

STUDY OF DM WATER FOR BETTER HEAT TRANSFER EFFICIENCY IN ROTARY VACUUM DRYER

Submitted in partial fulfillment of the requirements for the award of Bachelor of
Technology Degree in Chemical Engineering

by

ANUMOL PG (Reg.No.40690002)



**DEPARTMENT OF CHEMICAL ENGINEERING
SCHOOL OF BIO AND CHEMICAL ENGINEERING**

SATHYABAMA

**INSTITUTE OF SCIENCE AND TECHNOLOGY
(DEEMED TO BE UNIVERSITY)**

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OCTOBER - 2023



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CERTIFICATE

This is to certify that this Project Report is the bonafide work of
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I, **ANUMOL PG** (Reg.No.40690002) hereby declare that the Project Report entitled "**STUDY OF DM WATER FOR BETTER HEAT TRANSFER EFFICIENCY IN ROTARY VACUUM DRYER**" done by me under the guidance of **Dr. A. ANNAM RENITA, M.Tech., Ph.D.**, Professor, Department of Chemical Engineering, Sathyabama Institute of Science of Technology and **Mr. M.Sai Kousik, M.Tech.**, Engineer, SD SDSC SHAR, Sriharikota is submitted in partial fulfillment of Bachelor of Technology in Chemical Engineering.

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ABSTRACT

Ammonium Perchlorate is used as an oxidizer in the rocket propulsion system. The ammonium Perchlorate is available in the grade of 500 microns and reduced to 30 microns with the help of hammer mill system. The fine grinded Ammonium Perchlorate available has to dried as soon as received from the grinding unit. For the purpose of drying double cone rotary dryer system is used, that uses ordinary raw water for the heating, vacuum cycles. The rotary vacuum drier is used for reducing the moisture content. The initial moisture content is 0.2% that is reduced to final moisture content 0.02%. The process cycle takes place around 2 hours. The study emphasizes on reduction of the time approximately 30-35% of existing dryer operation time required for the total process to complete to 1 hour and 15 minutes. In order to study the reduction of time required for the total drying process Demineralized water is used instead of ordinary water. Thus the heat transfer efficiency was increased to 2% and the overall time required also was reduced.

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CHAPTER 1

INTRODUCTION

Ammonium perchlorate that is used as an oxidiser in the rocket propulsion system is received from the source in 500 microns size. The available Ammonium Perchlorate are reduced to fine size in the range of 30 microns with the help of grinder unit. After the grinding of Ammonium perchlorate the material has to be dried as soon as possible with the help of Double cone rotary drier. The process involved in the Double cone dryer is the Drying of Ammonium perchlorate is the Heating cycle around a period of 60 minutes or 90 minutes cycle based on the batch size. The heating cycle is followed by the vacuum cycle around 15 minutes.

The primary purpose of rotary vacuum dryer is for temperature rising of the AP content and to reduce the moisture content of the Ammonium Perchlorate by the heating cycle and the continuing vacuum cycle process. The total time required for the whole process is 2 hours based on the batch sizes. For the heating purpose the ordinary water is being used. The objective of the project is to save the time approximately 30-35% of existing dryer operation time. The primary purpose of using DM water as the medium of heat transfer inside the Rotary Vacuum dryer is to decrease the time required for achievement of the product specifying temperature of 72 degree Celsius. This is not achieved with the use of raw water as the source medium due to formation of scales inside the Rotary vacuum dryer which reduce the overall heat transfer efficiency.

Rotary Vacuum Dryers are one of the most energy efficient, cost effective and environmentally safe methods for indirect drying all solids from polymers and catalyst to food ingredients and pharmaceuticals.

The rotary vacuum dryer is used for batch operation for solid/powder materials. Raw materials is fed into dryer batch wise and heating process carried out.

Dryer conical shape is provided for proper and efficient circulation of hot fluid which is nothing but heating media. Heating fluid/media flows in jacket from outer to inner side of the dryer and vacuum is applied in inner portion by using vacuum devices. The principle of drying operation is to remove water or other volatile material from solid by reducing its boiling point. When vacuum is applied in the dryer the boiling point of the volatile material, say water, is reduced. The key parameter for effective drying is uniform heat transfer of the materials in the dryer through the entire batch operation and fast removal of moisture vapour from the material applied.

Generally the above said type of dryer is used for highly heat sensitive materials which are easily oxidized. It is more suitable for pharma products and for volatile compounds.

The rotational speed of the dryer has to be controlled and monitored properly for providing sufficient residence time for heat transfer.

Since this type of dryer does not have any mechanical device for mixing like agitation, therefore internal cleaning and inspections are very simple and economical.

For convenience of the operation, an automatic system can be provided for loading and unloading materials. This arrangement can save lots of time for each batch operation.

Drying process is the combination of heat and mass transfer operation in which water/volatile material is removed from a mixture and forms a solid product. Heat input is necessary for this operation to remove the moisture content. Since the latent heat of vaporization of water is around 2260 KJ/Kg, so a significant amount of heat input to the dryer is required for evaporating the water/moisture content. In the dryer, the mass transfer takes place by means of diffusion and heat transfer by means of convection. Heat transfer and other conditions like cooler design, cooling media used in cooler, product particle density, hardness, etc. also affect the drying process.

Absolute humidity is the mass ratio of water vapour to dry air. It is the ratio of the fugacity of a component in a system relative to the standard-state fugacity. In a drying system, it is the ratio of the vapour pressure of a solvent (e.g., water) in a mixture to the pure solvent vapour pressure at the same temperature.

In a solid is that liquid which exerts a vapour pressure less than that of the pure liquid at the given temperature. Liquid may become bound by retention in small capillaries, by solution in cell or fibre walls, by homogeneous solution throughout the solid, by chemical or physical adsorption on solid surfaces, and by hydration of solids.

Constant-rate period (unhindered) is that drying period during which the rate of water removal per unit of drying surface is constant, assuming the driving force is also constant. Critical moisture content is the average moisture content when the constant-rate period ends, assuming the driving force is also constant.

Dry basis expresses the moisture content of wet solid as kilograms of water per kilogram of bone-dry solid. Equilibrium moisture content is the limiting moisture to which a given material can be dried under specific conditions of air temperature and humidity. Falling-rate period (hindered drying) is a drying period during which the instantaneous drying rate continually decreases. Free moisture content is that liquid which is removable at a given temperature and humidity. It may include bound and unbound moisture. Moisture content of a solid is usually expressed as moisture quantity per unit weight of the dry or wet solid. Moisture gradient refers to the distribution of water in a solid at a given moment in the drying process. Unaccomplished moisture change is the ratio of the free moisture present at any time to that initially present. Unbound moisture in a hygroscopic material is that moisture in excess of the equilibrium moisture content corresponding to saturation humidity. All water in a non-hygroscopic material is unbound water. Vapour pressure is the partial pressure of a substance in the gas phase that is in equilibrium with a liquid or solid phase of the pure component. Wet basis explains the moisture in a material as a percentage of the weight of the wet solid. Use of a dry-weight basis is

recommended since the percentage change of moisture is constant for all moisture levels. When the wet-weight basis is used to express moisture content, a 2 or 3 percent change at high moisture contents (above 70 percent).

The basic kinetics information about drying can be obtained from drying curve. A drying curve explains that how the changes occurred during the drying.. The drying curve is affected by the drying parameters and conditions like materials properties and size of the products materials. The details about Experimental technique which is used to obtain the drying curve and characteristics of drying curve is explained below. The others details about drying curve is discussed in coming section named Drying Modelling Design and Scale-up.

The drying curve explains the different drying periods which occurred during the entire operation. The first period of drying is induction period where the material is heated early in drying.. The second period of drying is called the constant-rate period. In this period the vapour pressure of water on the surface is maintained so that the surface dries sufficiently. After the sufficient drying of the surface drying rate decreases and the falling rate periods start. This period is also known as Hindered drying. The transition between constant and falling rate periods of drying occurring at the critical point. The critical point explains the average moisture content of a material at this transition.

The kinetics of drying explains the design size of the industrial dryer which directly affects the capital investment (fixed costs) and operating costs. The desired product qualities are also influenced by the rate of drying and other factors like heat transfer and moisture losses.

1.1 CLASSIFICATION OF DRYERS

There are several criteria's are available to classify the dryer equipment's. The main type of dryer classifications is as follows:

1. Mode of operation—
 - a. batch

- b. continuous
- 2. Formation of feed and product
 - a. particulate (solid or liquid feed),
 - b. sheet
 - c. slab
- 3. Mode of heat transfer
 - a. convective
 - b. conductive
 - c. radiative and
 - d. dielectric
- 4. Condition of solids beds
 - a. static bed
 - b. moving bed
 - c. fluidized
 - d. dispersed
- 5. Gas-solids contacting flow pattern
 - a. parallel flow
 - b. perpendicular flow and
 - c. through-circulation
- 6. Gas flow pattern
 - a. cross-flow,
 - b. co-current,
 - c. counter current

Other important features of the drying system are the type of carrier gas (air, inert gas, or superheated steam/solvent), use of gas or solids recycle, type of heating (indirect or direct-fired), and operating pressure (atmospheric or vacuum).

The particulate type feed can include powders, granules, pastes, pellets, etc. and *liquid/slurry type feed* also includes solutions and sludges.

