Development and Manufacture of RDX Based an Insensitive Composition for Tetryl Replacement

By

Muhammad Soulaman Khan

School of Chemical and Materials Engineering
National University of Sciences and Technology
2020

Development and Manufacture of RDX Based an Insensitive Composition for Tetryl Replacement

Name: Muhammad Soulaman Khan
Regn.No. 0000027449

This thesis is submitted as a partial fulfillment of the requirements for the degree of

MS in Energetic Materials Engineering

Supervisor Name: Dr. Abdul Qadeer Malik

School of Chemical and Materials Engineering (SCME)
National University of Sciences and Technology (NUST)
H-12 Islamabad, Pakistan
July, 2020

BISMILLAH IR-RAHMAN IR-RAHIM

"(I START) IN THE NAME OF GOD, MOST GRACIOUS, MOST MERCIFUL"

Certificate

This is to certify that work in this thesis has been carried out by **Muhammad Soulaman Khan** and completed under my supervision in polymer laboratory, School of Chemical and Materials Engineering, National University of Sciences and Technology, H-12, Islamabad, Pakistan.

Supervisor: _______________

Prof. Dr. Abdul Qadeer Malik
Professor School of Chemical and
Materials Engineering Department, SCME
National University of Sciences and
Technology, Islamabad.

Co-supervisor_______________

Dr. Sarah Farrukh
Assistant Professor School of Chemical and
Materials Engineering Department, SCME
National University of Sciences and
Technology, Islamabad.

GEC Member _______________

Dr. Ahsan
Assistant Professor School of Chemical and
Materials Engineering Department, SCME
National University of Sciences and
Technology, Islamabad.

GEC Member _______________

Lt. Col. (Rted.) Nadeem Ahsan
Assistant Professor School of Chemical
and Materials Engineering Department,
SCME National University of Sciences and
Technology, Islamabad.

Submitted through

Principal/Dean,

School of Chemical and Materials Engineering Department, SCME

National University of Sciences and Technology, Islamabad.

Dedication

Dedicated to my beloved family

My parents, sister, wife & kids

Whose words of encouragement, appreciation and
push for firmness helped me achieving my task.

Acknowledgements

First of all I am thankful to **The Almighty Allah**, the creator of the whole world, the Merciful, for bestowing His favors upon me, because without His will we cannot do anything and so I complete this research study, which would otherwise have not been possible at all.

I am highly obliged to the scholarly and erudite guidance of my supervisor Sir **Dr. A.Q.Malik** whose immense command on the subject and vast experience proved like beckon of light to the present work.

I would also express my gratitude to co-supervisor Dr. Sarah Farrukh, Assistant Professor School of Chemical and Materials Engineering, GEC member Lt. Col. (Rted.) Nadeem Ahsan, Assistant Professor School of Chemical and Materials Engineering and GEC member Dr. Ahsan, Assistant Professor School of Chemical and Materials Engineering, SCME National University of Sciences and Technology, Islamabad.

I am very much thankful to my ex- and present bosses in my office whose support gave me a lot of confidence and courage for the research.

I am thankful to all the laboratory staff and technicians for all their support during the research work.

I am thankful to my prestigious organization for sparing me for higher studies and all-out support which was vital for my work.

My special thanks to my parents, wife Nusrat and specially my son Usman. This is due to you my son. Their faces always were the source of motivation and support for me to complete my work without which it would not have been possible.

Everyone played a team role and I am thankful to all.

(Muhammad Soulaman Khan)

Abstract

Tetryl is an extensively used as an explosive in leads and boosters of ordnance items. However, it has serious limitations like its greater tendency to explode when subjected to high temperatures like encountered in certain modern weapon environments. Its cook-off temperature is low having lesser output resulting in marginal initiation of the next charge besides being a skin irritant during handling. Due to these limitations it needs to be phased out.

RDX has better cook-off characteristics than Tetryl. It is preferable explosive for use in ordnance subjected to temperatures approaching and exceeding the cook-off temperature of Tetryl. RDX also has a greater output so that an RDX lead or booster would be effective under circumstances in which a Tetryl lead or booster give marginal initiation of the next charge.

An insensitive RDX based explosive composition (containing RDX, binder and small quantities of desensitizers) is needed to be developed. The binder improves spherical nature of shape of the RDX granules and holds the loaded component together with greater strength. Small percentage of graphite is added to increase the flow rate of the material and act as lubricant on the faces of the RDX particles and granules. The stearates act as dispersant for avoiding agglomeration of the particles of RDX and its granules during coating of RDX with binder during mixing process. It is manufactured through precipitation reaction between aqueous solutions so that it does not allow agglomeration of the granules of RDX to build up.

Subsequent to the characterization of requisite ingredients, a laboratory scale method has been developed during this work. The method has been translated into a pilot plant version. The upshot of this work is the development of an RDX based insensitive composition for Tetryl conducted successfully.

Table of Contents

S.No. Contents Page

Chapter 1 General Introduction

Chapter-2 Materials and Methods

Chapter-3 Experimental Techniques

Chapter-4 Results and Discussion

Chapter-5 Design for Scale Up

List of Figures

List of Tables

Abbreviations

AR	Analytical Reagent
CDB	Cast Double Base
C-4	C-4 Composition of RDX
CH-6	CH-6 Composition of RDX
CMCDB	Composite Modified Cast Double Base
DBP	Double Base Propellant
CE	Composition Exploding
CPU	Central Processing Unit of Computer
DDT	Deflagration-To-Detonation Transition
DOS	Dioctyl sebacate
DIOS	Diisooctylsebacate
DSC	Differential Scanning Calorimetry
EM	Electron Microscope
EMCDB	Elastomer Modified Cast Double Base
FAE	Fuel Air Explosive
FTIR	Fourier Transform Infrared Spectroscopy
HE	High Explosive
HMX	High Melting Explosive
IUPAC	International Union of Pure and Applied Chemistry

IR	Infrared
MW	Molecular Weight
NUPC-6	Name of RDX Based Insensitive Composition
OM	Optical Microscope
PBX	Plastic Bonded Explosives
PIB	Polyisobutylene
POF	Pakistan Ordnance Factories
RDX	Research Development Explosive
SEM	Scanning Electron Microscopy
SAE	Society of Automotive Engineers
SLIFER	Shorted Location Indication by Frequency of Electrical Resonance
TNT	Trinitrotoluene
TEM	Transmission Electron Microscope
TDR	Time Domain Reflectometry
US	United States of America
UK	United Kingdom of Britain
VOD	Velocity of Detonation

Chapter 1

General Introduction

1.1 Energetic Materials

The materials which liberate tremendous amount of energy in a very short interval of time are known as energetic materials whereas the materials which have greater energy stored for liberation over a greater interval of time are called high energy materials.

The concept is elaborated by giving an example of fighting mode of a boxer and a wrestler. Consider a smart body boxer and heavy weight wrestler. The energy of boxer is low as compared to energy of wrestler. But when the mode of liberation of energy during fight is considered, one comes to know that the boxer tends to concentrate his energy to attack his opponents and at times knocks out them with only one punch in very short interval of time. Whereas, the wrestler due to his tremendous body and weight tends to liberate his energy and knock out his opponents in long interval of time. Though, wrestler has more power than boxer but boxer is smarter to knock out his opponents.

Similarly, the body of boxer can be considered energetic material and the body of wrestler can be considered as high energy material. The energetic material decomposes at very rapid rate and liberates tremendous amount of energy to produce tremendous pressure of gases during the decomposition. This sort of decomposition produces jump condition in temperature and pressure of the system in just an interval of microseconds. The jump condition is very lethal and can destroy buildings and human beings instantly. On the other hand, the high energy material decomposes at much slower rate than energetic material. This doesn’t cause jump condition and is not lethal as energetic materials are.

The energetic materials are generally called explosives. [1-2]The Fig.1-1 shows the great boxer ‘Muhammad Ali with his coach Joe E. Martin (1960)’ & the great wrestler ‘Sumo with his heavy body. Fig.1-2[1-2] shows fighting modes of ‘Muhammad Ali’ and ‘Sumo’. The pictures in Figures are presented as an example to elaborate the concept of energetic materials and explain the lethality of these materials.

Fig.1-1 The Great Boxer ‘Muhammad Ali & The Great Wrestler ‘Sumo’

Fig.1-2 Fighting Modes of ‘Muhammad Ali’ and ‘Sumo’

1.2 Classification of Energetic Materials

Energetic materials or explosives may be classified based on characteristics such as chemical structure, sensitivity and stability. However, a detailed classification is shown in Fig.1-3 [3-4].

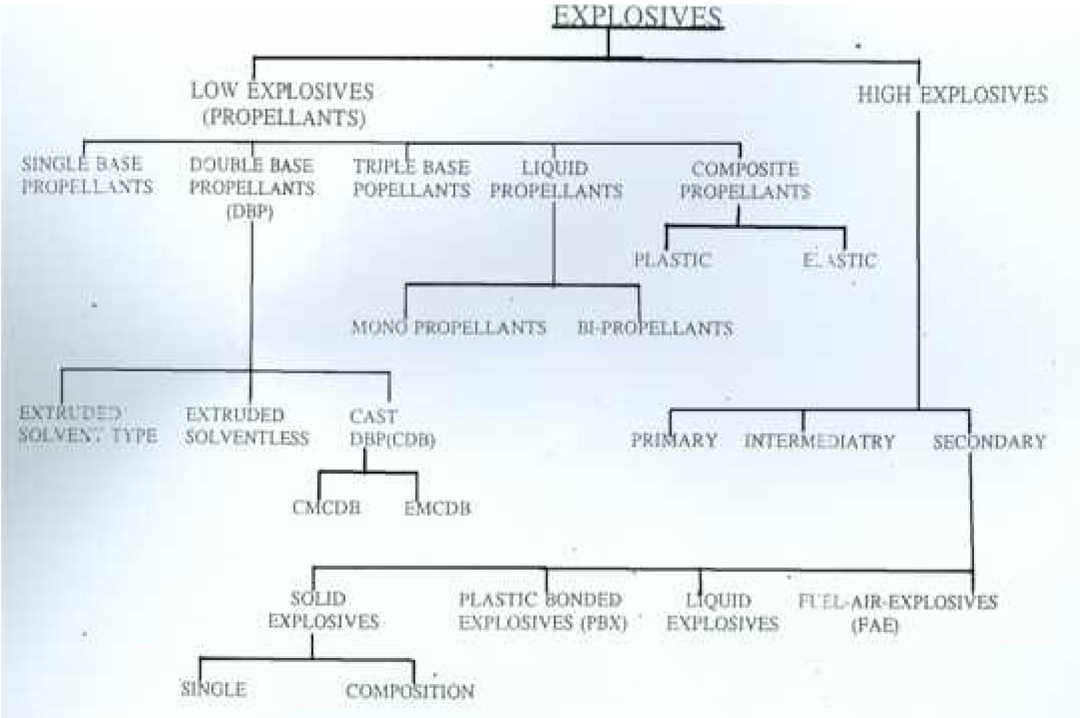


Fig.1-3 Classification of Explosives

1.2.1 Low Explosives or Propellants

A low explosive or propellant is any chemical substance or mixture that has burn velocity normally of order of m/sec. These deflagrate rather than detonate. A good example of these is black powder. Propellants are classified as single, double and triple base, liquid and composite propellants. The double base propellant (DBP) are further classified as extruded solvent type, extruded solventless type and cast double base (CDB) propellants. The CDB propellants are divided as composite modified caste double base (CMCDB) and elastomer modified caste double base (EMCDB) propellants. The liquid propellants are divided as mono and bi-propellants. The composite propellants are classified as plastic propellants and elastic propellants. [4-6]

1.2.2 High Explosives

A chemical or substance or mixture that has burn velocity of the order of Km/sec is known as high explosive. The process of burning or decomposition at such tremendous rate is called detonation and is always supersonic in nature. High explosives do not require oxygen for detonation and break down and are much more versatile than normal combustible materials. These are divided as primary, intermediatry and secondary explosives. The secondary are classed as solid, plastic bonded, liquid and fuel air explosives (FAE). Solid explosives are classified as single solid explosives and composition solid explosives. [4-6]

1.2.3 Pyrotechnics

Pyrotechnics are a particular group of explosive substances. These have versatile applications for flame and smoke production and signalling. These are characterised by burning velocity of the order of cm/s. [6]

1.3 Tetryl

An explosive called Tetryl was first developed in 1877 by Mertens and its structure was established by Romburgh in 1883[7]. It is one of the most toxic secondary explosives having molecular formula, $C_7H_5N_5O_8$ [8] Tetryl has also been known as “Composition Exploding (CE) Powder” [9]. It has been used as an intermediary demolition explosive [10]. It was initially used as an explosive in 1906 and later on was regularly used as the base charge of blasting caps [7]. It has been used as booster and donor charge or lead charge as well. [11]

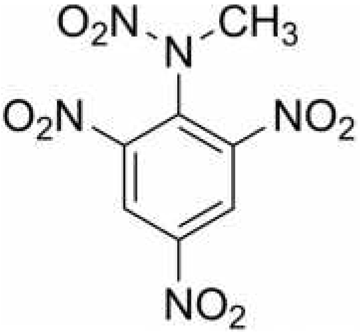


Fig.1-4 Tetryl [12]

1.3.1 Drawbacks of Tetryl

Tetryl has greater tendency to explode ammunition prematurely due to heat in the surrounding environment at about 140 ^{0}C. Its output energy is low as well which might fail in proper initiation of the next charge in different applications. It becomes very unsafe when it is used as booster explosive due to its sensitivity as well. [13] It causes yellowish colouring of exposed skin, acting as a skin irritant [14]. Tetryl workers have been historically known as “canaries” which is plural of “canary” known as yellowish bird. [14] Use of Tetryl in munitions filling operations in Western countries is obsolete now. The US has stopped its manufacturing and filling, and the UK has done it as well and has replaced all new stores with alternative ones. [15] Due to limitations associated with Tetryl such as low heat resistance, low output energy and hazards during its manufacture and filling operations it needs to be replaced.

1.3.2 Replacement of Tetryl

The key requirements for base material being sought as a replacement for Tetryl are; lesser impact sensitivity, comparable shock sensitivity, higher energy output, and local availability. [16] After World War I, major research programmes were conducted to find new and more powerful and energetic explosive material. One of these programmes came up with the result of cyclotrimethylenetrinitramine (RDX) which is also called Cyclonite. [17] RDX has no smell and is tasteless. It is commonly used as an explosive. It has a class of nitramide and is same in nature as HMX. It is powerful explosive and has great output energy. [18]

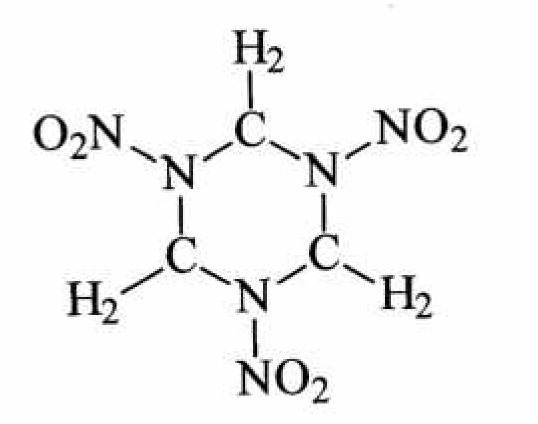


Fig.1-5   Structural Formula of RDX [12]

Its ammunition withstands up to 180 ^{0}C cook off temperature. Its output energy is higher than Tetryl as well which might ensure proper initiation of the next charge in different lead charge and booster applications. [13]

RDX booster pellets are abraded. It is slightly more sensitive than Tetryl as well. Since Tetryl is considered to be the most sensitive explosive practically useable in safety and arming device or fuze mechanism, therefore, pure RDX is not able to be used as replacement of Tetryl in this application.

A mixed desensitized composition of RDX with wax has very poor flow characteristics and is not entirely satisfactory. It adheres to the walls of molds when pressed for lead charges or booster pellets. Therefore, desensitized RDX with wax can’t be used to replace Tetryl as well. An explosive composition of RDX containing small quantities of desensitizers and matrix composition of binder can be used as replacement of Tetryl. [19]

The chief advantages of RDX based insensitive compositions are enhanced stability in hot environment applications and output for a given size of lead charge and booster for transfer of detonation. RDX becomes very safe explosive in desensitized form when used as booster explosive in boosting detonation and transferring it to main charges due to lesser sensitivity and safety to handle as well. [13]

1.4 Techniques for Coating and Desensitizing Explosives

There are three main techniques which have been used historically to achieve coating and desensitizing sensitive explosives. [20]

1.4.1 Solvent Slurry Technique

The solvent slurry technique involves an interaction of polymer, organic solvent and aqueous slurry of explosive. Polymer is dissolved in solvent and this is mixed as whole with aqueous slurry of explosive. Then solvent is recovered by distilling as a whole. The remaining slurry is filtered to give the product.

This technique has disadvantages during production such as flammability and toxicity issues linked with use of organic solvents in bulk amounts. This requires sophisticated processing equipment designed for distillation and often under vacuum operation at desired stage.

1.4.2 The 'Smeared Wax" Technique

This technique involves interaction of a fine wax and aqueous slurry of explosive. The stirred mixture is heated to evaporate water, melt the wax and get coated explosive with homogenously mixed wax.

This technique has safety issues like direct contact of explosive composition with heated surfaces when water is removed. This may cause severe consequences if process is uncontrolled and hot spot causes DDT phenomenon.

1.4.3 Aqueous-Emulsion or Dispersion Technique

The third main technique for preparing desensitized explosive composition is by mixing aqueous slurry of explosive with an aqueous emulsion or dispersion of the wax/polymer dissolved in solvent in slow or drop wise manner. The binder solution is added in slow manner is coated on grains of RDX and solvent with binder is removed by evaporation and is recovered in stage wise distillation.

At the end of this technique remaining additives are introduced in composition and hence required desensitized composition of explosive is achieved after filtering water and drying the filtrate in hot air circulated drying oven. This technique avoids the safety issues linked with the other two techniques and has been used historically for the preparation of explosive powder compositions.

Moreover, this technique is used when very small amount of binder is to be introduced in compositions. [10, 13, 15]

1.5 Roles of Additives

The amount of binder improves the binding characteristics at the expense of some decrease in flow characteristics of composition when introduced and pressed in molds. Graphite is added to increase the flow of the material and act as lubricant on the faces of the RDX grains. The stearates act as dispersant for avoiding agglomeration of the RDX grains during coating process of RDX with binder when solvent is evaporated. It is also as hydrophobic agent in composition. It can be introduced in composition by physical or chemical methods through precipitation reaction. These do not allow agglomeration of the grains of RDX to build up. These additives also improve sphericity and act as desensitizers of the RDX grains. [10, 13, 15]

1.6 Objectives of Present Study

The main objectives of this research work are:

- To characterise raw materials and to optimize and finalize the process parameters for development of an RDX based insensitive composition.
- To establish a laboratory scale method for development of an RDX based insensitive composition in defined system.
- To present scaled up design for production of an RDX based insensitive composition.

1.7 Short Methodology

These objectives will be achieved by characterising raw materials, optimizing and improving the parameters of historically used processes and carefully designing the pilot scale plant for production of composition. First of all, suitable solvent for process will be selected. In 2nd stage plasticization of binder shall be performed. The binder used in historically used processes is polyisobutylene but its grade with specified molecular weight and other parameters is not available. Research shall be conducted on developing the matrix composition of available grade of polyisobutylene binder which will help for attaining plasticized or matrix composition of binder. In 3rd stage selection of size range of RDX for homogenously mixed composition will be performed. In 4th stage, selection of method of introduction of calcium stearate in composition shall be performed. At the end method for mixing of graphite shall be selected and hence process shall be developed for preparation of a well-mixed homogeneous RDX based insensitive composition.

Chapter 2

Materials and Methods

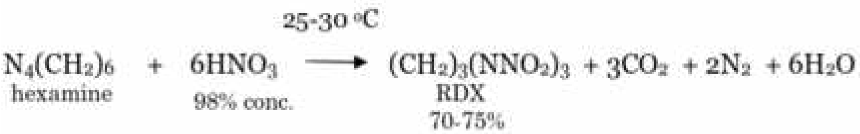
2.1 Materials

2.1.1 Cyclotrimethylenetrinitramine (RDX)

RDX which stands for “Research Development Explosive” (less common) and “Royal Demolition Explosive”, is the main ingredient of desired composition. RDX is easily available from POF as well. [21-22]

The manufacture of is performed by one of two methods, Woolwich or ‘nitric acid’ method and Bachmann or ‘aceto-nitric’ method.

The Woolwich method is also known as the direct nitrolysis method. This method produces a yield of RDX of 70-75%. Hexamine is nitrated with nitric acid to produce RDX [23] as follows;



$$\underset{\text{hexamine}}{N_4(CH_2)_6} + \underset{\text{98\% conc.}}{6HNO_3} \xrightarrow{25\text{-}30\ ^{o}C} \underset{\substack{\text{RDX}\\ \text{70-75\%}}}{(CH_2)_3(NNO_2)_3} + 3CO_2 + 2N_2 + 6H_2O$$

In Bachmann or ‘aceto-nitric’ method a mixture of ammonium nitrate and nitric acid is reacted in the presence of acetic anhydride at 75 ^{0}C. Its products are ca. 79% RDX, ca.6% HMX and ca.15% intermediates.

$$N_4(CH_2)_6 + \underset{\text{98\% conc.}}{4HNO_3} + 2NH_4O_3 + 6(CH_3CO)_2O \xrightarrow[\text{75°C, 1 atm.}]{} \underset{\text{70\% yield}}{2RDX + 12CH_3COOH}$$

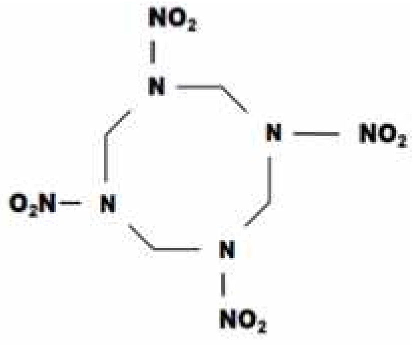


Fig.2-1 Structural Formula of HMX [24]

The type of production is used to classify RDX grades. [22,23,24,25] Class 1 RDX of POF is finely recrystallized has been finalised to be used in desired composition in this research. It consists of an intimate and uniform mixture of RDX and HMX obtained by co-precipitation and recrystallization from aqueous acetone. [26-27] The stability of RDX crystals is affected by impurities, particle size, and morphology. The particle size affects the mechanical and thermal sensitivity of RDX. [28] It detonates according to the following empirically determined equation [29]:

$$(CH_2.NNO_2)_3 \longrightarrow 3CO + 3H_2O + 3N_2$$

Table 2-1 Properties of RDX

Sr. No.	Property	Specification
1.	Appearance & Odour [18]	White powder & Odourless
2.	Method of Production [30]	Fine recrystallized
3.	Melting point [31]	204.38 to 204.49 ^oC
4.	Volatile matter [30]	11.95 % max.
5.	Retained on 100 Mesh BSS [30]	0.88 % max.
6.	Retained on 150 Mesh BSS [30]	95.41 % min.
7.	Size Range [30]	-100+150 mesh or 150 to 105 micron
8.	HMX Contents [26-27]	0.8 % max.
9.	RDX [26-27]	99.2 % min.
10.	Density [32]	1.796 (gm/cm^3) max.
11.	Specific Heat Capacity [33]	0.265 (cal/gm.K) max.
12.	Thermal Conductivity [33]	$4.88 \pm 0.19 \times 10^{-4}$ (cal/cm-s-°C)
13.	Viscosity of 10 % Water Slurry [34]	0.7 (Pa-s) max.

2.1.2 Polyisobutylene (PIB)

PIB is a rubber [35] and has linear structure. It has two methyl groups and one methylene group as in Fig.2-2.

