Feasibility of Re-Plasticization of Plastic Explosive (PE-3A) for Ammunition

By

Shoaib Ahsan

School of Chemical and Materials Engineering (SCME)

National University of Sciences and Technology

(NUST)

2020

Feasibility of Re-Plasticization of Plastic Explosive (PE-3A) for Ammunition

Name: Shoaib Ahsan

Reg.No: 00000281026

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

MS in Energetic Materials Engineering

Supervisor: 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"

DEDICATION

Dedicated to my beloved family

My parents, wife & kids

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

ABSTRACT

Explosives and ammunition being the precious commodity require proper care and preservation while in storage and transportation. PE-3A is an RDX based plastic explosive manufactured by Pakistan Ordnance factory. PE-3A Composition hardens at low temperatures and has undesirable volatility and hygroscopicity characteristics, therefore, in-situ re-plasticization of PE-3A is required at Ordnance Depots where the explosives is being stored to avoid loss of resources, safety and security issues related with transportation and handling to circumvent its routine re-plasticization at POF. The major advantages of re-plasticization of the composition is to increase the stability, consistent output pressure in the form of explosion while demolition. As this plastic explosive is RDX based, being sensitive it can be initiated by shock, impact and friction, therefore RDX crystals are coated with the mixture of plasticizing oil and paraffin liquid in the presence of lecithin which helps to coat RDX crystals evenly. Carbon black is also added to retain the RDX crystals for homogenity of the composition. Almost all the cases requiring re-plasticization observed at POF occurred due to the loss of three ingredients i.e. lecithin, paraffin liquid and plasticizing oil.

There are different methods of re-plasticization of PE-3A, one is conventional method which is in practice at Pakistan ordnance factory and cannot be adopted at Depot level as it is a full operational chemical plant and its commissioning and operational cost is very high. An alternate method has been devised in the present work while studying the whole process in Pakistan ordnance factory. The improvised method has been reported here comprises a mixer and rollers. Mixer is used to make the slurry of un-serviceable PE-3A through the addition of missing ingredients (Lecithin, Paraffin liquid and Plasticizing oil). Rollers are used to regain its plasticity, Rollers should be Teflon coated to avoid any unpleasant incident. Mixer thus designed can handle seven kilogram batch of explosive for re-plasticization at ammunition depot at a time. The upshot of this work is that a viable alternate technique for in-situ re-plasticization of PE-3A has been developed with low cost.

ACKNOWLEDGEMENTS

I am thankful to **Almighty ALLAH**, the creator of the beautiful world, the most beneficent and Merciful, for bestowing His blessings upon me, His never ending grace and His provision of countless help. I am highly obliged to the scholarly and erudite guidance of my supervisor **Dr. A.Q. Malik** whose immense command on the subject and vast experience proved like beckon of light to the present work. I would like to express my gratitude to all those individuals who supported us throughout from starting point to finishing line specially Dr. Sarah Farrukh and Dr. Tayyba Noor for their guidance, valuable and constructive criticism and friendly advice during the whole project.

I am very much thankful to Lt Col Ameer Mukhtar who gave me a lot of support and guidance for the research in Pakistan Ordnance factory and also my colleague Muhammad Suleman Khan, being a good scientist he always helped me out when it seems to be some difficulty.

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 family especially my mother and wife who motivated and supported me to complete my work otherwise it would not have been possible.

(SHOAIB AHSAN)

Table of Contents

Page

Chapter 1 Introduction

Chapter-2 Materials and Methods

Chapter-3 Experimental Techniques

Chapter-4 Design of PE-3A Pilot Plant

Chapter-5 Results and Discussion

LIST OF FIGURES

LIST OF TABLES

ABBREVIATIONS

PE	Plastic Explosive
RDX	Royal Demolition Explosive
SEM	Scanning Electron Microscopy
FTIR	Fourier Transform Infrared
HPLC	High Performance Liquid Chromatography
VOD	Velocity of Detonation
ANFO	Ammonium Nitrate Fuel Oil
AN	Ammonium Nitrate
AP	Ammonium Perchlorate
CDBs	Composite Double Base Propellants
DDT	Deflagration to Detonation Transition
DNT	Di-Nitro Toluene
DSC	Differential Scanning Calorimetry
EGA	Evolved Gas Analysis
EGD	Evolved Gas Detection
HE	High Explosive
HMX	High Melting Explosive
NC	Nitrocellulose
NG	Nitroglycerine
PETN	Pentaerythrite Tetranitrate
PSAN	Phase Stabilized Ammonium Nitrate
RDX	Research Development Explosive
POF	Pakistan Ordnance Factory
DDT	Deflagration to Detonation Transition
TNT	Trinitrotoluene

Chapter 1

General Introduction

1.1 Energetic Materials (EMs)

These are the materials having high amount of stored chemical energy, which can deflagrate or detonate accompanied with production of light, heat, sound and pressure. The rate of release of energy is a decisive factor. If the rate of release of energy is very fast, then it is termed as detonation (The wave travels in the material more than the speed of the sound); such energetic material is called High Explosives. Materials that deflagrate are called low explosives or propellants. Energetic materials are being utilized in a number of applications Energetic materials must be respected, being very sensitive by adhering to an SOP when using them. Energetic materials are in the form of solid, liquid or gaseous form containing high amount of chemical energy that can be released in very short time. EMs always require initiation by means of shock, mechanical, impact and thermal stimuli. Utilization of energetic materials is not only the domain of military but their huge utilization is also in the field of civil engineering, space exploration, mining, construction and rocket propulsion. [1][3]

1.2 High Energy Material

There is a difference between energetic material and high energy material; that difference is of rate of release of energy. In case it is too high than we can refer it as energetic material and if the rate of release is comparatively lower it is said to be high energy material e.g. Petrol has 7 times more energy than NG (nitroglycerine) but petrol is placed into the category of high energy materials whereas the NG is considered as energetic material because of the difference is in the rate of release of energy. [2]

1.3 Explosives

Such substances which can undergo exothermic chemical reaction at very high rate. The classification is based on its use, some explosives detonate (HE) and some of them deflagrate (propellants). Several parameters that might have great impact on classification of explosives are given below. [6]

1.3.1 Sensitivity

Sensitivity exhibit how explosives can be externally stimuli such as heat, spark, shock, friction and impact. On the basis of sensitivity explosive can be characterized into primary and secondary explosives. Primary explosives are more sensitive and can easily undergo deflagration to detonation transition as compared to secondary explosives. Secondary explosives are more powerful than the primary explosives. We can make an explosive train to make the advantage of primary as well as secondary explosives.

1.3.2 Heat of Explosion

Heat of explosion is the amount of heat that releases when decomposition of explosives take place during the process of explosion. This quantity is approximately equal to the difference between heat of formation of reactants and the products. Large heat of formation leads to higher explosive power.

1.3.3 Detonation Velocity

Detonation wave propagates with high speed and is directly related to the energy released by explosives. Detonation velocity depends upon packing density of the explosives in the column and it is correlated with detonation pressure. The shattering power can be a measure of brisance. Defined as the product of density and detonation velocity. Primary explosives have longer shelf life despite of their high sensitivities as compared to secondary explosives.

It is very important to have explosives of low sensitivity and high stability, storage and transportation capability which make an explosive easy to handle in bulk. Reproduction cost is another important issue which determine either any kind of explosive is practical or not. Complete classification of the explosives is given below in the form of table.

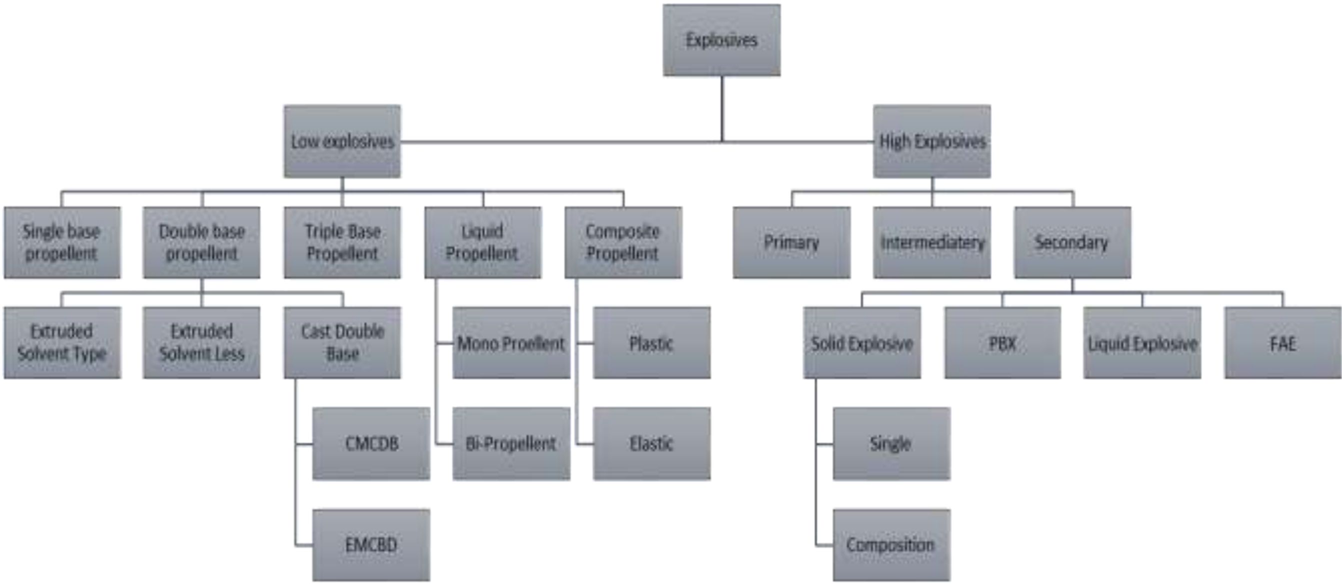


Fig 1.1 Classification of Explosives

1.4 High Explosive (HE)

High Explosive detonates when we initiate it with the production of pressure shock wave, can travel through any medium at supersonic speed. Depending upon the density and chemical properties detonation velocity varies from 3000 m/s to 9000 m/s e.g. detonation velocity of TNT 5870 m/s, RDX 8750 m/s and high melting explosive (HMX) 9100 m/s. These explosives can also be used in mining and for demolition purposes. On the basis of sensitivity high explosive are divided into two classes' primary explosives and secondary explosives. High explosives are being produced by different methods commonly by nitration process. Different additives such as stabilizer, plasticizers and curing agents are also incorporated during manufacturing process. For better shattering effect by the explosive it is necessary that explosive produce the shock wave greater than the speed of sound.

1.5 Low Explosives

Low Explosives can be classified on the basis of rate of reaction or decomposition. Their rate of decomposition is always less than the speed of sound. The shock wave produced in the high explosive is not there in this case as deflagration phenomenon occurs in the low explosives with the velocity ranging from few centimeters to 400 cm/s. When deflagration process commenced under confinement, we achieve more pressure and velocity as produced in the process of detonation. This process is also called deflagration to detonation transition (DDT).

Low explosive consists of fuel and oxidizer. Low explosive deflagrates where as high explosive detonate. The difference between these two is of rate of burning or decomposition. In military low explosive is mainly employed as propellants.

1.6 Propellants

Propellants have the capability to produce large volume of hot gases at controlled rate. All explosives on decomposition exhibit exothermic reactions. Energy released can be utilized in different applications where we needed, well controlled force for a relatively shorter period of time. Propellants are primarily employed to provide the working fluid for launching projectile from the guns, rockets and missiles system. Propellants may also be employed for various purposes e.g. in jet planes, turbines and pistons etc. [4]

1.7 Propellants Characteristics

Propellants are characterized by different properties given below. These properties are the deciding factors for the selection of the right propellant in the designing of any weapon where we are going to utilize propellants.