1.2 SELECTION CRITERIA FOR DRYERS

It is a very challenging task to select a suitable dryer which can handle the given task at similar cost. For example, for 500- μm particles, there may be several different dryer types available which are suitable to handle the task well, for 5- μm particles, there may be no dryers available which are fully suitable, and the task is to find the “least bad”!

- Batch dryers are used for small scale production approximately 50Kg/hr throughputs and continuous dryers are used for big scale production above 1 ton/h
- Liquid or slurry feeds, large artifacts, or continuous sheets and films require completely different equipment to particulate feeds.
- Dispersion or contact type dryer is suitable to handle the particles and powders type feed which is below 1 mm in size.
- Generally, through-circulation and agitated type dryers give better drying rates than non-agitated cross-circulated or contact tray dryers.
- Non agitated type dryers may be suitable for fragile particles where it is desired to avoid attrition.
- Contact dryers are often used for organic solvents, or solids which are highly flammable, and toxic, or decompose easily. An inert carrier gas (e.g., nitrogen) is often used for this type of dryer.
- For heat sensitive materials and high boiling point materials cocurrent, vacuum, and freeze dryers are used.

A step-by-step and simple procedure is given below.

1. Initial selection of dryers.

Select those dryers which is best suitable to handle the wet material and the dry product, and also produce a product as per our desired properties.

2. Initial comparison of dryers.

The dryers which is going to be selected must be evaluated and compare with the available cost and performance data. From this evaluation, those dryers which are uneconomical or unsuitable can be eliminated from further consideration.

3. Drying tests.

Drying tests must have conducted for those dryers which are under consideration. Drying tests will determine the optimum operating conditions and the product characteristics and will form the basis for firm quotations from equipment suppliers.

4. Final selection of dryer.

After obtaining the good results of the drying tests, quotations for the final selection of the suitable dryer can be made.

The most important factors which must be consider in the preliminary selection of a dryer are as follows:

- Properties of the material
 - a. Physical properties like flowability, adhesiveness, cohesiveness and stickiness when it is on wet condition
 - b. Physical properties when dry
 - c. Corrosiveness
 - d. Toxicity
 - e. Flammability
 - f. Particle size
 - g. Abrasiveness
- Drying characteristics of the material
 - a. Type of moisture like bound, unbound, or both.
 - b. Initial moisture content (maximum and range)
 - c. Final moisture content (maximum and range)

- d. Permissible drying temperature
- e. Probable drying time for different dryers
- f. Level of non-water volatiles
- Flow of material to and from the dryer
 - Quantity to be handled per hour (or batch size and frequency)
 - a. Continuous or batch operation
 - b. Process prior to drying
 - c. Process subsequent to drying
- Product quality
 - a. Shrinkage
 - b. Contamination
 - c. Uniformity of final moisture content
 - d. Decomposition of product
 - e. Over drying
 - f. State of subdivision
 - g. Product temperature
 - h. Bulk density
- Recovery problems

Followings are the main recovery problems which occurs in drying operations are:

 - a. Recovery of dust
 - b. Recovery of solvent
- Facilities availability at site for proposed installation
 - a. Sufficient and suitable space
 - b. Good quality of air like humidity and cleanliness
 - c. Availability of fuels
 - d. Availability of electric power
 - e. Permissible noise, vibration, dust, or heat losses
 - f. Source of wet feed
 - g. Exhaust-gas outlets

1.3 DEMINERALIZED WATER (DM WATER)

Demineralized Water (DM Water) which is also known as Deionized Water, is used in drying of Ammonium Perchlorate.

Since DM water is high purity water therefore having more advantages than the ordinary raw water.

DM Water is produced from raw water by mechanically filtered or processed. In this process the dissolved salts are removed and high purity of water is obtained. Presence of salts in water causes fouling and choking problems in process equipments like coolers, heat exchangers and dryers etc. Purification process removes contaminants like dissolved salts and minerals that may interfere with processes, or leave residues on evaporation.

The DM water or Distilled water is produced by followings process:

1. Deionization,
2. Reverse osmosis,
3. Carbon filtering,
4. Microfiltration,
5. Ultrafiltration and
6. Ultraviolet oxidation, or electrode ionization.

Nowadays Ultrapure water of high purity is produced by using the combinations of a number of processes and traces of contaminants if leftover is also measured in ppm level.

USES OF DM WATER:

The DM Water is largely used in pharmaceutical industry to produce the medicines, in laboratories, industries and also in beverage industries. Purified water is also suitable for many applications, including autoclaves, hand- pieces, laboratory testing, laser cutting, and automotive use

The followings impurities that must be removed are:

- Inorganic ions
- Organic compounds
- Bacteria
- Endotoxins and nucleases
- Particulates
- Gases

CHAPTER 2

AIM & SCOPE

2.1 AIM

The main aim of the project is to reduce the time required for the overall drying period from 2 hours to 1 hour and 15 minutes. In order to facilitate this process the ordinary water used for heating purpose of the present Rotary vacuum dryer system was replaced with DM water system. That reduced the time period for the drying cycle to attain the desired material temperature thus determining time and heat requirement.

2.2 SCOPE

- To calculate the heating cycle time.
- To calculate the vacuum cycle time.
- Comparing the results with the experimental data with the live trails that conducted.

2.3 EXISTING PROCESS EXPLANATION

2.3.1 Characteristics of Ordinary Water

It was found during the process cycle the ordinary water when was used for heating of Ammonium Perchlorate from 55 degree Celsius to 70 degree Celsius it took a time period of around 60 minutes. It was observed later on because of scale formation inside the rotary vacuum drier the heat transfer efficiency was reduced to a 2%.

Table 2.1 : Characteristics of Ordinary Water

Parameters	Unit	specification
pH		7.0-7.8
Conductivity@25°C	Mhos/cm	1400
TDS	mg/l	700
Silica as SiO ₂	mg/l	40
Chlorides as Cl	mg/l	350-460
Sulphates as SO ₄	mg/l	121
Total iron as Fe	mg/l	<0.3
Nitrates as NO ₃	mg/l	0.2
Total cation/anion CaCO ₃	mg/l	900
BOD	mg/l	NA
COD	mg/l	NA
Mo Alkalinity as CaCO ₃	mg/l	240
Ca Hardness as CaCO ₃	mg/l	176
Mg Hardness as CaCO ₃	mg/l	NA

2.3.2 Characteristics of DM water

The use of DM water proved to have better heat transfer efficiency than compared to ordinary water system because of non-formation of scale inside the rotary vacuum drier. The major advantage of using DM water is the declination of scale formation inside the RVD.

Table 2.2 : Characteristics of DM water

Parameters	Unit	Specification
pH		6.8-8.0
Conductivity@25°C	Mhos/cm	< 5
TDS	mg/l	< 0.2
Silica as SiO ₂	mg/l	0.02
Chlorides as Cl	mg/l	0.02
Sulphates as SO ₄	mg/l	NA
Total iron + copper	mg/l	<0.003
Nitrates as NO ₃	mg/l	0.2
Total cation/anion CaCO ₃	mg/l	NA
Total hardness	mg/l	NA
Calcium	mg/l	NA
Magnesium	mg/l	NA
Sodium	mg/l	NA
Pottassium	mg/l	NA
Mangnesium	mg/l	NA
Fluriode	mg/l	NA
Sulphate	mg/l	NA
Copper + Iron	mg/l	NA

CHAPTER 3

EXPERIMENTAL METHODOLOGY

3.1 EXPERIMENTAL METHODOLOGY FOR USE OF DM WATER

Since the Ammonium perchlorate material is a heat sensitive material , for the experiment to be conducted it was suggested to use chalk powder or sodium sulphate that has the same physical and chemical properties of Ammonium perchlorate. Thus, here the preferred choice of material choosing was sodium sulphate.

Pilot plant operation, lab testing report and plants scale experiments are playing key role in drying research.. Lab-scale experiments are often used to study about the product characteristics and physical properties; pilot-scale experiments are used for proof-of-concept and plant-scale experiments are needed to start or change a full-scale process.

There are three steps involved in sampling method In the first steps the samples are collected at different times of the drying. In second steps moisture analysis is done for each sample. In third steps data interpretation is done for final decisions..

Sample collecting techniques are very important factor for drying operation as improper sample may give wrong result which will affect the entire operation and drying curve.

The following consideration is to be taken into account for sampling

1. The fast and safe sampling. Samples must be sealed to avoid contact with air/moisture.
2. For heterogeneous mixture, bigger sample size required to analyze the correct composition of the mixture.

A consistent technique is required for measuring the moisture content because the moisture value depends on the measurement technique.

Gravimetric (loss on drying) is the most common method which is used to calculate the moisture content. A sample is weighed before the drying and put in to a sample tray. This sample tray is then kept into an oven at some high temperature for a given period of time. After the drying completion a sample is again weighed. The weight difference between these two sample is the weight of the moisture ie water content which is evaporated from the raw materials. The length of drying time, temperature and the sample size are the important factor. A very large or thick sample may not dry completely in the given time; a very small sample may not accurately represent the composition of a heterogeneous sample. A low temperature can fail to completely dry the sample, and a temperature that is too high can burn the sample, causing an artificially high loss of mass.

