at 100°C then at 10°C.

Potassium nitrate salpetre for the manufature of black powder must be of high purity. The technical specification for the synthematic product according to the U.S.R.S standard (Cost 1939 to 43) is sumarised in table 2.3 below.

		and the second
Requirement	CLASS	andar Artista Artista
	1	2
Appearance	White cystals	White cystals
Minimum content of KNo3	99.80	99.00
Max content of moisture %	0.10	0.20
Max content carbon as K_2Co_3	0.03	0.10
Max content of H2O insoluble substance %	0.03	0.05
Max content of substance insoluble in Hcl acide %	0.04	0.04
Max content of chloride calculated as Ncl %	0.005	0.02

Table 2

The technical specification for synthentic KNo₃ used in the manufacture of black powder (Gost 1939-43).

Potassium saltpetre manufatured from chillian saltpetre may also contain potasium perchlorate and potasium iodate which are ver determental since they increase the sensitiveness of black powder to friction and impact. A great many explosions which have occured during the manufacture of black powder have been ascribed to the

black powder. It must be soft, but not resinous and should be prepare from white light and porous wood [15]

For the pupose of this project the source of charcoal is limited to madar plant <u>calotropic proceral</u>. The use of this plant was not only base on the characristic mention above but also in addition, its contant a waste product and oftern irritating and poisnous substance so that it causes inflamination ([57)]

CARBONIZATION:- Before carbonization, the wood must be debacked and into pieces that could be feed into a sheet of iron reforts approximately 1m diameterand 1.5 by 3m long. One end of which is close with a air tight 1id and the other fitted with an off take for the gaseos products of distilation. These product are usually bumrr since it is not worth while recovering then. The combusition of Co is particularly important, other wise it may poison the atmospher. The retort are heated by passage of electric current.

The carbonization last for 3-8 hours depending on the construction of the furnaces and retort. The temprature and the types of material to be carbonized. After carbonization the retorts are taken out of the furnace and allowed to cold down (with the off take closed) the charcoal is removed from the rétort when it is cold. Hot charcoal easily lignite. The charcoal should not be milled untill after at least 24 hours from the removal itime since charcoal which is two fresh may catch fine during milling. The effect of temperature of carbonization on charcoal are shown in table 2.3 below.

Types of Charcoal	Temperature of (^O c)	carbonization	content of (c)%	carbon
Cocoa (red)	140-175		52-54	
" Brown	380-350		70-75	
" Black	350-450		80-85	

hree types officharcoal are obtain depending on the temperature of arbonization.

2.341 THE EFFECT OF CARBONIZATION TEMPERATURE AND GRINDING ON CHARCOAL.

The best structure of carbon required for the manufacture of black powder is aromatic structure and this is achieved during heating to a temperature above 300°C. The aliphate structure are breaks down in to aromatic ring through weaker and disappearance of the bound by hydrogen 33000cm from the water present 16140cm and bound between carbon and hydroxyl group.

The determination of aromatic ring in charcoal are entensively work on by different people such as Juctiner whom oxidized various types of coal including charcoal using various oxidizing agent such as potassim per maganate in an alkaline medium and nitric acid. On such oxidatism of charcoal obtained by carbonzation of wood at 1000 mellitic acid in a yield of 25% by weight results under similar condition flurene give mellitic acid in yield of about 45% while hexactly, benzene yield almost exclusively carbondioxide with out mellitic acide. Also this structure of carbondioxide with has definetly provide by infra-red spectroscopic analysis carried out by T.urbanski, Ostrowski and vitanowski,

The effect of grounding on charcoal provide the break of the bounds of unpaire electros which are polymers of convalent bound to a free radicals. These radicals considerable influnce the properties of black powder. The concentration of these free radicals considerably depend on temperature of carbonization.

-C-C- grinding -C- + -C. - (-2.7)

Charcoal here implies a component with variable properties, not a specific chemical. Hence, depending on the method by which the charcoal is prepated powder with various properties could be obtained. As early as 1848 this attracted the attention of violette who prepared different types of charcoal in a retort by employing different temperature of carbonization as shown below.

Table 2.4

	•				
Temperature of carbonization	Colour of charcoal	Yield	compos charco	sition Dal.	of
(oC)		¥	Nto	% C	H
280-300	Brown	'34.00	21.90	73.20	4.3
250-400	Black	28.31	-	77.81	
1000	Black	18.00	14.10	82.00	2.3
1250	Black	18.00	9.30	88.1	1.4
		1			

Effect of temperature of carbonization on the charcoal composition.

The relation between the ignition temperature of charcoal and the carbonization temperature of wood are show below.

Table 2.5

Temperature of carbonization (oC)	Ignition temperature of charcoal (oC)	
260-280	340-36	
270-350	36-370	
432	Ca-400	
1000-1500	600-800	

This shown that the power of powder containg black charcoal is greater because is less hygroscopic. Hence the powder from it is less hygroscopic ([6]]

2.40 THE BURNING PROPERTIES OF THE BLACK POWDER.

For a long time attempt have been made to explain why three non explosive substance viz potassiumnitrate, sulphur and charcoal, when combined together should form an explosive mixture. It was particularly incomprehensive that binary mixture of potassim nitrate and charcoal or with sulphur should be non explosive or only poorly if so due to the difficults in elusidate since reaction involved the solid pharse.

So that $1/3, \frac{1}{2}, \frac{1}{2}$ and the rest 1/24 of the powder decompose according to the equation respectively.

Much late K.A itHoftman resummed work on the mechanism of explosion of black powder. On examine: the mixture, he found that the reaction in black powder starts above the melting point of sulphur at approximately $150^{\circ}C$ with a reaction between hydrogen present in charcoal and sulphur. Hydrogen sulphur thus formed react at temperature between $285^{\circ}C-290^{\circ}C$ with potassim nitrate to §1eld K₂So4. Heat is then emitted and KNo₃ were course to melt (6)

The increment of suphur for the purpose of this project is to produce a powder with low initial decomposition and explosive temperature on the bases that;

i. Sulphur intensities the sensitiveness of mixture to inpact. Amixture of KNo₃ and charcoal does not explode while amixture of KNo₃ and sulphur decompose when struck by a 21g weight falling from 45-50cm on the other hand a mixture of KNo₃, charcoal and sulphur is explode by a 2kg weight from a drop of 70-80cm.

ii. It conteract the formation of carbon monoxide in the product of explosion Co and KCN occurs in the product of decomposition of a mixture of KNo_3 with charcoal, due to the reaction of charcoal with K_2Co_3 .