1. Propellant should have high density.
2. Good physical properties over operational temperature range.
3. Propellant should have low absorption of moisture.
4. Reproducible ignition qualities such as reasonable ignition over wide range of pressure.
5. High specific impulse.

6. Adjustable reproducible and predictable burning rates.
7. Burning rate exponent and temperature co-efficient should be small.
8. Safe, reproducible, low cost controllable and low hazard manufacturing of sample is required.
9. Good aging characteristics.
10. Not prone to combustion instantly.
11. Nontoxic exhaust gases.
12. Insensitive to certain energy stimuli.
13. Availability of the raw material.
14. Favorable history of prior applications.

1.8 Liquid Propellants

Liquid propellants consist of fuel component and oxidizer. Fuel and oxidizer stored in separate chamber and fed into combustion chamber to produce force/ thrust. Operationally liquid propellants are always complicated as compared to the solid propellants. These propellants serve the purpose to control the range of missile system because of achievement of high specific impulse, more restricted in use as compared to the solid propellants.

1.9 Solid Propellants

Solid propellants are highly utilized because of their great compactness, safety, handling and storability as compared to the liquid propellants. They are used in majority of the military weapons and other propellant actuated devices. The main ingredients of solid propellants are fuel and oxidizer. Other ingredients such as stabilizer, plasticizer, binder, curing agent and burn rate modifiers are added according to the requirement of the process/operations.

Fuel and oxidizer in some cases are mixed chemically and chemical connection established a new structure. In other cases, where both ingredients are mixed physically having no chemical linkage in their structure. First type called homogeneous propellant and other type is called heterogeneous propellants.

1.9.1 Single Base Propellants

These type of propellants contain nitrocellulose (NC) as main ingredient. Small quantities of stabilizers and other modifying agents are used in the formulation.

1.9.2 Double Base Propellants

These type of propellants contain nitrocellulose (NC) and nitroglycerine (NG) as major ingredients along with the stabilizers and moderators. They produce more energy as compared to single base propellants. Muzzle flash and excessive erosion of barrel caused by high gas temperature. It is the main drawback of double base propellants.

1.9.3 Triple Base Propellants

These contain nitrocellulose (NC), nitroglycerine (NG) and nitroguanadine as the main ingredients. They are as much powerful as double base propellants. The main advantage of triple base propellants is the reduction of hot gases and temperature. This is done by the third ingredient nitroguanadine.

1.10 Plastic Explosives (PE)

The first plastic explosive was invented by Alfred Nobel in 1875. Plastic explosives are the mixtures of finely powdered RDX and plasticizers. The mixture is putty like and can be moulded by hand to any desirable shape. Thus, plastic explosive is a soft and hand moldable solid form of explosive material. They consist of high brisance explosives such as RDX or PETN, plasticized with Vaseline or other plasticizers. Depending on the additives they contain, the PE are denoted as PE-1, PE-2 or PE-3, other components such as aluminum powder can also be incorporated. The products obtained can be of any desired size, and specified mechanical properties can be imparted to them, including rubber-like elasticity. Plastic explosives and plastic propellants are of great interest, if low thermal and impact sensitivity is needed. Some of the known plastic explosives are presented in Table-1 as follows; [7]

Table-1.1 Compositions of Different Plastic Explosives [5]

Composition	**Ingredients**	**% age**	**Role**
C-4	RDX	91.0	Demolition agent
	Di-(2-ethylhexyl sebacate	5.3	Plasticizer
	Polyisobutylene	2.1	Binder
	20-weight motor oil	1.6	Desensitizer
PE4	RDX	88.0	Demolition agent
	Pentaerythrite dioleate	1.0	Binder
	lithium stearate	2.2	
	paraffin oil BP	8.8	
PE7	RDX	88.0	Demolition agent
	2,3-dinitro-2,3-dimethylbutane (DMNB)	1.0	Taggant
	Hydroxyl-terminated polybutadiene	11.0	Binder
PE8	RDX	86.5	Demolition agent
	Di-(2-ethylhexyl sebacate	12.5	Plasticizer
	high molecular mass polyisobutylene		Binder
	2,3-dinitro-2,3-dimethylbutane (DMNB)	1.0	Taggant

1.11 Composition PE3-A

Composition PE3-A is quite extensively used as an explosive in ordnance items for demolition works by Pakistan Army in land services. It is black in color and greasy to touch. It is manufactured by Pakistan Ordnance Factories. The composition of PE-3-A is presented in Table-2 as follows;

Table-1.2 Composition of Plastic Explosive PE3-A

Composition	Ingredients	% age	Role
PE3-A	RDX	87.7± 0.5	Demolition agent
	Low Viscosity Oil	6.2 ± 0.5	Softener & Thinner
	Paraffin Liquid	4.1 ± 0.5	Binder & Plasticizer
	Lecithin	0.5 ± 0.2	Dispersant and sensitizer
	Carbon Black	1.5 ± 0.3	Desensitizer & colorant

1.12 Issues of PE3-A

The chemical stability of composition PE3-A is acceptable, but the physical stability is not entirely satisfactory. Composition PE3-A hardens at low temperatures and has undesirable volatility and hygoscopicity characteristics, so re-plasticization of PE3-A is required at Depot to avoid time constraints and security issues related with transportation of material back load to Pakistan Ordnance factory.

The chief advantages of re-plasticization of composition PE-3A is to increase stability, consistent output for a given size of charge and booster for transfer of detonation wave and safety to handle.

1.13 Objectives

Physical and chemical analysis of hardened PE-3A explosive.

The main objective of this research thesis is to re-plasticize the RDX based composition PE-3A at ammo depot keeping in view its safety, reliability and hence performance.

The third objective of this research is to design a pilot scale re-plasticization method of PE-3A composition. This has to be done as PE-3A is largely utilized explosive in Pakistan Army for demolition purposes. The third objective is to suggest this concept for plant scale operation.

Chapter 2

Materials and Compositions

2.1 RDX

It is also called as Royal Demolition explosives. It is a solid with white appearance without smell or taste. In modern world it is extensively used in making explosive. It has the molecular weight of 229 amu and having 37.84 % Nitrogen Contents. The VOD (velocity of Detonation) of RDX at 1.76 g/cm^3 density is 8750 m/sec. It starts to decompose when having temperature of 170 C$^\circ$ and melts at 204 C$^\circ$. Smaller in particle size results in more reactivity as we are increasing surface area and surface area is directly related to rate of reaction. [8]

It is very much stable at room temperature. It burns rather than explode. It only detonates when we make use of the detonator. This property makes it to be used for military purposes. RDX having figure of insensitivity of exactly 80.

Table 2.1 Properties of RDX

Sr. No.	Property	Specification
1.	Appearance	White crystalline powder
2.	Odour	Odorless
3.	Melting point	204.38 to 204.49 ^{o}C
4.	Retained on 100 Mesh	0.88 %
5.	Passed through 200 Mesh	------ %
6.	Size Range	
7.	Density	1.796 (gm/cm^3)
8.	Specific Heat Capacity	0.265 (cal/gm.K)
9.	Thermal Conductivity	$4.88 \pm 0.19 \times 10^4$ (cal/cm-s-°C)
10.	Viscosity of 10 % Water Slurry	0.00031–0.0111 (Pa-s)

Composition in which RDX particles are coated with wax are called Composition A. In mixture with TNT called companion B and blend with a non-explosive Plasticizer called Composition C. RDX is used in some base charge, blasting cap and also as an oxidizer**.**

2.2 Low Viscosity Engine Oil or Plasticizing Oil

In PE-3A manufactured in Pakistan Ordnance factory, 6% oil is used in composition as anti-wear protection agent. It makes the life of the explosive longer and thus ensuring max efficiency when subjected to high thermal stresses. This property minimizes decomposition or deformation that occur in Plastic Explosive when storage conditions are not feasible as it varies in different parts of the Pakistan. This oil usually produces extremely good lubricant stability even when subjected to high thermal or mechanical stresses. Mechanical stresses comes to an explosive while in transportation. It also prevents the composition from the moisture effect which results in oxidation and hence the reduction in shelf life of an explosive material. The oil possess density of 0.88 g/ cm^3 at 15˚C. They consist of minerals, semi or fully synthetic base oil plus amount of additives. The quality of low viscosity oil depends upon the additives. The low viscosity oil gives resistance against high temperature, yielding strength under compression and many more. Temperature impact the properties of the oil in explosive while it's paroled storage. [9] [10]

Table 2.2 Properties of Low Viscosity Oil

Ser. No.	**Property**	**Specification**
1.	Appearance	Dark brown
2.	Carbon Atoms	C_{18} to C_{34}
3.	Form	Liquid
4.	Melting point	ºC

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

2.3 Paraffin Liquid

In PE-3A manufactured in Pakistan Ordnance factory, Paraffin liquid used is approximately 4% which improves the handling Properties of the explosive e.g. brisance or detonation velocity etc. Paraffin liquid is a transparent, colorless nearly odorless oily liquid. It is composed of saturated hydrocarbons derived from the petroleum product. This oil is mainly composed of saturated alkanes. It is low reactive oil and insoluble in water. Paraffin oil has a wide range of industrial, medical and cosmetic use. A lot of noteworthy Utilization of liquid paraffin is as fuel and should not be used for the medical purposes. [12]

Paraffin liquid can have health hazards if it is inhaled or ingested because of repeated and prolonged skin exposure. Inhalation of paraffin oil can irritate the respiratory track causing cough, shortness of the breath and occasionally lead to hydrocarbon pneumatise. Prolonged skin exposure can cause skin disorder whereas ingestion causes upset of the intestinal tract. Paraffin oil which is not highly refined may be considered as cancer causing agent. Therefore, adequate precautions are required while using paraffin liquid. Ideally Paraffin oil should be stored in a cool, well-ventilated place and in a tightly closed container. As Paraffin being hydrocarbon are highly flammable. So, it should be kept away from the direct sunlight, impact and friction.

Paraffin oil is used as plasticizers and reduce swelling effect as well, should be handled and stored according to the instructions mentioned in security hazard classification.