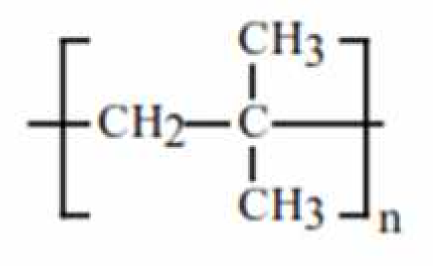


Fig.2-2 PIB

PIBs are classified as high and low molecular weight. Use of polyisobutylene is explosive composition formulations has a history to act as a binder. It is used in RDX based compositions like C-4 and CH-6 etc. The main features of PIB are low glass transition temperature and excellent moisture resistance. These features make PIB to be used as binder to achieve RDX based composition which can withstand low and high temperature tests, thermal shock or cycling test, low and high humidity tests, low and high pressure tests, mechanical shock test, acceleration test, transportation test and performance test. All these tests are essentially required for ammunition's military standards for qualification purpose. It is good option to achieve desired composition. It has physical properties as follows; [36-38].

Table 2-2 Properties of Polyisobutylene

Sr. No.	Property	Specification
1.	Appearance, odour and form	Colourless, odourless and Slab/chunk
2.	Weight average molecular weight	M_w ~4,200,000
3.	Refractive index	1.51
4.	Glass transition temp	-62^{o}C
5.	Density	0.92 (gm/cm^3) at 20°C
6.	Specific heat capacity	2 (KJ/Kg·K)
7.	Thermal conductivity	0.19 (W/K·m)

2.1.3 Diisooctylsebacate (DIOS)

Its chemical name is Diisooctyl Sebacate and is abbreviated as DIOS. Its molecular formula is $C_{26}H_{50}O_4$ and its structural formula is as follows;

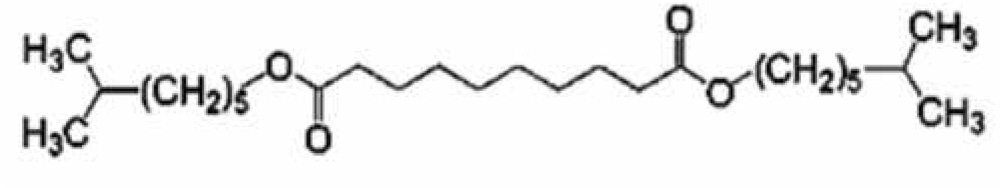
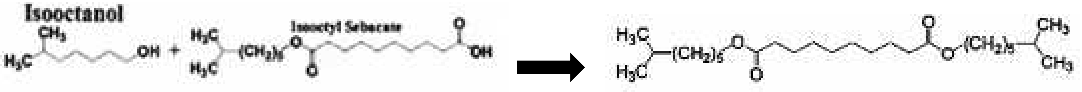


Fig.2-3 Diisooctylsebacate Structure

The raw materials required for its production are Sebacic Acid, 2-Ethylhexanol, Decanoic Acid and 1-Octanol. The chemical reaction between Isooctanol and Isooctyl sebacate which is intermediate product, [39-40] gives DIOS as shown;



Polymer plasticization can be achieved by the addition of DIOS plasticizer. DIOS is a low temperature plasticizer and is used as plasticizer for vinyl and other resins in very small quantities. Its addition results in a rapid increase in chain ends and free volume. It improves flow and strength of solution of polymer. [41-44]DIOS has been used as plasticizer for plasticization of the polyisobutylene. The properties of DIOS are as follows. [41-44]

Table 2-3 Properties of Diisooctylsebacate

Sr. No.	Property	Specification
1.	Appearance & odour	Colorless & odourless
2.	Molecular Formula	$C_{26}H_{50}O_4$ (73.24%C, 11.8%H, 14.96%O)
3.	Mol. Wt.	426.673 (gm/mol)
4.	Form	Liquid at room temperature
5.	Glass transition temp	-64°C

6.	Melting point	-55 °C
7.	Boiling Point	435.5 ± 0.0 °C at 760 mmHg
8.	Flash Point	189.7±18.2 °C
9.	Density[38]	0.92 gm/cm^3
10.	Specific Heat Capacity*	18.82 (J/mol.K)
11.	Thermal Conductivity*	69.67 (W/m.K)
12.	Viscosity	23 cP at 25 ºC

* Elemental weighted average properties

2.1.4 SAE 10 Engine Oil

In SAE-10, SAE stands for Society of Automotive Engineers and 10 stands for grade standardized by SAE. SAE-10 engine oils consist of oil and additives and have excellent flow. [45-46]

These have high quality lubrication and good anticorrosion properties which effectively protect from corrosion, from combustion moisture and acids. These additives inhibit the oxidation. The additives in SAE-10 engine oils make it possible to use these oils in explosive composition formulations. These can be used for developing polarity and reducing viscosity of binders. Hence these can be utilized to develop matrix compositions of binders for easy and smooth coating processes. The properties of SAE-10 engine oil used in this work are as follows; [47-49]

Table 2-4 Properties of SAE 10 Engine Oil

Sr. No.	Property	Specification
1.	Appearance	Dark brown
2.	Carbon Atoms	C_{18} to C_{34}
3.	Form	Liquid

4.	Flash Point	225
5.	Pour Point	-30
6.	Density	0.88 gm/cm^3
7.	Specific Heat Capacity	1.964 KJ/Kg. ^{o}C
8.	Thermal Conductivity	0.144 W/m. ^{o}C
9.	Viscosity Index	110
10.	Kinematic viscosity at 40 ^{0}C	44 cSt
11.	Kinematic viscosity at 100 ^{0}C	6.6 cSt
12.	Dynamic Viscosity at 50 ^{0}C	0.02 N s/m^2

2.1.5 Graphite

Carbon exists in three allotropic forms, diamond, fullerenes and graphite. Graphite is one of the softest solids having high lubricity. The properties of graphite used in this research work are as follows;

Table 2-5 Properties of Graphite

Sr. No.	Property	Specification
1.	Grade	Analytical Reagent Grade
2.	Appearance	Black
3.	Atomic Number	6
4.	Atomic Weight	12.011 gm/mol
5.	Form	Hexagonal crystals
6.	Sulphur Contents	0.2 % max.

7.	Density	2.25 gm/cm^3
8.	Specific Heat Capacity	2.03 Cal/degree mol
9.	Thermal Conductivity[42]	142 W/m.K

It is used for manufacturing explosive composition formulation. Its softness and lubricating features enable it to be used as lubricant and desensitizer in explosive composition formulations such that efficiently decreasing the sensitivities of explosive compositions and improving their flow characteristics. The main requirement by military specifications of graphite used in explosive composition formulation is minimum sulphur contents. [37, 50-52]

2.1.6 Sodium stearate

Sodium stearate has molecular formula is $C_{18}H_{35}O_2Na$ and molecular mass is 306.466 gm/mol. Its structure is as follows;

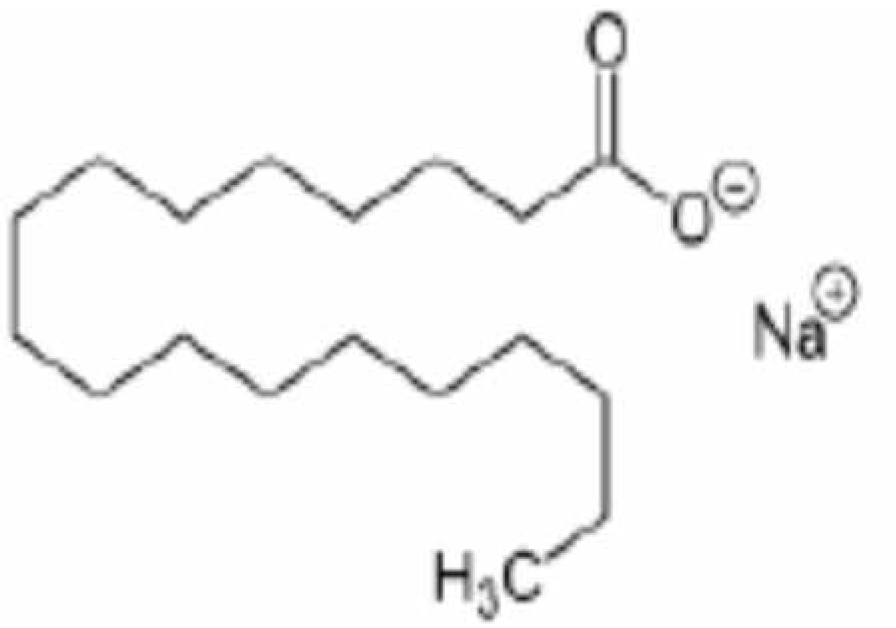


Fig.2-4 Sodium Stearate

Its percentage in soap depends on the fat contents in the oils and fats. It is white in colour and is an amorphous solid. It is soluble in warm water and has both hydrophilic and hydrophobic parts. It is also soluble in alcoholic water in cold state. The sodium stearate is used for precipitation reaction to produce calcium stearate to separate grains from agglomeration. Its properties are as follows; [53-55]

Table 2-6 Properties of Sodium Stearate

Sr. No.	Property	Specification
1.	Appearance	White
2.	State	Amorphous Powder
3.	Molecular Formula	$C_{18}H_{35}O_2Na$
4.	Molecular Mass	306.466 gm/mol
5.	Solubility in water	Soluble
6.	Melting point	245 to 255 °C
7.	Density	1.02 gm/cm^3
8.	Specific Heat Capacity	2.4 (J/g °C)
9.	Thermal Conductivity	59.05 W/m.K

2.1.7 Calcium Chloride

It is calcium salt of chloride and is an inorganic compound. It exists in mono-, di-, tetra- and hexahydrated forms. Its most important use is in controlling particle size development and reducing coalescence. It has been used to control the particle size of coated RDX grains in combination with the aqueous solution of sodium stearate. It is used to reduce coalescence of coated RDX grains and avoid agglomeration of RDX grains. It is white crystalline solid, powder or flake. It can absorb different amounts of water. [56-58] The properties of $CaCl_2$ used are as follows;

Table 2-7 Properties of Calcium Chloride

Sr. No.	Property	Specification
1.	Appearance	White
2.	Form	Powder

3.	Chemical Formula	$CaCl_2$
4.	Formula Wt.	110.99 g/mol
5.	Solubility in water	High
6.	Melting point	772–775 °C
7.	Density	2.15 gm/cm^3
8.	Specific Heat Capacity	17.42 cal/degree mol
9.	Thermal Conductivity	599.0 $x10^{-3}$ W/m.K
10.	Heat of solution	0 .16 Cal/g°C

2.1.8 Calcium Stearate

It is a carboxylate of calcium and is classified as calcium soap. Its chemical formula is $(C_{18}H_{35}COO)_2Ca$ or $C_{36}H_{70}CaO_4$ and formula mass is 607.030 gm/ mol.

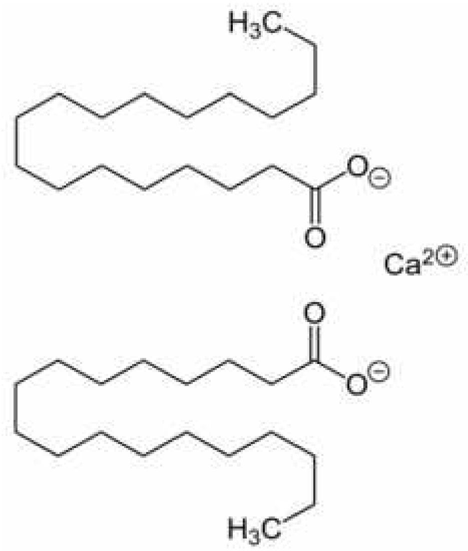


Fig.2-5 Calcium Stearate

It can be produced during explosive composition formulation to act as dispersant between aqueous solutions of $C_{18}H_{35}COONa$ and $CaCl_2$. The reaction equation is as follows;

$$2C_{18}H_{35}COONa_{\ (aq)} + CaCl_{2\ (aq)} \rightarrow (C_{18}H_{35}COO)_2Ca + 2NaCl_{\ (aq)}$$

It is used as a component of some surfactants in soaps for hard water areas. It is waxy material and is insoluble in water. It is applied as flow enhancing and surface conditioning agent due to its waxy nature.

It can play its role in water proofing of the explosive composition as well. It also acts as lubricant and mold releasing agent.

Its roles in this research are very crucial. It has been selected to play its main roles as; to act as dispersant during coating process of RDX grains, to perform as lubricant, to act as mold release agent for desired composition, to play its roles in making the desired composition hydrophobic, and enable composition for underwater applications as well.

These roles can be achieved by its direct mixing in small batches of explosive compositions to some extent. When it is produced during precipitation reaction it works better as a dispersant whether the batch size is small or large. It can perform its roles best when it is homogeneously mixed during mixing process. Its generation ensures complete mixing and control over RDX grains. [59-65] It has properties as follows;

Table 2-8 Properties of Calcium Stearate

Sr. No.	Property	Specification
1.	Appearance	White Waxy Powder
2.	Chemical Formula	$C_{36}H_{70}CaO_4$
3.	Formula Wt.	607.030 gm/mol
4.	Melting point	155 ^oC
5.	Density	1.08 gm/cm^3
6.	Specific Heat Capacity	2.33 (J/g °C)
7.	Thermal Conductivity	112.84 W/m.K
8.	Standard Heat of Formation	-2550.03 KJ/mol

2.1.9 n-Hexane

n-Hexane means normal hexane which is a straight chain alkane aliphatic or saturated carbon compound containing six carbon and fourteen hydrogen atoms. Its IUPAC nomenclature is n-Hexane or unbranched hexane.

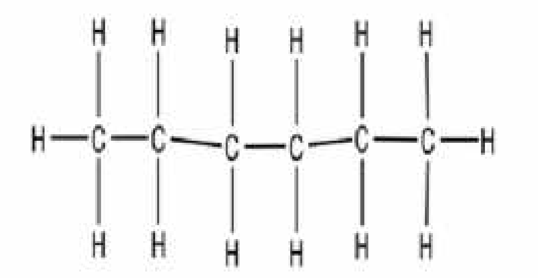


Fig.2-6 n-Hexane

It is colourless and is non-polar. It is in various industries. [64, 66-67] The properties of n-Hexane are as follows;

Table 2-9 Properties of n-Hexane

Sr. No.	Property	Specification
1.	Appearance & Odour	Colourless & Petrolic
2.	Molecular Formula	C_6H_{14}
3.	Molar mass	86.178 gm/mol
4.	State at Room Temperature	Liquid
5.	Melting point	−96 to −94 °C
6.	Boiling Point	68.5 to 69.1 °C
7.	Flash Point	−26.0 °C
8.	Explosive Limits	1.2–7.7%
9.	Autoignition Temperature	234.0 °C
10.	Vapour Pressure	17.60 kPa (at 20.0 °C)

11.	Vapour Pressure	76.36 kPa (at 20.0 °C)
12.	Refractive Index	1.375
13.	Density	0.66 gm/cm^3
14.	Specific Heat Capacity	265.2 J/K.mol
15.	Thermal Conductivity	0.1297 W/m.K
16.	Viscosity	0.3 mPa·s

2.1.10 Distilled Water

Distilled water is water boiled into vapour state and then condensed back into liquid state and is known as distillate. The other ingredients do not get boiled and remain in the original container. Only purified water is received through condenser. Water has polar nature and its molecular formula is H_2O and molar mass 18 gm/mol. RDX coating process and formulation into desired composition is done through usage of distilled water. It is used to prepare RDX water slurry in which the rest of ingredients are introduced one by one. The properties of distilled water are as follows; [68-69]

Table-2-10 Properties of Distilled Water

Sr. No.	Property	Specification
1.	Appearance	Colourless
2.	Odour	Odourless
3.	Molecular Formula	H_2O
4.	Formula Wt.	18. g/mol
5.	Form	Liquid
6.	Melting point	0 ^oC

7.	Boiling point	100 °C
8.	Density	0.997 gm/cm^3
9.	Specific Heat Capacity	4 .1813 (J/g °C)
10.	Thermal Conductivity	0.6072 W/m.K
11.	Viscosity	0. 890 mPa.s

2.2 Methods

2.2.1 Method for Selection & Processing of Solvent

For preparing desensitized compositions of RDX, solvents used were toluene, n-octane and n-Hexane. Toluene was a clear, water-insoluble liquid with typical smell of paint thinners. Its boiling point was about 110-111 °C. N-Octane was a hydrocarbon with the chemical formula C_8H_{18}, and the condensed molecular formula is $CH_3\ (CH_2)_6CH_3$. Its boiling point was about 125-126 °C. The higher processing temperature for evaporation associated with these solvents was a safety problem. In water slurry process, the solvent had to be recovered at the end. When these solvents were heated to their boiling points only then it was possible to recover these solvents. As the heating range was towards higher side, this could intrinsically cause safety problem. These solvents were very toxic and needed respirators during handling.

Considering these issues, n-hexane having better properties and lower toxicity was selected for developing RDX based composition. N-Hexane was a hydrocarbon with the chemical formula C_6H_{14}. Its boiling point was 68-69 °C and was removed rather easily at lower temperature without risking the process due to greater sensitivity of RDX at higher temperatures. It was used as economical, relatively safe, largely unreactive and easily evaporable non-polar solvent. It was also very safe from toxicity point of view using similar temperature control measures used for toluene and n-octane.

N-Hexane was selected as solvent for process for developing RDX based insensitive composition due to following reasons; low operational cost, low capital equipment cost, low energy requirement, low toxicity & greater safety for handling. It played its role as solvent during mixing, processing and dissolution to help in achieving homogeneously mixed composition of binder and the composition to be developed.

2.2.2 Method for Selection & Processing of Binder

Due to availability of high molecular weight polyisobutylene, several experiments were conducted to adjust its properties to prepare it for its role as binder in RDX based composition. This work lead us in reduction in mixing time and improving properties of high molecular weight PIB to play its role as binder. Tack property and low polarity of PIB was overcome by the addition of DIOS having polar nature to high molecular weight PIB. SAE 10 oil was added in limited amount to reduce viscosity of the raw high molecular weight polyisobutylene and DIOS. Hence a plasticized composition of polyisobutylene was developed. This composition was named as plasticized or matrix composition of polyisobutylene. This matrix composition consisted of ingredients as shown in Table 2-11.

Table 2-11 Composition of Plasticized PIB

Sr. No.	Ingredient	% (w/w)
1.	PIB	0.3334
2.	DIOS	0.5244
3.	SAE-10 Oil	0.1422

DIOS was used as plasticizer for plasticization of high molecular weight polyisobutylene. Plasticity of PIB was enhanced. Consequently, plasticization process of PIB induced plastic flow in PIB. DIOS increased intermolecular distance, thereby decreasing intermolecular bonding forces of PIB and resulted in long chain ends of PIB.

The plasticized composition of PIB was also considered improved the processability and coating process by reducing mixing time and final product. The DIOS molecules decreased intermolecular forces by increasing polarity and enhanced tack property.

SAE 10 engine oil introduced anti-oxidative property to enhance life and provided greater ease in coating process due to reduction in viscosity of polyisobutylene and DIOS blend. It improved coating and life of PIB and DIOS molecules. It helped in preventing sticking and kept easy coating of PIB plasticized with DIOS on face of RDX particles and granules. Thus plasticized PIB had lesser processing and mixing time for preparing an insensitive composition of RDX. The plasticized PIB had better flexibility, elongation, cohesive strength and polarity of high molecular weight polyisobutylene.

2.2.3 Method for Selection of Size Range of RDX

A series of experiments was performed for selecting RDX size range required for homogenously mixed RDX based composition. For this purpose initial work was started using -325 mesh RDX of POF Wah. After the microscopic and visual analysis of batches produced, the size range for desired composition was narrowed by separating portions of RDX of -325 mesh size through sieving. For the homogeneously mixed composition using RDX of different sizes experiments were performed. These experiments were performed using a recipe of 50 gm in one litre jacketed beaker using Teflon coated magnetic stirrer and hot plate. These experiments resulted in different compositions with different coating characteristics of binder. The size used for development of composition was -50----+150 mesh. When this size range of RDX was used the quality of coating and granulation of RDX crystals was very good. The size range of RDX adopted for the development of RDX composition was further narrowed. The optimized size range of RDX adopted for development of RDX composition was -100---+150 mesh. This size resulted in consistently uniformly distributed granules of RDX coated with plasticized PIB. When finer than 150 mesh particles were used for the preparation of composition it was seen that the binder and other ingredients of the composition with RDX do not give homogenously mixed well granulated composition. When particles greater than 100 mesh were used, it resulted in composition needing more and more binder for its digestion with binder due to large size grains. Using RDX of -100 ---+150 mesh size a well-mixed, well-digested and optimized composition from point of view of performance and mixing was developed. The size range was optimized using defined mixing system and homogeneity of composition.

The greater the percentage of binder to be mixed in the composition the greater was binding capability of composition. The lesser the percentage of the binder the lesser was the binding strength of the composition. When very small quantity of binder was mixed in the composition it needed a very high revolution per minute speed of the agitating system which can cause safety hazard in case of electrostatic charge accumulation, it can cause breakage of large particles into small particles to change the size range of the composition as well. If very fine particles are used for the composition a phenomenon known as agglomeration causes the initial amount of binder added to result in process difficulties for mixing of the composition.

In this case binder caused agglomeration of light particles in the form of large chunks. The moment it happened the whole composition was fluidized at the top of the mixing system and leaved stirring system to be ineffective. Sometimes when the process was uncontrolled the mixing of batch became impossible due to formation of large chunks causing total failure of mixing process. Greater homogeneity of composition was achieved by rapid mixing at high speed of the agitation system. This caused change in size range of the particles due to impact as well.

Mixing system, homogeneity of composition and performance of the composition were all dependent on the size range of RDX selected for composition. Finer the size ranges the harder and tougher was to get it well mixed homogeneously. The larger the particle sizes the more energy and speed was required by stirring system to get the particles in the suspended form. Even the manner of adding binder to certain size range of particles used in composition also caused disturbance of the mixing system. The finer the particles the slower was mixing of binder to avoid flooding by chunk formation of the whole mixture. An optimum size range of RDX was selected after a series of experimental work from -100---+150 mesh for developing an insensitive composition of RDX.

2.2.4 Method for Mixing of Calcium Stearate

Two types of mixing methods of the calcium stearate in composition, physical and chemical were used. In physical method calcium stearate was directly mixed in composition. In chemical method the calcium stearate was resulted in the precipitated form by chemical reaction. The best method for particle and granules size control during mixing process was chemical method of introducing calcium stearate. In this method solutions of sodium stearate and calcium chloride were prepared in distilled water in separate beakers. The calcium chloride solution was added to sodium stearate aqueous solution. The precipitation reaction of these solutions resulted in introduction of calcium stearate as dispersant in the composition. The chemical reaction occurring between aqueous solutions was;

$$2C_{18}H_{35}COONa_{\,(aq)} + CaCl_{2\,(aq)} \rightarrow (C_{18}H_{35}COO)_2Ca + 2NaCl_{\,(aq)}$$

$$\text{2 mol} \quad : \quad \text{1 mol} \qquad\qquad \text{1 mol} \quad : \quad \text{2 mol}$$

Its main role in the composition was acting as dispersant. The dispersant acted as an agent to disperse the granules prepared during distillation of solvent from emulsion of composition. This avoided agglomeration of the whole mix during mixing and helped in homogenizing the composition to large extent. Calcium stearate was insoluble in water and was a waxy material. Calcium stearate increased the flow of binder on to the surfaces of RDX granules. It also acted as an anticaking agent. It did not allow the caking of composition in the form of chunk mass and avoided flooding of the mixture during mixing. It made composition waterproof and hydrophobic. It did not allow wetting of the composition in water. During mixing when granules of RDX were developed calcium stearate acted as dispersant and avoided buildup of the grains causing failure of homogenizing process of composition.

2.2.5 Method for Mixing of Graphite

Graphite was mixed in the composition with sodium stearate aqueous solution then added to RDX-water slurry emulsion. The graphite acted as desensitizing agent in the composition. It also performed its role as lubricant and decreased the sensitivity of composition. It enhanced its stability. It also acted as an agent for improvement of flow characteristics of the composition. It converted into layers and spread throughout the composition after blending with graphite. It also reduced static charge sensitivity of the composition.