3.1.1 Trial without material inside Rotary vacuum dryer with Raw water

The initial trial was conducted by setting the controller temperature of RVD to a 72-degree Celsius. This is achieved with the help of water heater which are subsystems of the Rotary cone vacuum dryer. Once the Rotary vacuum dryer without the material was achieved to reach a temperature of 72 degree Celsius with using ordinary water, with the heating cycle process and the vacuum cycle process. The Rotary vacuum Dryer are designed to in and operated batch process with the process sequence of heating cycle, vacuum cycle that is followed by the blending cycle process. Thus, to attain the time required for the trail purpose the RVD was operated under no load condition. The details of this trial is mentioned below.

Table 3.1 : No Load Trial Details

Time (mins)	Temperature of inlet ,oulet and controller			RVD Inside temperature (Deg.C)
	Material temperatur e	Inlet	Outlet	
2	75.5	74.433	73.589	60.656
30	75.3	74.656	74.014	64.086
60	75.1	74.850	74.372	68.727z
120	74.9	74.488	74.399	70.281
150	74.9	74.536	74.400	72.119
180	75.1	74.598	74.391	73.261

3.1.2 Calculation of Normal Water For Heat Transfer Time Requirment

- 1) Taking the top con volume = bottom cone volume = 1047 lit
- 2) Thermal resistance offered by the RVD shell = thermal resistance offered by the cylindrical portion + thermal resistance offered by the RVD bottom cone+ thermal resistance offered by the RVD top cone

$$= h/ka \text{ (cylinder)} + h/ka \text{ (bottom)} + h/ka \text{ (top)}$$

3) $k = 0.668$ for ss

4) For water $N_{pr} = 2.3$

5) Velocity of water considering 2 inch inlet pipe =
 $0.008 / (3.14 / 4 * 0.0508 * 0.0508) = 3.947 \text{ m/s}$

6) $N_{re} = Deq V/v$ at 20 degree celcius

$$= ((2.25^2 - 2.134^2) / 2.134) * 3.947 / 1.004 * 10$$

$$= 0.2383 * 3.947 / 1.004 * 10$$

$$= 936844$$

7) Hence, $N_{nu} = 2837.9$

$$= h_{Deq} / k$$

$$= h = 2837.9 * .67 / .2383$$

$$= 7145 \text{ w/m}^2\text{k}$$

8) $R_{\text{cone}} = \ln 430/406 / (6.2832 * 16.2 * 1.527)$
 $= 0.000369$

9) $R_{\text{cylinder}} = \ln 2124/2100 / (6.2832 * 16.2 * 0.65)$
 $= 0.000712.$

10) Heat required to reduce moisture from 0.2% to 0.02% initial temperature

$$= 298 \text{ k}$$

final temperature = 345 k

$$\begin{aligned} Q1 &= M_w \gamma + M_w C_{Pw} T = 5.4 * 2336 + 5.4 * 4.187 * 47 \\ &\quad \text{Kg} \\ &= 12614.4 + 1062.66 \\ &= 13677.06 \text{ KJ} \end{aligned}$$

11) Heat required to raise the temperature of AP from 65 to 70 degree Celsius

$$\begin{aligned} Q2 &= M_{ap} C_p T \\ &= 3000 * 1.292 * 45 \\ &= 174535 \text{ KJ} \end{aligned}$$

Total heat required for the material to be dried = 188212 KJ

12) Heat required to raise the temperature of air inside RVD from 25 TO 70 degree Celsius.

$$\begin{aligned} \text{Quantity of air inside RVD} &= 0.4 * 4000 * 1.2 \\ &= 1.968 \text{ kg} \\ Q3 &= 1.968 * 1.005 * 45 \\ &= 89 \text{ KJ} \end{aligned}$$

13) Heat required to increase to raise the temperature of RVD shell from 25 to 70 degree Celsius.

Assumption RVD shell weight = 2000 kg

$$Q_4 = 2000 \times 5 \times 45$$

$$= 45000 \text{ KJ}$$

14) Neglecting the other losses,

Overall heat required = 2333101 KJ (overall heat that has to be supplied by water in one hour)

15) Calculation of mass of water required.

$$M_w C_{pw} T = 233301/3600$$

$$= 64.8 \text{ kw}$$

$$M_w = 66.816/(4.187 \times 2)$$

$$= 8.036 \text{ kg/sec}$$

$$M_w = 7.74 \text{ kg/sec}$$

$$= 464 \text{ kg/minute}$$

$$= 470 \text{ ltr/min}$$

16.) Calculation of Heat required to raise the Temperature of material to 72

°C .Heat required to raise the temperature of solids and liquids present.

$$Q_{hc} = m_{mat} [c_{ps} (T_{fmat} - T_{imat}) + X_a c_{pl} (T_v - T_{imat})$$

Where.

$$m_{mat} = \text{mass of material taken in RVD} = 600 \text{ kg}$$

$$c_{ps} = \text{specific heat of solid material} = 750 \text{ J/kg } ^\circ\text{C}$$

T_{imat} = Initial temperature of material(solids) = 30 °C

T_{fmat} = final temperature of material(solids) = 72 °C

T_v = vaporization temperature of liquid(water) = 72 °C

C_{ps} = specific heat of liquid(water) = 4182 J/kg °C

X_a = amount of water in kg / dry solids in kg

$$\begin{aligned} Q_{hc} &= 1200 * [750*(72-30) + 0.01*4182*(72-30)] \\ &= 1200 [31500 + 1756.44] \end{aligned}$$

$$Q_{hc} = 3990.0 \text{ J}$$

19) Time required to raise the feed temperature to 72 °C, t_{hc}

$$\begin{aligned} t_{hc} &= Q_{hc} / q_{material} \\ &= 3986 / 6347.9 \\ &= 5896.6 \text{ s} \\ t_{hc} &= 5896.6 \text{ s} = 91 \text{ min} \end{aligned}$$

20) Calculation of Heat required to raise the Temperature of material to 72 °C
Heat required to raise the vaporize liquids(water) present.

$$Q_{vc} = m_{mat} [\lambda (X_i - X_f)]$$

Where,

$$\begin{aligned} m_{mat} &= \text{mass of material taken in RVD} \\ &= 600 \text{ kg} \end{aligned}$$

$$\begin{aligned} \lambda &= \text{latent heat of liquid(Water)} \\ &= 2333 \text{ kJ/kg °C} \end{aligned}$$

X_i = Initial amount of water in kg/ wt of dry solids in kg

X_f = final amount of water in kg/ wt of dry solids in kg

$$Q_{vc} = 600 [2333 (0.01 - 0.001)]$$

$$= 600 * [20.997]$$

$$Q_{vc} = 12589 \text{ kJ} = 12589 \text{ J}$$

21) Time required to raise the feed temperature to 72 °C,

$$t_{vc} t_{vc} = Q_{vc} / q_{\text{material}}$$

$$= 12589 / 6347.9$$

$$t_{vc} = 2260 \text{ s}$$

$$t_{vc} = 2260 \text{ s} = 37 \text{ min}$$

22) Total time of process

$$\text{Total time} = \text{Heat cycle time} + \text{Vacuum cycle time}$$

$$t_{\text{tot}} = t_{\text{hc}} + t_{\text{vc}}$$

$$t_{\text{tot}} = 91 + 31 = 128 \text{ min}$$

The time required to heat 600 kg of AP in a 2000 LRVD from 35 to 72 degree Celsius was found to be 128 minutes. The value was found with the help of ordinary water used as a heating medium.

3.1.3 TRIAL WITH SODIUM SULPHATE USING DM WATER INSIDE ROTARY VACUUM DRYER

Experiment 2 was conducted with the Sodium Sulphate that has the similar properties to Ammonium Perchlorate in a 2000 L RVD so set the characteristics of temperature attainment with the help of DM water that is supplied from the in source manufacturing company. The details are furnished below.

Table 3.2 Sodium Sulphate data

Time (min.)	RVD Inlet Temperature (Deg.C)	RVD outlet temperature (Deg.C)	Material Temperature (Deg.C)
0	74.3	72.8	48.3
10	74.24	71.52	48.43
20	74.27	71.52	50.64
30	74.25	72.12	55.65
40	74.28	72.61	60.65
50	74.31	73.08	65.62
60	74.32	73.39	68.88

From the experiment that conducted with the sodium sulphate as the alternative material for Ammonium Perchlorate with the help of DM water to increase the temperature of the material from 43⁰C to 72⁰C was found out to be less than 60 mins

3.1.4 Trial with Ammonium Perchlorate Using DM Water Inside Rotary Vacuum Dryer

This trial was conducted with the oxidizer Ammonium Perchlorate itself with the material loading capacity of 1200 kg into Rotary Vacuum drier of capacity 2000 L. The details are furnished below.

Table 3.3 : Ammonium Perchlorate Data

Time (min.)	RVD Inlet Temperature (Deg.C)	RVD outlet temperature (Deg.C)	Material Temperature (Deg.C)
0	74.163	72.351	48.866
10	74.252	72.344	48.492
15	74.387	72.368	56.913
20	74.776	72.343	59.855
25	74.265	72.373	63.344
30	74.754	72.599	68.638
40	74.057	72.570	70.061
50	74.510	72.863	71.186
60	74.644	72.790	72.54

The above experiment shows the result of the attained of desired temperature of 72⁰C from 43⁰C was found out to be attained a temperature level of 50 minutes itself.

The individual results was analyzed with the live trial methodology with the experimental value collected for the Rotary vacuum dryer that was operated under no load condition. The whole process of trial running was taken under the SCADA operated system thus the results analysis with the SCADA system gave the appropriate drying and heating condition required individually for the ordinary raw water and the DM water system.