Table 2.3 Properties of Paraffin Liquid

Ser. No.	Property	Specification
1.	Appearance	Colorless
2.	Average Molecular Weight	> 500 g/mol
3.	Carbon Atoms	> C_{28}
4.	Form	Oily Liquid
5.	Melting point[E]	-17.78 ^{o}C
6.	Density	0.88 gm/cm^3
7.	Boiling Point	>350 ^{o}C
8.	Distillation Point	B.P > 422 ^{o}C at the 5% distillation point
9.	Flash Point	136 ^{o}C
10.	Pour Point	-57 ^{o}C
11.	Specific Heat Capacity	1.67 KJ/Kg. ^{o}C
12.	Thermal Conductivity	1.04 W/m. ^{o}C
13.	Viscosity	11 cSt
14.	Polycyclic Aromatic Hydrocarbons	Passes Test

Paraffin Liquid is also a hydrating and cleansing agent. Hence it is used in several cosmetics both for skin and hair product. Lastly it is used as an ingredient of wipes. [13] [15]

2.4 Lecithin

In PE–3A manufactured in the Pakistan Ordnance factory Lecithin is approximately 0.5% of the total composition. Lecithin acts to prevent the formation of large Crystals of RDX. It also increases the sensitivity of the explosive because if the formation of large crystals exist than active surface area for the reaction will be less hence large crystals reduce the rate of reaction and prevent the formation of large crystals. Lecithin describe a group of fatty substances found in plants and animals tissues and it is essential for proper biological functions. Lecithin is commonly used in the preparation of foods, cosmetics and in medication. In explosives its use also extend the shelf life and act as an emulsifier. Lecithin attract both water and fatty substances and so are both hydrophilic and lipophilic. It is also used for smoothing food texture, emulsifying and homogenising liquid mixture and also repelling sticking material. Lecithin is present in a variety of biological matters including blood in human lungs, bile, human brain tissues, fish eggs, chicken and sheep brain. Lecithin can easily be extracted chemically using solvent such as hexane, ethanol, petroleum ether or benzene. It is usually available from sources egg yolk, marine sources, soya bean, rape seed, cotton seed and sunflower oil. It has low solubility in water but it is an excellent emulsifier. It also act as surfactants. It is used as food supplement and dietary additives. In cooking it is sometimes used as an emulsifier and to prevent sticking. If explosive have Lecithin than it exhibits good deal of power and it is relatively non-toxic. [16] [17]

Table 2.4 Properties of Lecithin

Ser. No.	**Property**	**Specification**
1.	Appearance	Brown
2.	Form	Viscous Liquid
3.	Melting point	-25 ºC
4.	Boiling Point	110-160 ºC
5.	Density	0.97 gm/cm^3
6.	Specific Heat Capacity	0.75 KJ/Kg. ºC
7.	Viscosity	61.5 Pa.S
8.	Thermal Conductivity	0.094 W/m. ºC

2.5 Carbon Black

In Plastic Explosive PE- 3A composition manufactured in Pakistan Ordnance factory, 1.5% carbon black is added to provide stronger and deeper colour. Carbon Black in pure form is a fine black powder, essentially composed of elemental carbon. Carbon Black is a specific type of elemental Carbon in the form of colloidal particles that is generated or produced through incomplete combustion. It consists of more than 96% of amorphous Carbon and having small quantities of Oxygen, Hydrogen, Nitrogen and Sulphur. Carbon black may contain 15 % of the oxygen. The type of the Carbon black that have a high specific surface and particularly is conductive. These conductive carbon black are used in antistatic finishing of plastics. In many applications of carbon black it is subjected to after treatment even in paint and varnish. It enhances the colour as well as the subsequent oxidation. The size of Spherical Particles which are formed are called "Particle size" and size of the particle claim is called Structure of Carbon black. Beside this various types of functional Groups such as hydroxyl and Carbonyl are attached to the surface of the Carbon black thus their composition is called "Surface Chemistry". Three terminologies which have been described earlier i.e. "Particle Size", "Structure" and "Surface Chemistry" are the basic properties of Carbon black. These three properties have large effect on particle properties such as blackness and dispensability when they are mixed with resins, paint or ink. Smaller the particle size higher the blackness will be achieved. However dispersion becomes difficult due to increase in Coagulation force. Carbon black with a larger structure shows excellent conductive properties. Carbon black with large amount of hydroxyl group given with oxidation treatment shows an excellent dispensability. Particle size refers to the individual particle size of the Carbon black, the smaller the particle size, greater will be the specific surface area. Structure shows the state of particles adhere to other of similar size and also indicate oil absorption. The greater the oil absorption more complex the configuration. By modifying the surface properties various functional groups combine with the surface of the carbon black, thus changed the compound Characteristics. If the distribution is sharp the size of the aggregates varies, this indicates that there are many aggregates of the same size. Carbon Black (Graphite) has the following properties: [14]

Table 2.5 Properties of Graphite (Carbon Black)

Ser. 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	1.95 gm/cm^3
8.	Specific Heat Capacity	2.03 Cal/degree mol
9.	Thermal Conductivity	142 W/m.K

2.6 n-Hexane

Its maximum utilization is because of its cheap relatively safe, easily evaporated, highly unreactive and non-polar in nature. Its main utilization is to extract vegetable oil from crops such as soya bean. We may utilize it as a cleaning agent in printing, textile, furniture, electronics and shoe making industries. A lot of consumer products contain n-Hexane e.g. gasoline, quick drying glues and rubber cement. n-hexane is a straight chain saturated hydrocarbons containing six carbons and fourteen hydrogen atoms. Its molecular formula is C_6H_{14} and its molecular mass is 86 gm/mol. Its structure is shown in the figure below: [19]

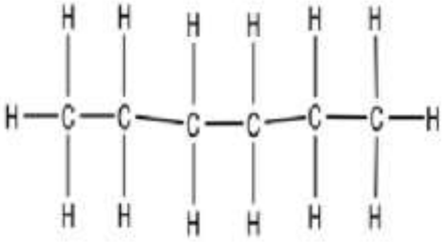


Fig. 2.1 n-Hexane Structure

Its main source is gasoline and it can also be produced as synthetic fuel through cracking of hydrocarbons. By refining of crude oil, hexane can be produced. The industrial product containing fifty percent by weight of straight chain isomers having boiling point 65-70 °C. It is colorless and has slightly disagreeable odor. It is highly flammable and its vapors can be explosive. [20]

Table 2.6 Properties of n-Hexane

Ser. No.	Property	Specification
1.	Appearance	Colorless
2.	Odour	Petrolic
3.	Molecular Formula	C_6H_{14}
4.	Molar mass	86.178 gm/mol
5.	State at Room Temperature	Liquid
6.	Melting point	−96 to −94 °C
7.	Boiling Point	68.5 to 69.1 °C
8.	Flash Point	−26.0 °C
9.	Explosive Limits	1.2–7.7%
10.	Auto ignition Temperature	234.0 °C
11.	Vapor Pressure	17.60 kPa (at 20.0 °C)
12.	Vapor Pressure	76.36 kPa (at 20.0 °C)
13.	Refractive Index	1.375
14.	Density	0.66 gm/cm^3
15.	Specific Heat Capacity	265.2 J/K.mol
16.	Thermal Conductivity	0.1297 W/m.K

17.	Viscosity	0.3 mPa·s

2.6.1 Experimental Work for Solvent

n-Hexane is a hydrocarbon with the chemical formula C_6H_{14}; that is, an alkane with six carbon atoms. Its boiling point is 68-69 ^{o}C and was removed rather easily at lower temperature without risking the process due to greater sensitivity of RDX at higher temperatures. It is widely used as economical, relatively safe, largely unreactive and easily evaporated non-polar solvent. It is also very safe from toxicity point of view.

n-Hexane has been selected as a solvent for RDX based composition due to low operational cost, low capital equipment cost, low energy requirement, low toxicity & greater safety for handling.

2.7 Distilled Water

Distill water can be defined as the water that boil into vapors and condense back into the liquid state in a separate container called distillate tank. Impurities having different boiling points than water can be separated and water is received through condenser. Schematic diagram is shown in the figure below:

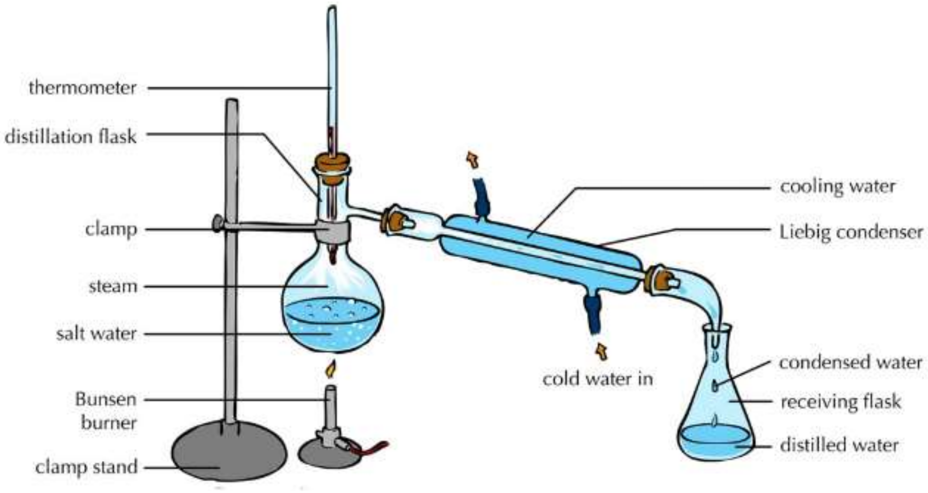


Fig 2.2 Schematic Diagram of Water Distillation Technique

Water is polar in nature having molecular formula H_2O and molar mass 18 gm/mol. Structure of water molecule is shown in the figure below

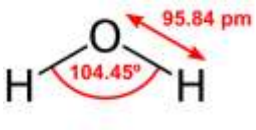


Fig 2.3 Structure of Water Molecule

Distilled water is used in industries, chemical and biological labs. 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 the ingredients are used one by one. The properties of distilled water are given below: [21]

Table-2.7 Properties of Distilled Water

Ser. 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 °C
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

2.8 Method of Manufacture of PE-3A in Pakistan Ordnance Factory

Dry RDX is fairly sensitive high explosive; it is too much sensitive to be used alone as a filling. In the manufacturing of PE-3A, crystals of RDX required to be desensitised are coated with mixture of liquid paraffin and shell oil. Beside this Lecithin is also used as a Wetting agent. It also performs the function of evenly distribution of coating materials to RDX crystals. Carbon black is also added in small quantity which helps to retain the oil coating. When Plastic explosive is manufactured it would be in such a state that it can be moulded to any required form which fulfil the purpose.

It is already mentioned that Lecithin serve the purpose of evenly distribution of the mixture of Paraffin oil and Shell oil on the RDX Crystal surface. So it is very pertinent that specification of these two must be tested and approved before utilization as it causes variation in plasticity from one batch to another.

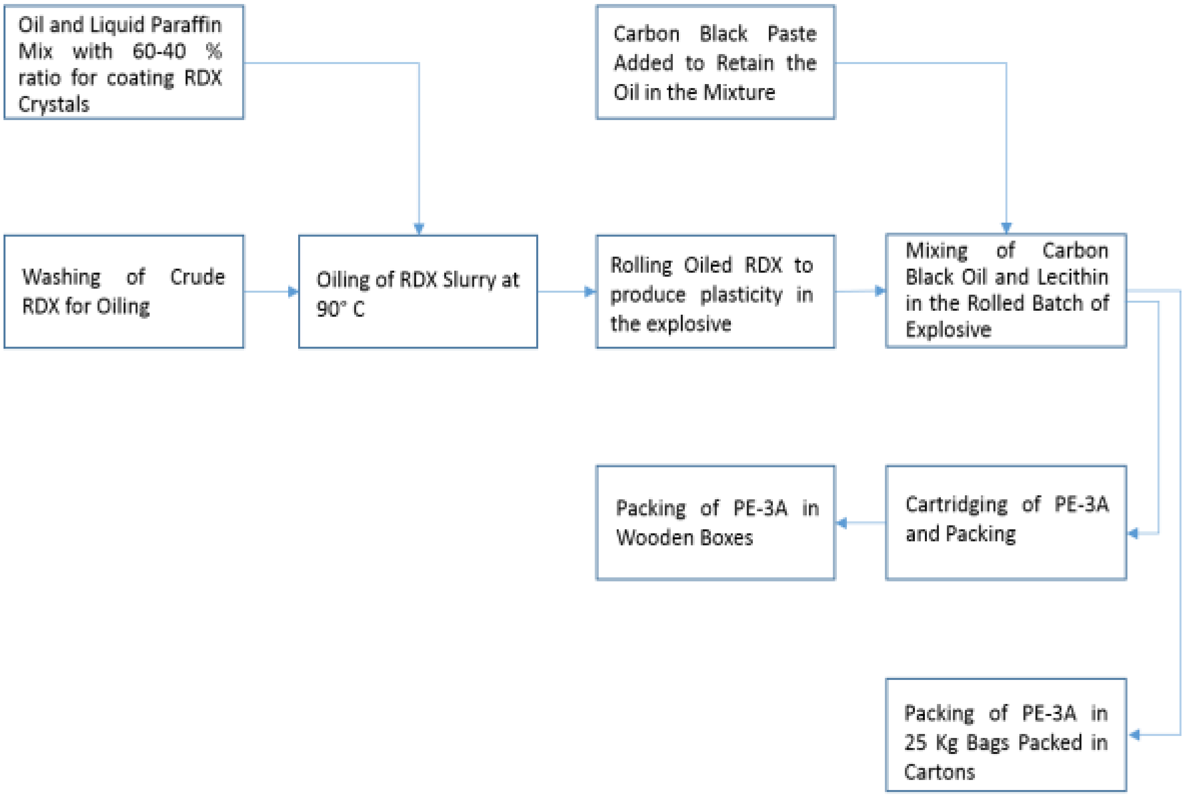
Second most important thing that should be considered is the particle size of the RDX crystals. Sometimes it happens that the RDX crystals combine to form aggregate as combination of these crystals reduces the effective surface area resultantly the rate of chemical reaction is reduced. For this purpose, RDX crystal happens to pass from a sieve of mesh number 200. If it is less than 62% than RDX Crystals are good for Plastic explosive.