2.3 Method for Lab Scale Preparation of Composition

The process developed for lab scale preparation of composition contained stages as;

i. In first stage, RDX containing 20% moisture contents was taken. The RDX was dried in air circulated oven at 70-80 ^{0}C. Then, a 10% RDX water slurry containing 10% RDX and 90% water by weight was prepared in jacketed beaker of one litre with Teflon coated magnetic stirrer and flameproof hotplate. The slurry was heated up to 50 ^{0}C.

ii. In second stage, the plasticized polyisobutylene was dissolved in the n-hexane and heated up to 45^{0}C. Then, this solution of polyisobutylene was added to RDX-water slurry in dropwise manner by maintaining the temperature of emulsion at 50 ^{0}C. Digestion of plasticized polyisobutylene solution was achieved in RDX-water slurry emulsion at 50 ^{0}C for 15 min.

iii. In third stage, sodium stearate was dissolved in distilled water and an aqueous solution containing 13% sodium stearate and 87% water was prepared. The graphite was mixed with sodium stearate solution and heated up to 60^{0}C. The graphite-sodium stearate solution was added to emulsion maintaining temperature of the emulsion at 50 ^{0}C. The whole emulsion was stirred for 25 min to distribute homogeneously.

iv. In fourth stage, calcium chloride was dissolved in solution containing 20% calcium chloride by weight % in distilled water. Calcium chloride aqueous solution was mixed with the above emulsion in step iii maintained at 50 ^{0}C. The precipitation reaction of these solutions resulted in introduction of calcium stearate as dispersant in the composition. The whole mixture was digested at 50 ^{0}C for 30 min.

v. In fifth stage, n-hexane was distilled from digested emulsion mixture. Distilling of n-hexane at 68-69 ^{0}C from emulsion left plasticized polyisobutylene coated on grains of RDX, calcium stearate and graphite composition. Hence composition containing RDX, graphite, calcium stearate, polyisobutylene, diisooctyl sebacate and SAE-10 engine oil was prepared. This composition was named as NUPC-6. This composition was washed from alkali contents along NaCl with distilled water to neutral pH, filtered cooled to room temperature.

The process flow diagram of method for lab scale preparation of NUPC-6 composition of RDX is shown in Fig.2-7.

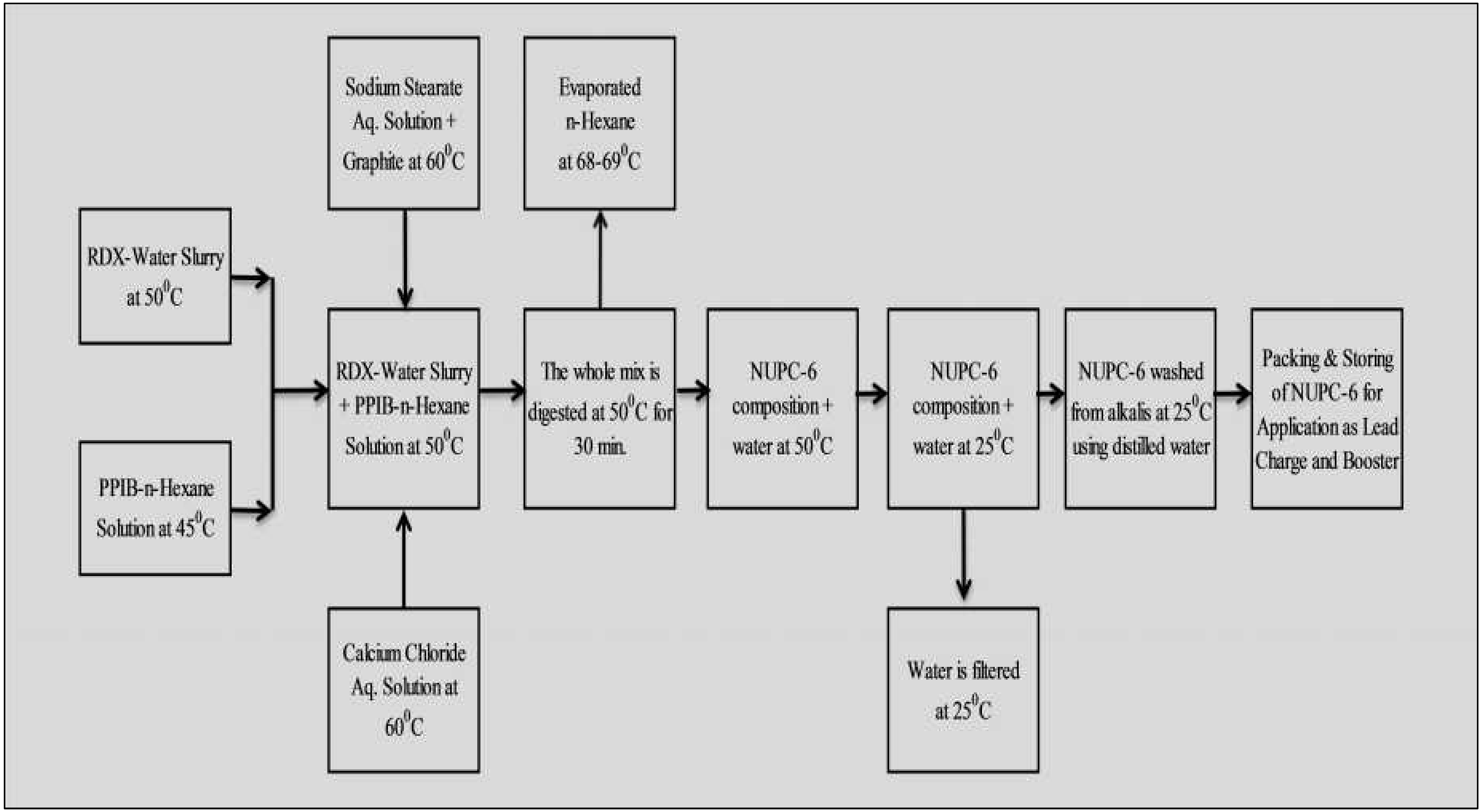


Fig.2-7 Process Flow Diagram of Method for Lab Scale Preparation of NUPC-6 Composition of RDX

Chapter 3

Experimental Techniques

3.1 Differential Scanning Calorimetry (DSC)

Calorimetry is used for measurement of thermal properties and thermal sensitivity of substances. It measures enthalpy of interest as well. DSC is a type of calorimetry. When temperature changes, DSC device measures heat liberated or absorbed by the sample. The size of sample is small of the size of a few milligrams. In this way, characteristics such as melting point, phase changes and decomposition temperature of sample are determined. Thermal sensitivity testing is also performed via differential scanning calorimetry. [70-72]

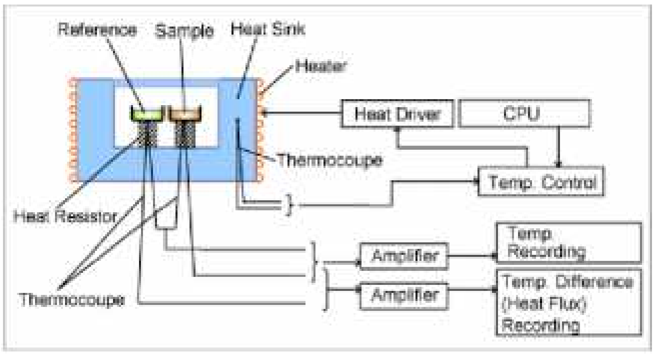


Figure 3-1 Differential Scanning Calorimetry

3.2 Scanning Electron Microscopy (SEM)

The distance which a normal human eye can resolve apart from each other, is called as the resolution power of the human eye. For resolution below 0.1–0.2 mm, magnification is needed. This can overcome limitation of human eye and for this purpose microscope was introduced having an arrangement for magnification of object as an efficient tool. It has a large range of magnification and is used for characterization of materials.

The power of the microscope depends upon quality of lenses, number of lenses and wavelength of the light source producing image. Microscope is classified in two categories, optical and electron microscope. Resolution power of electron microscope (EM) is much larger than the resolution power of optical microscope (OM). The accelerated electrons having short wavelength can help to study physical dimension at much smaller scale when used in EM cause diffraction effects. Types of electron microscope are transmission electron microscope (TEM) and scanning electron microscope (SEM). Schematic diagram of SEM is shown in Fig. 3-2. [73]

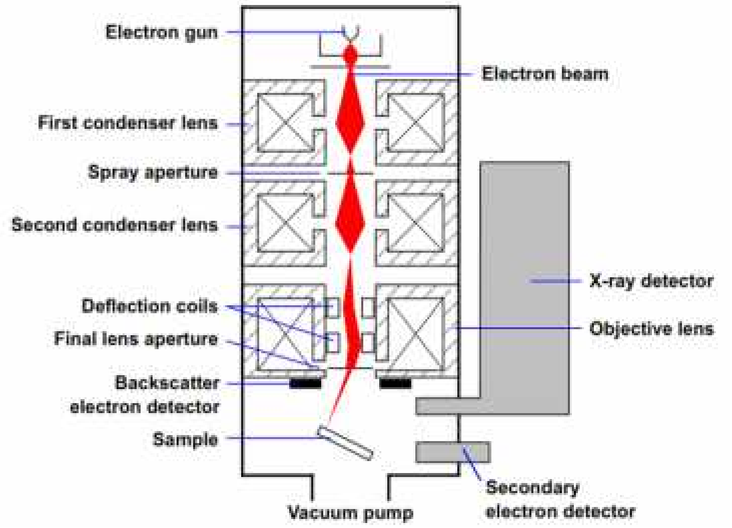


Fig.3-2 SEM

3.3 Sieve Analysis

Sieve analysis is used to find particle size range of a powder by allowing the powder to pass through a series of sieves with progressively smaller mesh size and weighing the mass fraction of material that is achieved at each sieve. The size distribution of powders is of importance. A sieve analysis can be performed on explosive powders with moderate to low sensitivity by using ex-proof analyser using non-ferrous and non-sparking sieves. The material is sieved in wet form if there is risk of high friction sensitivity due to vibration. Fig.3-3 shows a sieve analyser using non-sparking sieves and belt to tie up sieves with each other. [74]

Fig.3-3 Sieve Analyser

3.4 Fourier Transform Infrared (FTIR) Spectroscopy

FTIR spectroscopy uses IR rays which are passed through the sample. The rays received after absorbance of a particular part at receiver are analysed through spectrum analysis of these rays. The absorption and transmission indicates a fingerprint of the sample. This is a unique property of molecular structures. This property can be utilised for several types of analyses. IR spectroscopy is very common technique for the last seven decades or so in laboratories. A FTIR Spectrometer is shown in Fig.3-4. [75]

Fig.3-4 FTIR Spectrometer

Each compound has different combination of atoms having its own fingerprint in the form of IR spectrum. That's why IR spectroscopy can give positive identification of every kind of materials. FTIR spectroscopy is tool for qualitative and quantitative analysis. [76]

3.5 Refractive Index

Refractive index is commonly used to identify a particular substance, determine its purity or measure its concentration. It is dimensionless number and describes how fast light travels through the material. Fig.3-5 shows an automatic refractometer. [77]

Fig.3-5 An Automatic Refractometer

3.6 Visual and Microscopic Analysis

The visual and microscopic analysis means checking the sample visually and confirming the analysis with the help of microscope. It helps in assessing the degree of homogeneity of sample to be viewed. It helps in initial evaluation by the unaided eye and a low power metallurgical microscope. The physical characteristics that may be visible during the analysis include; homogeneity, colour, size, lumps, morphology etc. Fig.3-6 shows a metallurgical microscope.

Fig.3-6 A Metallurgical Microscope

3.7 Powder Flow Characteristics and Compressibility Index

Flow characteristics of powders mean the ability and rate of flow of powder composition in filling in explosive molds and conveyors when automatic filling of the material is performed. Whereas, the compressibility index is the estimated degree of compression possible for a material when automatic pelleting of sample material has to be achieved. Flow and compressibility measurement plays a critical role to design of modern explosive filling operations by pressing and melt casting processes. The powder flow characteristics and compressibility index are closely related with values of bulk density and tapped density. The bulk density of powder depends upon method of preparation, treatment and storage. The bulk density of a powder is often very difficult to measure. An apparatus for bulk density measurement of powders is shown in Fig.3-7. [78-79]

Fig.3-7 An Apparatus for Bulk Density Measurement of Powders

3.8 Composition Analysis

Composition analysis is analysis of percentage of each component present in the sample of composition. In this analysis, RDX is dissolved in hot acetone by dissolving the RDX present in sample in acetone, calcium stearate is dissolved in glacial acetic acid, polyisobutylene, diisooctyl sebacate and SAE-10 low viscosity oil are dissolved in the hot n-Hexane and the residue remaining is weighed for graphite present in the sample. This analysis completely defines the degree of mixing in samples collected from various locations of the lot and percent composition of each component in the samples. [80]

3.9 Sensitivity Tests

Sensitivity tests for impact friction electrostatic discharge and thermal sensitivity of explosives are discussed as follows; [81]

3.9.1 Impact Sensitivity Testing

In this testing a weights are dropped onto sample and the weight at which 50% of samples get detonated is determined in impact sensitivity test. This is known as Bruceton’s analysis. In this way impact sensitivity of explosive samples is determined. This testing may have weight of striker mass and height of striker mass as variable parameters. A schematic diagram of impact sensitivity testing is a follows; [81]

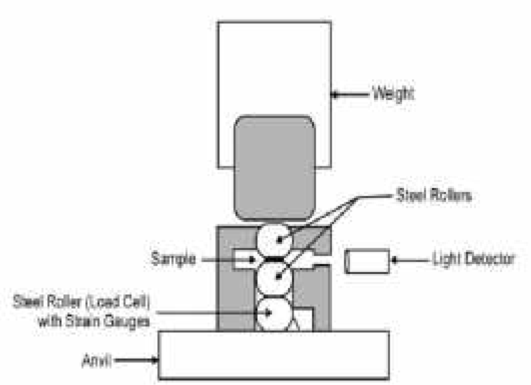


Fig.3-8 A Schematic Diagram of Impact Sensitivity Testing

3.9.2 Friction Sensitivity Testing

In friction sensitivity testing explosive sample between plate and wheel is squeezed by striking metal plate with pendulum. It is known as ABL friction sensitivity test. Using Bruceton’s analysis 50% initiation level of samples is determined. In BAM friction sensitivity test sample is placed on ceramic plate and is squeezed by using ceramic peg or pestle. A schematic diagram of friction sensitivity testing is as follows; [81]

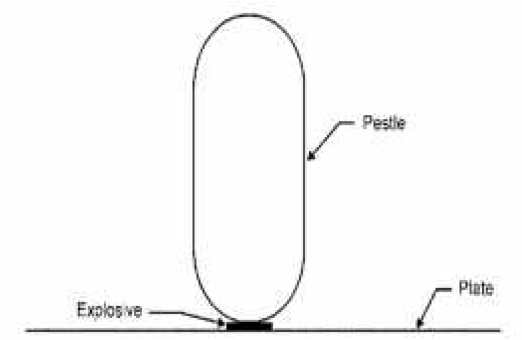


Fig.3-9 Friction Sensitivity Testing

3.9.3 Electrostatic Discharge Sensitivity Testing

Electrostatic discharge sensitivity testing measures the amount of energy discharged into through samples for 50% initiation level is determined using Bruceton's analysis. A schematic diagram of electrostatic discharge sensitivity testing is as follows; [81]

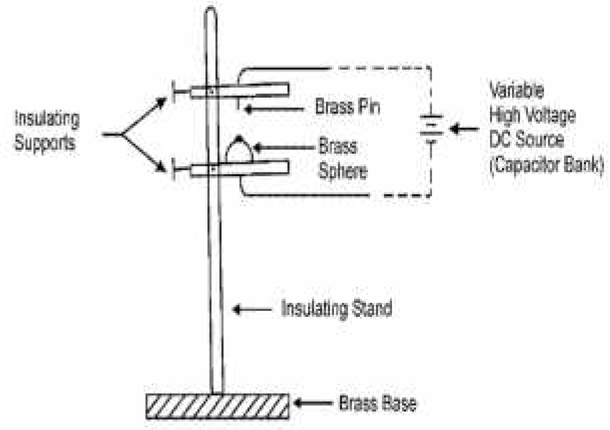


Figure 3-10 Electrostatic Discharge Sensitivity Testing

3.9.4 Thermal Sensitivity Test

Thermal sensitivity of explosive is the capability of explosive to detonate under thermal heating effect. It is determined for sample under confinement with thermal stress by immersing in a hot metal bath containing heating oil. The oil is heated with sample in it and the temperature and time to detonate of sample is measured. The heating is continued to higher unless the sample gets detonated. For this purpose high heating oil is used. A schematic diagram of thermal sensitivity testing is as follows; [81]

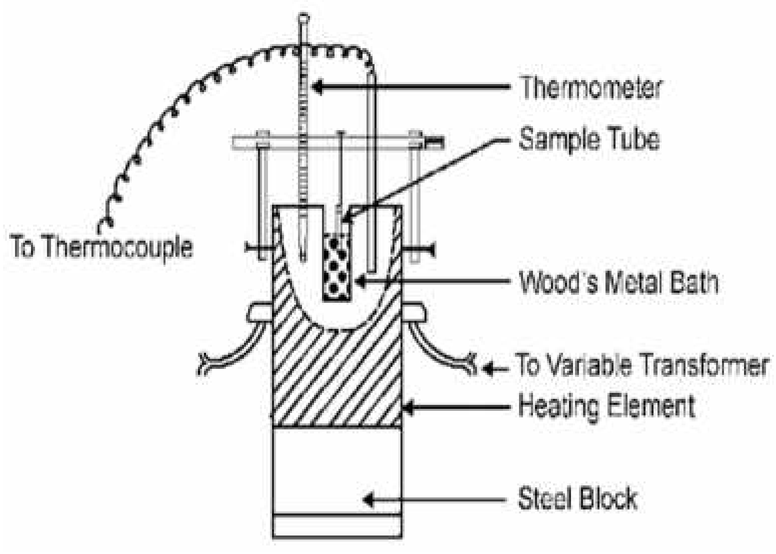


Fig.3-11 Thermal Sensitivity Testing

3.10 Velocity of Detonation (VOD) Measurement

Normally, explosives are characterised by strength, density and VOD. As the VOD is reduced the detonation pressure and available shock energy is reduced. VOD times density of explosive is called impedance of explosive which is another important parameter. A schematic diagram of detonation process is as follows;

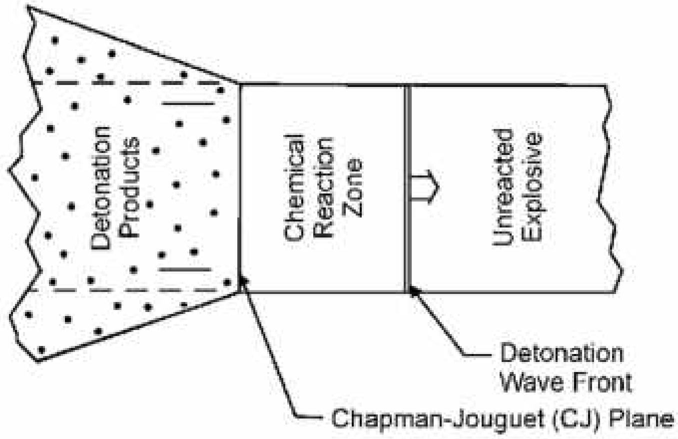


Fig.3-12 Detonation Process

The explosive velocities are higher than sound speed in the material. In case of confined explosive the force produced is massively intensified and focussed on much smaller area. This results in explosive velocity greater than if the explosive had been detonated in open air. Normally the VOD in open is 70-80% of the VOD in confined form. VOD is dependent on; particle dia, charge dia and degree of confinement. Normally, high explosives have VOD from 4000-10300 m/s. VOD data of explosives is given in confined and in open air. [82-83] A number of methods are used to measure velocity of detonation. These methods are as follows; [84]

- Dautriche Method
- Photographic Method
- Discrete Points (Point to point) Electric Method
- Resistance Wire Continuous VOD Method
- SLIFER Continuous VOD Method
- TDR (Time Domain Reflectometry) Continuous VOD Method
- Method Based on Optical Fiber

3.10.1 Method Based on Optical Fiber

In this method optical fiber is used for detecting and transmitting a light signal accompanied with the detonation wave front. The first cable signals the start clock whereas the second cable signals the stop clock. VOD is measured by fixed distance between sensor probes divided by timer clock. A schematic diagram of method based on optical fiber for VOD measurement is shown in Fig.3-13. [82-83]

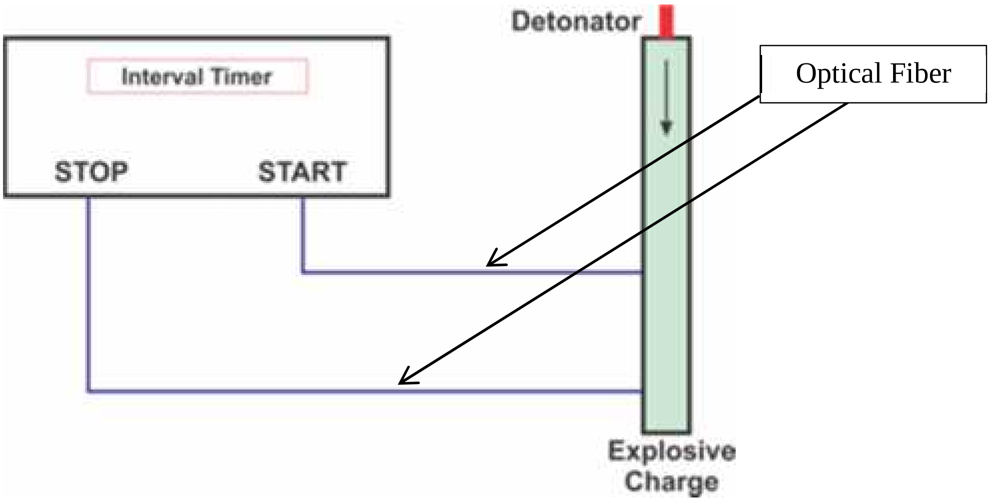


Fig.3-13 Schematic Diagram of Method Based on Optical Fiber for VOD

3.11 Skin Irritation Test

In this test, 500 mg of substance is applied to the skin of an animal. The observation of side reactions and signs of discolouration of skin are evaluated. [84-85]

Chapter 4

Results and Discussion

4.1 Results and Discussion of Characterization of Solvents

First of all different samples of xylene and n-hexane were used to dissolve PIB. The n-octane due to very high boiling point range was not used. It was found that the polyisobutylene was dissolved in almost the same time in both the solvents, whereas when evaporation was studied due to higher boiling point the xylene took more time to be evaporated then the low temperature boiling n-hexane. Moreover, xylene with very peculiar smell and high toxicity was very difficult to handle. To avoid the risk of exposing the RDX composition up to 111 ^{o}C which was boiling point of xylene and other issues associated with it, only n-Hexane was used to proceed further. The AR grades of toluene and n-hexane were used in experimental works having refractive index of 1.4802 and 1.3769 respectively. These values were measured by refractometer at 20^{0}C.

4.2 Results and Discussion of Characterization of Binder

The sample of PIB was analyzed by using FTIR to identify by functional group library of polyisobutylene from 500 to 3500 cm-1. It was needed to modify properties of PIB by hit and trial method. Tackiness of polyisobutylene was not good as was seen during experimental work on preparing NUPC-6 composition. It was therefore, modified by introducing DIOS which enhanced its polarity due to dipole-dipole interactions through external plasticization. It got wetting capability due to these forces and hence instead of instant solidification and causing composition to be fluidized the mixing and coating of the polyisobutylene was very much improved. Other ingredient which was needed to be added to the composition of PIB and DIOS was low viscosity engine oil of SAE-10 grade. This oil decreased the viscosity of the composition of PIB and DIOS dissolved in n-hexane and ensured homogeneous distribution of the binder on the face of RDX grains. The amounts of DIOS and SAE-10 oil were finalized after hit and trial method by preparing composition and checking it visually during manufacturing and after manufacture through microscopic and percent composition analysis. These analyses confirm the degree of coating of RDX grains and homogeneous mixing of binder in the composition. The percent addition of DIOS and SAE-10 oil in PIB needed to modify it for application in developing RDX based insensitive composition was 0.5244 and 0.1422 respectively. Fig.4-1 is of standard PIB [86] and Fig.4-2 is of sample PIB used which is same as that of standard PIB. This was confirmed by methyl and methylene group peaks.