 $K_2Co_3 - 2K + {}^{1}3Co - 14$ 2K + 2C + N₂ - 2KCN-15

On the other hand, in the presence of sulphur a reaction with K2504 takes places. $K_2So_4 + 2C - K_2S + 2Co_2 - 16$

iii. Sulphur reduce the inital decomposition temperature for instance a mixture of 2male of KNo₃ and 3g atoms of cabon (in the form of 71% charcoal) decompose at 320° and explode at 357° c while the same mixture but ligrance of sulphur decompose at 310° C and explode at 450° C. The latest studies of H. Thomos quote the following figures for normal "Gun powder G 12" [5]

Table 2.7

Potassim nitrate	75.3%
Sulphur	10.3%
Charcoal	14.4%
and the second	

While for this project the composition of the Black powder are

as shown below.

Table 2.8

Potassim Nitrate	67.20%
Chārcoal	11.50%
Sulphur	21.30%

This powder were proportionaly as above base on the masses of the component that gives the completely buring of the powder. To avoid unreacted deposition that could be found on the side of the gun bore barel which may block the hole to the ignition cap and result in shutt failure if not regularly washed.

The normal Gun powder has the following charateristic heat of explosition is 735 ± 15 Kcal/kg at a moisture content of 0.85% and 740± 15 Kcal/kg at dry place according to Kast, the volume Vo is 280 L/kg; the specific pressure (f) is 2800m and the temperature of explosion (t) is 2380°C while the specific gravity of Gun powder may vary within the limit of 1.50-180 depending on it intended use. Its appearent density is 0.900-0.980.

The reaction between the time of buring of black powder and density for grain 3.5-10.5mm dia are shown below.

time o (mil se	
120 80 40	→ 10.5mm dia → 3.5mm dia
(0,1.	6) 1.7 1.8 1.9 2.0

Finaly, the mixture of the 3 component at the proportion above decompose at 290°_{C} and explodes at 311°_{c} . The black wood and Bowden formulate the following mechanic for the ignition and buring reaction of black powder. First sulphur react with organic substance present in charcoal.

S4 organic compound - H2S- 17

Almost simultanously, salt petre reacts with these compound

KNo3 + organic compound -No2 - 18

The follow reaction may also occur.

 $2KNo_3 + S - K2504 + 2No-19$ $KNo_3 + 2Ncl - KNo_2 + No + No_2 - 20$ $H_2S + No_2 - H_{20} + S + No - 21$

Reaction (last) proceeds until all the hydrogen sulphide is used up when No3 appears and begin to reactwith the still unconsume sulphur according to equation below.

 $2No_2 + 2S - 2So_2 + N_2 - 22$

The sulphue dioxides so formed may immdiately reactpwithspotassiume nitrate. 2KNo₃ + So₂ - K₂So₄ + 2No₂ - 23

According to Black wood and Bowden, the chief reaction when powder begin to burn is the oxidation of charcoal by potassin nitrate from a comprhensive analysis of the product of explosive of black powder Kast drived the following equations.

74 KNO_3 + 30S + 16C6H₂O (charcoal)

 $56Co_2 + 14Co + 3EH_4 + 2H_2S + 4H_2 + 35N_2 + 19 K2Co_3 + 2K_2S + 8K_2S_2O_3 + 2KCN_3 + (NH_4)2Co_3 + C + S + 665Kca1/kg - 24$

The heat of explosion and gas volume naturally depend on the decomposition of the powder. Noble and Abel in the work quoted above gives the following figures.

Table	2.	6
-------	----	---

Black powder	C	omposition		Heat of explosion Kcal/kg	Gas volume vocm3/g
	KN03	Sulphur	charcoa	1	
course grains ad	75.00	10.00	15.00	726.00	274.00
Fined graine sport- ting.	75.00	10.00	15.00	764.00	241.00
Blasting	62.00	16.00	22.00	516.00	360.00
Cocoa powder	80.00	2.00	18.00	837.00	198.00

THE MILLING OF THE INGREDENTS.

The manufature of black powder consist thoroughly mixing and well milled of ingredient to obtain the required particular size and density (fast buring black powder is fine grained while the slower buring types has larger grainss of higher density the manufacture consit of the following operation.

Milling of the ingredient i.

ii. Mixing of the ingredient

iv. Corning and finishing (drying, polishing and screening). iii. Pressing The method of manufacture depend upon the trend of development tradition in a give factory oir country and for the purpose of this project pressing is done by the stone.

2.600

FINISHING

The powder from the milling is then finished, this consist mainly of polishing, drying and grading the grains depend on the required structure.

POLISHING: - The grains have an irrigular and rough surface which prevent them from flowing strecly and from filling aspace with out voids, so that their density is rather low. To over come this the proces of polishing were employed and it takes places as follows. At first, the work is carried out with the side door closed so that the powder retains enough moisture to facilitate the process. After a few hours 0.1 to 0.5% of graplite is added to the charge and polished for several more hours. The graphite fills the pores in the surface of the grains and coat with a thin large giving then characteristic of brightness.

GRADING:- Finally the polishing dried powder is finally graded by sieving out the dust through revolving or the types of vibrating sieves often, hand sieving and commonly used for this purpose. The sieve frames are made of woodd to avoid the danger of friction and impact.

2.500

SAFETY IN BLACK POWDER

2.700

The manufacture of black powder is one of the most dangerous in the production of explosive, shelter for the work should be set up in the vicinity of each building. There are huts covered with earth at the side of the operating building. If the building have three stout walls, the shelter may be located behind one of them. Doors and windows should be large to gives planty of light.