It is an important factor which affect directly the plasticity of the Plastic explosive. It can be over-rolling or it can be under-rolling. Both results in low plasticity beside this roll clearance and spring compression is very important. Plasticity has been found minimum after just adding lecithin because of a "flopping" but continuous mixing results in steady rise of plasticity. Plasticity also depends on the PH of the oiling liquor. If RDX is oiled in acidic liquor than plasticity will be less and if it is neutral the result will be vice versa.

First of all slurry is made by adding water to RDX and stirred continuously. Than it is heated to 90 ˚C by steam. Add oil and Lecithin to the slurry and continue stirring. Batch should not be over oiled or under oiled rather oil can be adjusted in early stage. Slurry is continuously stirred until small RDX pellets are formed. Than slurry is cooled to about 50 ˚C by the addition of cold water and discharge oiled RDX for further utilization. Oiled RDX rolled is between two water cooled phosphor bronze rollers for several time until good plasticity can be achieved. Each roll can apply the force of 2000 lb by the compressed spring.

At the end oiled RDX comes to steel pan where further carbon black is added, which retain the oil in the RDX crystals. Mixing continues in this until the required product is obtained in which all the ingredients are well mixed and retained. At the end Stage the prepared PE-3A is converted into required shape (cartridge). This Cartridge is 8 inch long with the weight of 8 oz. It is wrapped and packed with greaseless paper and stored in the storage locations.

Fig 2.4 Flow Chart of PE-3A Production



2.9 Method of Re-Plasticization of PE-3A at POF

As it is in our Knowledge that Chemical stability of PE-3A is acceptable but Physical stability of PE- 3A is not satisfactory as it hardens at low temperature and have undesirable hygroscope characteristics so re-plasticization is required which is done keeping in view the following steps.

- Wetting in mixer to make it soft with the help of steam.
- Sampling to check the composition.
- Rolling to achieve plasticity.
- Addition of deficient oil and mixing.
- Sampling for Plasticity.
- Cartridging on automatic machine
- Packing.
- Detonation test.
- Despatch.

Plastic explosive hardens because of the plasticity reduced to min level. To retain plasticity back the missing ingredients are added. A sample taken to determine the composition. Add wetting agent (Lecithin) and plasticizing agents (Paraffin liquid, plasticizing oil). Put it into the mixer for the purpose of homogeneous mixture. PE-3A hardens in majority of cases because of lack in plasticity. Perform the process of rolling as described earlier.

We utilize the Lecithin as a wetting agent. It also serves the purpose of evenly distribution of the mixture of Paraffin liquid and Plasticizing oil over RDX crystals and Carbon black helps to retain mixture of paraffin liquid and plasticizing oil in the crystals of RDX. After this process again take the sample to find out Chemical Composition if it is satisfactory automatic Cartridging process becomes in contact followed by packing and despatched for reuse at the respective locations.

2.10 Method of Analysis of PE-3A in Laboratory

When the hardened PE-3A was analysed than the first question which arises is that what are the conclusions made from the analysis, what kind of deficiencies are there, how we can get rid of that deficiencies to make it able to be re-used for the required purpose. There are two kinds of analysis Physical analysis and Chemical analysis. For physical analysis, Visual Contact with the Sample of 50 g of hardened PE-3A gives the conclusion that it is hardened. To find out the reasons behind, composition analysis have to be performed. So, according to the original manufacture of PE-3A 50 g of sample should contain the following chemical composition.

Table 2.8 Table of 50 gm Sample Tested in Laboratory

Ingredients	Chemical Composition by Original Manufacturer	Amount in 50g Sample
RDX	87.7 %	43.40 Grams
Plasticizing Oil	6.2 %	3.10 Grams
Paraffin Liquid	4.1 %	2.50 Grams
Lecithin	0.5 %	0.25 Grams
Carbon Black	1.5	0.75 Grams

To perform this experiment, 50 gm sample was taken in the beaker and slurry was made by adding 950 ml distilled water. Slurry was formed because of the continuous stirring and the temperature was maintained at 50°C for better mixing and results. Then all the ingredients were separated and weighed to find out the deficiency. For separation of ingredients the solvent was added which can dissolve the specific chemical in the composition. n-hexane was added drop by drop. n-hexane dissolved Lecithin, paraffin liquid and plasticizing oil and because of the density difference two separate layers were formed. One was of water slurry containing the R.D.X crystal and Carbon black and other was of n-hexane containing Lecithin, Paraffin liquid and Plasticizing oil. By difference in their boiling points they all were separated to find their percentage composition. RDX and carbon black in Slurry form was obtained. By evaporating water powder form of RDX Plus carbon black was obtained. RDX was segregated by dissolving it in acetone. As RDX Crystals dissolved in acetone leaving the residues of the carbon black at the bottom which were separated by using filter paper. Now by getting all the ingredients % age composition was obtained and then the deficiency was traced. In the above experiment the Composition had deficiency in the Lecithin and Plasticizing Oil.

2.11 Step Wise Experimental Work for Lab Scale Re-Plasticization

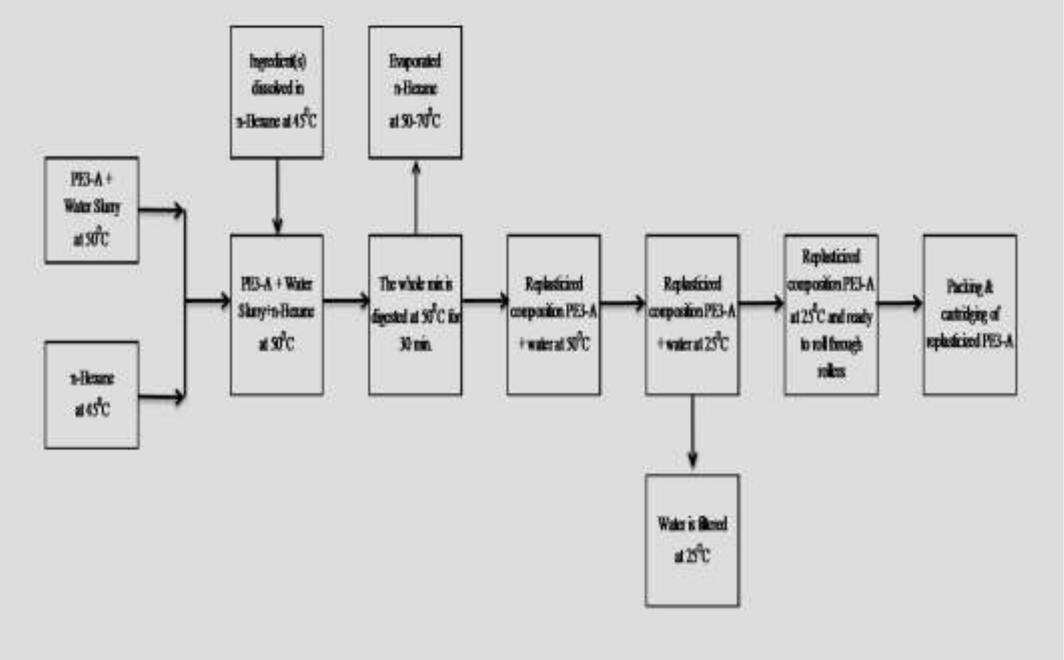
The process developed for lab scale preparation of composition contains following steps;

i. First of all take the PE3-A to re-plasticize in required batch size. Prepare a 10% PE3-A water slurry containing 10% PE3-A and 90% water by weight. The slurry is heated up to 50 ^{0}C. Add n-Hexane in required proportion to dissolve the lecithin, plasticizing oil and paraffin liquid of PE3-A. Keep stirring through Teflon coated ex-proof marine propeller mixer and heat contents up to 50^{0}C. Lecithin, plasticizing oil and paraffin liquid in PE3-A is dissolved in n-Hexane.

ii. Dissolve required lecithin in n-hexane and heat up to 45^{0}C. Add solution of lecithin to PE3-A-water slurry slowly and maintain temperature at 50 ^{0}C. Digest the lecithin n-Hexane solution in PE3-A-water slurry emulsion at 50 ^{0}C for 15 min.

iii. Dissolve required plasticizing oil and paraffin liquid in n-Hexane and heat up to 45^{0}C. Add solution of the plasticizing oil and paraffin liquid to RDX-water slurry slowly. Maintain the temperature of emulsion at 50 ^{0}C. The whole mix is digested at 50 ^{0}C for 30 min.

iv. Distil n-hexane from digested emulsion mixture. The distillation range of n-hexane is from 50-70 ^{0}C. Distilling n-hexane from emulsion leaves lecithin, plasticizing oil and paraffin liquid coated on grains of RDX. The whole mixer is cooled to room temperature and filtered. The filtrate is passed through brass rollers and is rolled to achieve plastic properties and evaporate water from mixer. Hence re-plasticized composition PE3-A is achieved. The process flow diagram for process developed is presented as follows;



Chapter 3

Experimental Techniques

3.1 Introduction

The surface characterization is the most powerful means to quantify surface under investigation. Now a days, there are various techniques developed to seal down the surfaces at the Nano level. Flaws detection is very much necessary to prevent the greater damages at the end of any process. While thinking explosive being a precious commodity if there is any deformation or wear and tear occurring before completing its shelf life than these valuable characterization techniques give us the reason behind these deformation or determination. To be more specific if we are considering PE-3A explosive, it happened to be deteriorated because unsuitable storage conditions. At depot level if we want to sort it out than characterization techniques which are given below can help us what type of deficiencies exist in compositions of PE-3A to re-utilize the most important and precious commodity i.e. explosive. These characterization techniques include:

- Scanning Electron Microscopy
- Fourier Transform Infrared Spectroscopy
- High Performance Liquid Chromatography
- Velocity of detonation
- Sensitivity Test
- Differential Scanning Calorimeter
- Melting Point

3.2 Scanning Electron Microscopy

There is some resolution power of human eye i.e. 0.2 mm and if we add a lens it can be reduced between 0.1-0.2 mm, beyond this if some more resolution needed than some magnification equipment will be required. Microscopes are used for such purposes; its resolution power depends on the quality and the quantity of the lenses which are in use but also depends on the source to produce image. Therefore, on the basis of the source the microscope can be divided into two categories.