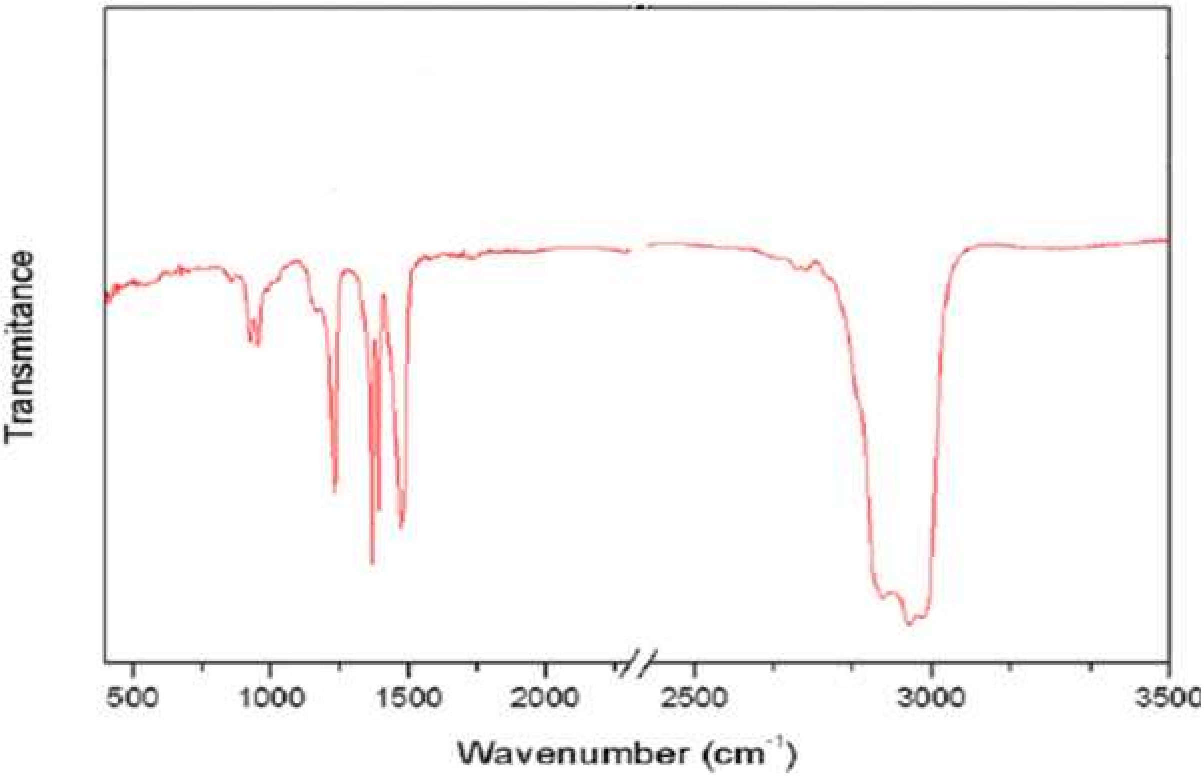


Fig.4-1 FTIR spectrum of Standard PIB

Fig.4-2 FTIR spectrum of PIB Sample by using Perkin Elmer Spectrum 100

4.3 Results and Discussion of Characterization of RDX

RDX of Thermal stability and melting point of RDX was studied by DSC. The DSC of sample was performed using Mettler Toldo DSC with open pan. The DSC curve of the sample used was completely similar to the DSC curve of standard sample of RDX except melting point of pure RDX. The DSC curve of pure RDX is shown in Fig.4-3[87] at a heating rate of 10.0 °C/min. This shows the melting starts at 204°C, whereas the melting point of used sample was 204.38°C to 204.49°C from the DSC curves shown in Fig.4-4 and Fig.4-5. Moreover, single vivid peak ensures that the used RDX is highly purified having negligible HMX contents. It also ensures that the RDX is thermally stable and can be used to proceed further for research work and experimental work.

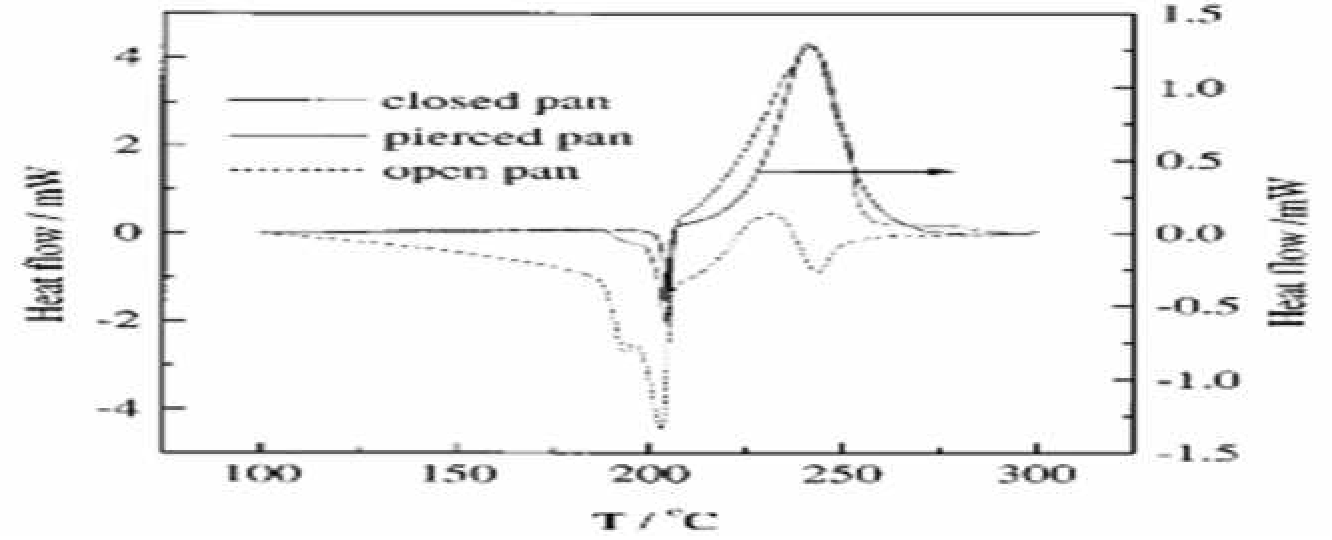


Fig.4-3 DSC curves for RDX decomposition in open, pierced and closed pans

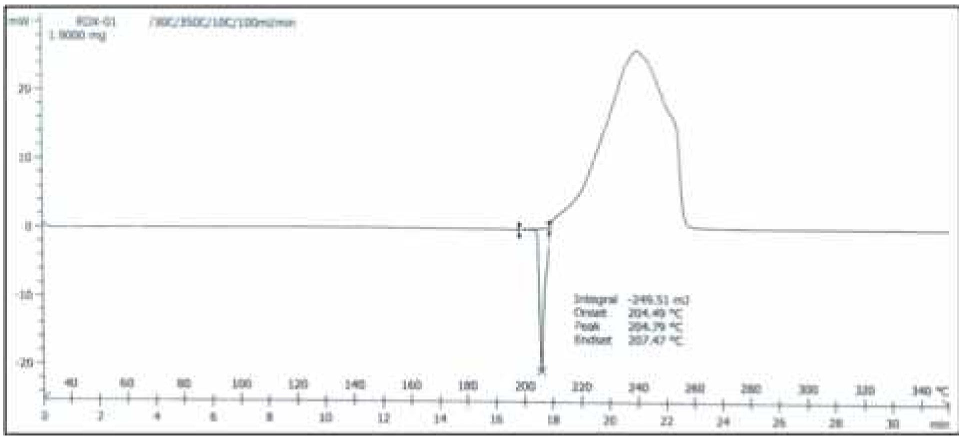


Fig.4-4 DSC Thermograph of Sample 1 of RDX in open pan

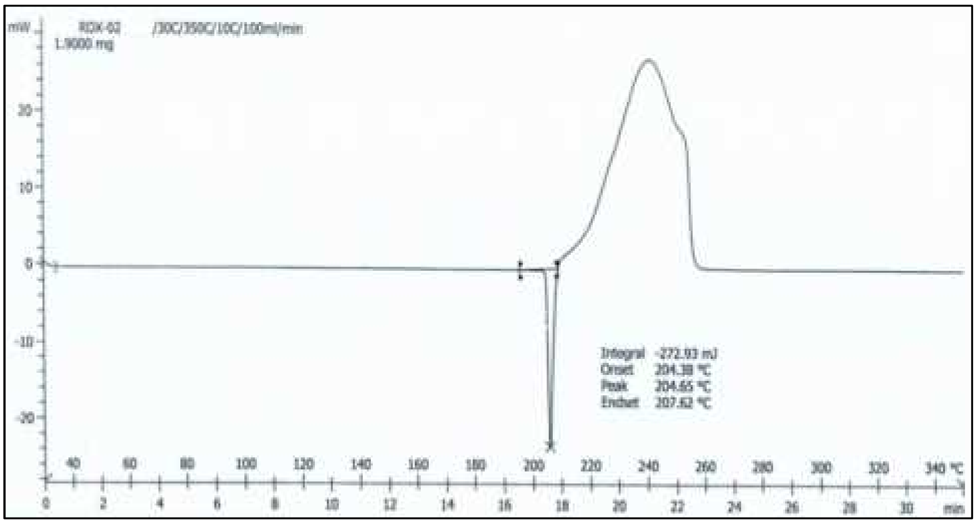


Fig.4-5 DSC Thermograph of Sample 2 of RDX in open pan

The sieve analysis and volatile matter analysis of finely recrystallized and purified RDX was performed for gradation of RDX by using Meinzer II sieve shaker and hot air circulated drying oven indirectly heated through hot water for best prepared recipe which is completely and homogenously mixed using the setup as defined. The sieve analysis of optimized size range RDX is given in Table 4-1. As the used grade of RDX was fine recrystallized grade which was needed for conducting research on the development of RDX based insensitive composition. The finer the RDX the easier is to control the particle size and greater shall be uniformity of the product composition. It reduces sensitivity of the composition as well. A lot of experimental work was done to reach a result of -100 to +150 mesh size required for developing homogeneously mixed composition of RDX. This experimental work was gradation of -325 mesh RDX. The following table shows that the size distribution of RDX samples.

Table-4-1 Sieve Analysis of Samples of RDX

Sample	% V.M	% Sieving	
		Retained 100 BSS	Retained 150 BSS
RDX-1	11.95	0.88 max.	95.41 min.
RDX-2	11.85	0.85 max.	95.50 min.

The RDX grains were visually and microscopically viewed to check the size range by using metallurgical Olympus Meiji microscope. The Figure 4-6 indicates the microscopic picture of RDX grains from Olympus Meiji Metallurgical Microscope.

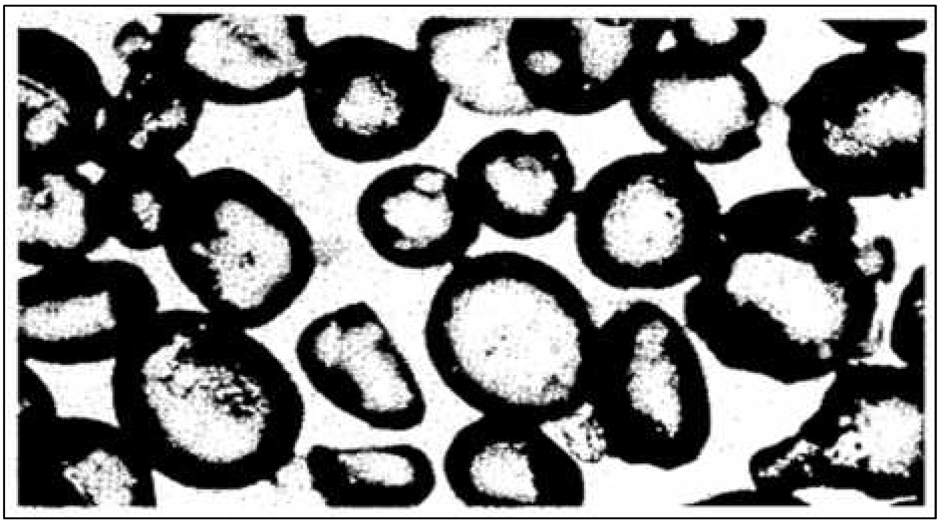


Fig.4-6 Picture of RDX grains from Olympus Meiji Metallurgical Microscope

SEM of RDX was carried out to study morphology of RDX grains. It showed RDX grains are octahedral. The SEM of RDX grain is shown in Fig.4-7.

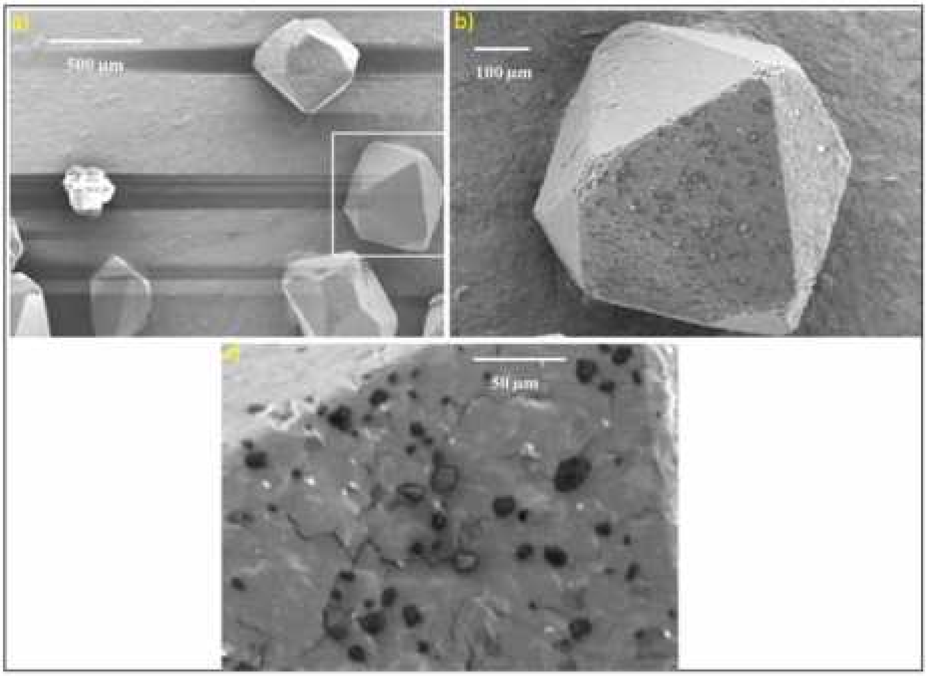


Fig.4-7 SEM of RDX Grain Sample at (a) 50× magnification, (b) 150× magnification, and (c) 500× magnification (working distance: 40 mm; voltage: 5 kV)

4.4 Results and Discussion of Characterization of Calcium Stearate

During the method development for introducing by physical and chemical methods it was found that due to very fine it was very difficult to suspend calcium stearate in the slurry form. That’s why it was not able to play its role as dispersant as it was not homogeneously distributed in the emulsion. During chemical method for introducing calcium stearate on the face of RDX grains, it was found that calcium stearate was homogenously mixed in the emulsion and it played its role as dispersant to control size of RDX grains. It was possible due to access of calcium stearate to each RDX grain suspended in the slurry phase in emulsion.

In experiments to study kinetics of the reaction between aqueous solution of sodium stearate and calcium chloride precipitates of calcium stearate obtained were filtered and dried on whatman filter papers. It was found that % conversion of reaction to calcium stearate was 98.6%. The filtrate was washed thoroughly to neutral pH before drying. The resultant product after drying was checked for purity by performing DSC, the DSC curve showed consistent result regarding purity of the calcium stearate as shown by Fig.4-8. The results of distribution of calcium stearate in the developed composition were checked through visual and microscopic analysis for homogeneous distribution a well.

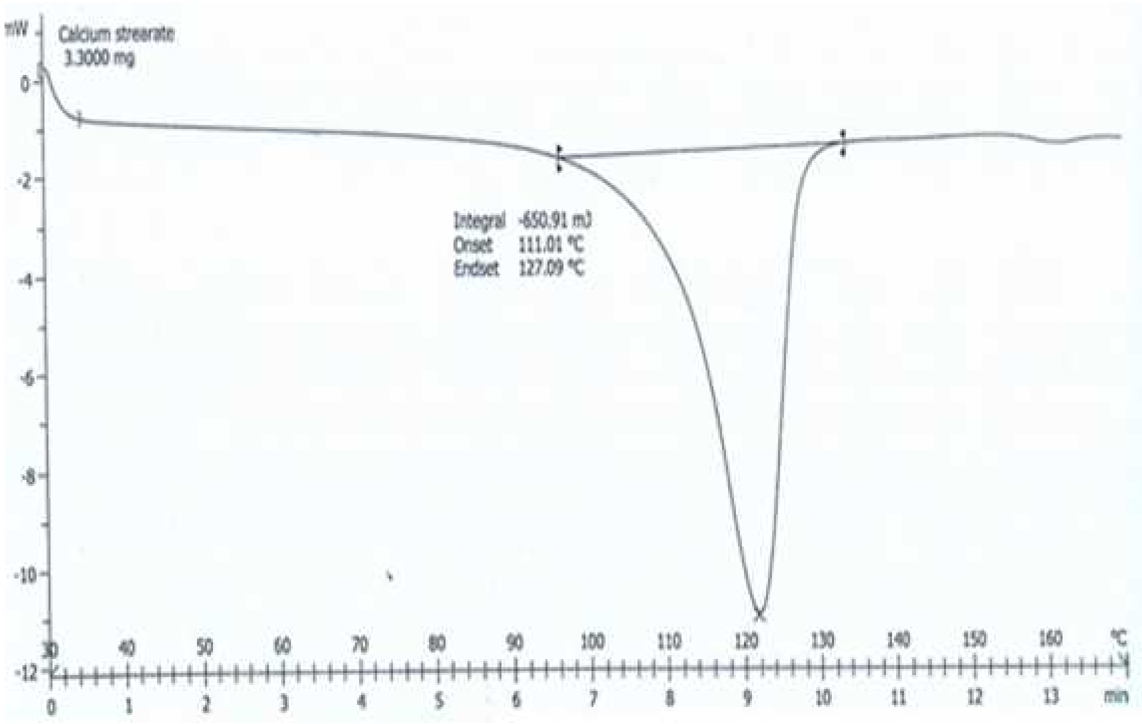


Fig.4-8 DSC Thermograph of Sample of Stearate

RDX of Thermal stability and melting point of RDX was studied by DSC. The DSC of sample was performed using Mettler Toldo DSC with open pan. The DSC curve of the sample used was completely similar to the DSC curve of standard sample of RDX except melting point of pure RDX. The DSC curve of pure RDX is shown in Fig.4-3[87] at a heating rate of 10.0 °C/min. This shows the melting starts at 204^{o}C, whereas the melting point of used sample was 204.38^{o}C to 204.49^{o}C from the DSC curves shown in Fig.4-4 and Fig.4-5. Moreover, single vivid peak ensures that the used RDX is highly purified having negligible HMX contents. It also ensures that the RDX is thermally stable and can be used to proceed further for research work and experimental work.

4.5 Results and Discussion of Characterization of Graphite

Mixing of graphite was achieved in a stage when it was suspended in aqueous solution with sodium stearate. The solution was heated and introduced in to the suspension of RDX, matrix PIB and distilled water. The moment graphite comes into contact with rains of RDX, the colour of whole mass suspend to change from white to light grey. The graphite got coated on to the face of grains of RDX homogenously. This process goes to completion and the whole of graphite gets converted in the form of layered covering on the RDX. This makes the whole composition independent of the size of particles of graphite powder.

In experiments to perform selection of solvent, development of binder for required application, gradation of RDX and selection of the right size range to develop required composition, research on reaction kinetics between aqueous solution of sodium stearate and calcium chloride to yield calcium stearate and selection of chemical method for introducing calcium stearate in the required composition and the method of introducing graphite into the required composition, a new high explosive composition was developed. This composition is named as NUPC-6 composition of RDX. By the way of developing this composition, a complete methodology for its preparation on laboratory scale was also developed. Moreover, a detailed design work on the basis of process developed is also performed to adopt it for production on pilot scale.

4.6 Results and Discussion of Characterization of NUPC -6

The successfully developed composition NUPC-6 through characterized materials and methods was needed to be further characterized for use as lead and booster charges. For the use of NUPC-6, it was characterized by visual and microscopic analysis and percent composition analysis. A homogenously mixed and coated sample of developed composition is shown in Fig.4-9. The results of these analyses are tabulated in Table 4-2.

Fig.4-9 Picture of Sample of NUPC-6 taken from Sony Camera

Table 4-2 Composition Analysis Results of NUPC-6 Samples

Sr. No.	Ingredient	% (w/w)
1.	RDX	97.5±0.5
2.	Graphite	0.5±0.01
3.	PIB	0.1667±0.0001
4.	DIOS	0.2622±0.0002
5.	SAE-10 Oil	0.0711±0.0001
6.	Calcium Stearate	1.5±0.01

Characterization of NUPC-6 composition was performed to assess a measurement of its capability to flow in automatic filling operations and pressing operations. This was achieved by measuring flow rate and compressibility of NUPC-6 in self-developed experimental setups. Rate of flow for RDX and NUPC-6 was determined using apparatus consisting of essentially a miniature hopper and receiving bin. Dimensions of the hopper body were 83.5 mm and 250 mm for diameter and length respectively. The diameter of discharge hole at the base of the hopper was 17.7 mm. Flow times were recorded for discharge of same mass of each sample through timer. A summary of results is given in Table 4-3. The results showed that NUPC-6 has good flow characteristics and can be used easily in the automatic pelleting process. The error in mass flow rate was ±1.0776 gm/sec.

Table 4-3 Flow Rate Determination of RDX and NUPC-6

Sr. No.	Parameters of Sample	RDX	NUPC-6
1.	Mass	200 gm	200 gm
2.	Time of Flow	3 sec	5 sec
3.	Mass Flow Rate	66.67 gm/sec	40 gm/sec

The bulk density measurement setup consisted of a funnel, petri dish, scale and divider. The bulk density of the NUPC-6 was measured by a freely dropping the sample on to the petri dish. The height of sample heap was measured using scale. Dia of heap was determined using divider. The mass of sample dropped was known. By using mathematical relation for volume of cone, volume of heap was determined. Then by dividing mass of heap with volume of the heap, bulk density of the sample was determined. The results are tabulated in Table 4-4. By knowing value of bulk density of powder, filling mold is easily designed for its automatic filling operation process. The error in bulk density was ±0.1 gm/cm^3.

Table 4-4 Comparative Results of Bulk Density of RDX and NUPC-6

Sr. No.	Parameters of Sample	RDX	NUPC-6
1.	Mass	200 gm	200 gm
2.	Height of Heap	9.8 cm	10.74 cm
3.	Dia of Heap	10.87 cm	11.93 cm

4.	Bulk Volume of Heap	303 cm^3	400 cm^3
5.	Bulk Density of Heap	0.66 gm/cm^3	0.5 gm/cm^3

Similarly tapped density of samples of RDX and NUPC-6 was calculated by equally tapping the samples in cylinders of known volume. The samples were tapped by dropping from 10 mm height and the process was repeated 100 times. The finally achieved volume by tapped samples was recorded. This is known as tapped volume of sample tapped. By dividing masses of the samples tapped with its tapped volumes, their tapped densities were determined. The results are tabulated in Table 4-5

Table 4-5 Comparative Results of Tapped Density of RDX and NUPC-6

Sr. No.	**Parameters of Sample**	**RDX**	**NUPC-6**
1.	Mass	20 gm	20 gm
2.	Tapped Volume of sample	25 cm^3	33 cm^3
3.	Tapped Density of Heap	0.80 gm/cm^3	0.61 gm/cm^3

By knowing value of tapped density of powder, pressing mold is easily designed for its automatic pressing operation process. The error in tapped density was ±0.08 gm/cm^3.Compressibility indexes and Hausner’s ratios give measures of compressibility and flow property of the samples. The results are tabulated in the Table 4-6.