Electric cables should be carried in steels condunit on the extension of the walls. In candescent lamps inside the building should be recessed in to the wall and protected by safety glass(7)

The three component was then mill together in a mortar and water was added to maintain a moisture content of 1 to 1.5% The mill of the three component was carried out for 1½ hour to insure proper incoporation of potassiumnitrate in to charcoal. The mill cake after 90 minute was removed and dried useindistunlight

The dried cake was broken by pressing used stone in to finished grain and the gains was sieve in to 25,20, 15, 10 and 5mm diameter particles size with the respectively sieving diameter.

3.40 · TESTING

The testing of the powder was carried out in two stages in the first stage, 0.5% of the ungraded powder produced was measure and test for the heat of explosion used a calori meter of bomb heat capacity 120.86 (KJ), surrounded by water of mass 1.2kg and heat capacity 4.184 kg/g.k and a change of 23.9°C was observe when it burns.

While, in the second stage 0.04 of each from the respectively particle size diameter was measure and spread over 1.50 centimeter long of iron sheet and was spark, the time taken for each particle to explode was measure and was used to calculate the rate of explosion at each particle diameter. The result of the testing were show oh chapter four.

The identification of these raw material could be through their physical propeties such as colour, structure and observation of certain charteristics. The potassim nitrate and sulphur could be indentify through their colour and structure, sulphur asuadyellow cylindmical fod and KNo3 in a fine ground silverlish colour. While charcoal as a black stuff material from show buring of wood usually from a white light and porbus wood.

The use of pottasimmnitrate saves as a primary source that gives the powder an explosive charateristics. Other additives such as sulphur as a catalyst and charcoal as a base compound as catalyst and are of significate important. Although, the source of charcoal has notlämit to those mention above but may also be obtain from any other plant base on the same characteristic and this formm the base of this project.

1.20

THE OBJECTIVES OF STUDY

The objectives of this project could be sumarized as follows. Despite that this powder utilize sulphur & KNO3as its base, the aim is

- i. To produce the same type of Gun powder but from a difference source of charcoal. And with difference proportionlity of ingredient.
- ii. To determine the particle size that will gives high rate of explosion.

1.30

METHOD AND SCOPE.

The method and scape of this project involves milling of the 3 compound together in mortar using pestle. Water is added through out the process to maintain moisture content of 1 to 1.5% to reduce ingnities and incoporation of KNm in to charcoal. The result cake is broken by pressing using stone to increase initimaty contact of the ingredients and improve strength and buring properties of the powder.

41 part	Salt petre
29.5 part	charcoal
29.5 part	sulphur.

Table 2.1

It was not untill the invention of the fire aims that the manufacture and use of black powder ready began to developed up to the present day.

The first published reference to use of black powder in mining was the paper read to the royal society in london by Sir R. Moray in 1665. He refered to "away to break easily and spreedily the hardest rock" according to the same paper. The method was invented by duson in france from that time the use of explosive for various engineering operations such as mining, road building, dam building, land improvement etc beging. Becames general, black powder are employed in the manufacture of safety fuse in which the cores consist of black powder and the use of gun powder for rain making by D.A Dauris. The change consiting of 15g of black powder plus 1.5% of silva lodide and send by balloon in to a clound where they are exploded by a time fuse the particles of silva lodide thus released act as nuclei on which the water vapour is in a rain clound coagulate to fall as rain drops. In great Britain, the manufacture start in 14 century and by the 10 century was introduces in to Nigeria and late became over Shadow by high explosives in 1833 to 1846.

2.20 THE BLACK POWDER AND IT RAW MATERIAL (CLASSIFICATION)

There are infact, two types of black powder clasify according to their intended uses.

Typer 1 for filling fuse.

Typer 2 for blasting charger.

The diffence between the two type lies mainly in their different rate of burning. For type 1, the rate of buring should be as high as posible and is achieved by making the grains of powder of the density as low as posible. Example of such are powder used in small In great Britain for instance until relatively recenly, the only source of potassium nitrate was salt petre from India.

At one time potassium saltpetre was also obtained from manure wood ash. Here potassium nitrate forms as a result of microbiblegical processes follow by reaction with potassium carbonate. Description from various period of history may also be found that refer to the collection of salt petre from the wall of cellars situated in the vicinity of serves. In this way for instance, saltpetre was acqinied in poland for the manufacture of black powder during the insurrections of 1830 and 1863 [7].

From the middle of the ninetees century potassium nitrate began to be manufactured from chillian salt petre containing 20-35% NaNo3 10 90% and after wards subjected to be exchange reaction.

NA NO3 + Kd1 KNO3 + Nad1 - (2)2)

Reaction 2 is conducted in an agnous solution at a temperature of 100°C. It takes the desired course due to the poor solubility of Naclat this comparation. The soddium chloridedes is then prcipitated and the reaction there fore shift to the right The potassim nitrate so obtained was termedid converted saltpetre. And ther method of manufacture of kNo₃ was base on double decompolition with potassim cambonate [8]

At present, reaction 2 is most frequently used to obtain potassim nitrate although the sodium nitrate used tisis now of synthertic origin. Another method now exployed consist in the treatment of potapsiumimcarbonate or canstic potash with nitrogen dioxide. The courses of the reaction is:

 $N_204 + H_20 + No_3 - (\rho_3)$

 HNO_2 + HNO_3 + 2KOH $-KNO_2$ + H_2O + KNO_3 + H_2O - ((2).4) Amixture of potassim nitrate and potassim nitrate is heated with nitric acid to oxidize nitrite to nitrate.

 $2KNO_2 + 2HNO_3 - 3KnO_3 + 2NO + H_2O - (2(.5))$

pressure of these salts in salts petralt petre, although, it has been ascentained experimentaly that small admixtures of K^{cbQ}_{4} increase the sensitives of black powder to impact and friction. Never the less, for the sake of safety potassim nitrate manufacture from chillias saltpetre must not contain perchlorates and iodates in analytically detectable quantities other wise should be remove through method of purification before used [5].

However, for fasts and easily production of black powderepotapossium nitrate could be readily purchase from the market in its purest town.