- Optical Microscope (OM)
- Electron Microscope (EM)

In electron microscope, a beam of highly accelerated electrons is focused on a surface of material, which in terms helps to resolve atomic feature ranging from nanometer to micrometer particle size.

A scanning electron Microscope scans a focused electron beam over a surface to create an image. The electrons in the beam interact with the sample, producing various signals that can be used to obtain information about the surface topography and composition. [24]

Fig 3.1 Scanning Electron Microscope

3.2.1 Working Principle

A scanning electron microscope scans a beam of the electrons over a specimen to produce a magnified image of an object. Electrons from the beam hit the surface of the object and bounce back. A detector registers these scattered electrons and turns them to a picture. We use scanning electron microscopy to produce various signals that can be used to obtain signal and tell us about the surface topography. The compositions accelerated electrons carry significant amount of kinetic energy and this energy dissipated as a variety of signals produce by electron interactions.

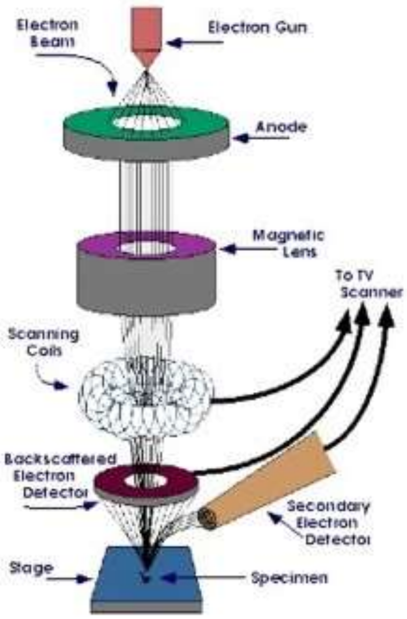


Fig 3.2 Working principle of SEM

Above all scanning electron microscopy analysis provide high resolution imaging useful for evaluating various materials for surface fractures, flaw, contamination or corrosion.

Magnification of scanning electron microscopy is about 300000 times; therefore, it provides qualitative information of the specimen including its topography, morphology composition and crystallographic information. In other words, it provides the information about surface feature, texture, shape, size and arrangements of the particles lying on the surface of the sample. Thus, SEM is multipurpose instrument with ability to examine the material with high resolution.

3.3 Fourier-Transform Infrared Spectroscopy (FTIR)

Fourier-Transform Infrared Spectroscopy is a technique used to obtain an infrared spectrum of absorption or emission of a solid, liquid or gas. An FTIR spectrometer simultaneously collects the high spectral resolution data over a wide spectral range.

Fig 3.3 FTIR

This confers a significant advantage over a dispersive spectrometer which measures intensity over a narrow range of wavelengths over a time. [25]

The term Fourier-Transform Infrared Spectroscopy originates from the fact that a mathematical process is required to convert the raw data into actual spectrum.

3.4 High Performance Liquid Chromatography (HPLC)

HPLC is a technique in analytical chemistry used to separate, identity and quantify each component in the mixture. It depends upon the pumps to pass pressurized liquid solvent containing the sample mixture through a column filled with a solid absorbent material. Each component in the sample interact slightly differently with the absorbent material, causing different flow rates for different materials thus leading the separation of the components as they flow out of the column.

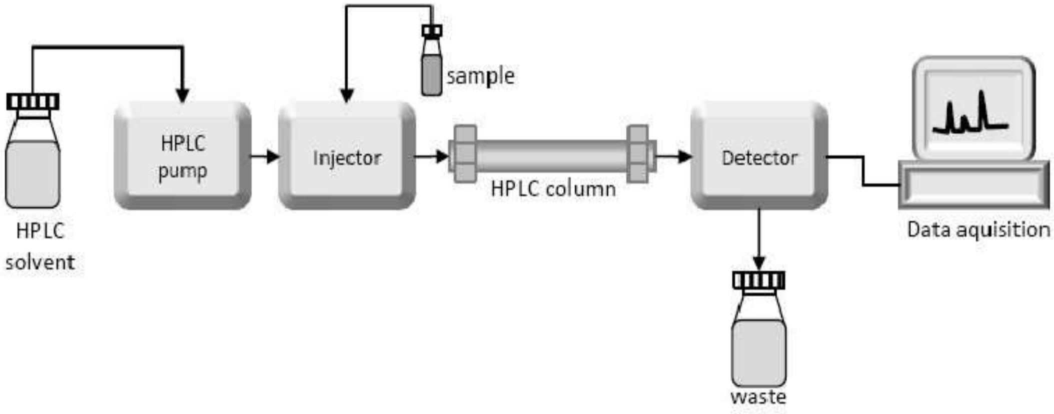


Fig 3.4 HPLC

Variation in HPLC is that it can be normal phased or reversed phase. In normal phase HPLC the chemical structure linked to the solid support within the column are more polar than the solvent. In reversed phase HPLC the solvent is more polar than the chemical structures of the solid support. [27]

HPLC is most powerful of all the chromatographic technique. It can easily often be a separation and analysis that could be difficult or impossible by other forms of chromatography. [28]

3.5 Velocity of Detonation (VOD)

The rate at which detonation wave travels through an explosive column is called the velocity of detonation. All the explosives can be characterized by their properties such as strength, density, viscosity, composition and VOD. Hence, velocity of detonation is one of the most important features of the explosive as pressure generated by an explosive is directly proportional to VOD. It also decides the performance of the explosives in real time. A reduction in VOD will result in decrease in pressure and hence the performance of the explosive reduces and resultantly availability of the shock energy will also reduce. It is important that explosives detonate at its optimum rate and induces its sufficient optimum pressure to produce fragmentation. Detonation velocity of an explosive can be used to calculate the impedance of an explosive which is defined as the product of density and the velocity of an explosive.

Confinement of an explosive results in more intensity of the velocity as happened in the arty shell. Explosive velocities are always greater than the velocity of sound in the material and if we provide confinement, it always results in more pressure. In this aspect particle size matters a lot, smaller particle size means more surface area available for reaction that is why if we keep on decreasing particle size than as a result rate of reaction will continue to increase. Solid explosive often has the detonation velocities ranging beyond 4000m/s to 10300m/s. The confined detonation velocity measures the speed at which the detonation travels through a column of explosive within a confined place. The unconfined velocities indicate this relation when explosive is detonated in the operation. Explosives are generally used under some degree of confinement.

3.6 Sensitivity Test

In case of an explosive sensitivity is the key factor. Explosive should have moderate sensitivity such that it can bear small impact and friction while its storage and transportation. Reliability and sensitivity are two different terminologies; more sensitivity means lesser reliability. There are different parameters that fulfill the criteria of sensitivity. In these tests, the most common are given below:

- Impact test
- Frictional test

- Electrostatic discharge test
- Thermal sensitivity

This test allows user to determine 50% initiation level and standard deviation from the initiation. In other words it gives us the distance at which 50% of the sample will “go”. Basically, this is impact testing method. In this procedure fixed weight used to be dropped from a known distance to a sample of explosive. The distance and weight calculation gives us sensitivity by using different method. One is given above.

Explosives may be tested by frictional test by using several techniques, ultimately gives us the sensitivity of that particular explosives. ABL Technique is most commonly used, in it we utilize line of explosive on a prepared metal plate, placed in front of specially prepared metal wheel that is forced down on the plate with a hydraulic press. Initiation is determined hence, sensitivity determined.

Electrostatic discharge occurs when differently charged objects are brought closer. In other words, it is a sudden flow of the electricity between two electrically charged objects caused by contact. ESD can be harmful in case of explosives. ESD can be measured by a machine design to discharge from the capacitor through a prepared sample.

Thermal sensitivity is determined by a point at which a compound is capable of detonating under confinement. In this a fixed quantity of the explosives placed in an aluminum blasting cap shell and then pressed to determine the time for detonation. In this way we can determine the temperature at which an explosive can detonate.

3.7 Differential Scanning Calorimeter (DSC)

This technique is based on the temperature. Basically its thermo analytical technique in which difference in the amount of heat required to increase the temperature of the sample and reference is measured as a temperature function. Both the sample and the reference are going to be maintained at the same temperature throughout the experiment. In DSC analysis, sample holder temperature increases linearly as a function of time. There are two types of DSC.

- Power- compensated DSC in which power supply remains constant.
- Heat-Flux DSC in which heat flux remains constant.

In DSC we have different curves as a function of the temperature. These curves can be used to calculate enthalpies of the transitions. The enthalpy transition can be shown using following equation:

$\Delta H = KA$

$\Delta H = Enthalpy\ of\ the\ Transition$

$K = Calorimetric\ Constant$

$A = Area\ under\ the\ curves$

Thermal safety testing may also be performed via differential scanning calorimetry in which a small sample is placed in a sample cell and temperature is increased slowly. The calorimeter determines how much energy is required to increase the temperature of the sample. Using this device, we can find out characteristics such as melting point, phase transition and decomposition temperature of an explosive**.** The power-compensation DSC has two nearly identical measuring cell, one for the sample and one for the reference holder. Both cells are heated with different heaters, their temperatures are measured with separate sensors. The temperature of both cells can linearly increase as a function of the time. Small temperature difference occurring in both cells due to exothermic/endothermic effects of the sample recorded as a function of the temperature.

3.8 Melting Point

Explosives can be manufactured, destroyed, stored and transported even in the case we know how it reacts, in other words we have to come across the characteristics of the explosives. The most important and decisive characteristic is the melting point e.g. if the explosive has low melting point than its storage is always a problem because if that temperature can be achieved while storage as it normally happens than explosive can be initiated. So, we can store such explosive in temperature control igloo or make composition that can raise its temperature. Melting point is significant property of the energetic materials especially for melting cast explosive. In the melting process the structural transition from ordered arrangement to molecules is observed. Melting point determines the molding approach of explosive.

Melting point is determined by Thomas Hoover Capillary Melting Point Apparatus using a heating rate of approximately 2^{o}C/min. consisting of oil bath with electric stirrer, light weight heating element controlled by autotransformer built in capillary vibrator. Normally 100ml glass beaker used as a melting point bath.

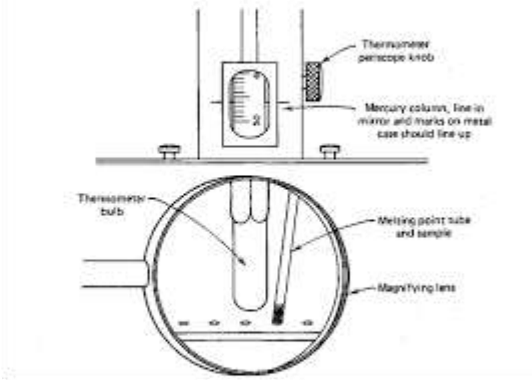


Fig 3.5 Capillary Vibrator

Second method is of Thiele tube, which is a laboratory glassware designed to contain heat and an oil bath. The shape of the Thiele tube allows for formation of the convection currents in the oil when it is heated. This current maintains a uniform temperature distribution throughout the oil in the tube. A sample with sealed capillary attached to the thermometer with a rubber band is immersed in a tube. Heating is commenced and the temperature ranges at which the sample melts can be observed.