Table 4-6 Comparative Compressibility and Flow Property of RDX and NUPC-6

Sr. No.	Sample	Bulk Density gm/cm^3	Tapped Density gm/cm3	Compressibility Index	Hausner’s Ratio	Flow Property
1.	RDX	0.66	0.80	17.5	1.21	Fair
2.	NUPC-6	0.50	0.61	18.0	1.22	Fair

The performance of NUPC-6 composition was evaluated by using fiber optical method for measurement of VOD. The samples of Tetryl and NUPC-6 were evaluated for VOD. The specifications of samples and their respective VOD’s are tabulated in the

Table 4-7. The error in VOD was ± 10 m/s. As the VOD of NUPC-6 is greater than the VOD of Tetryl, therefore, output energy of NUPC-6 and pressure of detonation is also greater than the output energy and pressure of detonation of the Tetryl. This result shows that application of NUPC-6 as lead and booster applications is superior to Tetryl.

Table 4-7 Comparative Results of VOD of Tetryl and NUPC-6

Sr. No.	Parameters of Sample	Tetryl	NUPC-6
1.	Mass	18 gm	18 gm
2.	Dia of Pellet	2.98 cm	2.98 cm
3.	Height of Pellet	1.6 cm	1.6 cm
4.	Volume of Pellet	11.18 cm^3	11.18 cm^3
5.	Loaded Density	1.61 gm/cm^3	1.61 gm/cm^3
6.	Loading Pressure	20000 Psi	20000 Psi
7.	VOD	7570 m/s	8290 m/s

Sensitivity and skin toxicity tests were performed to characterize handling and storage requirements from point of application of developed composition as military explosive. The comparative results of the tests performed on the samples of Tetryl and NUPC-6 sample composition are tabulated in Table 4-8.

Table 4-8 Comparative Results of Sensitivity of Tetryl and NUPC-6

Sr. No.	Test	Parameters	Tetryl	NUPC-6
1.	Impact Sensitivity	For 50% initiation of sample height of 3 Kg weight	27 cm	26 cm
2.	Friction Sensitivity	The tester has nine different positions on the arm 0.5 to 36 kg	60 N	100 N

3.	Electrostatic Sensitivity	Samples were exposed to static charge through capacitor	2.321 J	2.543 J
4.	Thermal Sensitivity	Samples were heated in bomb calorimeter	115-160 °C	196-207 °C
5.	Skin Toxicity Test	Causes yellowing of skin?	Yes	No

These results show that NUPC-6 has greater thermal stability, lesser impact sensitivity, lesser friction sensitivity, lesser electrostatic charge sensitivity and lesser toxicity than the Tetryl. This shows that NUPC-6 can be perfectly used to replace Tetryl in military applications as per requirements of military standards.

Chapter 5

Design for Scale Up

For manufacture of NUPC-6 composition of RDX scale up design has been given in detail in this chapter. This scale up design is given in following sequence;

- Material balance
- Energy balance
- Equipment design

The design work has been given for manufacture of 7 Kg of NUPC-6 composition of RDX on pilot scale level. The detailed design calculations are given in appendices.

5.1 Material Balance

The pilot scale production is based on 7 Kg batch of explosive composition NUPC-6. Material balance is carried out as follows;

Basis of Calculation: 7000 gm of NUPC-6

Table 5-1 Composition of NUPC-6

Sr. No.	Ingredient	% (w/w)
1.	RDX	97.5±0.5
2.	Graphite	0.5±0.01
3.	PIB	0.1667±0.0001
4.	DIOS	0.2622±0.0002
5.	SAE-10 Oil	0.0711±0.0001
6.	Calcium Stearate	1.5±0.01

The raw materials required for 7000 gm of NUPC-6 are;

i. Moisture free RDX = 7000 X .975 = 6825.000 gm

ii. Graphite = 7000 X 0.005 = 35.000 gm

iii. PIB = 7000 X 0.005 = 11.669 gm

iv. DIOS = 7000 X 0.2622 = 18.354 gm

v. SAE-10 Oil = 7000 X 0.0711 = 4.977 gm

vi. Calcium Stearate = 7000 X 0.015 = 105.000 gm

5.1.1 Chemical Reaction of Sodium Stearate and Calcium Chloride

Calcium stearate is manufactured as a chemical reaction b/w sodium stearate and calcium chloride with 98.5 % conversion [88]. The reaction equation is;

$$\begin{array}{ccccccc} C_{18}H_{35}COONa_{\,(aq)} & + & CaCl_{2\,(aq)} & \rightarrow & (C_{18}H_{35}COO)_2Ca & + & 2NaCl_{\,(aq)} \\ 2\text{ mol} & & 1\text{ mol} & & 1\text{ mol} & & 2\text{ mol} \end{array}$$

Formula weight of calcium stearate is 607.017 gm/mol. Hence no. of moles of calcium stearate required in composition is 105/607.017 or 0.173 moles. Calcium chloride and sodium stearate required for 0.173 moles of calcium stearate 0.173 moles and 2x 0.173 moles or 0.346 moles respectively. As the reaction conversion is 98.5% therefore amounts of calcium chloride and sodium stearate is to be supplied in the excess is 0.173/0.985 or 0.1756 moles and 2x0.1756 moles or 0.3512 moles respectively.

vii. Formula weight of $CaCl_2$ is 110.984 gm /mol; hence the $CaCl_2$ supplied for reaction is 0.1756x10.984 or 19.5 gm.

viii. Formula weight of sodium stearate is 306.459 gm/mol; hence the sodium stearate supplied is for reaction is 0.3512x306.459 or 107.63 gm.

ix. Weight of $CaCl_2$ unconverted and remaining unreacted is (0.1756-0.173) x 110.984 or 0.289 gm.

x. Similarly unreacted sodium stearate is (0.3512-0.346) x306.459 or 1.6 gm.

xi. Formula weight of NaCl is 58.443 gm / ml, No. of moles of NaCl produced are 0.173 X 2 or 0.346 moles; hence sodium chloride produced is 0.346x 58.443 or 20.2212 gm.

xii. Specific gravity of n-hexane is 0.660, 1 gm of plasticized polyisobutylene is 35x50 or 1750 ml, specific gravity of plasticized polyisobutylene is 0.92, volume of plasticized polyisobutylene is 35/0.92 or 38.04 ml, volume of mixing vessel required for plasticized polyisobutylene solution is 1750+38.04 or 1788.04 ml; hence mass of n-hexane used is 0.66 X 1788.04 or 1155 gm.

xiii. Density of water at 50^0C is 1 gm /ml, 10% RDX slurry; hence water in 10 % RDX slurry is 90 % which is (90/10) X 6825 or 61425 gm.

xiv. Mass of water in 13 % sodium stearate aqueous solution is 87 % which is (87/13) x 107.63 or 720.293 gm.

xv. Mass of water in 20 % $CaCl_2$ aqueous solution is 80 % which is (80/20) x 19.5 or 78 gm.

Material balance of n-Hexane distillation and filtration steps of recipe from water for scale up production of 7 Kg of explosive composition NUPC-6 has been given only in Fig.5-1 and Fig.5-2 respectively. Detailed mass balance calculations are given in Appendix-A.

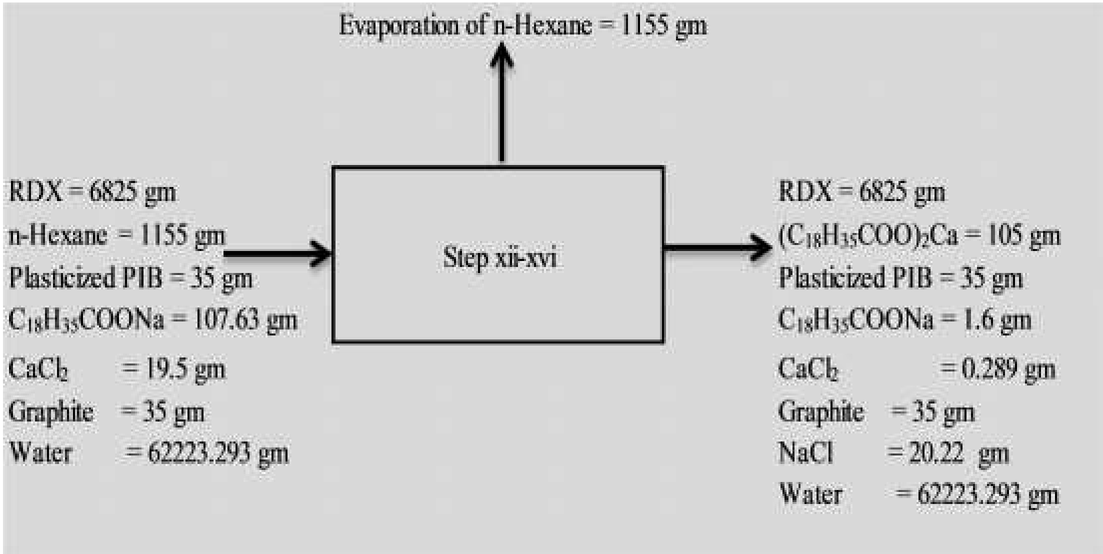


Fig.5-1 Mass Balance of n-Hexane Distillation Step

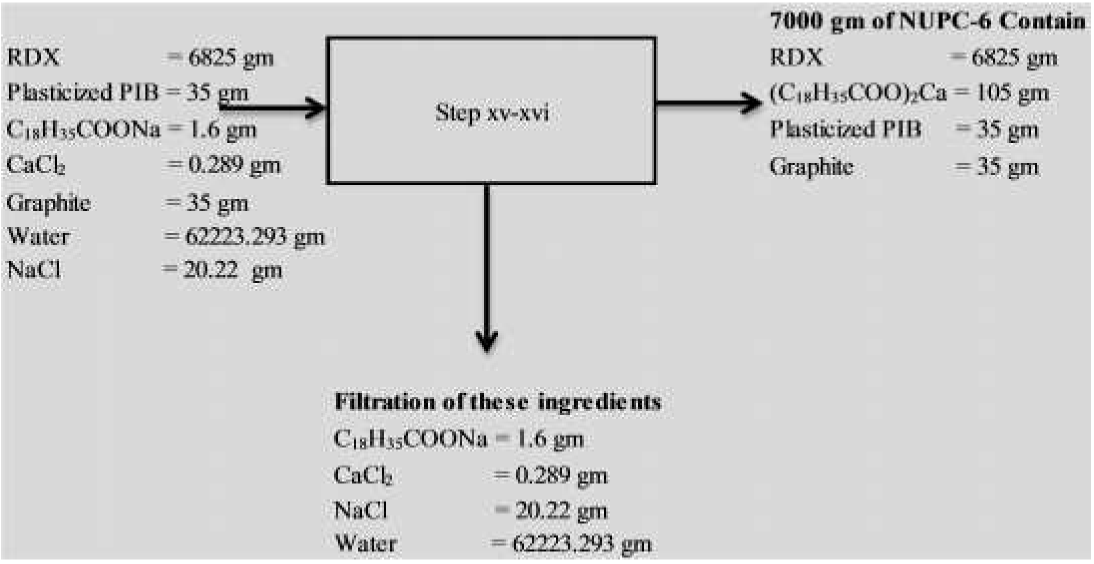


Fig.5-2 Mass Balance of Recipe Filtration Step

5.2 Energy Balance

Energy balance of cooling and filtration steps of recipe from water for production of 7 Kg of explosive composition NUPC-6 is given. Detailed energy balance calculations are given in Appendix-B.

Basis of Calculation: 7000 gm of NUPC-6

Energy balance Cooling and Filtration Steps of Recipe;

Mass of RDX, M_{RDX} = 6825 gm

Mass of PIB, M_{PIB} = 11.669 gm

Mass of DIOS, M_{DIOS} = 18.354 gm

Mass of SAE-10 Oil, M_{SAE-10} = 4.977 gm

Mass of graphite blended, M_G = 35 gm

Mass of Calcium Stearate produced, M_{CSP} = 105 gm

Mass of Sodium Stearate unreacted, M_{SSU} = 1.6 gm

Mass of calcium chloride unreacted, M_{CCU} = 0.289 gm

Mass of NaCl produced, M_{SCP} = 20.2212 gm

Mass of distilled water, M_W = 62223.293 gm

Total mass of remaining whole contents, M_{RWC} = 69245.40 gm

Average specific heat capacity of RDX, C_{RDX} = 1.1095 J/gm^{0}C

Average specific heat capacity of PIB, C_{PIB} = 2 J/gm^{0}C

Average specific heat capacity of DIOS, C_{DIOS} = 0.0441 J/gm^{0}C

Average specific heat capacity of SAE-10 Oil, C_{SAE-10} = 1.964 J/gm^{0}C

Average specific heat capacity of graphite blended, C_G = 0.7076 J/gm^{0}C

Average specific heat capacity of calcium stearate, C_{CS} = 2.33 J/gm^{0}C

Average specific heat capacity of sodium stearate, C_{SS} = 2.4 J/gm^{0}C

Average specific heat capacity of calcium chloride, C_{CC} = 0.657 J/gm^{0}C

Average specific heat capacity of NaCl, C_{NaCl} = 0.864 J/gm^{0}C

Average specific heat capacity of distilled water, C_w = 4.000 J/gm^{0}C

Wt. Avg. specific heat capacity of rest of whole contents, C_{RWC} = 3.71 J/gm^{0}C

Initial temperature of emulsion whole contents, T_1 = 70 ^{0}C

Final temperature of emulsion whole contents, T_2 = 25 ^{0}C

Temperature change of remaining whole contents, ΔT_{EWC} = 45 ^{0}C

Heat required to be removed to 25 ^{0}C, $Q_{11}=M_{RWC}XC_{RWC}X\Delta T_{RWC}$ = 3.21 kWH

5.3 Equipment Design

Equipment design based on RDX based explosive composition NUPC-6 has been described here. The pilot scale production plant is based on 7 Kg batch of explosive composition NUPC-6. The equipment design of pilot plant for process developed for composition is described.

õ Calculations of % Slurry Contents and Slurry Density

Graphite = 35 gm

Calcium Stearate = 105 gm

Total mass of solid phase components in emulsion, M_s = 6965 gm

SAE-10 Oil = 4.977 gm

PIB = 11.669 gm

Na-St = 1.6 gm

$CaCl_2$ = 0.289 gm

NaCl = 20.2212 gm

H_2O = 62223.293 gm

n-Hexane = 1155 gm

Total mass of liquid phase components in emulsion, M_L = 63435.4032 gm

% Slurry or % Solid Contents in Slurry, B = 100 x (M_S/M_L) = 11 %

õ Calculations of Slurry Density

Component	Mass (gm)	Density ρ (Kg/m^3)	Weighted Density, ρ_w (gm/cm^3)
RDX	6825	1.796	0.17411406
Graphite	35	2.25	0.001118602

Ca-St	105	1.08	0.001610786
DIOS	18.354	.92	0.000239852
SAE-10 Oil	4.977	0.88	6.22121E-05
PIB	11.669	0.92	0.000152492
Na-St	1.6	1.02	2.31817E-05
$CaCl_2$	0.289	2.15	8.82594E-06
NaCl	20.2212	2.165	0.000621856
H_2O	62223.293	0.997	0.881196986
n-Hexane	1155	0.66	0.010828063
Density of emulsion whole contents, ρ_S (Kg/m^3)			1069.976916

õ Volume of Emulsion Whole Contents = 0.065796189 m^3

= 65.796 litres

5.3.1 Design of Jacketed Mixing Vessel System

Basis for design of jacketed mixing vessel system is taken as 68 litres of volume of whole emulsion contents and 3.5 kWh heat load on jacket side.

Design of mechanically stirred vessel for heating the slurry of RDX, precipitation reaction of the sodium stearate solution with the calcium chloride solution and for sodium stearate solution with the calcium chloride solution and for distilling the n-hexane from the suspension has been carried out. The total capacity required for all the contents in the recipe production is about 68 litres.

Diameter of impeller, D_a = 152.4 mm

Diameter of vessel, $D = 3 X D_a$ = 457.2 mm

Height of vessel, H is $3 X D_a$ = 457.2 mm

Volume of vessel = V = $\left(\frac{\pi D^2}{4}\right) \times H$ = $75021945\ mm^3$

= 75.021945 Litres

One third of volume of mixing vessel should be left empty in order to compensate the upward movement of vessel contents, therefore the volume of vessel for design should be; = 75.021945 +1/3 (75.021945) ~ 100.0 Liters. [89]

Height of vessel for final design is = $\frac{[illegible]}{(\pi D^2)}$ 609.42 mm = 24 inch

Impeller diameter = 6.00 inch = 152.40 mm = 0.15240 m

Vessel diameter = 18.00 inch = 457.20 mm = 0.45720 m

Vessel height = 2400 inch = 609.42 mm = 0.60942 m

Vessel volume = 6102 $inch^3$ = 100.0 Liters = $0.10000\ m^3$

5.3.2 Selection of Flow Pattern and Impeller

For scale up of composition pouring of n-hexane polyisobutylene solution in RDX slurry with water, evaporation of n-hexane occurred leaving polyisobutylene which solidify being coated on RDX particles. At this very moment the bunch of RDX particles gathers around the polyisobutylene and the downward circulation of these particles is very crucial for proper mixing. If a few bunches of particles of RDX coated with polyisobutylene set together to coalesce, in spite of impeller movement the particle are not able to be separated. Therefore, to overcome this phenomenon of coalescence of granules of RDX particles coated with polyisobutylene, position of impeller is needed to be off-centre and more importantly the impeller is positioned at this point at an angle which causes the movement of granules of RDX particles coated with polyisobutylene in downward and axial flow manner. Pouring of n-hexane-polyisobutylene solution on RDX slurry is consistently needed at impeller centre to control the size of granules.

A basic classification of impellers is into those that circulate the liquid axially and those that achieve primarily radial circulation. With commercially available motors and speed reducers, standard speeds are 37, 45, 56, 68, 84, 100, 125, 155, 190, and 320 RPM. For off-bottom suspension of solids, an impeller location of 1/3rd the impeller diameter off the bottom is satisfactory. A rotating impeller in a fluid imparts flow and shear to it, the shear resulting from the flow of one portion of the fluid past another. Limiting case of flow is in the axial or radial directions so that impellers are classified conveniently according to which of these flows is dominant. The three-bladed mixing propeller is modelled on the marine propeller but has a pitch selected for maximum turbulence. They are used at relatively high speeds up to 1800 RPM with low viscosity fluids, up to 4000 cP. With axial flow impellers, an angular off-centre position may be used. The impeller is mounted approximately 15-17^0 from the vertical, as shown in Fig. 8.1. For tanks less than 1.8 m in diameter, the camp or flanged mounted angular, off-centre axial-flow impeller without baffles should be used for a wide range of process requirements (refer to Fig.5-3). The volume of liquid should not exceed 4 m^3. Marine propeller (as shown in Fig.5-4) is selected. [90]

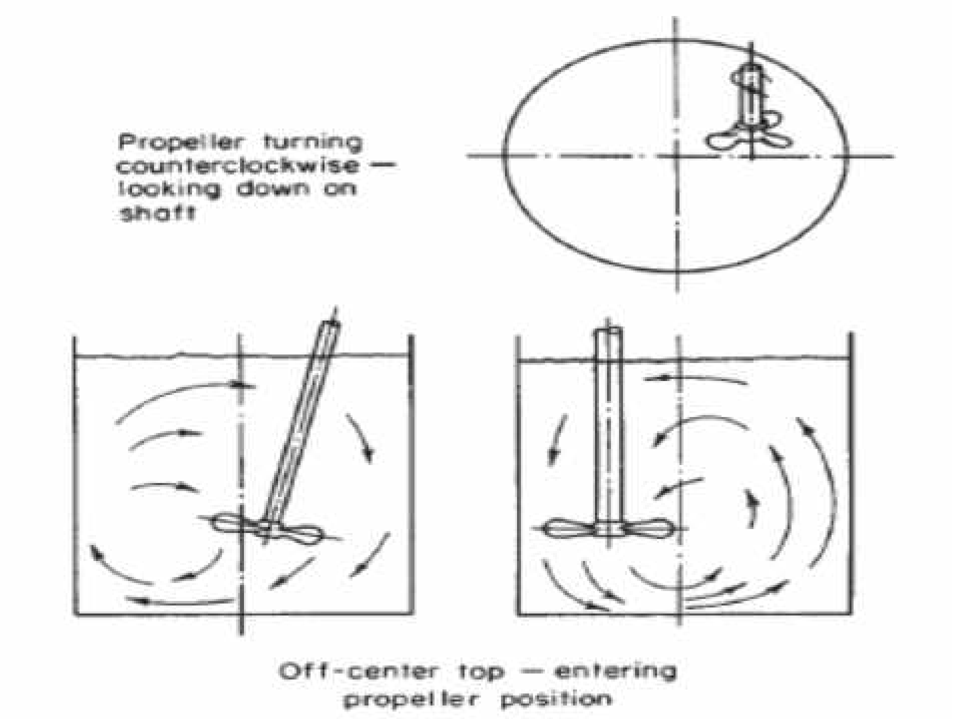


Fig.5-3 Flow Dynamics of Angular Off-Centre Position Propeller without Baffles

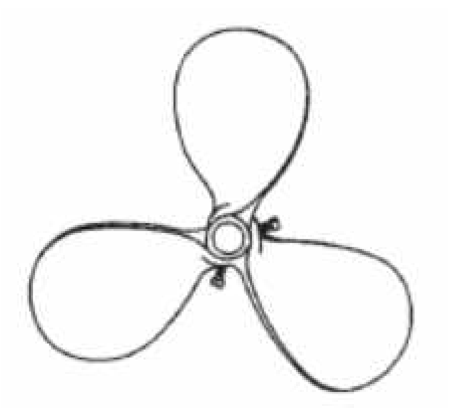


Fig.5-4 Marine Propeller [91]

5.3.3 Critical Speed of Impeller (N_C)

Zwietering's correlation [91] which is based on data for five types of impellers in six tanks from 6 inch to 2 feet diameter has been used as required for complete suspension of composition. The critical stirrer speed is given by the dimensionless equation;

$$N_c \text{ x } D_a^{0.85} = S \text{ X } v^{0.1} \text{ x } Dp^{0.2} \text{ x } g \text{ x } (\Delta\rho/\rho) \text{ x } B^{0.13}$$

Critical stirrer speed, $N_c = S \text{ X } v^{0.1} \text{ x } Dp^{0.2} \text{ x } g \text{ x } (\Delta\rho/ \rho_L) \text{ x } B^{0.13} \text{ x } 1/D_a^{0.85}$, where

Agitator diameter, D_a = 0.15240 m

Shape factor, S = 6.5

Viscosity of suspension, μ = 700 cP

= 0.7 Pa-s

= 0.7 Kg/m.s

Density of liquid phase, ρ_L = 991 Kg/m^3

Density of slurry or suspension phase, ρ_S = 1070 Kg/m^3

Density difference of phases, $\Delta\rho = \rho_S - \rho_L$ = 79 Kg/m3

Kinematic viscosity of suspension, $\nu = \mu/ \rho_S$ = 0.7/1070

= 0.000654 m^2/s

Average particle size, D_p = $101.5 \text{ x } 10^{-6}$ m

Gravitational acceleration, g = 9.8 m/s^2

B = 11%

$$N_c = 6.5 \text{ x} 0.000654^{0.1} \text{x } (101.5 \text{ x } 10^{-6})^{0.2} \text{ x } 9.8 \text{ x } (79/ 993.804) \text{ x } (11)^{0.13} \text{ x } 1/0.15240^{0.85}$$

$$= 2.62 \text{ RPS}=157 \text{ RPM}$$

The critical speed of the impeller for comes out to be 157 RPM for just off-bottom suspension of the composition. The RPM range for design should be 50% greater than the critical speed which is $\simeq$ 235 RPM. A variable speed drive motor having range of 200-250 rpm is sufficient for the mixing and granulation process of composition.