2.32

SULPHUR

For the manufacture of black powder the suphur used should be of higher putity, refined by distillation crude sulphur (which usually contain 2.5% of inputitive) is distilled from retorts heated to a temperature of $400^{\circ \text{C}}$. The recieves should be maintained at a temperature above $115^{\circ \circ}$ (120-130°^c) i.e above the melting point of sulphur (114-115°^c). Under these condition, the distillate condenses to liquid which is then cast in to stickes or blocks. This is the only form of sulphur suitable for the manufacture of black powder. If the reactive temperature is lower, the sulphur distilled from the refort condences as flowers of sulphur which always contains a little S°2, and even traces of H₂ S°4. (the substance is easily oxidized to its large surface area). Sulphur is this form is therefore slightly hygroscopic and acidic and is unsuitable for the manufacture of black powder.

Unlike potassim nitrate, sulphur could also be found readily available in the market and this is the only easist way of obtaining sulphur for fasts manufacture of black powder (3)

2.33 <u>CHARCOAL</u>

The Agnition temperature of charcoal highly after the Agnition temperature of the black powder and so also the typey of the wood used as a source of charcoal. It is very important to select a suitable types of wood for the manufacture of the charcoal used in

CHAPTER THREE

3.00 <u>THE EXPERIMENTAL</u> 3.10 <u>THE EQUIPMENT FOR THE EXPERIMENT</u> i. The Electric furnaces ii. Mortar and pestle iii. Electronic weighting balance

- iv. Sieving of 25mm dia, 20, 15, 10 and 5 diametre.
 - v. Stop watch.
- vi. Stone.

3.20

RAW MATERIALS

i. Wood (From madar colotopic proceral)

ii. Sulphur

iii. Potassim nitrate.

3.21 CARBONIZATION OF THE WOOD.

The wood from madar <u>calotopic proceral</u> was deback and cut into 0.8-25m long. The cut pieces was feed to the sheet of iron retorts of 1m diameter and 1.5-3metres long of a furnaces with one end close and other fitted with an off taken for the gascous products of distillation. The furnace was then switch on and the retort heated by the electric current to a temperature of $450^{\circ}c$ to $1000^{\circ}c$ and was maintain at this temperature for a period of 3 hours the retort was taken out and allowed to cool for 24 hours.

3.30

THE EXPERIMENTAL PROCEDURE

The cystals of sulphur and the charcoal was first ground in to powder seprately used mortar and pestle. 32 grains of sulphur and 17.4 grames of charcoal was measure from the samples of each respectively used electric weighing balance. The two sample were mill together in mortar used pestle for 30 minute 101gram of KNo_3 was measure and added to the mixture and mix throughly with rod like wood.

CHAPTER FOUR

4.00

THE RESULT.

The percentage of sulphur = <u>Amount of sulphur</u> x100% total amount of mixture.

$$= 32 \times 100 = 21.3\%$$

$$\overline{150.40}$$

Percentage of charcoal = $\frac{17.4 \times 100}{150.4}$ = $\frac{17.5 \times 100}{150.4}$

Percentage of KNo₃ = $\frac{101 \times 100}{150.4}$ = 67.2%

The heat od explosion is give by $Hu = H_b D_T + h_w M_DT$ where $h_b =$ heat capacity of the steel bomb in calorimetre = 120.86x10JK

 $h_{\tilde{W}}$ = heat capacity of water = 4.184x10J³/gk

 $m_{\tilde{tw}} = mas of water = 1.2kg$

 D_{T} = change bf temperature = 48.9-25023.9° c

 $Hu = 4.184 \times 10^{3} K/g.k \times 1.2 Kh \times 23.9^{\circ}c + 120.86 \times 10 J/K \times 23.9^{\circ}c$

$= 3008.6 \times 10^{3} \text{kJ}$ = -719.06 Kcal/kg

The rate of explosion U =<u>distance covered</u> time taken

for 25mm dia = 1.5/0.038mm =39.47cm/min

" $20mm = \frac{1.5cm}{0.023min} = 65.20cm/mm$

for $15mm = \frac{1.5cm}{0.013min} = 115.38cm/mm$

for $10mm = \frac{1.5cm}{0.005min} = 300.00cm/mm$

for 5mm = 1.5cm = 312.500cm/mm0.004min

The rate of explosion with particles sixes are.

	+ · · · · ·			
Particle size (mm dia)	Mass (g)	distance covered (cm)	Time taken (min)	rate of explo sion (cm/mm).
25.00	0.04	1.50	0.0380	39.470
20.00	n	n de la construcción de la constru La construcción de la construcción d	0.0230	65.200
15.00	11	II.	0.0130	115.380
10.00	11	T	0.0050	300.000
5.00	17		0.0048	312.500

Table 5.1

The composition of the powder produces.

Table 5.2

4

Component	Composition by mass (g)	Composition by percertage (%)
Potassium nitrate	101.00	67.20
Sulphur	3.00	21.30
Charcoal	17.40	11.50

The heat of explosion of the powder was found to 719Kcal/kg exodemic because heat is release to the surrounding.