Chapter 4

Design of PE-3A Pilot Plant

4.1 Material Balance

PE-3A is basically RDX based explosive because RDX present in the explosive is of 87.7 % by weight. In this chapter efforts are being made to design a pilot scale. Re-plasticization/Production plant with the production capability of 7 Kg composition in one batch. The material balance for pilot scale plant can be carried out as follows;

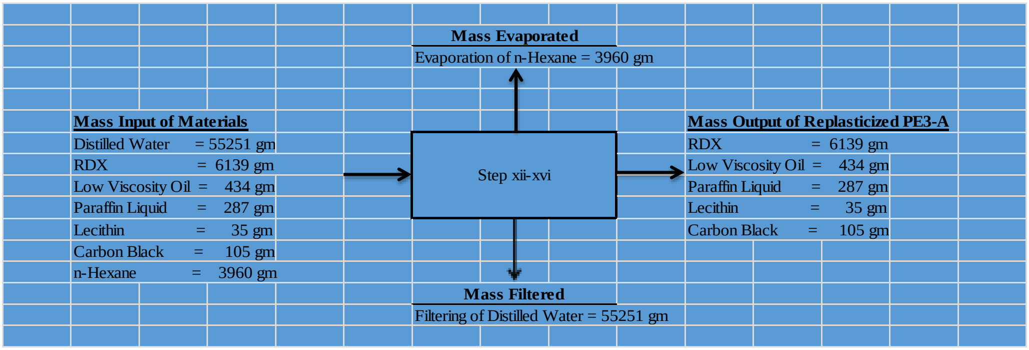
Basis of Calculation: 7000 gm of PE3-A

Table 4.1 Composition of PE3-A

Ser. No.	Ingredient	% (w/w)
1.	RDX	87.7± 0.5
2.	Low Viscosity Oil	6.2 ± 0.5
3.	Paraffin Liquid	4.1 ± 0.5
4.	Lecithin	0.5 ± 0.2
5.	Carbon Black	1.5 ± 0.3
6.	n-Hexane Required	6000 mL

i. RDX Required = 7000 X .877 = 6139 gm
ii. Distilled Water Required = (90/10) x 6139 = 55251 gm
iii. Low Viscosity Oil Required = 7000 X 0.062 = 434 gm
iv. Paraffin Liquid Required = 7000 X 0.041 = 287 gm
v. Lecithin Required = 7000 X 0.005 = 35 gm
vi. Carbon Black Required = 7000 X 0.015 = 105 gm
vii. N-Hexane Required = 6000 X 0.66 = 3960 gm
viii. The overall material balance is given as below:

Fig 4.1 Material Balance



Mass Evaporated
Evaporation of n-Hexane = 3960 gm

Mass Input of Materials
Distilled Water = 55251 gm
RDX = 6139 gm
Low Viscosity Oil = 434 gm
Paraffin Liquid = 287 gm
Lecithin = 35 gm
Carbon Black = 105 gm
n-Hexane = 3960 gm

Step xii-xvi

Mass Output of Replasticized PE3-A
RDX = 6139 gm
Low Viscosity Oil = 434 gm
Paraffin Liquid = 287 gm
Lecithin = 35 gm
Carbon Black = 105 gm

Mass Filtered
Filtering of Distilled Water = 55251 gm

ix. Hence 7000 gm of PE3-A composition are produced.

4.2 Energy Balance

Energy balance of RDX based explosive composition PE-3A on pilot scale has been described here. The pilot scale production plant is based on 7 Kg of explosive composition PE-3A. The energy balance around production steps as mentioned in process developed for lab scale preparation can be carried out as follows;

Basis of Calculation: 7000 gm of PE-3A

4.3 Overall Energy Balance

Mass of RDX, M_{RDX} = 6139 gm

Mass of Low Viscosity Oil, M_{LVO} = 434 gm

Mass of Paraffin Liquid, M_{PL} = 287 gm

Mass of Lecithin, M_{L} = 35 gm

Mass of Carbon Black, M_{CB} = 105 gm

Mass of n-Hexane, $M_{n\text{-}Hexane}$ = 3960 gm

Mass of distilled water = 55251 gm

Total mass of emulsion of whole contents, M_{EWC} = 66211 gm

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

Average specific heat capacity of Low Viscosity Oil, C_{LVO} = 1.964 J/gm^{0}C

Average specific heat capacity of Paraffin Liquid, C_{PL} = 1.67 J/gm^{0}C

Average specific heat capacity of Lecithin, C_{L} = 0.75 J/gm^{0}C

Average specific heat capacity of Carbon Black, C_{CB} = 0.70762 J/gm^{0}C

Average specific heat capacity of n-Hexane, $C_{n\text{-Hexane}}$ = 3.07735 J/gm^{0}C

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

Average specific heat capacity of emulsion whole contents, C_{EWC} = 3.642355 J/gm^{0}C

Initial temperature of emulsion whole contents, T_1 = 25 ^{0}C

Final temperature of emulsion whole contents, T_2 = 70 ^{0}C

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

Heat required to heat mix to 70 ^{0}C, $Q_1 = M_{EWC} X C_{EWC} X \Delta T_{EWC}$ = 10852378 J

= 10852 KJ

= 3.014 kWH

Molar heat of evaporation of n-Hexane, H_{eva} = 28.9 KJ/mol

Moles of n-Hexane, n = 46 moles

Heat required for evaporation of n-Hexane, Q_2 =n H_{eva} = 28.9 KJ/mol

= 1328 KJ

= 0.370 kWH

Overall heat required heat required, $Q_3 = Q_1 + Q_2$ = 3.014 +0.370

= 3.38 kWH

4.4 After Distillation Heat Removed

Heat removed from n-Hexane, Q_2 =n H_{eva} **=** 0.370 kWH

Heat Required to be Removed from Remaining Mix to Cool to 25^0**C**

Mass of RDX, M_{RDX} **=** 6139 gm

Mass of Low Viscosity Oil, M_{LVO} = 434 gm

Mass of Paraffin Liquid, M_{PL} = 287 gm

Mass of Lecithin, M_L = 35 gm

Mass of Carbon Black, M_{CB} = 105 gm

Mass of distilled water = 55251 gm

Total mass of mix, M_M = 62251 gm

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

Average specific heat capacity of Low Viscosity Oil, C_{LVO} = 1.964 J/gm^{0}C

Average specific heat capacity of Paraffin Liquid, C_{PL} = 1.67 J/gm^{0}C

Average specific heat capacity of Lecithin, C_L = 0.75 J/gm^{0}C

Average specific heat capacity of Carbon Black, C_{CB} = 0.70762 J/gm^{0}C

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

Average specific heat capacity of mix, C_M = 3.637 J/gm^{0}C

Initial temperature of mix, T_1 = 70 ^{0}C

Final temperature of mix, T_2 = 25 ^{0}C

Temperature change of mix, ΔT_M = -45 ^{0}C

Heat required to cool mix to 70 ^{0}C, $Q_4 = M_M X C_M X \Delta T_M$ = -10189640 J

= -10190 KJ

= -2.83 kWH

Heat Load on Vessel, Q_{Load} = 3.5 kWH

Mass of Low Viscosity Oil, M_{LVO} = 434 gm

Mass of Paraffin Liquid, M_{PL} = 287 gm

Mass of Lecithin, M_L = 35 gm

Mass of Carbon Black, M_{CB} = 105 gm

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

4.5 Mass of Solid Components

RDX = 6139 gm

Carbon Black = 105 gm

Total mass of solid phase components in emulsion, M_s = 6244 gm

4.6 Mass of Liquid Components

Low Viscosity Oil = 434 gm

Paraffin Liquid = 287 gm

Lecithin = 35 gm

n-Hexane = 3960 gm

H_2O = 55251 gm

Total mass of solid phase components in emulsion , M_L = 59967 gm

% Slurry or % Solid Contents in Slurry, B = 100 x (M_S/M_L) = 100 x 6244/59967

= 100 x 0.10412

= 10.412 %

Volume of Emulsion Whole Contents

Total mass of emulsion contents, M_{EWC} = 66211 gm

Density of emulsion whole contents, ρ_S (Kg/m^3)

Component	**Mass (gm)**	**Density ρ (Kg/m^3)**	**Wt. Density ρ_w (gm/cm^3)**
Water	55251	0.997	= 0.831965187
RDX	6139	1.796	= 0.166522844
LVO	434	0.88	= 0.005768226
PL	287	0.88	= 0.003814472
Lecithin	35	0.97	= 0.000512755
CB	105	1.95	= 0.003092386
n-Hexane	3960	0.66	= 0.039473803
Density of slurry, ρS			= 1.051149673
Total mass of emulsion contents, M_{EWC}			= 1051 Kg/m^3

Volume of whole contents, $V_{EWC} = M_{EWC}/\rho_S$ = 66211/(1.0511x1000)

= 63 liters

4.7 Equipment Design

Equipment design based on RDX based explosive composition PE-3A has been described here. The pilot scale plant is based on 7 Kg batch of explosive composition PE-3A. The equipment design of pilot plant for process developed for composition is described as follows.

4.8 Design of Jacketed Mixing Vessel System

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

4.9 Sizes of Impeller and Vessel

For the purpose of re-plasticization continuous mechanically stirred vessel is required which is heated by steam and make a well homogeneous slurry to take the sample which on further analysis of that homogeneous slurry give us the exact deficiencies. The total capacity required for all the contents in the recipe production is about 68 liters. [29] [30]

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 = $\left(\frac{\pi D^2}{4}\right) \boldsymbol{X\ H}$ = 75021945 mm^3

= 75.021945

Liters

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) $\simeq$ 100.0 Liters

Height of vessel for final design is = $\left(\frac{4V}{\pi D^2}\right)$ = 609.42 mm

$\simeq$ 24 inch

Specification of Sizes of Impeller and Vessel

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

4.10 Selection of impeller

Impeller Types: which can circulate the liquid axially and can achieve primarily redial circulations.

Impeller Speed

Standard impeller speeds are 37, 45, 56, 68, 84, 100, 125, 155, 190, and 320 RPM. [31]

4.11 Selection of Flow Pattern Mechanism

Tanks less than 1.8 m in diameter, axial-flow impeller without baffles should be used for a wide range of process requirements. The volume of liquid should not exceed 4 m^3.

4.12 Critical Speed Calculation

According to Zwietering’s correlation based on data for five types of impellers in six tanks from 6 inch to 2 feet diameter used for complete suspension of composition. The critical stirrer speed is given by the dimensionless equation; [33]

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

Critical stirrer speed, $\boldsymbol{Nc} = S \text{ X } v^{0.1} \text{ x } Dp^{0.2} \text{ x } g \text{ x } (\Delta\rho/ \rho_L) \text{ x } B^{0.13} \text{ x } 1/D_a^{\,0.85}$, 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 = 973 Kg/m^3

Density of slurry or suspension phase, ρ_S = 1051 Kg/m^3

Density difference of phases, $\Delta\rho = \rho_S - \rho_L$ = 78 Kg/m3

Kinematic viscosity of suspension, $\nu = \mu/ \rho_S$ = 0.7/1051

= 0.000666 m^2/s

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

Gravitational acceleration, g = 9.8 m/s^2

B = 10.412%

$\boldsymbol{Nc} = 6.5 \text{x} 0.000666^{\,0.1} \text{x } (75\text{x}10^{-6})^{0.2} \text{ x } 9.8\text{x}(78/ 973) \text{ x } (10.412)^{0.13} \text{ x } 1/0.15240^{0.85}$

= 2.47 RPS=148 RPM

The critical speed of the impeller comes out to be 148 RPM for just off-bottom suspension of the composition. The RPM range for design should be 50% greater than the critical speed which is ~ 222 RPM. A variable speed drive motor having range of 200-250 rpm is sufficient for the mixing and granulation process of composition.

4.12.1 Factors

Following factors should be considered while selecting the type of jacket.

Cost

A simple jacked has been selected with spiral baffles to reduce the cost of the equipment.

Heat Transfer Rate

A spirally baffled jacket for high rates of heat transfer has been selected.