5.3.4 Power Transmitted By Impeller Shaft (P)

$P = N_p N_C^3 D_a^5 \rho_S$

Power Number, Np [92] = 0.8

Critical stirrer speed, N_C = 2.617 RPS

Diameter of impeller or agitator, Da = 0.1524 m

Density of vessel contents at average temperature, ρ_s = 1070 Kg/m3

$P = N_p N_C^3 Da^5 \rho_S = 0.8 \times 4.17^3 \times 0.1524^5 \times 1036.676$ $\underline{\sim}$ 1.26 w

5.3.5 Selection of Jacket Type of Mixing Vessel

The most commonly used type jacket is that shown in Fig.5-5. It consists of an outer cylinder which surrounds part of the vessel. The heating or cooling medium circulates in the annular space b/w the jacket and vessel walls and heat is transferred through the wall of the vessel. Circulation baffles usually in the annular space increase the velocity of the liquid flowing through the jacket and improve the heat transfer co-efficient, see Fig.5-6. The spacing between the jacket and vessel wall depend on the size of the vessel, but typically range from 50 mm for small vessels to 300 mm for large vessels.

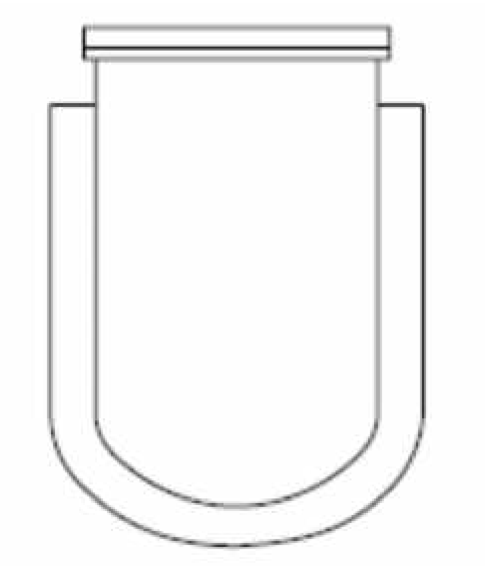


Fig.5-5 Jacketed Vessel

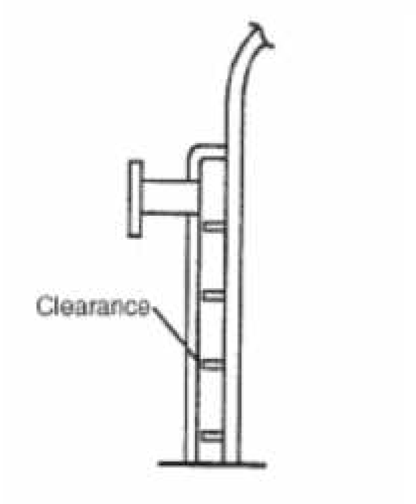


Fig.5-6 Spirally Baffled Jacket

In terms of cost the designs can be ranked, from cheapest to most expensive, as; simple, no baffles, agitation nozzles, spiral baffle, dimple jacket and half-pipe jacket. A simple jacket with spiral baffle to reduce cost of manufacturing has been selected. A spirally baffled jacket for high rates of heat transfer has been selected. The pressure rating of the design is taken as up to 10 bars for simple spirally baffled jacket. [93]

5.3.6 Design Calculations of Jacket

5.3.6.1 Physical Properties of Vessel Side Contents

Initial temperature, T_i = $25\ ^0C$

Final temperature, T_f = $70\ ^0C$

Average temperature = $(T_i + T_f)/2$ = $(25 + 70) / 2$ = $47.5\ ^0C$

Viscosity of slurry or suspension phase, [illegible] = 0.7 Kg/m.s

Specific heat capacity of emulsion whole contents, C_{EWC} = $3.7\ J/gm^0C$

Thermal Conductivity of emulsion whole contents, k_{EWC} = $0.82\ J/m.s.^0C$

Density of emulsion whole contents, ρ_S (Kg/m^3) = $1070\ Kg/m^3$

5.3.6.2 Physical Properties of Jacket Side Fluid

Initial temperature, T_i = $85\ ^0C$

Final temperature, T_f = $80\ ^0C$

Average temperature = $(T_i + T_f)/2$ = $(85 + 80) / 2$ = $82.5\ ^0C$

Viscosity of water (Kg/m.s) = 0.0003445 Kg/m.s

Specific heat capacity of water ,C_w = $4.1868\ J/g.^0C$

Thermal conductivity of water, k_w = $0.667\ J/m.s.^0C$

Density of water, ρ_w = $970\ Kg/m^3$

5.3.6.3 Heat Transfer Co-efficient of Vessel Side (hi)

Impeller Reynolds number (N_{Re}) of vessel side is;

$N_{Re} = N_C \times D_a^2 \times \rho_S / \mu$, where

Critical stirrer speed, N_C = 157 RPM = 2.617RPS

Impeller or agitator diameter, D_a = 6.0inch = 0.1524 m

Density of slurry, ρ_S = $1070\ Kg/m^3$

Viscosity of slurry, μ = 0.7 Kg/m.s

$N_{Re} = 2.617 \times 0.15242 \times 1070 / 0.7$ = 610

Prandtl number (N_{Pr}) of vessel side is;

$N_{Pr} = C_{EWC}\ \mu / k_{EWC}$, where

Specific heat capacity, C_{EWC} = 3.7 J/g.^{0}C

Viscosity, μ = 0.7 Kg/m.s

Thermal conductivity, k_{EWC} = 0.82 J/m.s.^{0}C

Npr =3.7 x 1000 x 0.7/0.582652 = 3158

Nusselt Number (N_{Nu}) of vessel side is;

$N_{Nu} = 0.54\ N_{Re}^{\ 0.67}\ N_{Pr}^{\ 1/3}$, where

Vessel side Reynolds number, N_{Re} = 93

Vessel side Prandtl number, N_{Pr} = 3158

$Nu = 0.54\ X143.325^{\ 0.67}\ X\ 4574.60^{1/3}$ = 165

Heat transfer co-efficient (hi) of vessel side is;

$h_i = N_{Nu}$ x k / D, where

Diameter of vessel, D = 0.4572 m

Nusselt number, N_{Nu} = 165

Thermal conductivity, k_s = 3.7 J/m.s.^{0}C

$h_i = N_{Nu}$ x k / D= 165 x 14.4 / 0.45720 = 1335.3 W/m^2.^{0}C

5.3.6.4 Heat Transfer Co-efficient of Jacket Side (h_j)

Spacing between jacket and vessel wall, S = 75 mm

Clearance, C = 25 mm

Pitch, P = 50 mm

Height of vessel, H = 0.609 m

No. of spirals of spiral baffle in jacket, n = 552.45 /50 = 11

Length of channel, L = πn D = 3.14 x 11 x 0.4572 = 16 m

Hydraulic mean diameter, D_e

$D_e = \left(\frac{4 \times S \times P}{2(S+P)}\right) - \left(\frac{4 \times 0.075 \times 0.050}{2(0.075 + 0.050)}\right) - \left(\frac{0.015}{0.25}\right)$ = 0.06 m

Cross-sectional area of the channel, A = 0.075 x 0.05 = 0.00375 m^2

Velocity of water through channel (assumed), u = 0.6 m/s

Mass velocity = 970 x 0.6 x 0.00375 = 2.1825 Kg/s

Flow rate of water required = 0.6 x 0.00375 x 264.2 x 60 = 35.667 GPM

Jacket side Reynolds number (N_{Re}) is;

$N_{Re} = D_e\, u\, \rho_W / \mu$, where

Hydraulic mean diameter, D_e = 0.06 m

Velocity of water through channel (assumed), u = 0.6 m/s

Density of water at average temperature, ρ_W = 970 Kg/m^3

Viscosity of water at average temperature, μ = 0.0003445 Kg/m.s

$N_{Re} = \left(\frac{0.06 \times 0.6 \times 970}{0.0003445}\right)$ = 101364

Jacket side Prandtl number (N_{Pr}) is;

$N_{Pr} = C\, \mu / k$, where

Specific heat capacity of water at average temperature, C = 4.1868 J/g.^{0}C

Viscosity at average temperature , μ = 0.0003445 Kg/m.s

Thermal conductivity of water at average temperature, k = 0.667 J/m.s.^{0}C

N_{Pr} = 4.1868 x 1000 x 0.0003445 / 0.667 = 2.16245

Jacket side Nusselt number (N_{Nu}) is;

$N_{Nu} = 0.023\, N_{Re}^{\ 0.8}\, N_{Pr}^{\ 1/3}$, where

Vessel side Reynolds number, N_{Re} = 101364

Vessel side Prandtl number, N_{Pr} = 2.16245

$N_{Nu} = 0.023 \times 101364^{\ 0.8} \times 2.16245^{\ 1/3}$ = 301

Jacket side heat transfer co-efficient (h_j) is;

$h_j = N_{Nu} \times k / D_e$, where

Hydraulic mean diameter, D_e = 0.06 m

Jacket Side Nusselt Number (N_{Nu}) = 301

Thermal conductivity of water at average temperature, k = 0.667 J/m.s.^{0}C

$h_j = N_{Nu} \times k / D_e = 301 \times 0.667 / 0.06$ = 3346 W/m^2.^{0}C

= 3346 W/m^2.K

5.3.6.5 Overall Heat Transfer Co-efficient (U)

$1/U = 1/h_i + 1/h_j + 1/(k/x) + 1/h_{di} + 1/h_{dj} = \frac{1}{1335.3} + \frac{1}{3346} + \frac{10^{-2}}{16} + \partial + \frac{1}{1500}$

Overall heat transfer co-efficient is; $U^{[94]}$ = 427.5W/m^2.K

5.3.6.6 Minimum Time for Heat Transfer (t)

Temperature Difference of Hot Fluid, (ΔT) = $T_2 - T_1$ = 80 – 85= -5 ^{0}C

Minimum time for required heat to be given off, Q_s = (m/s) C_p Δ T = 45688.455 J/s

Q = 12600000 J, Q_s = 45688.455 J/s , it implies that; t = Q /Q_s = 12600000/45688.455

= 275.78s = 4.6 min

5.3.6.7 Log Mean Temperature Difference (ΔT_m)

$\Delta T_m = ((\frac{(t2-T2)-(t1-T1)}{\ln((t2-T2)/t1-T1))}),$ where ΔT_m is log mean temperature difference

Hot fluid inlet temperature, T_1, = 85 ^{0}C

Hot fluid outlet temperature, T_2, = 80 ^{0}C

Cold fluid inlet temperature, t_1, = 25 ^{0}C

Cold fluid outlet temperature, t_2 = 70 ^{0}C

Temperature Difference of Vessel Fluid (Δt) = $t_2 - t_1$ = 70 – 25 = 45 ^{0}C

$\Delta T_m = ((\frac{(70-80)-(25-85)}{\ln((70-80)/(25-85))}) = \frac{(-10)-(-60)}{\ln((-10)/(-60))}) = \frac{(-10)+60}{\ln(1/6)} = \frac{50)}{-1.79176}$ = -27.9055 ^{0}C

5.3.6.8 Time to Heat Contents of Jacketed Vessel (θ)

Time needed to heat contents of jacketed vessel (θ) is calculated as follows:

$\theta = \ln\frac{(T1-t1)}{(T1-t2)} \times (\frac{M \times Cp \times k)}{WC(k-1)})$, where

Time needed to heat contents, θ

Hot fluid inlet temperature - Cold fluid inlet temperature = $T_1 - t_1$ = 85-25 = 60 ^{0}C = 60 ^{0}F

Hot fluid inlet temperature - Cold fluid outlet temperature = $T_1 - t_2$ = 85-70 = 15 ^{0}F

Mass within vessel, M_{EWC} = 154.387 lb

Specific heat capacity of mixture, C_{EWC} = 0.884 Btu/lb.^{0}F

Mass flow rate of water in jacket, W = 17321.7 lb/h

Specific heat capacity of water at average temperature, C = 1.00327 Btu/lb.^{0}F

Overall heat transfer co-efficient, U , = 43.298 Btu/ft^2.h.^{0}F

Height of liquid & slurry contents in vessel, H_L = 18 inch = 1.5 feet

Area in contact through periphery, A_1= [illegible] H_L x H_L = 3.14x1.5x1.5

= 7.065 ft^2

Area in contact with vessel contents through floor, $A_2 = \pi D^2/4$ = $3.14 \times 1.5^2/4$

= 1.76625 ft^2

Total heat transfer area, $A = A_1 + A_2$ = 8.83125 ft2

$k = e^{(UA/WC)} = e^{(47.405)(8.83125)/(17321.7)(1.00327)}$ = 1.024382754

[illegible]

$\theta = \ln\frac{(T1-t1)}{(T1-t2)} \times \frac{(M \times Cp \times k)}{WC(k-1)} = \ln\frac{60}{15} \times \frac{(154.387 \times 0.884 \times 1.024382754)}{17321.7 \times 1.00327 \times (1.024382754-1)}$ = $\ln\frac{4}{1} \times 0.33$

= 0.4753 hours

= 28.52 min

5.3.6.9 Pressure Drop through Jacket (ΔP)

At N_{Re} of 101364, $j_f = 2.8 \times 10^{-3}$ (from Appendix-C)

$\Delta P = 8\ j_f \times L \times \rho w \times u^2/2D_e$

Length of channel, $L = \pi n D = 3.14 \times 11 \times 0.4572$ = 16 m

Hydraulic mean diameter, D_e = 0.06 m

Density of water at average temperature, ρ_W = 970 Kg/m^3

Velocity of water through channel (assumed), u = 0.6 m/s

$\Delta P = 8j_f xLx\rho_w xu^2/2D_e = 8x2.8x10^{-3}x16x970x0.6^2/(0.06 \times 2)$ = 1043 N/m^2

5.3.7 Mechanical Design of Vessel

For vessels under internal pressure, the design pressure is normally taken as the pressure at which the relief device is set. This is taken 10 percent above the normal working pressure, to avoid spurious operation during minor process upsets. The design temperature at which the design stress is evaluated is taken as the 150 ^{0}C. Design Stress (Nominal Design Strength) is determined by applying a suitable “design stress factor” (factor of safety) to the maximum stress that the material could be expected to withstand without failure under test conditions. Design stress at 150 ^{0}C for austenitic stainless steels is 130 (N/mm^2). Design stress factor of mean stress to produce rupture at 10^5 h at the design temperature for austenitic stainless steels is 1.5. The corrected design stress is thus 225 (N/mm^2). [95]

5.3.7.1 Static Pressure of Vessel Contents (Ps)

$Ps = Pa + \rho_W\ g\ H_L$, where

Atmospheric pressure, Pa = 101325 N/m^2

Density of vessel contents at avg. temperature, ρ_W Kg/m3 = 1036.676

Gravitational acceleration, g = 9.8 m/s^2

Height of liquid & slurry contents, H_L = 0.4572 m

$Ps=Pa+\rho_W gH_L=101325+1036.7 \times 9.8 \times 0.4572=105970$ N/m^2 = 15.3696 Psi

5.3.7.2 Internal pressure of vessel contents (P_i)

Taking P_i as 10 times greater than static pressure of vessel contents then

Internal pressure of vessel contents, Pi = 1.1 x 105970 = 116567 N/m^2

= 16.9113 Psi

5.3.7.3 Minimum Wall Thickness of Vessel (e)

Minimum wall thickness of vessel, $e = P_iD_i/(2f-P_i)$, where

Internal pressure of vessel contents, Pi = 116567 N/m^2

Internal diameter of vessel, Di = 0.4572 m

Ultimate tensile strength, f = 225 N/mm^2

e = 116567x0.4572 (2 x 225 x 10^{-6} – 116567) =1.146x10^{-4} m = 0.12 mm

The “the corrosion allowance is the additional thickness of metal added to allow for material lost by corrosion and erosion, or scaling. The allowance to be used (as severe corrosion is not expected) is 2.0 mm. There will be minimum wall thickness required to ensure that vessel is sufficiently rigid to withstand its own weight, and any incidental loads. Minimum wall thickness of vessel taken from table is 5 mm; the value includes a corrosion allowance of 2 mm. [96]

5.3.8 Design of Water Circulating Centrifugal Pump for Jacket

Fluid enters axially through the eye of the casing, is caught up in the impeller blades, and is whirled tangentially and radially toward up till it leaves through all circumferential parts of the impeller into the diffuser part of the casing. The fluid gains both velocity and pressure while passing through the impeller. The doughnut-shaped diffuser, or scroll, section of the casing decelerates the flow and further increases the pressure. A schematic diagram of working of centrifugal pump is shown in Fig.5-7. [97]

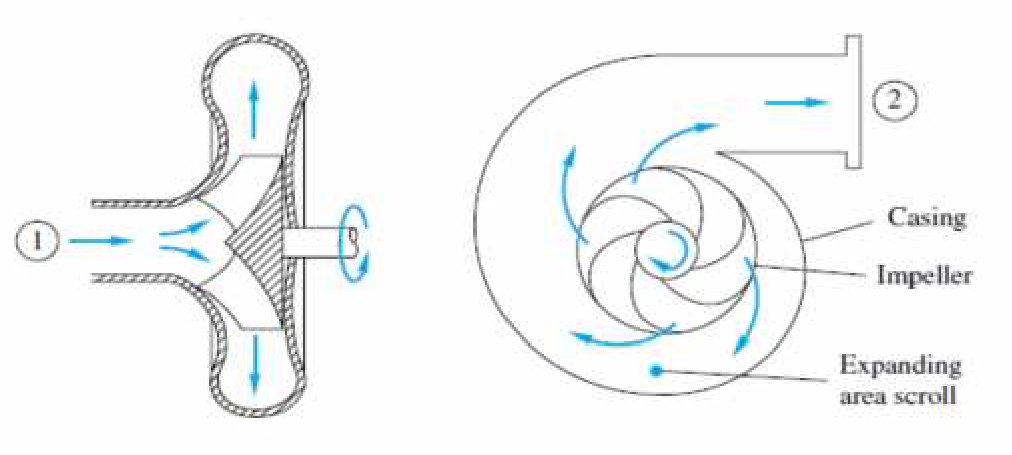


Fig.5-7 Schematic Diagram of Working of Centrifugal Pump

Static elevation change (ΔZ) = Z_2-Z_1 is taken as 3.5 ft. or 1.0668 m and correction factor due to loss of kinetic head in pipe length and fittings from values of K is; [98]

Table 5-2 Summation of K Values

Sr. No.	Valve or fitting	Nos.	Loss (K)/Unit	ΣK
1.	Sharp entrance	1	0.5	0.5
2.	Open globe valve (2 inch)	1	6.0	6.0
3.	90^0 elbow, standard	3	0.45	1.35
4.	Gate- valve half open	5	4.0	20
5.	Sharp exit	5	1.0	5
6.	Sum of additional friction loss, eq. no. of velocity heads, K		ΣK	32.25

5.3.8.1 Head of System Required (H_{Sys})

Volumetric flow rate of water in jacket, q = = 0.00225024 m^3/s

Diameter of pipeline, D = 2 inch = 0.0508 m

Cross-sectional area of pipe, $A = \pi D^2/4$ = 3.14 × 0.0508$^2/4$ = 0.002193544 m^2

Velocity of water, V = q/A = 0.00225024 / 0.002193544 = 1.02585 m/s

Reynolds number of water in pipeline is;

$N_{Re} = D\ V\ \rho_W / \mu$, where

Diameter of pipeline, D = 2 inch = 0.0508 m

Velocity of water, V = 1.02585 m/s

Density of water at average temperature, ρ_W = 970 Kg/m^3

Viscosity of water at average temperature, μ = 0.0003445 Kg/m.s

$N_{Re} = \left(\frac{0.0508 \times 1.02585 \times 970}{0.0003445}\right)$ = 146734

From Appendix-C At N_{Re}=146734 , fanning friction factor, f = 0.00425

Length of pipeline, L = 100 ft = 30.48 m

Head of system required (H_{Sys}) is;

The head of system required for circulation of water for heating purpose contains a static-elevation change Z_2-Z_1 plus friction losses in pipes and fittings and is given as follows:

$$H_{Sys} = \frac{\left(Z_2 - Z_1 + V^2\left(\sum_{K=i,1}^{K=f\,n}(Kxn) + f.L/D\right)\right)}{2g} = \frac{\left(1.0668 + 1.02585^2(32.25) + 0.00425x\frac{30.48}{0.0508}\right)}{2 x 9.8} = 1.0668 + 1.87 = 3\ m$$

5.3.8.2 Total Head of System Required (H_T)

Head loss through jacket (H_L) is;

$\Delta P = \rho_W\ g\ H_L$, where

Pressure drop through jacket, ΔP = 1043 N/m^2

Density of water at average temperature, ρ_W = 970 Kg/m^3

Gravitational acceleration, g = 9.8 m/s^2

Head loss through jacket, $H_L = \Delta P/\rho_W\ g$=1043 / (970 x 9.8) = 0.11 m

$H_T = H_L + H_{Sys}$ = 0.11 + 3 = 3.11 m

5.3.8.3 Pressure Increased by Pump (ΔP_P)

$\Delta P_P = \rho_W\ g\ H_T$, where

Density of water at average temperature, ρ_W = 970 Kg/m^3

Gravitational acceleration, g = 9.8 m/s^2

Total head of system required, H_T = 3.11 m

$\Delta P_P = \rho_W\ g\ H_T$ = 970 x 9.8 x 3.11= 29563.66 N/m^2 = 4.3 Psi

5.3.8.4 Power Required To Operate Pump

$P = \rho_W \, g \, H_T \times q$, where

Density of water at average temperature, ρ_W = 970 Kg/m^3

Gravitational acceleration, g = 9.8 m/s^2

Total head of system required, H_T = 3.11 m

Volumetric flow rate of water, q = 0.00225024 m^3/s

$P = \rho_W \, g \, H_T \times q = 970 \times 9.8 \times 3.11 \times 0.00225024$ = 67 Watt

5.3.8.5 Net positive Suction Head (NPSH) Required

$NPSH = \dfrac{\dfrac{(P_a - P_v)}{\rho_W} - f_s}{g - Z_a}$, where

Atmospheric pressure, Pa = 101325 N/m^2

Vapour pressure at 85 ^{0}C, P_v = 58116 N/m2

Density of water at average temperature, ρ_W = 970 Kg/m^3

Ground level elevation, Z_a = 0

Skin friction factor, f_s (assumed) = 0

Gravitational acceleration, g = 9.8 m/s^2

$NPSH = \dfrac{\dfrac{(101325 - 58116)}{970} - 0}{9.8 - 0} = \dfrac{(101325 - 58116)}{970 \times 9.8}$ = 4.545 m

5.3.9 Design of Water Tank Reservoir for Hot Water Supply

Capacity = 70 gallon, Power of Heater (02 Nos.) = 1.75 kW & dimensions of water reservoir are; L = 707 mm, W = 707 mm, H = 707 mm. The details are given in appendix-E. Hence, the final specifications of equipment for pilot scale production of composition are given in appendix-F.

Conclusion

No explosive or explosive composition is perfect and with developments in technologies, the improvement of existing explosives and explosives compositions is the need of the hour. Accordingly, the present work is undertaken to replace Tetryl with better explosive composition with lower sensitivity and better performance. For this purpose RDX was selected and following main objectives set forth were achieved:

- Characterisation of raw materials and optimization & finalization of the process parameters for development of an RDX based insensitive composition.
- Establishing a laboratory scale method for development of an RDX based insensitive composition in defined system.
- Presenting scaled up design for production of an RDX based insensitive composition.

The nutshell conclusion of this work is that a safe and better explosive performance wise RDX based insensitive composition has been developed for production up to pilot plant scale level.

Future Recommendations

It is recommended that the developed methodology be adopted for the preparation of this composition at pilot plant scale by POF. Further research can be undertaken to develop similar explosive composition based on HMX.

References

[1] https://en.wikipedia.org/wiki/Muhammad_Ali, (2020).

[2] https://en.wikipedia.org/wiki/Sumo, (2020).

[3] George P. Sutton, Oscar Biblarz, Rocket Propulsion Elements, 7th Edition, p.474-476 (2001).

[4] S. Fordham, High Explosives and Propellants, 2nd Edition, p.3 (1980).

[5] Paul W. Cooper, Explosives Engineering, p.51-66 (1997).

[6] M. Suceska, Test Methods for Explosives, p.1-3 (1995).