CHAPTER FIVE

DESIGNUPROJECT

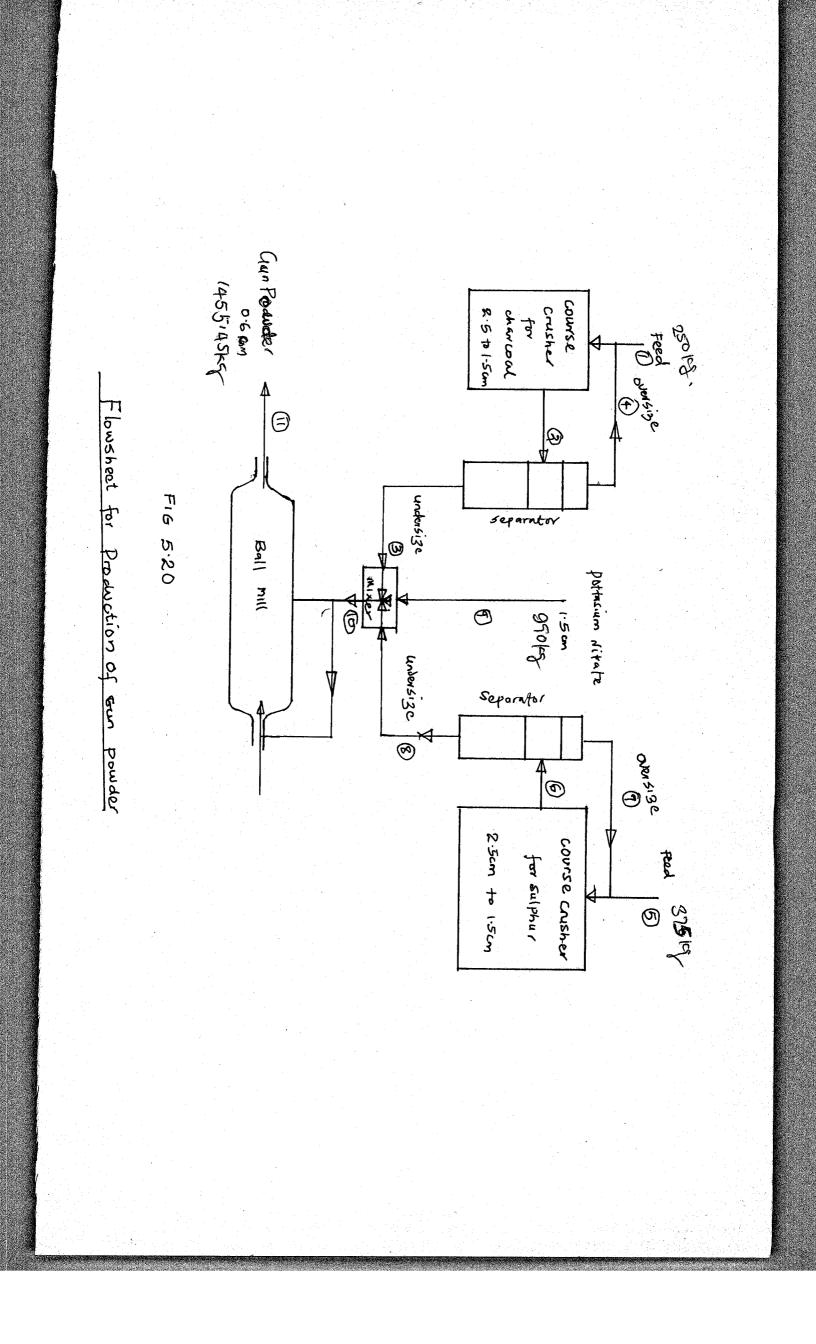
5.100

5.100

INTRODUCTION

The production of Gun powder has a number of process of which different machine are employed. The charcoal and sulphur are usually found with large particle size and for intermitely contanct of the ingredient these are reduce to the smallest possible size each to provide effective chamical reaction.

The two component are crushed or grinding seperatly using any size reduction machine such as crushers. The crushed sulphur and charcoal are then mixed with KNo₃ which ais already, in its fine particle size in a mixer thoroughly. The mixture of the 3 component are then send to the ball mill. There the component come together and the chmical reaction takes place between the component such as iron exchange reaction.



MATERIAL BALANCE OVER THE PROCESS

Material in = material out+loss Basis 250kg/hr of charcoal Balance over charcoal crusher. Stream (1) = 250kg of charcoal. Stream (1) = stream (2) + lossloss = 1% of stream (1) stream (2) = $250 - 1 \times 250$ 100 = 247.5 kgInitial particle size = 2.5cm final particles size = 1.5cm If 80% of the crushed charcoal is the under size = 1.5cm 20% of the crushed charcoal is the over size = 1.5 cm Undersize stream (3) $= 80 \times 247.5$ kg = 198kg 100Over size stream (4) = $\frac{20}{100}$ x 247.5kg = 49.5kg Balance over surphur crusher (The ratio of the three component are 10:3:2 potassim sulphur and charcoal respectively.) Since stream (3) = 198kg stream $8 = 3/2 \times 198$ kg = 297kg If stream (8) = 80% of stream 6 then stream (6) $=1/0.8 \times 297 = 371.25$ kg stream (6) = stream 8 + stream (7) stream (7) = stream (6) - stream (8)=371.25 0 297 = 74.25kg. Stream (5) = stream (6) + lossloss = 1% of stream (5) stream (5) = 371.25 + 1/100 stream (5) stream (5) x 99/100 = 371.25stream (5) = $100 \times 371.25 = 375$ kg

5.30

Particle size = 2.5cm 601.5cm Amount of stream (9) = $10 \times$ stream (3) = $5 \times$ stream (3)

= 990 kg.

Balance over mixer

stream (10) = stream (9) - loss. loss = 1% of stream (9) = stream (8) - loss. loss = 1% of stream (8) = stream (3) - loss. loss = 1% of stream (3) = 990kg - 1 x 990 = 980.1kg of KNo₃ + 297 - 1 x 297 = 294.03kg of sulphur + 198 - 1 x 198 = 196.02kg of charcoal with particlessize = 1.5cm Balance over Ball mill Stream (11) = stream (10) -loss. loss = 10% of each stream = 980.1 - 1 x 980.1 = 970.3kg of KNo₃ + 294.03 - 1 x 294.03 = 291.09kg of sulphur

+ 196.02 - $\frac{1}{100}$ x 196.0.2 = 194.06kg of charcoal

with particles size = 0.6cm

Then the amount of gun powder produces is equal to

970.30 291.09 194.06 1455.45kg.

POWER REQUIREMENT

The power requirement by charcoal crusher is given by

$$P = 0.3162 \quad \text{Wi}_{c} \sqrt{\frac{1}{D_{p_{b}}}} \sqrt{\frac{1}{D_{p_{a}}}}$$

where P = Power requirement.

 Wi_c = working index of charcoal = 11.37kw/ton D_{Pb} = paticles size of the product (mm)=15 D_{Pb} = particle size of the feed (mm)=25

P = 0.209 Kw.

The power requirement by the sulphur crusher is given by

$$P = 0.3162 \text{ Wis} \int_{D_{p_b}}^{1} \int_{D_{p_b}}^{1} \int_{D_{p_b}}^{1}$$

where Wi_s = working index of sulphur = 11.5kw/ton Dp_b & Dp_a are the same as for charcoal.

$$P = 0.211 \text{ KW}$$

Power require by the ball mill is given by

$$E = [(1.64L -1) k + 1][(1.64D)^{25}E_{2}]$$
where L = inside length of the mill (m) = 1.8m
$$D = \text{inside Diameter of the mill (m) } 0.9m$$

$$E_{2} = \text{power require by } 0.6 \text{ by } 0.6m \text{ Laboratory}$$
mill over 1.5m long.