CHAPTER 4

RESULTS AND DISCUSSION

4.1 STUDIES ON DM WATER OVER ORDINARY RAW WATER

The DM water is having much more advantages than using ordinary raw water for the heat transfer to the Ammonium Perchlorate that is to raise the temperature of the material from 43 to 72 required as specified for the product specification. The DM water that prepared in in house and supplied to the RVD has another advantage of economy consideration.

The DM water studies relieved that the heat requirement also varies with the type of material and also the amount of material added to the Rotary Vacuum dryer.

The calculated time is based on the heat supplied and amount of moisture absorbed by the material.

Table 4.1 : Specification Raw Water and DM Water

Sl. No.	Water	Moisture	Heat cycle time	Vacuum cycle time
1.	Raw water	0.1% to 0.01%	60 min	15 min
2.	DM water	0.1% to 0.01%	55 min	15 min

The above clearly represents that time varies with water content and amount of moisture present in the material. The time also depends on amount of heat transferred, which in turns depend the thermal conductivity of the material. The higher the thermal conductivity the more heat transferred and

lesser the time for drying. In the above table ammonium perchlorate has higher thermal conductivity, 4.03 W/m K compared to sodium sulphate which has thermal conductivity of 0.09 W/m K.

The product specification that requires the moisture to be removed from 0.1% to 0.01% the RVD was run on trial basis and the following data was collected.

4.2 PROCESS DIFFICULTIES OF ORDINARY RAW WATER

It was found that during the previous process activities the use of ordinary raw water the scale formation was observed in the shell of RVD that hindered the transfer of heat to the material inside RVD. The problem of scale formation was rectified once in every 3 months, with the help of acid flushing. The time required for achievement of 72 Degree Celsius as required by product specification was attained after 60 minutes of heating and 15 minutes of vacuum cycle process.

Ammonium Perchlorate material since it is a hygroscopic material as well as it is heat sensitive material the acid flushing was not a correct method option for cleaning the RVD.

4.3 RESULTS WITH GRAPH REPRESENTATIONS

4.3.1 RVD trial run with no load using ordinary water as heating medium

Table 4.2 : TRIAL 1 with no load using Raw water

Material loaded	600 kg
Drying cycle time	60 Min
Vacuum cycle time	15 Min
Material temperature at the end of Heating Cycle	71.0
Material temperature at the end of vacuum cycle	72.0
Moisture content	0.02%

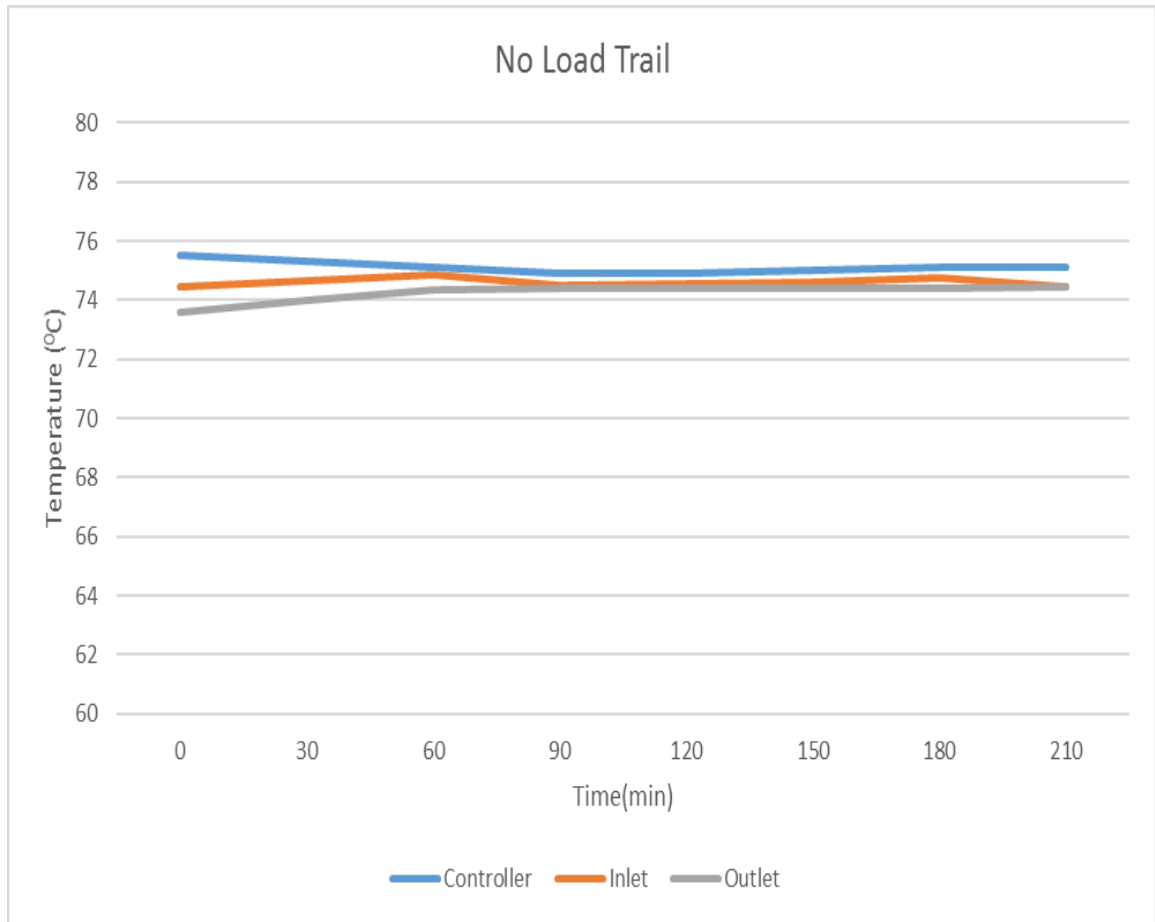


Figure 4.1 : Drying data curve Temp vs Time

4.3.2 RVD trial run with Sodium Sulphate using DM water as heating medium

Table 4.3 : Trial with sodium sulphate using DM water

Material loaded	1200 kg
Drying cycle time	55 Min
Vacuum cycle time	15 Min
Material temperature at the end of Heating Cycle	70.5
Material temperature at the end of vacuum cycle	72.3
Moisture content	0.02%

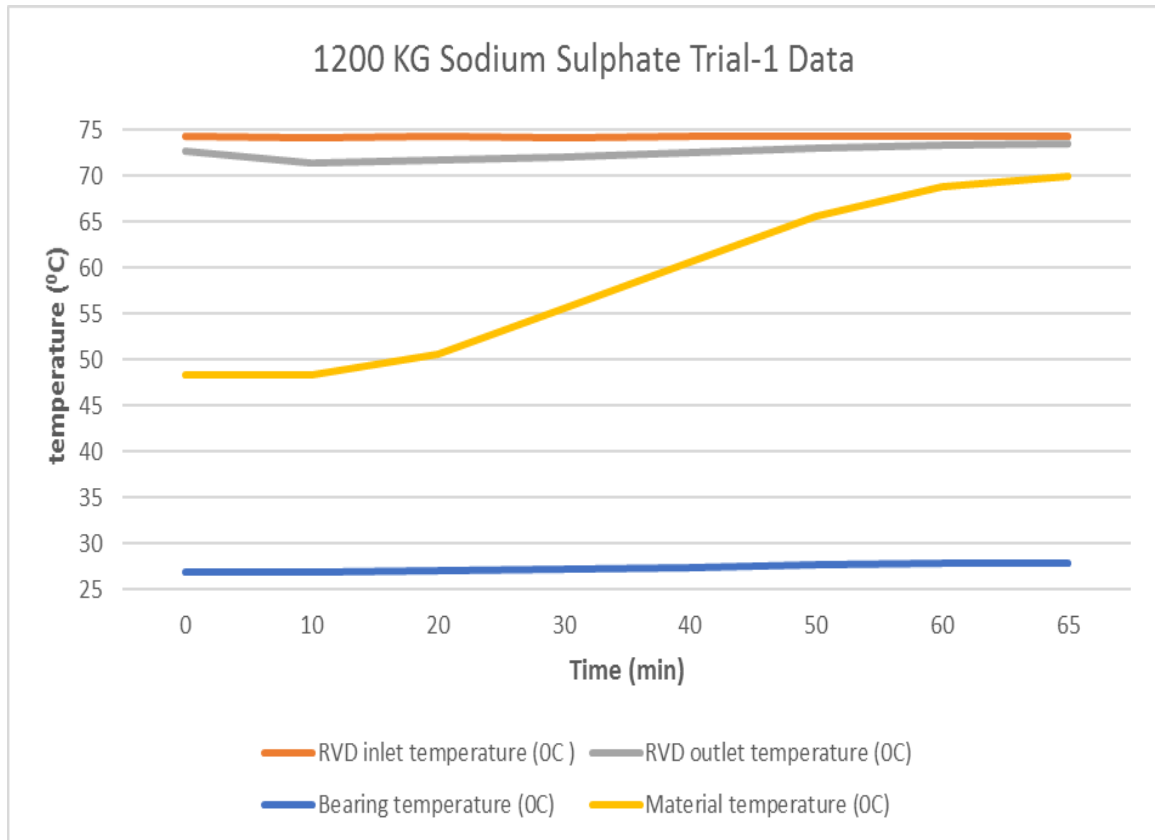


Figure 4.2 : Sodium Perchlorate drying data curve Temp vsTime

4.3.3 RVD trial run with Ammonium Perchlorate using DM water as heating medium

Table 4.4 : Ammonium perchlorate drying data

Material loaded	600 kg
Drying cycle time	50 Min
Vacuum cycle time	15 Min
Material temperature at the end of Heating Cycle	71.0
Material temperature at the end of vacuum cycle	72.0
Moisture content	0.01%