[7] J. Akhawan, The Chemistry of Explosives, 2nd Edition, p.8 (1998).

[8] https://en.wikipedia.org/wiki/Tetryl, (2020).

[9] H. B. Troup, Clinical Effects of Tetryl (CE Powder), The Royal Ordnance Factory, Bridgend (Wales), p.20 (1946).

[10] Robert J. Spear, Victor, et al, Recommended Replacements for Tetryl in Australian Production Fuzes and Related ordnance, p.1 (1987).

[11] R. Meyer, J. Köhler, et al, Explosives, Explosives. 6th Edition. p.40, 85 (2007).

[12] Paul W. Cooper, Explosives Engineering, p.33 (1996).

[13] L. D. Hampton, Development of RDX Composition CH-6, p.1 (1960).

[14] Walla A. Alfaraj, Brian McMillan, et al, Tetryl Exposure: Forgotten Hazards of Antique Munitions, Annals of Occupational and Environmental Medicine, p.3 (2016).

[15] Robert J. Spear, Victor Nanut, A Comparative Assessment of US and UK Explosives Qualified as Replacements for Tetryl, p.1 (1987).

[16] Robert J. Spear, Victor, et al, Recommended Replacements for Tetryl in Australian Production Fuzes and Related ordnance, p. 2 (1987).

[17] J. Akhawan, The Chemistry of Explosives, 2nd Edition, p.9 (1998).

[18] https://en.wikipedia.org/wiki/RDX,(2020).

[19] L. D. Hampton, Development of RDX Composition CH-6, p.1-2 (1960).

[20] Robert J. Spear, Victor, et al, Recommended Replacements for Tetryl in Australian Production Fuzes and Related ordnance, p.3 (1987).

[21] Baxter III, p. 27, 42, 255–259 (1968).

[22] https://www.britannica.com/technology/dynamite.

[23] William G. PROUD, Stephen M. Walley, et al, Recent Trends in Research on Energetic Materials at Cambridge, Central European Journal of Energetic Materials, p.86-87 (2009).

[24] R. J. HUDSON, Cranfield University Department of Applied Science and Engineering, Ph.D Thesis “Investigating the Factors Influencing RDX Shock Sensitivity”, p.2-4 (209-2012).

[25] J. Akhawan, The Chemistry of Explosives, 2nd Edition, p.10 (1998).

[26] Veera M. Boddu, Abburi Krishnaiah, Preparation of Insensitive RDX from Crude RDX using an Antisolvent Phase Separation Process, p.1 (2014).

[27] M. Mokhtar, Recrystallization as a Technique for Lowering the Sensitivity of RDX, p.1-2 (2008).

[28] Jennifer L. Gottfried, Frank C. De Lucia Jr., et al, RDX Characterization of the Morphology of RDX Particles Formed by Laser Ablation, p.1,3,5 (2012).

[29] Paul W. Cooper, Explosives Engineering, p.23 (1996).

[30] Factory Report.

[31] DSC Report.

[32] Sasuqiao Wang, Xin Li, et al, Preparation and Characterization of Cyclotrimethylenetrinitra-mine (RDX) with Reduced Sensitivity, p.5 (2017).

[33] Martin S. Miller, Thermophysical Properties of RDX, p.3, 5 (1997).

[34] S. Jianke Wang, RMIT University Melbourne Master Thesis “Suspension of High Concentration Slurry in Agitated Vessels”, p.21 (2010).

[35] https://en.wikipedia.org/wiki/PIB, (2020).

[36] István Benedek, Mikhail M. Feldstein, Handbook of Pressure-Sensitive Adhesives and Products, p.4.1- 4.4 (2009).

[37] Military Specification, MIL-C-7123B 8 February (1994).

[38] James E. Mark, Physical Properties of Polymers Handbook, 2nd Edition, p.94 (2007).

[39] Monice M. Flume, HBart eldreth, et al, Final Report of the Cosmetic Ingredient Review Expert Panel on the Safety Assessment of Dicarboxylic Acids, Salts, and Esters, p.11S, 41S(2012).

[40] https://www.chemicalbook.com/ChemicalProductProperty_EN_CB8505859.htm.

[41] Jan W. Gooch, Encyclopedic Dictionary of Polymers, p.299, 307 (2007).

[42] W. V. Titow, PVC Plastics: Properties, Processing, and Applications, p.177-178 (1990).

[43] Robert O. Ebewele, Polymer Science and Technology, p.121, 236 (2000).

[44] Tony Whelan, Polymer Technology Dictionary, p.535 (1994).

[45] https://wiki.anton-paar.com/en/engine_oil

[46] Source: Society of Automotive Engineers (SAE), December 1999.

[47] lubricants@pumaenergy.com

[48] Bernard J. Hamrock, Steven R. Schmid, Bo O. Jacobson, Fundamentals of Fluid Film Lubrication, 2nd Edition, p.100 (1994).

[49] S. B. WRIGHT, Method of Preparing a Cyclotrimethylenetrinitramine and Cyclotetramethyl-ene Tetranitramine Plastic Bonded Explosives, USP 3,138,501, (1964).

[50] Pradyot Patnaik, Handbook of Inorganic Chemicals, p.180-183 (2003).

[51] Military Specification, MIL-G-155A (1962).

[52] https://en.wikipedia.org/wiki/Carbon,(2020)

[53] H. Mojiri, M. Aliofkhazraei, Sodium Stearate, p.6 (2017).

[54] Klaus Schumann, Kurt Siekmann, "Soaps" in Ullmann's Encyclopedia of Industrial Chemistry, 2005.

[55] Ali Karaipekli, Ahmet Sar, Kamil Kaygusuz, Thermal Conductivity Improvement of Stearic Acid Using Expanded Graphite and Carbon Fiber for Energy Storage Applications, p.2201-2210 (2007).

[56] Pradyot Patnaik, Handbook of Inorganic Chemicals, p.161-163 (2003).

[57] M. J. Assael, E. Charitidou, J. Ch. Stassis, W. A. Wakeham, Absolute Measurements of the Thermal Conductivity of Some Aqueous Chloride Salt Solutions, p.888 (1989).

[58] www.oxycalciumchloride.com

[59] Nora A, Szczepanek A, Koenen G (2001) "Metallic Soaps".

[60] Weingarten H, Franck EU, et al (2000) "Water".

[61] www.cement.org/masonry/cc_fn_preventing_efflorescence.asp.

[62] L. D. Hampton, Development of RDX Composition CH-6, p.4, 7 (1960).

[63] Military Specification, MIL-C-7123B 8 February (1994).

[64] Lide, David R., CRC Handbook of Chemistry and Physics, 90th Edition, (2009).

[65] www.schoolmykids.com.

[66] https://en.wikipedia.org/wiki/Hexane,(2020).

[67] B. Orge, M. Iglesias, et al, Mixing properties of (methanol, ethanol, or 1-propanol) with (n-pentane, n-hexane, n-heptane and n-octane) at 298.15 K, p.214 (1997).

[68] https://en.wikipedia.org/wiki/Distilled_water.

[69] W. M. Haynes, CRC Handbook of Chemistry and Physics, 95th Edition, (2014-15).

[70] G. Pooria et al, Journal of Biomolecular Techniques, Differential Scanning Calorimetry Techniques: Applications in Biology and Nanoscience, 167 (2010).

[71] https://en.wikipedia.org/wiki/Differential scanning calorimetry,(2020).

[72] W.M. GROENEWOUD, Characterisation of Polymers by Thermal Analysis, Differential Scanning Calorimetry, (2001).

[73] Kalsoom Akhtar, Shahid Ali Khan, et al, Scanning Electron Microscopy: Principle and Applications in Nanomaterials Characterization 113-114 & 116 (2019).

[74] Donald Mcglinchey, Characterisation of Bulk Solids, p.231 (2005).

[75] www.thermonicolet.com, Introduction to Fourier Transform Infrared Spectrometry, (2001).

[76] DA-Wen Sun, Infrared Spectroscopy for Food Quality Analysis and Control, 146-147 (2009).

[77] https://en.wikipedia.org/wiki/Refractive_Index,(2020).

[78] J. Akhavan, L.J. Welsh, The Flow Characteristics of High Explosives, p.221-222 (1992)

[79] Document QAS/11.450 FINAL, Final text for addition to The International Pharmacopoeia, Bulk Density and Tapped Density of Powders, World Health Organization, 1 & 4-6 (2012).

[80] Military Specification, MIL-C-7123B 8 February, p.8-10 (1994).

[81] Jai Prakash Agrawal, High Energy Materials, p.19-20 (2010).

[82] J. Mesec, Stjepan, et al, International Journal of Mining Science and Technology, In-hole velocity of detonation (VOD) measurements as a framework for the selection type of explosive, 675 & 680 (2015).

[83] https://en.wikipedia.org/wiki/Detonation velocity,(2020)

[84] Curren R.D., Harbell J.W, In Vitro Alternatives for Ocular Irritation, p.106:485-492 (1998) 1998.

[85] York M, Steiling W., A Critical Review of the Assessment of Eye Irritation Potential using the Draize Rabbit Eye Test, p.18:233-40 (1998).

[86] P. Alexandra, D. Valentina, et al, Matrix-assisted pulsed laser evaporation of Chemoselective, 656 (2011).

[87] Gregory T. Long, Sergey Vyazovkin, et al, Competitive Vaporization and Decomposition of Liquid RDX, p.2572 (2000).

[88] Mehmet Go¨ nen, Serdar O, et al, Preparation and Characterization of Calcium Stearate Powders and Films Prepared by Precipitation and Langmuir-Blodgett Techniques, p.1732 (2010).

[89] Warren L. McCabe, Julian C. Smith, et al, Unit Operations of Chemical Engineering, 5th Edition, p.242-243 (1993).

[90] Don W. Green, Robert H. Perry, Perry's Chemical Engineers' Handbook, 8th Edition, 18-10:18-19 (2008).

[91] (Steven) Jianke Wang, A Thesis Submitted for the Degree of Master of Engineering, Suspension of High Concentration Slurry in Agitated Vessels, RMIT University Melbourne, p.54 (2010).

[92] Warren L. McCabe, Julian C. Smith, et al, Unit Operations of Chemical Engineering, 5th Edition, p.248 (1993).

[93] R. K. SINNOTT, Coulson & Richardson's Chemical Engineering, Volume 6, 4th Edition, Chemical Engineering Design, p.776-777 (2005).

[94] R. K. SINNOTT, Coulson & Richardson's Chemical Engineering, Volume 6, 4th Edition, Chemical Engineering Design, p.776-781 (2005).

[95] R. K. SINNOTT, Coulson & Richardson's Chemical Engineering, Volume 6, 4th Edition, Chemical Engineering Design, p.781 (2005).

[96] R. K. SINNOTT, Coulson & Richardson's Chemical Engineering, Volume 6, 4th Edition, Chemical Engineering Design, p.814 (2005).

[97] Frank M. White, Fluid Mechanics, 4th Edition, p.714 (1991).

[98] R. K. SINNOTT, Coulson & Richardson's Chemical Engineering, Volume 6, 4th Edition, Chemical Engineering Design, p.204 (2005).

Appendices

Annexure-A

Detailed Material Balance

Method for Lab Scale Preparation of Composition

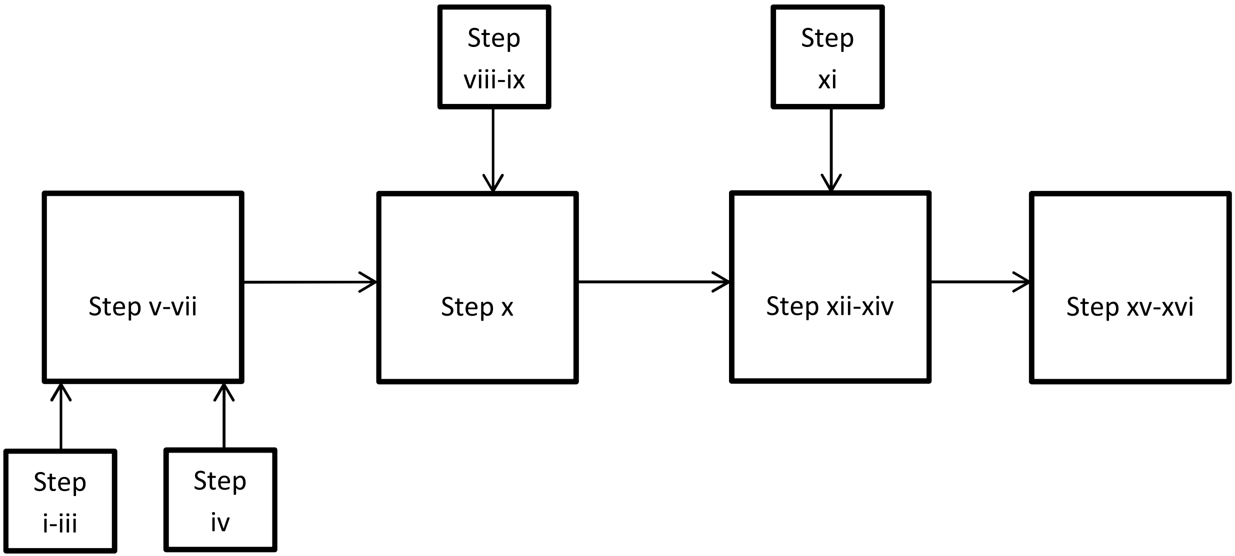
The process developed for lab scale preparation of composition contains following steps;

i. First of all RDX which contain 20% moisture contents is taken.
ii. The RDX is dried in air circulated oven at 70 -80 ^{0}C.
iii. A 10% RDX water slurry containing 10% RDX and 90% water by weight is prepared in jacketed mixing vessel. The slurry is heated up to 50 ^{0}C.
iv. The plasticized polyisobutylene is dissolved in the n-hexane and heated up to 45^{0}C.
v. Add solution of polyisobutylene to RDX-water slurry in dropwise manner.
vi. Maintain the temperature of emulsion at 50 ^{0}C.
vii. Digest the plasticized polyisobutylene in RDX-water slurry emulsion at 50 ^{0}C for 15 min.
viii. Sodium stearate is dissolved in distilled water and an aqueous solution containing 13% sodium stearate and 87% water is prepared.
ix. The graphite is mixed with above solution and heated up to 60^{0}C
x. The graphite-sodium stearate solution to emulsion maintained at 50 ^{0}C. The whole is stirred for 25 min to distribute homogeneously.
xi. Calcium chloride is dissolved in distilled water containing 20% calcium chloride and 80% distilled water.
xii. Calcium chloride aqueous solution is mixed with the above emulsion maintained at 50 ^{0}C. The precipitation reaction of these solutions results in introduction of calcium stearate as dispersant in the composition. The chemical reaction equation is given as;

$$C_{18}H_{35}COONa_{\ (aq)} + CaCl_{2\ (aq)} \rightarrow (C_{18}H_{35}COO)_2Ca_{(aq)} + 2NaCl_{\ (aq)}$$

xiii. The whole mix is digested at 50 ^{0}C for 30 min. Distil n-hexane from digested emulsion mixture. Distilling n-hexane from emulsion leaves plasticized polyisobutylene coated on grains of RDX, calcium stearate and graphite composition.
xiv. The distillation range of n-hexane is from 50-70 ^{0}C.
xv. Hence composition of RDX/graphite/calcium stearate/plasticized polyisobutylene is prepared. This composition is named as NUPC-6.
xvi. This composition is washed from alkali contents along NaCl with distilled water to neutral pH, filtered cooled to room temperature.

xvii. The process flow diagram for process developed is presented as follows;



Basis of Calculation: 7000 gm of NUPC-6

Composition of NUPC-6

Sr. No.	**Ingredient**	**% (w/w)**
1.	RDX	97.5
2.	Plasticized Polyisobutylene	0.5
3.	Calcium Stearate	1.5
4.	Graphite	0.5

Composition of Plasticized PIB

Sr. No.	**Ingredient**	**% (w/w)**
1	PIB	0.3334
2	DIOS	0.5244
3	SAE-10 Oil	0.1422

Raw materials required for 7000 gm of NUPC-6 are;

xvi. Moisture free RDX = 7000 X .975 = 6825.000 gm

xvii. Graphite = 7000 X 0.005 = 35.000 gm

xviii. PIB = 7000 X 0.005 = 11.669 gm

xix. DIOS = 7000 X 0.2622 = 18.354 gm

xx. SAE-10 Oil = 7000 X 0.0711 = 4.977 gm

xxi. Calcium Stearate = 7000 X 0.015 = 105.000 gm

Calcium stearate is manufactured as a chemical reaction b/w sodium stearate and calcium chloride with 98.5 % conversion [81]. The reaction equation is mentioned as follows;

$$\underset{2\text{ mol}}{C_{18}H_{35}COONa_{(aq)}} + \underset{1\text{ mol}}{CaCl_{2\,(aq)}} \rightarrow \underset{1\text{ mol}}{(C_{18}H_{35}COO)_2Ca} + \underset{2\text{ mol}}{2NaCl_{(aq)}}$$

Formula weight of calcium stearate is 607.017 gm / mol. Hence no. of moles of calcium stearate required in composition is 105/607.017 or 0.173 moles. Calcium chloride and sodium stearate required for 0.173 moles of calcium stearate 0.173 moles and 2 x 0.173 moles or 0.346 moles respectively. As the reaction conversion is 98.6% therefore amounts of calcium chloride and sodium stearate is to be supplied in the excess is 0.173/0.985 or 0.1756 moles and 2 X 0.1756 moles or 0.3512 moles respectively.

xxii. Formula weight of $CaCl_2$ is 110.984 gm / mol; hence the $CaCl_2$ supplied for reaction is 0.1756 X 110.984 or 19.5 gm.

xxiii. Formula weight of sodium stearate is 306.459 gm / mol; hence the sodium stearate supplied is for reaction is 0.3512 X 306.459 or 107.63 gm.

xxiv. Weight of $CaCl_2$ unconverted and remaining unreacted is (0.1756-0.173) X 110.984 or 0.289 gm.

xxv. Similarly unreacted sodium stearate is (0.3512-0.346) X 306.459 or 1.6 gm.

xxvi. Formula weight of NaCl is 58.443 gm / ml, No. of moles of NaCl produced are 0.173 X 2 or 0.346 moles; hence sodium chloride produced is 0.346 X 58.443 or 20.2212 gm.

xxvii. Specific gravity of n-hexane is 0.660, 1 gm of plasticized polyisobutylene is 35 X 50 or 1750 ml, specific gravity of plasticized polyisobutylene is 0.92, volume of plasticized polyisobutylene is 35 / 0. 92 or 38.04 ml, volume of mixing vessel required for plasticized polyisobutylene solution is 1750 + 38.04 or 1788.04 ml; hence mass of n-hexane used is 0.66 X 1788.04 or 1155 gm.

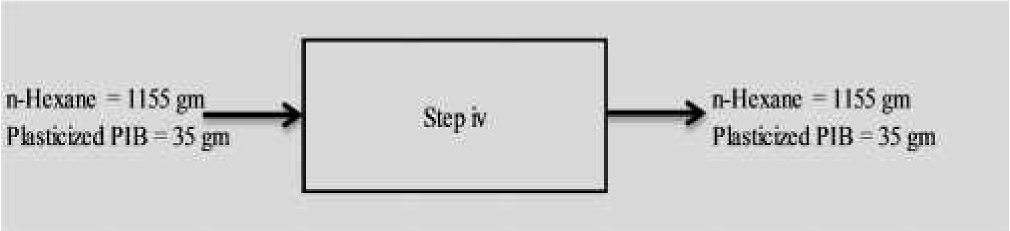
xxviii. Density of water at 50^0C is 1 gm / ml, 10% RDX slurry; hence water in 10 % RDX slurry is 90% which is (90/10) X 6825 or 61425 gm.

xxix. Mass of water in 13% sodium stearate aqueous solution is 87% which is (87/13) X 107.63 or 720.293 gm.

xxx. Mass of water in 20% $CaCl_2$ aqueous solution is 80% which is (80/20) X 19.5 or 78 gm.

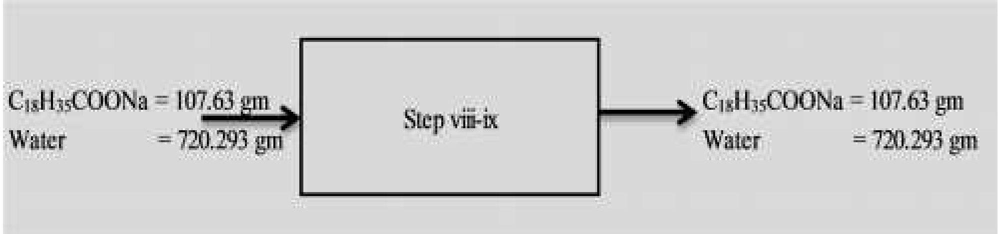
xxxi. The overall mass balance across step i-iii for preparing RDX-water slurry is given as;

xxxii. The overall mass balance across step iv is given as ;

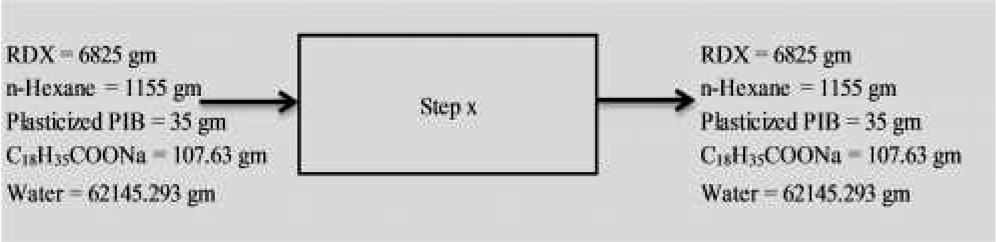


xxxiii. The overall mass balance across step v-vii is given as ;

xxxiv. The overall mass balance across step viii-ix is given as ;

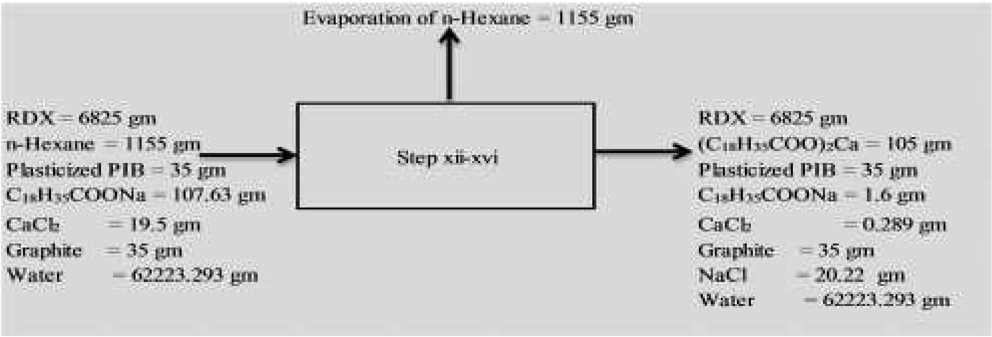


xxxv. The overall mass balance across step x is given as ;

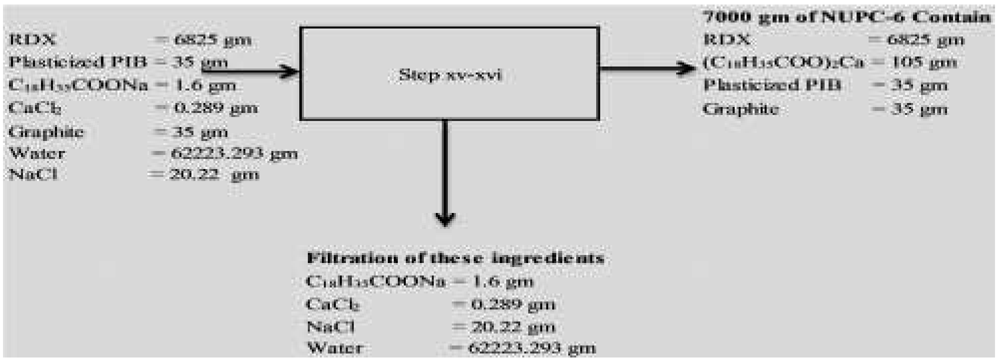


xxxvi. The overall mass balance across step xi is given as ;

xxxvii. The overall mass balance across step xii-xiv is given as ;



xxxviii. The overall mass balance across step xv-xvi is given as ;



xxxix. Hence 7000 gm of NUPC-6 composition are produced

Annexure-B

Detailed Energy Balance

Basis of Calculation: 7000 gm of NUPC-6

Energy required for heating slurry of RDX & across step i-iii;