 $E = 52336.02E_2$ Kw

5.40

BALL MILL DESIGN (DETAIL DESIGN)

This ball mill is to be design to mill 250 kghr of charcoal with 8 working hour planned per day.

5.21 DETERMINATION OF MATERIAL REQUIREMENT

The production of ciGunscal powder is a continous process. The anual productivity of the technological line is calculated as;

$$G = Q - tq - 5.1$$

Where q = productivity of reactor mill kg/hr = 250kg/hr t = planned working hour per year = 8x365hr Q = Annual productivity = 8x365x250 = G = 8760x250kg/hrx2920kg = 2190000kg-730000kg = 146 0000kg.

5.2.2

CHOICE OF GRINDING MILL

The factor determing the size of the grinding ball are finess of material being ground and maintance cost for the ball charge. Acoarse feed requires a larger ball than fine feed, a relation has been proposed.

 D_b^2 = Kxp-5.3 where D_b = Diameter of the ball cm Xp = size of coarse feed particle cm =2.5cm K = guidability costant = 90

$$= \sqrt{90 \times 2.5}$$
$$= 15 \text{ cm}$$

 D_{b}

The criteries by which the ball action in mills of various size may be compared in the concept of critical speed at which the centrifugal force on the ball in contact with mill sheet and the height of its path equals the forces on it due to the gravity.

For ball mill, the critical (Nc) and working number of revolution are determine as follows.

5.20

Nc =
$$\frac{42.3}{\sqrt{D}}$$
 - 5.4
L = 2D - 5.5
where D = Diameter of the mill (m)
L = lenght of the mill = 1.8m
D = $\frac{1.8 \text{ cm}}{2 \text{ cm}}$ = 0.9m
Nc = $\frac{42.3}{\sqrt{0.9}}$ = 44.5cm
N = 0.76 Nc = $\frac{32.15}{\sqrt{D}}$ = 33.84

For grinding using small balls.

N = (0.71-0.70) NC $= \frac{0.423}{0.95} = 0.445$

The maximum ball diameter (D_b)

$$D_{\rm b} = 283/d - 5.6$$

where d is the maximun diameter of coarsefeed to the mill =4cm

 $D_{bmx} = 28\sqrt[3]{4} = 44.4$ cm

Actual mill speed is equal 75% of critical speed for dry grinding

= 0.75Nc = 33.39

The weight of grinding ball in the mill (g) in kg is determine using

$$g = \frac{ND^2L\rho P}{4} = 0.785 D^2P \rho - 5.7$$

where D = internal diameter of the mill in (m) = 0.0009m L = mill length in = 0.0018m p = coefficient of loading = 0.3of = ball density Kg/m³ = 0.0048Kg/m[¶] of = 0.785 x (0.9) ²x 0.0048x0.3 = 1.0742 secondd compartment g = 0.785 x(0.9) x 0.0043x0.45 = 0.00358x0.45 = 1.61g first compartment.

of = 0.00358×0.27

= 0.967g third compartment

The number of revolution perminate can be calculated as $n = 8/\sqrt{D} (5\phi + 2) -5.8$

 $= \frac{8}{\sqrt{0.95}} (5 \times 0.45 + 2)$

 $= 8 \times 4.25$ $\sqrt{0.95}$ = '35 .79 first compartment.

n = 8.42x(5x0.3+2)

= 29.47 Second compartment

n = 8.42x(5x0.27+2)

= 28.21 third compartment.

The specific loss of milling balls and limiting material per 1Kg of grinded material can be calculated as

$$P = Ps 0.04 -5.9$$

$$kg$$

 $P = 0.25 \times 0.04/90 \times 0.8 = 0.01 = 1.39 \times 10^{-4}$

The mill productivity $Q = 6.4 \text{m/B} (P/V) \frac{0.8}{\text{gkn} -5.10}$

where n=coeffeicient of powder utilization for closed loop=1.1 = P open loop= 1.

q = specific productivity kg/kd hr

k = corective coeffecient for grinding finess=0.85

v = mill internal volume $m^3 = AD2/4 = 1.145m^3$

p =mass of the mill grinding ball (kg)

=0.967kgx, 10^{-4} 1.61 x10x10kg and 1.074x10³

For particle size of 20-25mm material to be grinded at ventilation air velocity of 0.3mm/s Act velocity = 0.7m/s the mill productivity increases 15-20%

- Q = 6.4m (0.95) 0.0003x250x1.1
 - = 4.41 third compartment
- Q = 6.4m (0.95) 0.0046 x 250 x 0.85 x 1.1
 - = 6.54 first compartment.
- Q = 6.4m (0.95) 0.0033 x 250 x 0.85 x 1. 1
 - = 4.74 second compartment.

Electric motor powder consumption (N) required to rotate the mill it given by

 $N = \frac{0.2pDn}{n_p} ns -5.55$

where P =mass of grinding ball (kg) = 0.001074,0.00161 and 0.000967.

D =mill diameter (m) =0.9m

n = number of revolutions peruminuteu=35.79%2947aand028%21
np =mechanical coefficient of usefull work done considering
loss due to friction and starting np = 0.85

ns = coefficient of increase in powder due to starting
 initia =1.1

 $N = 0.2 \times 0.00161 \text{kg} \times 0.9 \text{m} \times 35.79 \times 1.1$

0.85

= 1.34×10^{-2} first compartment

 $N = \frac{0.2 \times 0.0001074 \text{kgx } 0.9 \text{m x } 29.47 \times 1.1}{0.85}$

 $=7.4 \times 10^{-3}$ second compartment.