Pressure

The pressure rating of the design is taken as up to 10 bars for simple spirally baffled jacket. [35]

4.12.2 Design of Jacket

Physical Properties of Vessel Side Contents

Initial temperature, T_i = 25 ^{0}C
Final temperature, T_f = 70 ^{0}C
Average temperature = $(T_i + T_f)/2$ = (25 + 70) / 2 = 47.5 ^{0}C
Viscosity of slurry or suspension phase, μ = 0.7 Kg/m.s
Specific heat capacity of emulsion whole contents, C_{EWC} = 3.64 J/gm^{0}C
Thermal Conductivity of emulsion whole contents, k_{EWC} = 0.764 J/m.s.^{0}C
Density of emulsion whole contents, ρ_S (Kg/m^3) = 1051 Kg/m^3

Properties of Jacket Side Fluid

Initial temperature, T_i = 85 ^{0}C

Final temperature, T_f = 80 ^{0}C

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

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

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

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

Density of water, ρ_w = 970 Kg/m^3

Heat Transfer Co-efficient of Vessel Side (h_i)

Impeller Reynolds Number or Vessel Side Reynolds Number (N_{Re})

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

Critical stirrer speed, N_C = 148 RPM = 2.467RPS

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

Density of slurry, ρ_S = 1051 Kg/m^3

Viscosity of slurry, μ = 0.7 Kg/m.s

N_{Re} = 2.467 x 0.15242 x 1051 / 0.7 = 599

Vessel Side Prandtl Number (N_{Pr})

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

Specific heat capacity, C_{EWC} = 3.64 J/g.^{0}C

Viscosity, μ = 0.7 Kg/m.s

Thermal conductivity, k_{EWC} = 0.764 J/m.s.^{0}C

Npr =3.64 x 1000 x 0.7/0.764 = 3.33

Vessel Side Nusselt Number (N_{Nu})

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

Vessel side Reynolds number, N_{Re} = 599

Vessel side Prandtl number, N_{Pr} = 3.33

Nu = 0.54 X$599^{0.67}$ X $3.33^{1/3}$ = 58.55

Vessel Side Heat Transfer Co-efficient (hi)

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

Diameter of vessel, D = 0.4572 m

Nusselt number, N_{Nu} = 58.55

Thermal conductivity, k_s = 14.4 J/m.s.^{0}C

h_i = N_{Nu} x k / D= 58.55 x 14.4 / 0.45720 = 1844 W/m^2.^{0}C

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 the jacket, n = 552.45 / 50 = 11

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

Hydraulic mean diameter, D_e

$D_e = \left(\frac{4 \text{ x S x P}}{2(S+P)}\right) = \left(\frac{4 \text{ x } 0.075 \text{ x } 0.050}{2(0.075+0.050)}\right) = \left(\frac{0.015}{0.25}\right)$ = 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

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

Jacket Side Reynolds Number (N_{Re})

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

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

Jacket Side Prandtl Number (N_{Pr})

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

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

Vessel side Reynolds number, N_{Re} = 101364

Vessel side Prandtl number, N_{Pr} = 2.16245

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

Jacket Side Heat Transfer Co-efficient (h_j)

$h_j = N_{Nu} \text{ x } k / D_e$, 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 $W/m^2.{}^0C$

= 3346 $W/m^2.K$

Thermal conductivity of steel at average temperature, k = 16 W/m.K

Thickness of vessel wall, $x = 10$ mm $= 10 \times 10^{-3}$ = 10^{-2} m

Fouling factor for jacket side fluid (steam condensate), h_{dj} = 1500 $W/m^2.K$

Fouling factor for vessel side due to complete cleaning after every run i.e. $h_{di} = 0$

Overall Heat Transfer Co-efficient (U)

$1/U = 1/h_i + 1/h_j + 1/(k/x) + 1/h_{di} + 1/h_{dj} = \frac{1}{1844} + \frac{1}{3346} + \frac{10^{-2}}{16} + 0 + \frac{1}{1500}$

U = 469 $W/m^2.K$

Minimum Time for Heat Transfer (t)

Temperature Difference of Hot Fluid, $(\Delta T) = T_2 - T_1$ = 80 – 85

= $-5\ {}^0C$

Minimum time for required heat to be given off, $Q_s = (m/s)\ C_p\ \Delta T$ = 2.1825x4.19x1000x5

= 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

Log Mean Temperature Difference (LMTD)

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

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

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

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

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

Temperature Difference of Vessel Fluid $(\Delta t) = t_2 - t_1$ = = 45 ${}^0C$

70 – 25

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

$\frac{50)}{-1.79176}$

Time to Heat Contents of Jacketed Vessel (θ)

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

$\theta = ln\frac{(T1-t1)}{(T1-t2)}$ x x $\frac{(M \text{ x } Cp \text{ x } k)}{WC(k-1)}$), 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} = 145.199 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 , = 82.63Btu/ft^2.h.^{0}F

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

Area in contact through periphery, $A_1 = \pi$ x H_L x H_L = 3.14x1.5x1.5

= 7.065 ft^2

Area in contact with vessel through floor, $A_2 = \pi xD^2/4$ = 3.14 x 1.5^2/4

= 1.76625 ft^2

Total heat transfer area, A = $A_1 + A_2$ = 7.065 + 1.76625

= 8.83125 ft^2

$k = e^{(UA/WC)} = e^{((82.63)(8.83125)/(17321.7)(1.00327)}$ = 1.042884

$\theta = ln\frac{(T1-t1)}{(T1-t2)}$ x $\frac{(M \text{ x } Cp \text{ x } k)}{WC(k-1)}$)

$\theta = ln\frac{60}{15}$ x $\frac{(145.199\text{x } 0.884 \text{ x } 1.042884)}{17321.7 \text{ x } 1.00327 \text{ x } (1.042884-1)} = ln\frac{4}{1}$ x 0.1796 = 1.3173 x 0.1796

= 0.2366 hours

= 0.2366 x 60

= 14.196 min

Pressure Drop Through Jacket (ΔP)

At N_{Re} of 101364, $j_f = 2.8 \times 10^{-3}$

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

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

$\Delta P = 8\, j_f \times L \times \rho w \times u^2/2D_e = 8 \times 2.8 \times 10^{-3} \times 16 \times 970 \times 0.6^2/(0.06 \times 2) =$ 1043 N/m^2

Power Transmitted By Impeller Shaft (P)

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

Power Number, Np = 0.8

Critical stirrer speed, N_C = 2.467 RPS

Diameter of impeller or agitator, Da = 0.1524 m

Density of vessel contents at average temperature, ρ_s = 1051 Kg/m3

$P = N_p N_C^3 Da^5 \rho_S = 0.8 \times 2.467^3 \times 0.1524^5 \times 1051$ $\simeq$ 1.04 watt

4.13 Mechanical Design of Vessel

4.13.1 Design Pressure

This is taken 10 percent above the normal working pressure, to avoid spurious operation during minor process upsets.

4.13.2 Design Temperature

The design temperature at which the design stress is evaluated is taken as the 150 ^{0}C.

4.13.3 Design Stress (Nominal Design Strength)

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).

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 average 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

$Ps = Pa + \rho_W\, g\, H_L$ =101325+1036.676 x 9.8 x 0.4572=101325+4645 = 105970 N/m^2

= 15.3696 Psi

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

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 = 116567 x 0.4572 (2 x 225 x 10^{-6} – 116567)= 53294.4324/44983433	=	1.146x10^{-4} m
	=	0.12 mm

4.14 Corrosion Allowance

It is the additional thickness added to metal for the material lost by corrosion. The allowance to be used is 2.0 mm.

4.15 Minimum Practical Wall Thickness

Minimum wall thickness required for any vessel to withstand its own weight, minimum wall thickness of vessel taken from table is 5 mm; the value includes a corrosion allowance of 2 mm.

Static Elevation Change (∆Z)

Static Elevation Change (∆Z) = Z_2-Z_1 is taken as 3.5 ft. or 1.0668 m

Pipe Length and Fittings

Ser.No.	Valve or fitting	Nos.	Loss (K)
1.	Sharp entrance	01	0.50
2.	Open globe valve (2 inch)	01	6.00
3.	90 0 elbow, standard	03	2.25
4.	Gate- valve half open	05	22.50
5.	Sharp exit	05	22.50
6.	Sum of additional friction loss,	32.25	

eq. no. of velocity heads, K

Velocity of water (V)

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, $\boldsymbol{A = \pi D^2}/4$ = = 0.002193544 m^2

$\boldsymbol{A = 3.14\ x\ 0.0508^2}/4$

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

Reynolds Number of Water in Pipeline

$\mathbf{N_{Re}}$ **=** D V ρ_W **/** μ , 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

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

From Appendix-E 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})

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}

=

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

$\simeq$ 3 m

Head Loss Through Jacket (H_L)

$\Delta P = \rho_W \, g \, H_L$, 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, $H_L = \Delta P / \rho_W \, g = 1043 / (970 \text{ x } 9.8)$ = 0.11 m

Total Head of System Required (H_T)

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

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

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

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

Pump 0.5 HP motor is more than sufficient for circulation of water for heating system.

Net positive Suction Head (NPSH) Required

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

Sin friction factor, f_s (assumed) = 0

Gravitational acceleration, g = 9.8 m/s^2

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

4.16 Design of water tank reservoir for hot water supply

4.16.1 Capacity

The volumetric flow rate required for 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.

4.16.2 Power of Heater

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 x 10^7 J

Time of operation, t = 3600 s

Power required, P = 1.26 x 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.

4.16.3 Dimensions of Water Reservoir

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 x (1/3)

= 0.265 x 1/3

= 0.0883 m^3

Water tank reservoir's volume, V = V_w + V_a = 0.265 + 0.0883 = 0.3533 m^3

Hence , dimensions of water reservoir are ; L = 353 mm, W = 353 mm, H = 33 mm.

Chapter 5

Results and Discussion

5.1 Results and Discussion of Characterization of Solvents

n-hexane was used as a solvent in solvent extraction because it can be recovered easily. It has non polar nature and above all it has low latent heat of vaporization. Because of this property it was utilized as a solvent in RDX based plastic explosive composition PE-3A. There were other solvents available for RDX based explosive but to avoid the risk of exposing RDX composition to the higher temperature n-hexane was selected. n-hexane when used as a solvent in PE-3A it dissolved the components e.g. Lecithin, plasticizing oil and Paraffin liquid. n-hexane when dissolved, these components could be distilled easily because it evaporates at low temperature. n-hexane has the refractive index of 1.3769, this value is measured by refractometer at 20^o C. Another solvent I used for re-plasticization of PE-3A is acetone as it dissolves the RDX crystals to separate it from carbon black. Acetone is a good solvent as it dissolves polar and non-polar substances means it can dissolve both organic and inorganic substances. Experiments were performed on number of solvents the results of each solvent is given below:

Table 5.1 Solvent Index at 40° C

Solvent	**Flashpoint °C**	**Paraffin Dissolved Per 100 gms Solvent**
Pentane	< - 48	8.94
Cyclohexane	- 22	8.04

Heptane	- 4	9.26
n-Hexane	- 20	22.18

5.2 Results and Discussion of Visual Analysis, Rolling and Moulding

PE-3A is a soft and mouldable explosive within its shelf life. As time passes its plasticity reduces because of many reasons including major cause of storage conditions, resultantly it becomes hard and non-mouldable. The hardened PE can be visually analysed. Investigations revealed that this is the beginning of deformation as it produces cracks in the plastic explosive PE-3A. This will affect the propagation of the shock wave.