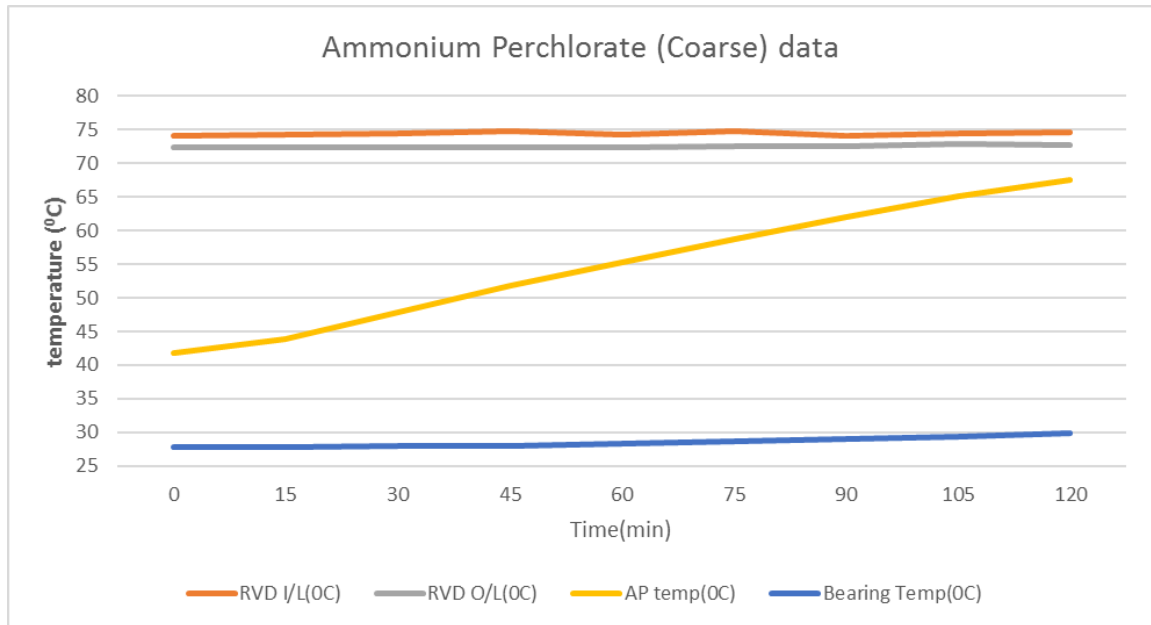


Figure 4.3 : Ammonium Perchlorate drying data curve Temp vsTime

CHAPTER - 5

CONCLUSION

The required heat transfer for the material, Ammonium Perchlorate was achieved in 2000 lit RVD from 43 degree Celsius to 72 degree Celsius with the DM water instead of ordinary raw water and through which the usage of DM water for the better heat transfer efficiency was calculated. The design parameters are matched with available RVD. The heating and vacuum cycles were calculated for the given materials loaded and the experimental results were matched. The test was carried out with the different levels. The RVD initially in Heat cycle, where the material temperature was raised and controlled at temperature of 72, vacuum cycle was initiated.

The heat associated for raising and maintenance of material temperature during two cycles was provided by hot water that was considered with the ordinary water and DM water supply and it was found out the total time consumed for the overall process was reduced to 15 to 20 minutes, based on the quantity of the material loaded to the RVD. These results in the minimum time required for the batch process that is carried out in house process activities.

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STUDY ON DM WATER FOR BETTER HEAT TRANSFER EFFICIENCY IN ROTARY VACUUM DRYER

² Submitted in partial fulfillment of the requirements for the award of bachelor of
Technology Degree in Chemical Engineering

by

ANUMOL PG (Reg.No.40690002)



² **DEPARTMENT OF CHEMICAL ENGINEERING
SCHOOL OF BIO AND CHEMICAL ENGINEERING
SATHYABAMA**

**INSTITUTE OF SCIENCE AND TECHNOLOGY
(DEEMED TO BE UNIVERSITY)**

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BONAFIDE CERTIFICATE

This is to certify that this Project Report is the bonafide work of **ANUMOL PG**
(**Reg.No.40690002**) Who carried out the project entitled "STUDY OF DM WATER
FOR BETTER HEAT TRANSFER EFFICIENCY IN ROTARY
VACUUM DRYER" under my/our supervision from July 2023 to November 2023.

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Internal Examiner

External Examiner

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DECLARATION

I, ANUMOL PG (Reg.No.40690002) hereby declare that the Project Report entitled "STUDY OF DM WATER FOR BETTER HEAT TRANSFER EFFICIENCY IN ROTARY VACUUM DRYER" ² done by me under the guidance of Dr. A. ANNAM RENITA, M.Tech., Ph.D., Professor, Department of Chemical Engineering Sathyabama Institute of Science of Technology and M . Sai Kousik, M.Tech., Engineer, SD SDSC SHAR, Sriharikota is submitted in partial fulfillment ² of Bachelor of Technology in Chemical Engineering.

DATE: November, 2023

PLACE: Chennai

SIGNATURE OF THE CANDIDATE

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ABSTRACT

Ammonium Perchlorate used as an oxidizer in the rocket propulsion system. The ammonium Perchlorate is available in the grade of 500 microns and reduced to 30microns with the help of hammer mill system. The fine grinded Ammonium Perchlorate available has to dried as soon received from the grinding unit. For the purpose of drying double cone rotary dryer system is used, that uses ordinary raw water for the heating, vacuum cycles. The rotary vacuum drier is used for reducing the moisture content. The process cycle takes place around 2 hours. The study emphasises on reduction of the time approximately 30-35% of existing dryer operation time required for the total process to complete to 1 hour and 15 minutes. In order to study of reduction of time required for the total drying process Demineralised water is used instead of ordinary water. Thus the heat transfer efficiency was increased to 2% and the overall time required also was reduced.

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CHAPTER 1

1. INTRODUCTION

Ammonium perchlorate that is used as a oxidiser in the rocket propulsion system is received from the source in 500 microns size. The available Ammonium Perchlorate are reduced to fine size in the range of 30 microns with the help of grinder unit. After the grinding of AP the material has to be dried as soon as possible with the help of Double cone rotary drier. The process involved in the Double cone dryer is the Drying of Ammonium perchlorate is the Heating cycle around a period of 60 minutes or 90 minutes cycle based on the batchsize. The heating cycle is followed by the vacuum cycle around 15 minutes.

The primary purpose of rotary vacuum dryer is for temperature rising of the AP content and to reduce the moisture content of the Ammonium Perchlorate by the heating cycle and the continuing vacuum cycle process. The total time required for the whole process is 2 hours based on the batch sizes. For the heating purpose the ordinary water is being used. The objective of the project is to save the time approximately 30-35% of existing dryer operation time. The primary purpose of using DM water as the medium of heat transfer inside the Rotary Vacuum dryer is to decrease the time required for achievement of the product specifying temperature of 72 degree celcius. This is not achieved with the of raw water as the source medium due to formation of scales inside the Rotary vacuum dryer. That reduce the overall heat transfer efficiency.

Rotary Vacuum Dryers are one of the most energy efficient, cost effective and environmentally safe methods for indirect drying all solids from polymers and catalyst to food ingredients and pharmaceuticals.

The rotary vacuum dryer is used for batch operation for solid/powder materials. Raw materials is fed into dryer batch wise and heating process carried out.

Dryer conical shape is provided for proper and efficient circulation of hot fluid which is nothing but heating media. Heating fluid/media flows in jacket from outer to inner side of the dryer and vacuum is applied in inner portion by using vacuum devices. The principle of drying operation is to remove water or other volatile material from solid by reducing its boiling point. When vacuum is applied in dryer the boiling point of the volatile material say water is reduced. The key parameter for effective drying is uniform heat transfer of the materials in the dryer through the entire batch operation and fast removal of moisture vapour of the material applied.

Generally the above said type of dryer is used for highly heat sensitive materials which are easily oxidized. It is more suitable for pharma products and for volatile compounds.

The rotational speed of the dryer to be controlled and monitored properly for providing sufficient residence time for heat transfer.

Since this type of dryer is not having any mechanical device for mixing like agitation, therefore internal cleaning and inspections are very simple and economical.

For convenience of the operation automatic system can be provided for loading and unloading materials. This arrangement can save lots of time for each batch operation.

CHAPTER 2

LITREATURE SURVEY

Drying process is the combination of heat and mass transfer operation in which water/volatile material is removed from a mixture and formed a solid product. Heat input is necessary for this operation to remove the moisture content. Since the latent heat of vaporization of water is around 2260 KJ/Kg so significant amount of heat input to dryer is required for evaporating the water/moisture content. In dryer the mass transfer takes place by means of diffusion and heat transfer by means of convection heat transfer other conditions like cooler design, cooling media used in cooler, product particle density, hardness are also affects the drying process. Heat for design a rotary dryer following study/calculations must be carried out.:

1. Material and energy balance
2. Thermodynamics Kinetics
3. Rate of Mass- and heat-transfer
4. Consideration of product specification.