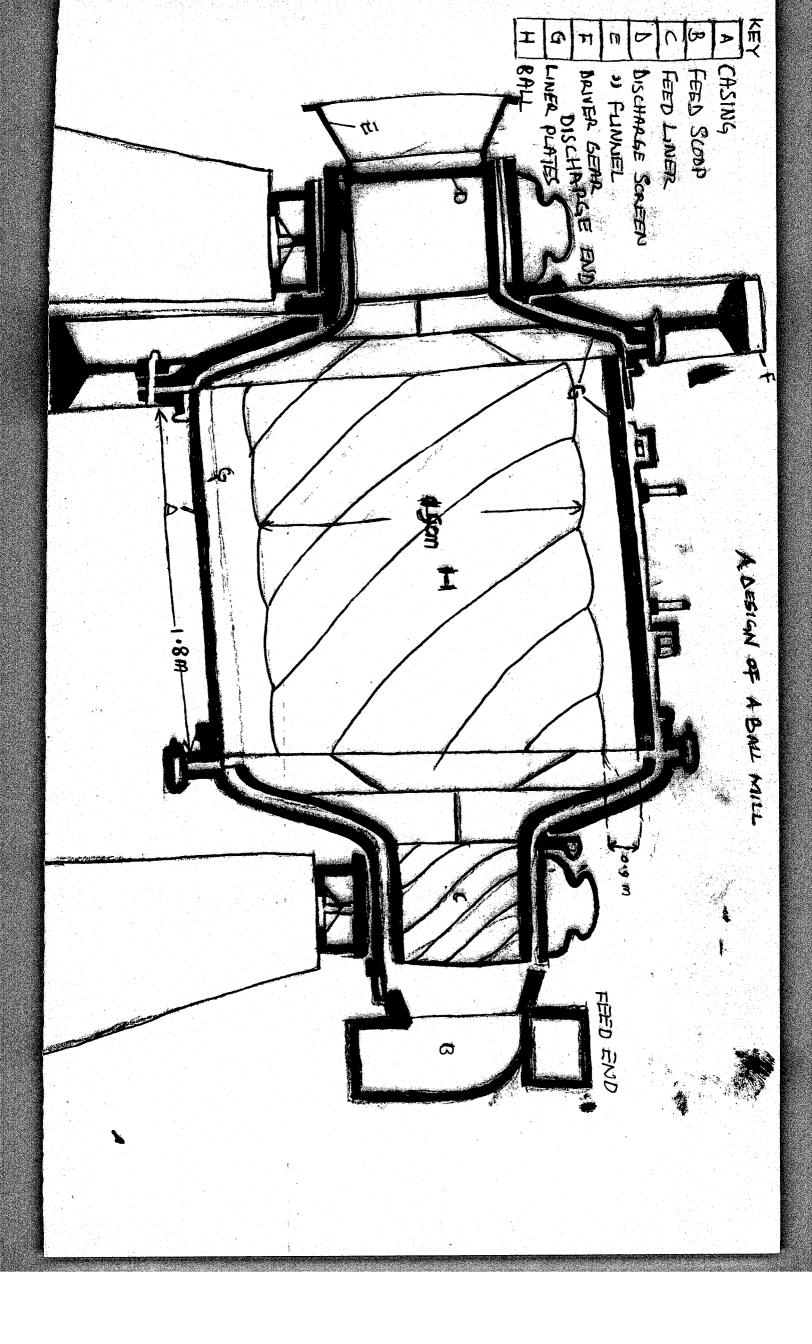
$$N = 0.2 \times 0.000967 \text{kg} \times 0.9 \text{m} \times 28.21 \times 1.1$$

= 0.85

=6.35x10 third compartment

Then, productivity of the ventilator during grinding $(v)m^3/hr$ V= 2818 D² (1- $\not q$) wkn - 5.12

where W0=velocity of ventilation air =0.3m/s
Kn =coefficient of air draght = 2



MATERIAL FOR CONSTRUCTION

Many factor: have to be considered in selecting materials for construction but for chemical process plant the over iding consideration is usually the ability to resist corrosion strength, and easily to operate. It should be a material that will gives lowest possible cost over the working live.

Material of construction should therefore be of great mechanical strength as with stand vibration stress, But longitudinal or axial, it should be one that is not suceptible to deformation inder any condition unless it life time is exceeded. Availability of standard should also forged steel, cast.

The Grinding balls can be made of forged steel balls general give optimun wear characteristics. Ball vary considerably in hardness with soft balls having briness hardness in range of 350 to 450 and hard balls having hardness in excess of 700 the cylindrical or conical shell is made of cyclindrical steel shell.

5.700

COSTING OF EQUIPMENT

Equipment costing can be obtain from the relation below.

Ce = $CS^n - 5$ where Ce = purchased equipment cost in (£2) S = costant = 1600 N = index = 0.4 Rate = N125 to £2 1

The costing of the crusher for charcoal the characteristic power parameter is

0.209 Kw

 $Ce = 1600 (0.209)^{0.4}$

46 855.42 = N106,927.28

The costing of the crusher for sulphur The characteristic power parameter

is 0.211kw

30

5.60

 $Ce = 1600 (0.211)^{0.4}$

= EQ 958.68 = N107335.14

The costing of ball mill The characteristic size parameter is 134.00 cm^3 Ce = 1600 (134)^{0.4} = EQ 11347.11 = N 1,418639.10

5.800

SAFETY CONSIDERATION

Any organisation has legal and moral obligation to safeguard the health and welfare of employer and the general public. Good management practices need to ensure safe operation will also ensure effecient operation.

Manufacture process are to sure extent hhazardous, but in chemical process, the application of sound engineering practices that risks are reduced to aceptable level.

The most likely hazardi expose to the employee by mill plants are dust explosion, and noise Release of dust to the atmosphere and people working around the plant, ways of minimising such dust should be considering during the design of ball mills. Noise is also harzard to health by exposure to high level of noise can cause permanent demage to hearing. At lower lever noise distraction and cause fatigue.

Industrial sound meter include a filter net work to give the meter the respose that correspond roughly to that of human error and attention should be given to noise level when specifing and when laying out the plant.