Fig 5.1 Sample PE-3A

After visual analysis all the samples were observed on scanning electron microscopy and results are given below in Fig 4.2 and illustration crystal shape and sizes. The crystals of RDX in composition PE-3A ranges in size from a few micrometres to a few millimetres. Some of the large crystals were extracted with tweezers from sample and scraped clean of binder. Experiments were performed on four samples named A, B, C and D and tested for its plasticity, moulding and rolling capabilities. Sample A lot was freshly manufactured and its elasticity was excellent whereas sample B, C and D had less elasticity than sample A. All samples when subjected to equal stretching force for plasticity than sample A broke after 3 cm, sample B broke after 1.5 cm, sample C broke after 0.73 cm and sample D was brittle enough could not pass it. Result is shown in tab 4.2. Rolling is important as it affects directly plasticity by distributing the additives homogeneously. If number of rolling increases than plasticity increases but a stage came when plasticizing ingredients started to leave and stuck with rollers. Hence optimum number of rolling is required to achieve maximum plasticity. Optimum number of rolling is five with 2.5 ft. diameters of rollers applying 2000 lbs. force, result is shown in tab 4.2

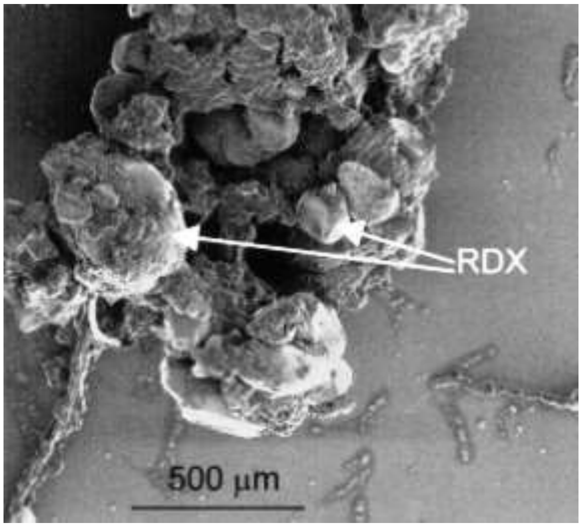


Fig 5.2 SEM image of sample PE-3A

Plastic explosives are soft and mouldable, to apply moulding test a circular pipe shaped mould was devised with 5x cm length and 8x cm diameter. All the samples were put in it one by one and force was applied with circular shaped piston. Sample A was mouldable and 99.2 % recovered from mould, sample B recovered 95 % and could not adopt completely the shape of mould, sample C recovered 91 % approximately and was less mouldable than sample B. Sample D was non mouldable.

Table 5.2 Stretching and Rolling of Different Lots of PE-3A

Ser No.	Explosive Type	Stretching with equal Force for Moulding	Equal no of Rolling	Conclusion
1.	PE-3A (A)	Soft Material	Achieve Good plasticity	Serviceable Explosive
2.	PE-3A (B)	Hardened Material	Plasticity is less	Unserviceable Explosive
3.	PE-3A (C)	Very Rigid	Very less plasticity	Unserviceable Explosive
4.	PE-3A (D)	Brittled	No plasticity	Unserviceable Explosive

For sample evaluation the sample was observed in metallurgical microscope and surfaces of all four samples were observed. The main difference lies in disengagement of the surface, in sample A, there was no disengagement and number of spots were very less, in sample B, C and D there were more disengagement and no of spots respectively.

5.3 Results and Discussion of Composition Analysis Technique

When the hardened PE-3A was analysed than the first question which arises is that what are the conclusions made from the analysis, what kind of deficiencies are there, how these deficiencies can be managed to make it able to be re-used for the required purpose. There are two kind of analysis Physical analysis and Chemical analysis. For physical analysis, Visual Contact with the Sample of 50 g of hardened PE-3A gives the conclusion that it is hardened. To find out the reasons behind Composition analysis have to be performed. So, according to the original manufacture of PE-3A 50 g of sample should contain the following chemical composition.

Table 5.3 Table of 50 gm Sample Tested in Laboratory

Ingredients	**Chemical Composition by Original Manufacturer**	**Amount in 50g Sample**
RDX	87.7 %	43.40 Grams
Plasticizing Oil	6.2 %	3.10 Grams
Paraffin Liquid	4.1 %	2.50 Grams
Lecithin	0.5 %	0.25 Grams
Carbon Black	1.5	0.75 Grams

To perform this experiment, 50 gm sample was taken in the beaker and slurry was made by adding 950 ml distilled water. Slurry was formed because of the continuous stirring and the temperature was maintained at 50°C for better mixing and results. Then all the ingredients were separated and weighed to find out the deficiency. For separation of ingredients the solvent was added which can dissolve the specific chemical in the composition. n-hexane was added drop by drop. n-hexane dissolved Lecithin, paraffin liquid and plasticizing oil and because of the density difference two separate layers were formed. One was of water slurry containing the R.D.X crystal and Carbon black and other was of n-hexane containing Lecithin, Paraffin liquid and Plasticizing oil. By difference in their boiling points they all were separated to find their percentage composition. RDX and carbon black in Slurry form was obtained. By evaporating water powder form of RDX Plus carbon black was obtained. RDX was segregated by dissolving it in acetone. As RDX Crystals dissolved in acetone leaving the residues of the carbon black at the bottom which were separated by using filter paper. Now by getting all the ingredients % age composition was obtained and then the deficiency was traced. In the above experiment the Composition had deficiency in the Lecithin and Plasticizing Oil.

It is observed with great concern that during the process of re plasticization particle size f RDX become very small because of continuous mixing as a result we are unable to obtain our required result of detonation. In that case we have to characterize RDX by using differential scanning calorimeter (DSC). In other case if RDX is not desensitized homogeneously when coated it with mixture of paraffin liquid, plasticizing oil and lecithin than it fails to achieve its required viscosities and Fourier transform infrared (FTIR) is performed in Ordnance Depots to overcome this problem.

5.4 Results and Discussion of Feasibility of Re-plasticization of PE-3A at Ammunition Depot

PE-3A being the demolition explosive is highly utilized in Pakistan Army. It is used in bulk that is why its quantity cannot be utilized during its shelf life and remain in storage. Sometimes storage conditions are not ideal for this kind of explosive resultantly deformation or deterioration take place at higher rate and explosive loses its softness, ability of moulding and plasticity before its shelf life. Such Explosives cannot serve the purpose because of multiple reasons e.g. propagation of shock wave phenomenon is hindered because of appearance of the cracks in explosive body, these cracks appear just for a reason of the explosive hardness. Secondly detonation velocity achieved is less than the expected one etc. Such explosives are loaded back to Pakistan ordnance factory for the purpose of re-plasticization and this is a burden in terms of cost and handling because it involvess loading/unloading charges of explosives, labour charges, charges of the fuel of the vehicles involved in this transportation of explosives, wear and tear of the vehicles involved in transportation and individuals have to be spared for security of the convoy beside this explosive being the dangerous commodity , lot of risk is involved in its transportation. To overcome these problems, need of the hour is to establish some mechanism for re-plasticization of PE-3A explosives in depots. After having detailed study of the process of re-plasticization any one of the following process can be used.

1. Conventional method of re-plasticization.
2. Non-conventional method suggested for re-plasticization.

When conventional method is discussed, it involves a lot of investment to build a plant for re-plasticization as in Pakistan ordnance factory. In conventional method pumps, valves, heat exchangers, evaporator, condensers, mixers etc. have to be designed and it will be very costly. This whole procedure of manufacturing and re-plasticization is discussed in chapter 2 with all necessary details. Beside all this if conventional method is applied it requires skilled labour which can operate a full scale plant and lots of other requirements like its shut down and cleaning activities. If pumps are not working than requirement of a skilled mechanic having at least mechanical diploma in subject field and if there is some problem in DCS system, an electrical diploma holder will be required. So a great arrangement of resources will be required to fulfil the purpose of re-plasticization at ammunition depot. So, cost effectiveness analysis for re-plasticization of PE-3A at Ammo Depots by applying conventional method is not suggested.

Non-conventional method is devised and suggested to adopt at ammunition depot for re-plasticization of PE-3A explosives. Plastic explosives in almost all the cases become hardened because of absence of three ingredients as observed in Pakistan ordnance factory. These are Lecithin, paraffin liquid and plasticizing oil. By adding these missing ingredients we can have our explosive back in action and can be utilized for the required purpose. The procedure involved is very much elaborated in chapter 2. So, a mixer was designed (chapter 5) which can handle a batch of 7 kilograms of plastic explosive at a time and a brass or copper roller having Teflon coating to avoid any initiation of the explosive through impact, friction and any other means while rolling process. This rolling and mixing result in gaining of plasticity and thus re-plasticization of PE-3A can be done at depot level. This cost effective method definitely saves the Army resources as well as danger involved while handling and transportation of the explosives. This small unit can be ran by labours already available at depot by training them through run a cadre as they already know how to handle and give respect to ammunition.

Conclusion

An explosive is the form of material that develops with the world progress; new formulations and techniques are being introduced in the international market with enhanced performance, economical cost with better safety to lesson threat to human life. Plastic explosives when start deforming or deteriorate and become hardened because of temperature variations need to be re-plasticized. PE-3A when hardened or loses its plasticity, its detonation efficiency is affected. PE-3A composition is currently utilized in bulk by Pakistan Army for demolition of obsolete ammunition. When there is a need for re-plasticization Pakistan Ordnance factory is approached. It is neither economical nor safe as loading and transportation is involved. Accordingly the objectives set in chapter one have been achieved viz.

1. The hardened PE-3A explosive was analyzed through physical and chemical analysis.
2. A new method has been developed for in-situ re-plasticization PE-3A for Ordnance Depot.
3. Further in this research work a pilot plant is designed to handle 07x Kg batch of explosive for re-plasticization at a time and Teflon coated rollers applied to achieve required plasticity.

In conclusion, a viable and cheap alternate technique for in-situ re-plasticization has been developed during this work.

Future Recommendations

It is recommended to develop an improvised PE-3A composition with enhanced shelf life and better explosion affect by incorporating better plasticizer and filler being the need of the hour.

REFERENCES

[1] http://www.globalsecurity.org/military/systems/munitions/explosives.htm

[2] https://en.wikipedia.org/wiki/Explosive_material#high explosives

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

[4] http:/en.wikipedia.org/wiki/propellant

[5] R. Meyer, J. Köhler, et al, Explosives, Explosives. Sixth Edition. 40, 85 (2007).

[6] *The Chemistry of Explosives* 2nd edition by Jacqueline Akhavan

[7] *Cooper paul W. (1996) "chapter 4:Use forms of explosives".*

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

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

[10]. lubricants@pumaenergy.com

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

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

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

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

[15] “*Magnesium & Liquid Paraffin”. Patient UK Archived from the original on 2009-04-14*

[16]”Lecithin”. Merriam Webster Dictionary online.

[17] “*Composition of soybean lecithin” journals of American Oil Chemist Society. Scholfield, C.R. (1981)*

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

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

[20]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, 214 (1997).

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

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

[23] *Stanjek,H: houler,W. “Basics of X-ray Diffraction” (2004-06-01)*

[24]”Nano Fabrication by Advance Microscopy Using Intense and Focused Beam”. Science And Technology Of Advanced Materials. Article 014110

[25] Fourier Transform Infrared Spectroscopy (2nd ed.) , (18 May 2007), Wiley-Blackwell.

[26] “The Infrared Double-Beam Spectrophotometer”. Clinical Science 16(2). 1957.

[27] “The Early Days of HPLC at Dupont”. (1 February 2009) By Henry, Richard A.

[28] “Milestones in Chromatography: The Birth of Partition Chromatography “ (PDF).LCGC.19(5):506512 2016-02-26

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

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

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

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

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

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

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