The calculations details are explained in below sections.

TERMINOLOGY

The terminology used here is explained in downstream section.

Absolute humidity

¹ Absolute humidity is the mass ratio of water vapor to dry air.

Activity:

¹ It is the ratio of the fugacity of a component in a system relative to the standard-state fugacity. In a drying system, it is the ratio of the vapor pressure of a solvent (e.g., water) in a mixture to the pure solvent vapor pressure at the same temperature.

¹ Bound moisture :

In a solid is that liquid which exerts a vapor pressure less than that of the pure liquid at the given temperature. Liquid may become bound by retention in small capillaries, by solution in cell or fiber walls, by homogeneous solution throughout the solid, by chemical or physical adsorption on solid surfaces, and by hydration of solids.

¹ **Constant-rate period (unhindered)** is that drying period during which the rate of water removal per unit of drying surface is constant, assuming the driving force is also constant.

¹ **Critical moisture content** is the average moisture content when the constant-rate period ends, assuming the driving force is also constant.

⁴ **Dry basis** expresses the moisture content of wet solid as kilograms of water per kilogram of bone-dry solid.

Equilibrium moisture content is the limiting moisture to which a given material can be dried under specific conditions of air temperature and humidity.

Falling-rate period (hindered drying) is a drying period during which the instantaneous drying rate continually decreases.

Free moisture content is that liquid which is removable at a given temperature and humidity. It may include bound and unbound moisture.

4

Moisture content of a solid is usually expressed as moisture quantity per unit weight of the dry or wet solid.

Moisture gradient refers to the distribution of water in a solid at a given moment in the drying process.

1

Unaccomplished moisture change is the ratio of the free moisture present at any time to that initially present.

Unbound moisture in a hygroscopic material is that moisture in excess of the equilibrium moisture content corresponding to saturation humidity. All water in a non hygroscopic material is unbound water.

Vapor pressure is the partial pressure of a substance in the gas phase that is in equilibrium with a liquid or solid phase of the pure component.

Wet basis explains the moisture in a material as a percentage of the weight of the wet solid. Use of a dry-weight basis is recommended since the percentage change of moisture is constant for all moisture levels. When the wet-weight basis is used to express moisture content, a 2 or 3 percent change at high moisture contents (above 70 percent).

2.1 DRYING KINETICS

¹ The kinetics of drying explains the design size of the industrial dryer which directly affects the capital investment (fixed costs) and operating costs. The desired products qualities are also influenced by the rate of drying and other factors like heat transfer and moisture losses.

2.2 PERIODS OF DRYING

¹ The basic kinetics information about drying can be obtained from drying curve. A drying curve explains that how the changes occurred during the drying. ¹ The drying curve is affected by the drying parameters and conditions like materials properties and size of the products materials. The details about Experimental technique which is used to obtain the drying curve and characteristics of drying curve is explained below. The other details about drying curve is discussed in coming section named Drying Modeling Design and Scale-up.

¹ The drying curve explains the different drying periods which occurred during the entire operation. The first period of drying is induction period where the material is heated early in drying. ¹ The second period of drying is called the constant-rate period. In this period the vapour pressure of water on the surface is maintained so that the surface dries sufficiently. After the sufficient drying of the surface ¹ drying rate decreases and the falling rate periods start. This period is also known as Hindered drying.

⁷ The transition between constant and falling rate periods of drying occurring at the critical point. The critical point explains the average moisture content of a material at this transition.

2.3 EXPERIMENTAL METHODS

Pilot plant operation, lab testing report and plants scale experiments are playing key role in drying research. ¹ Lab-scale experiments are often used to study about the product characteristics and physical properties; pilot-scale experiments ¹ are used for proof-of-concept and plant-scale experiments are needed to start or change a full-scale process.

2.3.1 Sampling Methods

There are three steps involved in sampling method. In the first steps the samples are collected at different times of the drying. In second steps moisture analysis is done for each sample. In third steps data interpretation is done for final decisions.

Sample collecting techniques are very important factor for drying operation as improper sample may give wrong result which will affect the entire operation and drying curve.

The following consideration is to be taken into account for sampling

1. The fast and safe sampling. Samples must be sealed to avoid contact with air/moisture.
2. For heterogeneous mixture, bigger sample size required to analyze the correct composition of the mixture.

A consistent technique is required for measuring the moisture content because the moisture value depends on the measurement technique.

Gravimetric (loss on drying) is the most common method which is used to calculate the moisture content. A sample is weighed before the drying and put in to a sample tray. This sample tray is then kept into an oven at some high temperature for a given period of time of time. After the drying completion a sample is again weighed. The weight difference between these two sample is the weight of the moisture ie water content which is evaporated from the raw materials. The length of drying time, temperature and the sample size are the important factor. A very large or thick sample may not dry completely in the given time; a very small sample may not accurately represent the composition of a heterogeneous sample. A low temperature can fail to completely dry the sample, and a temperature that is too high can burn the sample, causing an artificially high loss of mass.

2.4 CLASSIFICATION OF DRYERS

There are several criteria's are available to classified the dryer equipments. The main type of dryer classifications are as follows:

1. Mode of operation—
 - a. batch
 - b. continuous
2. Formation of feed and product
 - a. particulate (solid or liquid feed),
 - b. sheet
 - c. slab

- 1 3. Mode of heat transfer
 - a. convective
 - b. conductive
 - c. radiative and
 - d. dielectric
4. Condition of solids beds
 - a. static bed
 - b. moving bed
 - c. fluidized
 - d. dispersed
5. Gas-solids contacting flow pattern
 - 1 a. parallel flow
 - b. perpendicular flow and
 - c. through-circulation
6. Gas flow pattern
 - a. cross-flow,
 - b. concurrent,
 - c. countercurrent

Other important features of the drying system are the type of carrier gas (air, inert gas, or superheated steam/solvent), use of gas or solids recycle, type of heating (indirect or direct-fired), and operating pressure (atmospheric or vacuum).

The particulate type feed can include powders, granules, pastes, pellets, performs, etc. and liquid/slurry type feed also includes solutions and sludges.

2.5 SELECTION CRITERIA FOR DRYERS

It is a very challenging task to select a suitable dryer which can handle the given task at similar cost. For example, for 500- μm particles, there may be several different dryer types available which are suitable to handle the task well, for

1

5- μm particles, there may be no dryers available which are fully suitable, and the task is to find the "least bad"!

- Batch dryers are used for small scale production approximately 50Kg/hr throughputs and continuous dryers are used for big scale production above 1 ton/h
- ¹ Liquid or slurry feeds, large artifacts, or continuous sheets and films require completely different equipment to particulate feeds.
- Dispersion or contact type dryer is suitable to handle the particles and powders type feed which is below 1 mm in size.
- Generally, through-circulation and agitated type dryers ³ give better drying rates than non-agitated cross-circulated or contact tray dryers.
- Non agitated type dryers may be suitable for fragile particles where it is desired to avoid attrition.
- ¹ Contact dryers are often used for organic solvents, or solids which are ¹ highly flammable, and ¹ toxic, or decompose easily. An inert carrier gas (e.g., nitrogen) is often used for this type of dryer.
- For heat sensitive materials and high boiling point materials cocurrent, vacuum, and freeze dryers are used.

¹ A step-by-step and simple procedure is given below.

1. Initial selection of dryers.

Select those dryers which is best suitable to handle the wet material and the dry product, and also produce a product as per our ⁴ desired properties.

2. Initial comparison of dryers.

The dryers which is going to be selected must be evaluated and compare with the available cost and performance data. From this evaluation, those dryers which are uneconomical or unsuitable can be eliminated from further consideration.

3. Drying tests.

Drying tests must have conducted for those dryers which are under consideration. Drying tests will determine the optimum operating conditions and the product characteristics and will form the basis for firm quotations from equipment suppliers.

4. Final selection of dryer.

After obtaining the good results of the drying tests, quotations for the final selection of the suitable dryer can be made.

The most important factors which must be consider in the preliminary selection of a dryer are as follows:

- Properties of the material
 - a. Physical properties like flowability, adhesiveness, cohesiveness and stickiness when it is on wet condition
 - b. Physical properties when dry
 - c. Corrosiveness
 - d. Toxicity
 - e. Flammability
 - f. Particle size
 - g. Abrasiveness
- Drying characteristics of the material
 - a. Type of moisture like bound, unbound, or both.
 - b. Initial moisture content (maximum and range)
 - c. Final moisture content (maximum and range)
 - d. Permissible drying temperature
 - e. Probable drying time for different dryers

f. Level of non-water volatiles

- Flow of material to and from the dryer

Quantity to be handled per hour (or batch size and frequency)

- a. Continuous or batch operation
- b. Process prior to drying
- c. Process subsequent to drying

- Product quality

- a. Shrinkage
- b. Contamination
- c. Uniformity of final moisture content
- d. Decomposition of product
- e. Over drying
- f. State of subdivision
- g. Product temperature
- h. Bulk density

- Recovery problems

Followings are the main recovery problems which occurs in drying operations are:

- a. Recovery of dust
- b. Recovery of solvent

- Facilities availability¹ at site for proposed installation

- a. Sufficient and suitable space
- b. Good quality of air like humidity and cleanliness
- c. Availability of fuels³
- d. Availability of electric power
- e. Permissible noise, vibration, dust, or heat losses
- f. Source of wet feed
- g. Exhaust-gas outlets

2.6 DEMINERALIZED WATER (DM WATER)

Demineralized Water (DM Water) which is also known as Deionized Water, is used in drying of Ammonium Perchlorate.