CHAPTER SIX

6.00 Discusion of results

Despite that Gum powder can be produce from any Light. White, and porous wood gs sources of charcoal was utilized to produce this powder, of which it almost have the same characteristic as normal powder. However, it was found that the mass proportion of the component that provide completely buning of the powder was the same with the relative masses of the components of the mixture but for carbon is a little high. The excess amount correspond to the masses of its inpurities (oxygen and hydrogen) and this gives us or corresponding to 67.2% Kno₃ 21.3\% sulphur and 11.5\% of charcoal.

The must important characteristic of a good Gum powder is to have a relatively low of heat of explosion this is highly achieve on test: for its heart of explosion and was found to be -719 Kcal/kg which was abit higher than that of normal powder (-735 Kcal/kg) by 16 Kcal/kg. Although, this might be as a result of encalorimeter use to takes the measurement and that of normal powder was calculated from the heat: capacity of stack gases of the produce of decomposition.

The comphensive analysis of the grain of the powder produce base on 5unit interval, it was seen that the grain of the powder do not burn by parallel layer but all over the mass of charge of their particles size is lower than 15mmdia. Above this, there exist a parallel layer buring of the powder and the buring time depend on the grains size. The particles Size of 5mm shows a faster rate of buring of about 312.50cm/min while that of 25mm has a slow buring effect (buring by parallel layer) of about 39.41cm/min.

This observation was also was ploted using rate against particle size and was notice on the graph from particle size above 15mm their exist a parallel layer buring and below it a non parallel buring. This characteristic tend to reduce as the size change below 10mm (from 15 to 10mm the change in rate was 184.62cm/min and from 10 to 5mm it was 12.5cm/min. Then, apoint may be reach of which any attempt decrease size further may enventually gives a negative rate. This properties was in correspondance with the observation made of normal powder of which the rate of 3.5mm is high than that of 10.5mm particle size.

In scale up, production in chapter five, it was found that for every 250kg/m feed of the charcoal 297 kg and 990 kg of sulphur and potassium nitrade are require base on the ratio of 2:3:10 respectively to produce 1455.45kg of the Gum powder. The power require by the sulphur crusher was 0.002kg higher than that require by the charcoal crusher be cause of difference in the working index of the materials and that ball mill has the highest power requirement and therefore has the highest cost.

343

.../2..

CHAPTER SEVEN.

7.00 CONCLUSION, RECOMMANDATION, NUMENCLATURE AND REFERENCE.

7.10 CONSLUSION AND RECOMMENDATION.

This project was carried out to explore possible way of manufacture Gum powder from local raw materials. The characteristic indicated that charcoal from madar <u>calotropic proceral</u> has high inflamenable property for Gum application. The result and other analysis shown that, the Gum powder can not withstand high pressure rathersplosions will result. Therefore, a moderate drying temperature below 60 $^{\circ}C$ is recommended for a good powder and raw material should of highly pure, as impurity may increase sensitive to friction impart.

moreover, different ratio of the component of the powder may be use to determine the best composion that will give the best properties when ever different application is required. The content of sulphur is high to reduce the dager of corbonomoxide form and release when little amount are used. While charcoal is low to avoid unreadted deposition of carbon on the barel after shuting. This result showns that gun produce posses excellent properties that are needed in most of our Guns. And that the particle size between 15 to 10mm die should always be used when ever employed.

However lack of availability of datas was a mojor constraint to this research project. Some of the important propaties of this powder were not determine as a result. Therefore I recomanded that effort should be made to provide datas needed in this areas of research. Further research should be to incoporate Gun powder with water proof to avoid shut failure at low temperature dur to its deliquence behavours.

For scaling up production of this powder any amount of initial feed of charcoal may be use and other proportions determine from the ratio of 2:3:10 for charcoal sulphur and kNo₃ respectively and that the size of a ball mill and other parameters are a function of length of the mill and diameter of the feed particles only.

Therefore, I recommanded that for any readjustment after fabri cation should be restrict to either length of the mill of the diameter of the feed particles. Therefore this shows that the high the diameter of the feed particles the bigger is the diameter of the ball.

.30 REFERENCES.

T.urbanski, chemistry and technology of explosive Vol) Sybol parkes, cyclopidia of chemistry mc Grain Hill) Sfordham, High explosive and propellants 2 revised edition) T. Urbanski, chemistry and technology of explosive Vol II ₫) Vol III 11 11 11 11 11 (5) "IV 11 11 11 11 11 (6) Botary for degree standent fifth edition A.C. Du (7) N.L. Glnkan, General Chemistry Vol 2 (8) M. Hughes, Physical chemistry. (9) David A. Tillman, Amaded J. Rossi, Miham D. kitto wood combustion (10) principle processes and economic. Rene Descartes, principle de la philosophie elsevier Amsterdam (1644) (11) T. Gay Lussac, Rapponts de la comite des pondre at sulpetre 1823 (12) M. Cherreuil according to R. E scales (13) Andrew Noble and F. Abel pril trans toy soc 165. (14) J.D Black wood and F.P. Bowden Proc. Roy Soc. (15) Perry chemical engineering hand book 6th Roberth Perry bongreen. (16)

7.20 NUMECLATURE

Db	=	Diameter of the ball (m)
Wi	=	Working index
E ₂	=	Power use by 0.6 by 0.6cm laboratory mill under the same
		condition
Nc	=	Ball critical revolution (rpm).
Хр	=	Size of coarse feed particles (cm).
N	=	Ball mill working Number (rpm).
0	=	Coefficient of loading.
Hu	=	Useful heat released (kcal/kg).
Ср	=	Specific heat capacity (kj/g).
Ac	 	Heat loss from stack gases (kj/g).
IIHw	=	Higher heat value (kcal/kg).
		Ball density (kg/m^3) .
D	- - -	Mean inside diameter of mill (m)
L	=	Inside length of the mill (m)
H ₂ °		Heat of varorization of water. (kj/g)
C	. · · · . .	Carbon.
ĸ		Potassim
S ¹³	÷:	Sulphur.
N	-	
0	=	
hw	-	the of water (ki/gk).
mw	=	
hb	-	the state of bomb (ki).
	_	- Total mass (g).
^m T		± 1 - 1 - 1
۷ _T	•	ai in tomporating
DT		f modulet (kegl/kg).
Нр		
U		= Rate of explosion (cm/min).
Т		= Temperature (°C).