Mass of RDX, M_{RDX} = 6825 gm
Mass of distilled water, M_{Water} = 61425 gm
Total mass of contents of slurry in vessel, M_{Slurry} = 68250 gm
Initial temperature of slurry, T_{1S} = 25 ^{0}C
Final temperature of slurry, T_{2S} = 50 ^{0}C
Temperature change of slurry, $\Delta T_{Slurry} = T_{2S} - T_{1S}$ = 25 ^{0}C
Average specific heat capacity of RDX, C_x = 1.1095 J/gm^{0}C
Average specific heat capacity of water, C_w = 4.000069 J/gm^{0}C
Average specific heat capacity of the slurry, C_S = 3.711 J/gm^{0}C
Heat need to raise RDX water slurry, $Q_1 = M_{Slurry} X C_S X \Delta T_{Slurry}$ = 6331914.737 J
= 6331.914737 KJ
= 1.76 kWH

Energy required for heating binder solution across step iv;

Mass of PIB, M_{PIB} = 11.669 gm
Mass of DIOS, M_{DIOS} = 18.354 gm
Mass of SAE-10 Oil, M_{SAE-10} = 4.977 gm
Mass of n-Hexane, $M_{n-Hexane}$ = 1155 gm
Total mass of solution of binder & n-Hexane in vessel, M_{BS} = 1190 gm
Initial temperature of solution, T_{1S} = 25 ^{0}C
Final temperature of solution, T_{2S} = 45 ^{0}C
Temperature change of solution, ΔT_{Slurry} = 20 ^{0}C
Average specific heat capacity of PIB to 45 ^{0}C, C_{PIB} = 2.0 J/gm^{0}C
Average specific heat capacity of DIOS to 45 ^{0}C, C_{DIOS} = 0.04411 J/gm^{0}C
Average specific heat capacity of SAE-10 Oil to 45 ^{0}C, C_{SAE-10} = 1.964 J/gm^{0}C
Average specific heat capacity of n-Hexane, C_{hexane} = 3.08 J/gm^{0}C
Average specific heat capacity of solution, C_{BS} = 3.01535 J/gm^{0}C

Heat required to heat solution of binder, $Q_2 = M_{BS} X C_{BS} X \Delta T_{BS}$ = 71765.30 J

= 71.765 KJ

= 0.020 kWH

Energy required to keep RDX-water slurry-binder solution at 50^0C across step v-vii;

Mass of PIB, M_{PIB} = 11.669 gm

Mass of DIOS, M_{DIOS} = 18.354 gm

Mass of SAE-10 Oil, $M_{SAE\text{-}10}$ = 4.977 gm

Mass of n-Hexane, $M_{n\text{-}Hexane}$ = 1155 gm

Total mass of solution of binder & n-Hexane in vessel, M_{BS} = 1190 gm

Initial temperature of solution, T_{1S} = 45 ^{0}C

Final temperature of solution, T_{2S} = 50 ^{0}C

Temperature change of solution, ΔT_{Slurry} = 5 ^{0}C

Average specific heat capacity of PIB, C_{PIB} = 2.0 J/gm^{0}C

Average specific heat capacity of DIOS, C_{DIOS} = 0.04411 J/gm^{0}C

Average specific heat capacity of SAE-10 Oil, $C_{SAE\text{-}10}$ = 1.964 J/gm^{0}C

Average specific heat capacity of n-Hexane, C_{hexane} = 3.08 J/gm^{0}C

Average specific heat capacity of the solution, C_{BS} = 3.01535 J/gm^{0}C

Heat required to heat binder solution to 50 ^{0}C, $Q_3 = M_{BS} X C_{BS} X \Delta T_{BS}$ = 17941.32 J

= 17.94 KJ

= 0.00498 kWH

Energy required to heat aq. sodium stearate with graphite across step viii-x;

Moles of sodium stearate to be dissolved, n = 0.3512 moles

Mass of sodium stearate to be dissolved = 107.6284 gm

Heat of solution of sodium stearate, H_{soln} = -239.7 KJ/mol

Heat of solution of sodium stearate evolved, $Q_4 = n\ H_{soln}$ = -84182.64 J

= -84.18264 KJ

Mass of graphite blended, M_G = 35 gm

Mass of distilled water for dissolution of sodium stearate, M_w = 720.2823 gm

Total mass of solution of sodium stearate & graphite, M_{GSS} = 862.9108 gm

Initial temperature of slurry, T_{1S} = 25 ^{0}C

Final temperature of slurry, T_{2S} = 60 ^{0}C

Temperature change of slurry, ΔT_{BS} = 35 ^{0}C

Average specific heat capacity of graphite, C_g = 0.708 J/gm^{0}C

Average specific heat capacity of sodium stearate, $C_{Na\text{-}St}$ = 0.008 J/gm^{0}C

Average specific heat capacity of the water, C_w = 4.000069 J/gm^{0}C

Average specific heat capacity of slurry, C_{sg} = 3.667 J/gm^{0}C

Heat required to heat solution ,$Q_5 = M_{GSS}XC_{GSS}X\Delta T_{GSS} + Q_4$ = 26566.25001J

= 26.566 KJ

= 0.00738 kWH

Energy balance across step xi;

Moles of calcium chloride to be dissolved = 0.1756 moles

Mass of calcium chloride to be dissolved = 19.5 gm

Heat of solution of calcium chloride, H_{soln} = -239.7 KJ/mol

Heat of solution of calcium chloride evolved, $Q_6 = n\ H_{soln}$ = -127924.6 J

Mass of distilled water for dissolution of calcium chloride, M_w = 78 gm

Total mass of solution of calcium chloride, M_{CCS} = 97.5 gm

Initial temperature of slurry, T_{1S} = 25 ^{0}C

Final temperature of slurry, T_{2S} = 60 ^{0}C

Temperature change of slurry, ΔT_{BS} = 35 ^{0}C

Average specific heat capacity of solution of $CaCl_2$, C_{CC} = 0.6571 J/gm^{0}C

Average specific heat capacity of the water, Cw = 4.000069 J/gm^{0}C

Average specific heat capacity of slurry, C_{CCS} = 3.331 J/gm^{0}C

Heat removed from solution, $Q_7 = M_{CCS}XC_{CCS}X\Delta T_{CCS} + Q_6$ = -116555.9253 J

= -116.556 KJ

= -0.0324 kWH

Energy balance across step xii-xiv;

Heat of reaction of sodium stearate and calcium chloride, Q_{Rexn} = -221.523 KJ

= -0.0615 kWH

Mass of RDX, M_{RDX} = 6825 gm

Mass of PIB, M_{PIB} = 11.669 gm

Mass of DIOS, M_{DIOS} = 18.354 gm

Mass of SAE-10 Oil, $M_{SAE\text{-}10}$ = 4.977 gm

Mass of graphite blended, M_G = 35 gm

Mass of Calcium Stearate produced, M_{CSP} = 105 gm
Mass of Sodium Stearate unreacted, M_{SSU} = 1.6 gm
Mass of calcium chloride unreacted, M_{CCU} = 0.289 gm
Mass of NaCl produced, M_{SCP} = 20.2212 gm
Mass of n-Hexane, $M_{n\text{-}Hexane}$ = 1155 gm
Mass of distilled water, M_W = 62223.293 gm
Total mass of emulsion whole contents, M_{EWC} = 70400.4032 gm
Average specific heat capacity of RDX, C_{RDX} = 1.109502 J/gm^{0}C
Average specific heat capacity of PIB, C_{PIB} = 2 J/gm^{0}C
Average specific heat capacity of DIOS, C_{DIOS} = 0.0441 J/gm^{0}C
Average specific heat capacity of SAE-10 Oil, $C_{SAE\text{-}10}$ = 1.964 J/gm^{0}C
Average specific heat capacity of graphite blended, C_G = 0.7076 J/gm^{0}C
Average specific heat capacity of calcium stearate, C_{CS} = 2.33 J/gm^{0}C
Average specific heat capacity of sodium stearate, C_{SS} = 2.4 J/gm^{0}C
Average specific heat capacity of calcium chloride, C_{CC} = 0.6571 J/gm^{0}C
Average specific heat capacity of NaCl, C_{NaCl} = 0.864 J/gm^{0}C
Average specific heat capacity of n-Hexane, $C_{n\text{-}Hexane}$ = 3.077 J/gm^{0}C
Average specific heat capacity of distilled water, C_w = 4.000 J/gm^{0}C
Average specific heat capacity of whole contents, C_{EWC} = 3.698 J/gm^{0}C
Initial temperature of emulsion whole contents, T_1 = 50 ^{0}C
Final temperature of emulsion whole contents, T_2 = 70 ^{0}C
Temperature change of emulsion whole contents, ΔT_{EWC} = 20 ^{0}C
Heat required to heat contents, $Q_8 = M_{EWC} X C_{EWC} X \Delta T_{EWC}$ = 5206980 J
= 52370 KJ
= 1.4464 kWH
Molar heat of evaporation of n-Hexane, H_{eva} = 28.9 KJ/mol
Moles of n-Hexane, n = 13.4 moles
Heat required for evaporation of n-Hexane, $Q_9 = n\ H_{eva}$ = 28.9 KJ/mol
= 387.26 KJ
= 0.1076 kWH
Net heat required in step xii-xiv, $Q_{10} = Q_{Rexn} + Q_8 + Q_9$ = -0.062+1.45+0.11
= 1.498 kWH

Energy balance across step xv-xvi;

Mass of RDX, M_{RDX} = 6825 gm

Mass of PIB, M_{PIB} = 11.669 gm

Mass of DIOS, M_{DIOS} = 18.354 gm

Mass of SAE-10 Oil, M_{SAE-10} = 4.977 gm

Mass of graphite blended, M_G = 35 gm

Mass of Calcium Stearate produced, M_{CSP} = 105 gm

Mass of Sodium Stearate unreacted, M_{SSU} = 1.6 gm

Mass of calcium chloride unreacted, M_{CCU} = 0.289 gm

Mass of NaCl produced, M_{SCP} = 20.2212 gm

Mass of distilled water, M_W = 62223.293 gm

Total mass of remaining whole contents, M_{RWC} = 69245.40 gm

Average specific heat capacity of RDX, C_{RDX} = 1.109502 J/gm^{0}C

Average specific heat capacity of PIB, C_{PIB} = 2 J/gm^{0}C

Average specific heat capacity of DIOS, C_{DIOS} = 0.0441 J/gm^{0}C

Average specific heat capacity of SAE-10 Oil, C_{SAE-10} = 1.964 J/gm^{0}C

Average specific heat capacity of graphite blended, C_G = 0.7076183 J/gm^{0}C

Average specific heat capacity of calcium stearate, C_{CS} = 2.33 J/gm^{0}C

Average specific heat capacity of sodium stearate, C_{SS} = 2.4 J/gm^{0}C

Average specific heat capacity of calcium chloride, C_{CC} = 0.6571228 J/gm^{0}C

Average specific heat capacity of NaCl, C_{NaCl} = 0.864 J/gm^{0}C

Average specific heat capacity of distilled water, C_w = 4.000 J/gm^{0}C

Average specific heat capacity of remaining whole contents, C_{RWC} = 3.71 J/gm^{0}C

Initial temperature of emulsion whole contents, T_1 = 70 ^{0}C

Final temperature of emulsion whole contents, T_2 = 25 ^{0}C

Temperature change of remaining whole contents, ΔT_{EWC} = 45 ^{0}C

Heat required to be removed, $Q_{11} = M_{RWC} X C_{RWC} X \Delta T_{RWC}$ = 11555760 J

= 11555.76 KJ

= 3.21 kWH

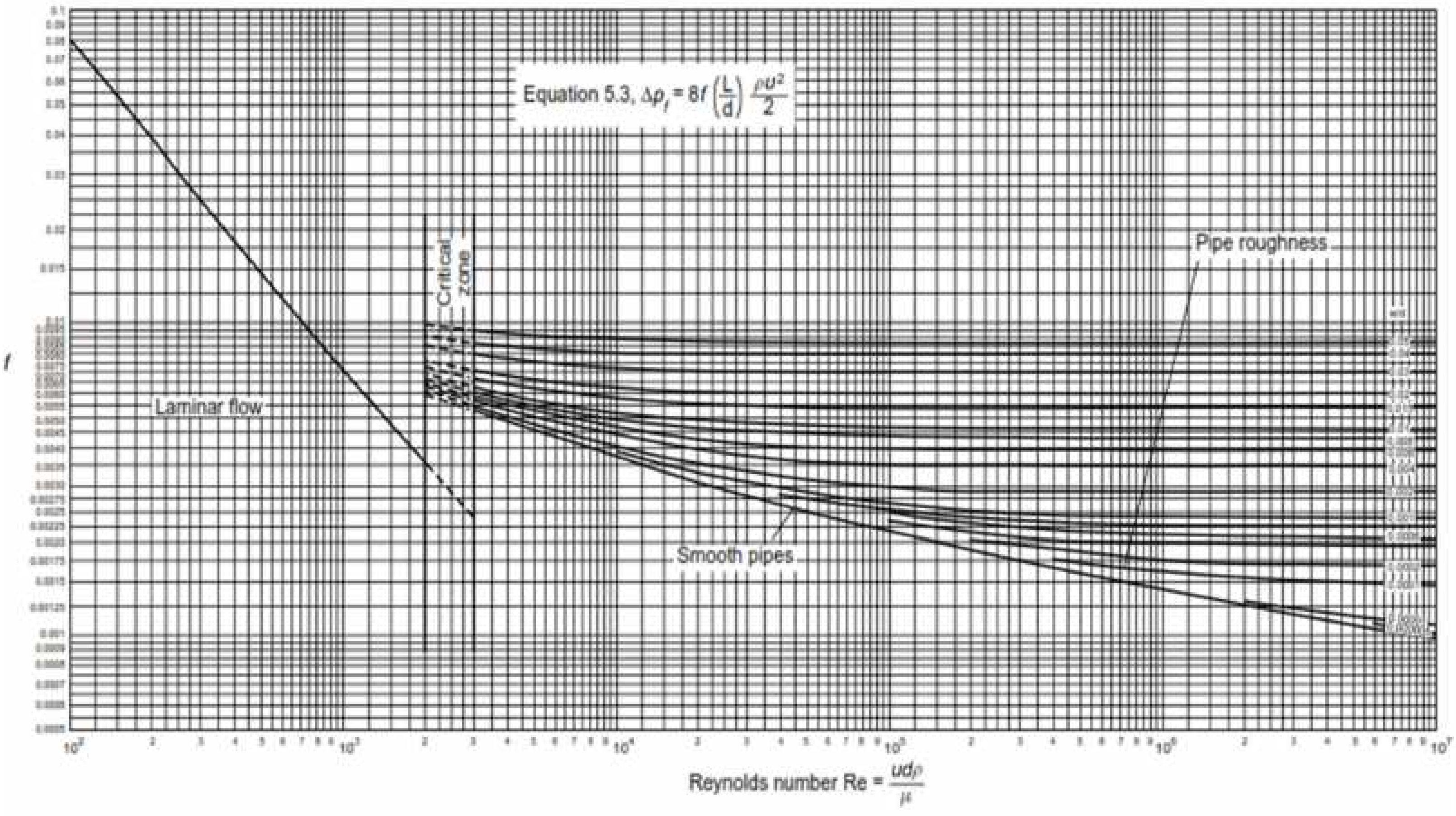


Figure 5.7. Pipe friction versus Reynolds number and relative roughness

R. K. SINNOTT, Coulson & Richardson's Chemical Engineering, Volume 6, 4th Edition, Chemical Engineering Design, p.203 (2005).

Annexure-D

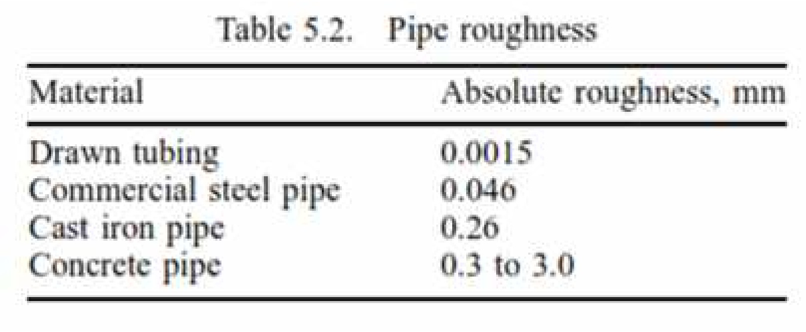


Table 5.2. Pipe roughness

Material	Absolute roughness, mm
Drawn tubing	0.0015
Commercial steel pipe	0.046
Cast iron pipe	0.26
Concrete pipe	0.3 to 3.0

R. K. SINNOTT, Coulson & Richardson's Chemical Engineering, Volume 6, 4th Edition, Chemical Engineering Design, p.202 (2005).

Annexure-E

Design of Water Tank Reservoir for Hot Water Supply

The volumetric flow rate required of water to be circulated in jacket of mixing vessel is 35.667 gallon (US)/min. therefore; water reservoir's capacity is selected two times of volumetric flow rate which comes out to be 70 gallon (US). This much capacity is sufficient to run the process in smooth manner.

Total heat load on jacket is 3.5 kWh (1.26 x 10^7 J). Mathematical relationship between heat and power required is given as follows:

$P = W/t$, where

Heat load (Joules), W = 1.26×10^7 J

Time of operation, t = 3600 s

Power required, P = 1.26×10^7 J / 3600 s = 3500 Watts

If we use two heaters of 1.75 kW for one hour the required heat load is supplied to our water reservoir as a whole. As we have to proceed in step wise manner to transfer heat to system in two steps of one hour each. Therefore, two heaters of 1 kW are more than sufficient to run operation in smooth and stepwise manner.

Dimensions of water reservoir for 70 gallon (0.265 m3) water tank reservoir with allowance of one third of capacity to be kept empty for evaporation and splashing during refilling of water tank reservoir are given as follows;. Therefore, capacity to be supplied is 0.353 m3 (93.2527 gallon).is given as follows:

Volume of water, V_w = 70 gallon
= $0.265\ m^3$

Allowance for evaporation and splashing, V_a = $V_w \times (1/3)$
= $0.265 \times 1/3$
= $0.0883\ m^3$

Water tank reservoir's volume, $V = V_w + V_a = 0.265 + 0.0883$ = $0.353\ m^3$

Hence , dimensions of water reservoir are ; L = 707 mm, W = 707 mm, H = 707 mm.

Annexure-F

Final Specifications of Equipment

Specifications of Mixing Vessel

Sr. No. Specifications

1. Capacity of mixing vessel is 98 liters (26 Gallon)
2. Volume of contents to be handled in vessel is 68 liters
3. Empty volume of mixing vessel to be supplied in extra is 30 liters
4. Diameter of mixing vessel is 457.2 mm
5. Height of mixing vessel is 555.45 + 74.725 = 629.92 mm
6. Height of liquid contents in vessel will be 457.2 mm
7. Thickness of mixing vessel is 7 mm
8. Material of construction of mixing vessel is SS-304
9. Teflon coating is required throughout mixing vessel
10. Thickness of Teflon coating required is 0.2 mm
11. Finish of mixing vessel will be mirror polished
12. Mixing vessel will be ASME dish bottom cylinder
13. Height added to mixing vessel due to ASME dish bottom is 74.725 mm
14. Mixing vessel will be sealed with collars having O-rings
15. Vessel having top cover for sealing
16. Top cover of vessel should be same as bottom of vessel depth wise i.e. 74.725 mm
17. Top cover having 2” connection for exhaust of vapours at centre of top cover
18. An elbow of 2” and 45^0 welded at 2” connection for exhaust vapours to condenser
19. Top cover having one thermo well up to center of liquid level towards agitator side
20. Top cover having agitator mounted through explosion proof gear mechanism
21. Top cover with agitator at off center position at an angle of $15\text{-}17^0$ with horizontal
22. Top cover having one feed point tube of 0.5” up to center of liquid level
23. Feed point tube at opposite side of agitator
24. Feed point will be in line with end center of impeller blade vertically downwards
25. Top cover with one reflux point tube of 0.5” for solvent up to center of liquid level
26. Reflux point will be towards feed point side of top cover
27. Top cover with 1” point for attachment to vacuum pump blinded with blind nut
28. Blind point will be located in between the feed point and exhaust point at center

29. Top cover fixed to hydraulic mechanism for its upwards and downwards movement
30. Hydraulic mechanism operate able from remote location up to 100’ distant
31. Top cover hermetically sealable with easy to remove fittings after every run
32. Vessel having tilt able mechanism to remove contents of vessel after every run
33. Vessel having provided with legs able to be grouted permanently
34. Thermo well having coated with Teflon 0.2 mm thick layer up to tip of well
35. Feed and reflux point tubes having coated with Teflon 0.2 mm thick layer up to end

Specifications of Jacket

Sr. No. Specifications

1. Heat load on jacket is 3.5 kWH
2. Spiral baffle welded on outer side of vessel peripherally up to bottom of vessel
3. Spacing between jacket and vessel wall is 75 mm
4. Clearance between vessel wall and spiral baffle 25 mm
5. Pitch is distance between two consecutive rounds of baffle vertically downwards and is 50 mm
6. Thickness of spiral baffle is 3 mm
7. Material of spiral baffle is SS-304
8. Number of rounds of spiral baffle around vessel is 11
9. Length of spiral channel is 16 m
10. Volumetric flow rate of water through jacket is 36 GPM
11. Inner diameter of jacket around vessel is 471.2 mm
12. Height of jacket around vessel is 552.45 +74.725 + 75 = 704.92 mm
13. Thickness of jacket is 7 mm
14. Outer diameter of jacket around vessel is 635.2 mm
15. Material of construction of jacket is SS-304
16. Polystyrene insulation is required throughout jacket
17. Thickness of insulation required is 2”
18. Encapsulation of insulation with 3 mm SS-304
19. Finish of jacket is mirror polished
20. Jacket will be ASME dish bottom cylinder
21. Height added to jacket due to ASME dish bottom is 74.725 mm
22. Jacket will be welded with vessel
23. Jacket having 2” top entering and 2” top leaving points for fluid in jacket

24. Jacket having 2” drain point for fluid in jacket at the bottom of jacket

Specifications of Impeller and Agitator

Sr. No. Specifications

1. Impeller type is marine propeller
2. Square pitch 1:1
3. Finishing of propeller is mirror polished
4. Coating of 0.2 mm thick layer of Teflon on propeller
5. Diameter of propeller is 152.4 mm
6. Height of center of propeller from bottom of vessel is 50.8 mm
7. Length of shaft of propeller is 457. Mm
8. Diameter of shaft is 19 mm
9. Material of construction of shaft and sleeve is SS-304
10. Teflon coating is required throughout shaft and sleeve
11. Thickness of Teflon coating required is 0.2 mm
12. Finish of impeller and sleeve is mirror polished
13. Impeller and agitator motor will be flameproof
14. Agitator will be installed at off-center position at an angle of 15-17^0
15. Mechanism of motion of agitator will be from remote location up to 100’ distance
16. 0.5 HP, single phase, ex-proof motor will be installed with agitator
17. Impeller RPM will be variable from 200-250 RPM through agitating mechanism
18. Agitator mechanism should be installed on separate bed

Specifications of Centrifugal Pump, Heaters, Fittings and Water Tank Reservoir

Sr. No. Specifications

1. Centrifugal pump flow rate is up to 40 GPM
2. Power of pump motor is 0.5 HP
3. NPSH required is 4.545
4. Two heaters of kW
5. 100’ length of pipe SS-304
6. One globe valve of dia 2”
7. Three 90^0 elbows, standard

8. Five gate-valves
9. Length of water tank reservoir is 707 mm
10. Width of water tank reservoir is 707 mm
11. Height of water tank reservoir is 707 mm