Since DM water is high purity water therefore having more advantages than the ordinary raw water.

DM Water is produced from raw water by mechanically filtered or processed. In this process the dissolved salts are removed and high purity of water is obtained.

Presence of salts in water causes fouling and choking problems in process equipments like coolers, heat exchangers and dryers etc. Purification process removes contaminants like dissolved salts and minerals that may interfere with processes, or leave residues on evaporation.

The DM water or Distilled water is produced by followings process:

1. Deionization,
2. Reverseosmosis,
3. Carbonfiltering,
4. Microfiltration,
5. Ultrafiltration and
6. Ultraviolet oxidation, or electrode ionization.

Nowadays Ultrapure water of high purity is produced by using the combinations of a number of processes and traces of contaminants if leftover is also measured in ppm level.

USES OF DM WATER:

The DM Water is largely used in pharmaceutical industry to produce the medicines, in laboratories, industries and also in beverage industries. Purified water is also suitable for many applications, including autoclaves, hand-pieces, laboratory testing, laser cutting, and automotive use

The followings impurities that must be removed are:

- Inorganic ions
- Organic compounds
- Bacteria
- Endotoxins and nucleases
- Particulates
- Gases

CHAPTER 3

AIM & SCOPE

² 3.1 AIM

The main aim of the project is to reduce the time required for the overall drying period from 2 hours to 1 hour and 15 minutes. In order to facilitate this process the ordinary water used for heating purpose of the present Rotary vacuum dryer system was replaced with ordinary water system. That reduced the time period for the drying cycle to attain the desired material temperature thus determining time and heat requirement.

3.2 SCOPE

- To calculate the heating cycle time.
- To calculate the vacuum cycle time.
- ²⁴ Comparing the results with the experimental data with the live trials that conducted.

3.3 EXISTING PROCESS EXPLANATION

3.3.1. Heating process with ordinary water.

It was found during the process cycle the ordinary water when was used for heating of Ammonium Perchlorate from 55 degree celcius to 70 degree celcius it took a time period of around 60 minutes. It was observed later on because of scale formation inside the rotary vacuum drier the heat transfer efficiency was reduced to a 2%.

Table 3.3.1 characteristics of Ordinary water

Parameters	Unit	specification
pH		7.0-7.8
Conductivity@25°C	Mhos/cm	1400
TDS	²⁶ mg/l	700
Silica as SiO ₂	mg/l	40
¹⁰ Chlorides as Cl	mg/l	350-460
Sulphates as SO ₄	mg/l	121
¹⁸ Total iron as Fe	mg/l	<0.3
Nitrates as NO ₃	mg/l	0.2
Total cation/anion CaCO ₃	¹⁶ mg/l	900
BOD	mg/l	<30
COD	mg/l	<250
¹¹ Mo Alkalinity as CaCO ₃	mg/l	240
Ca Hardness as CaCO ₃	mg/l	176
Mg Hardness as CaCO ₃	mg/l	NA

3.3.2. Use of DM water instead of ordinary water.

The use of Dm water proved to have better heat transfer efficiency than compared to ordinary water system because of non formation of scale inside the rotary vaccum drier. The majour advantage of using Dm water is the declination of scale formation inside the RVD.

Table 3.3.2 Characteristics of DM water

Parameters	Unit	specification
pH		6.8-8.0
Conductivity@25°C	Mhos/cm	< 5
TDS	mg/l	< 0.2
Silica as SiO ₂	mg/l	0.02
10 Chlorides as Cl	mg/l	0.02
Sulphates as SO ₄	mg/l	NA
Total iron as Fe	mg/l	<0.003
Nitrates as NO ₃	mg/l	0.2
9 Total cation/anion CaCO ₃	mg/l	NA
Total hardness	mg/l	NA
Calcium	mg/l	NA
Magnesium	mg/l	NA
Sodium	mg/l	NA
Pottassium	mg/l	NA
Mangnesium	mg/l	NA
Fluriode	mg/l	NA
Sulphate	mg/l	NA
Copper + Iron	mg/l	NA

HAPTER 4

EXPERIMENTAL METHOLOGY

4.1. EXPERIMENTAL METHOLOGY FOR USE OF DM WATER

Since the Ammonium perchlorate material is a heat sensitive material , for the experiment to be conducted it was suggested to use chalk powder or sodium sulphate that has the same physical and chemical properties of Ammonium perchlorate. Thus, here the preferred choice of material choosing was sodium sulphate.

4.1.1 Trial without material inside Rotary vacuum dryer with Raw water

The initial trial was conducted by setting the controller temperature of RVD to a 72-degree Celsius. This is achieved with the help of water heater which are subsystems of the Rotary cone vacuum dryer. Once the Rotary vacuum dryer without the material was achieved to reach a temperature of 72 degree Celsius with using ordinary water, with the heating cycle process and the vacuum cycle process. The Rotary vacuum Dryer are designed to in and operated batch process with the process sequence of heating cycle, vacuum cycle that is followed by the blending cycle process. Thus, to attain the time required for the trail purpose the RVD was operated under no load condition. The details of this trial is mentioned below.

Table 4.1 No Load Trial Details

Time (mins)	Temperature of inlet ,oulet and controller			RVD Inside temperature (Deg.C)
	Material temperature	Inlet	Outlet	
0	75.5	74.433	73.589	60.656
30	75.3	74.656	74.014	64.086
60	75.1	74.850	74.372	68.727
120	74.9	74.488	74.399	70.281
150	74.9	74.536	74.400	72.119
180	75.1	74.598	74.391	73.261

**4.1.1.1 CALCULATION OF NORMAL WATER FOR HEAT TRANSFER TIME
REQUIRMENT**

1) Taking the top con volume = bottom cone volume = 1047 lit

2) Thermal resistance offered by the RVD shell = thermal resistance offered by
the cylindrical portion + thermal resistance offered by the RVD bottom cone+
thermal resistance offered by the RVD top cone

$$= h/ka \text{ (cylinder)}+ h/ka \text{ (bottom)} +h/ka \text{ (top)}$$

3) k = 0.668 for ss

4) For water $N_{pr} = 2.3$

5) Velocity of water considering 2 inch inlet pipe = $0.008/(3.14/4 * 0.0508 * 0.0508)$
= 3.947 m/s

6) $N_{re} = Deq V/v$ at 20 degree celcius

= $((2.25*2.25-2.134*2.134)/2.134) * 3.947/1.004*10$

= $0.2383*3.947/1.004*10$

= 936844

7) Hence, $N_{nu} = 2837.9$

= $hDeq/k$

= $h = 2837.9*.67/.2383$

= 7145 w/m²k

8) $R_{cone} = \ln 430/406/(6.2832*16.2*1.527) = 0.000369$

9) $R_{cylinder} = \ln 2124/2100/(6.2832*16.2*0.65) = 0.000712.$

10) Heat required to reduce moisture from 0.2% to 0.02%

initial temperature = 298 k

final temperature = 345 k

$$Q_1 = M_w \gamma + M_w C P_w T = 5.4 * 2336 \frac{KJ}{Kg} + 5.4 * 4.187 * 47$$

$$= 12614.4 + 1062.66$$

$$= 13677.06 \text{ kJ}$$

11) Heat required to raise the temperature of AP from 65 to 70 degree celcius

$$Q_2 = M_{ap} C_p T$$

$$= 3000 \times 1.292 \times 45$$

$$= 174535 \text{ KJ}$$

Total heat required for the material to be dried = 188212 KJ

12) ¹⁷ Heat required to raise the temperature of air inside RVD from 25 TO 70 degree Celsius.

$$\text{Quantity of air inside Rvd} = 0.4 \times 4000 \times 1.2$$

$$= 1.968 \text{ kg}$$

$$Q_3 = 1.968 \times 1.005 \times 45$$

$$= 89 \text{ KJ}$$

13) Heat required to increase to raise the temperature of RVD shell from 25 to 70 degree celcius.

$$\text{Assumption RVD shell weight} = 2000 \text{ kg}$$

$$Q_3 = 2000 \times .5 \times 45$$

$$= 45000 \text{ KJ}$$

14) Neglecting the other loses,

Overall heat required = 2333101 KJ (overall heat that has to be supplied by water in one hour)

15) Calculation of mass of water required.

$$M_w C_{pw} T = 233301/3600$$

$$= 64.8 \text{ kw}$$

$$M_w = 66.816/(4.187 \times 2)$$

$$= 8.036 \text{ kg/sec}$$

$$M_w = 7.74 \text{ kg/sec}$$

$$= 464 \text{ kg/minute}$$

$$= 470 \text{ ltr/min}$$

16.) Calculation of Heat required to raise the Temperature of material to 72 °C .

Heat required to raise the temperature of solids and liquids present.

$$Q_{hc} = m_{mat} [c_{ps} (T_{fmat} - T_{imat}) + X_a c_{pl} (T_v - T_{imat})$$

Where.

$$m_{mat} = \text{mass of material taken in RVD} = 600 \text{ kg}$$

$$c_{ps} = \text{specific heat of solid material} = 750 \text{ J/kg } ^\circ\text{C}$$

$$T_{imat} = \text{Initial temperature of material(solids)} = 30 \text{ } ^\circ\text{C}$$

$$T_{fmat} = \text{final temperature of material(solids)} = 72 \text{ } ^\circ\text{C}$$

$$T_v = \text{vaporization temperature of liquid(water)} = 72 \text{ } ^\circ\text{C}$$

$$c_{ps} = \text{specific heat of liquid(water)} = 4182 \text{ J/kg } ^\circ\text{C}$$

$$X_a = \text{amount of water in kg / dry solids in kg}$$

$$Q_{hc} = 1200 * [750*(72-30) + 0.01*4182*(72-30)]$$

$$= 1200 [31500 + 1756.44]$$

$$Q_{hc} = 3990.0 \text{ J}$$

19) Time required to raise the feed temperature to 72 °C, t_{hc}

$$t_{hc} = Q_{hc} / q_{\text{material}}$$

$$= 3986 / 6347.9$$

$$= 5896.6 \text{ s}$$

$$t_{hc} = 5896.6 \text{ s} = 91 \text{ min}$$

20) Calculation of Heat required to raise the Temperature of material to 72 °C

Heat required to raise the vaporize liquids(water) present.

$$Q_{vc} = m_{\text{mat}} [\lambda (X_i - X_f)]$$

Where,

$$m_{\text{mat}} = \text{mass of material taken in RVD} = 600 \text{ kg}$$

$$\lambda = \text{latent heat of liquid(Water)} = 2333 \text{ kJ/kg } ^\circ\text{C}$$

$$X_i = \text{Initial amount of water in kg/ wt of dry solids in kg}$$

$$X_f = \text{final amount of water in kg/ wt of dry solids in kg}$$

$$Q_{vc} = 600 [2333 (0.01 - 0.001)]$$

$$= 600*[20.997]$$

$$Q_{vc} = 12589 \text{ kJ} = 12589 \text{ J}$$

21) Time required to raise the feed temperature to 72 °C, t_{vc}

$$t_{vc} = Q_{vc} / q_{\text{material}}$$

$$= 12589 / 6347.9$$

$$t_{vc} = 2260 \text{ s}$$

$$t_{vc} = 2260 \text{ s} = 37 \text{ min}$$

22) Total time of process

Total time = Heat cycle time + Vacuum cycle time

$$t_{tot} = t_{hc} + t_{vc}$$

$$t_{tot} = 91 + 31 = 128 \text{ min}$$

The time required to heat 600 kg of AP in a 2000 LRVD from 35 to 72 degree Celsius was found to be 128 minutes. The value was found with the help of ordinary water used as a heating medium.

4.1.2 Trial with Sodium sulphate using DM water.

Experiment 2 was conducted with the sodium sulphate that has the similar properties to Ammonium Perchlorate in a 2000 L RVD so set the characteristics of temperature attainment with the help of DM water that is supplied from the in source manufacturing company. The details are furnished below.

Table 4.2 Sodium Sulphate data

Time (min.)	RVD Inlet Temperature (Deg.C)	RVD outlet temperature (Deg.C)	Material Temperature (Deg.C)
0	74.3	72.8	48.3
10	74.24	71.52	48.43
20	74.27	71.52	50.64
30	74.25	72.12	55.65
40	74.28	72.61	60.65
50	74.31	73.08	65.62
60	74.32	73.39	68.88

From the experiment that conducted with the sodium sulphate as the alternative material for Ammonium Perchlorate with the help of DM water to increase the

temperature of the material from 43°C to 72°C was found out to be less than 60 minutes.

4.1.3 Trial with Ammonium Perchlorate using DM water.

This trial was conducted with the oxidiser Ammonium Perchlorate itself with the material loading capacity of 1200 kg into Rotary Vacuum drier of capacity 2000 L. The details are furnished below.

Table 4.3 Ammonium Perchlorate data

Time (min.)	RVD Inlet Temperature (Deg.C)	RVD outlet temperature (Deg.C)	Material Temperature (Deg.C)
0	74.163	72.351	48.866
10	74.252	72.344	48.492
15	74.387	72.368	56.913
20	74.776	72.343	59.855
25	74.265	72.373	63.344
30	74.754	72.599	68.638
40	74.057	72.570	70.061
50	74.510	72.863	71.186
60	74.644	72.790	72.54

The above experiment shows the result of the attained of desired temperature of 72°C from 43°C was found out to be attained a temperature level of 50 minutes itself.

The individual results was analysed with the live trial methodology with the experimental value collected for the Rotary vacuum dryer that was operated under no load condition. The whole process of trial running was taken under the SCADA operated system thus the results analysed the SCADA system gave the appropriate drying and heating condition required individually for the ordinary raw water and the DM water system.

RESULTS AND DISCUSSION

5.1 STUDIES ON DM WATER OVER ORDINARY RAW WATER

The DM water is having much more advantages than using ordinary raw water for the heat transfer to the Ammonium Perchlorate that is to raise the temperature of the material from 43 to 72 required as specified for the product specification. The DM water that prepared in in house and supplied to the RVD has an other advantage of economy consideration.

The DM water studies relieved that the heat requirement also varies with the type of material and also the amount of material added to the Rotary Vacuum dryer.

The calculated time is based on the heat supplied and amount of moisture absorbed by the material.

Table 5.1 : Specification Raw water and DM Water

Sl. No.	Water	Moisture	Heat cycle time	Vacuum cycle time
1.	Raw water	0.1% to 0.01%	60 min	15 min
2.	DM water	0.1% to 0.01%	55 min	15 min

The above clearly represents that time varies with water content and amount of moisture present in the material. The time also depends on amount of heat transferred, which in turns depend ¹ the thermal conductivity of the material. The

higher ²¹ the thermal conductivity the more heat transferred and lesser the time for drying. In the above table ammonium perchlorate has higher thermal conductivity, 4.03 W/m K compared to sodium sulphate which as thermal conductivity of 0.09 W/m K.

The product specification that requires the moisture to be removed from 0.1% to 0.01% the RVD was run on trial basics and the following data was collected.

5.2 PROCESS DIFFICULTIES OF ORDINARY RAW WATER

It was found that during the previous process activities the use of ordinary raw water the scale formation was observed in the shell of RVD that hindered the transfer of heat to the material inside RVD. The problem of scale formation was rectified once in every 3 months , with the help of acid flushing. The time required for achievement of 72 Degree celcius as required by product specification was attained after 60 minutes of heating and 15 minutes of vacuum cycle process.

Ammonium Perchlorate material since it is a hygroscopic material as well as it is heat sensitive material the acid flushing was not a correct method option for cleaning the RVD.

5.3 RESULTS WITH GRAPH REPRESENTATIONS

5.3.1 RVD trial run with no load using ordinary water as heating medium

TABLE 5.3.1 TRIAL 1 with no load using Raw water

Material loaded	600 kg
Drying cycle time	60 Min
Vacuum cycle time	15 Min
Material temperature at the end of Heating Cycle	71.0
Material temperature at the end of vacuum cycle	72.0
Moisture content	0.02%

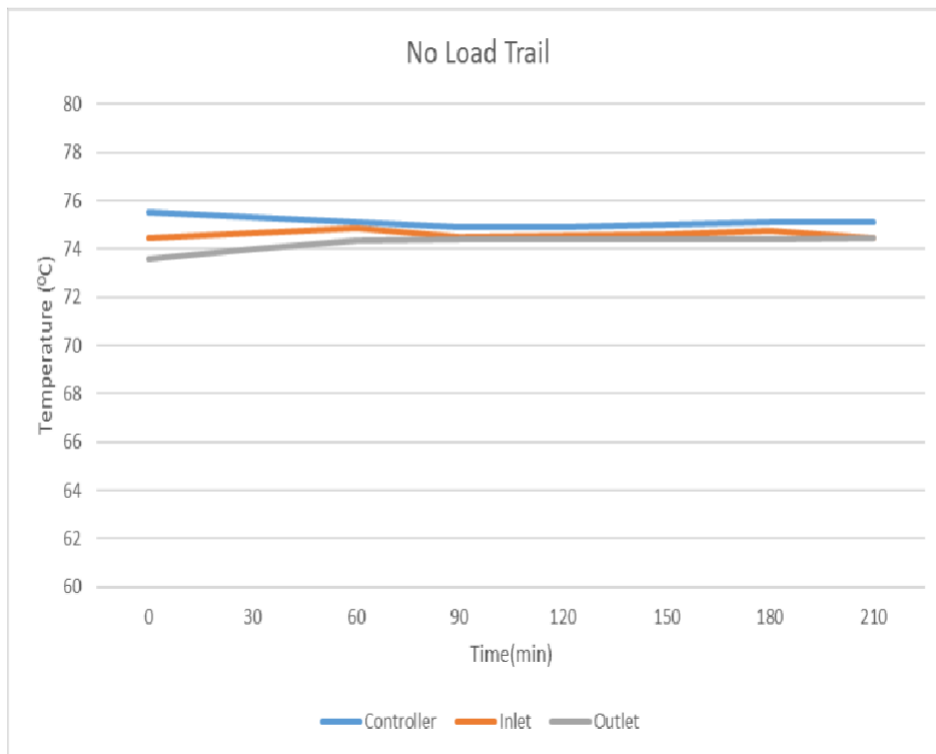


Figure 5.3.1 Drying data curve Temp vs Time

5.3.2 RVD trial run with Sodium Sulphate using DM water as heating medium.

TABLE 5.3.2 Trial with sodium sulphate using DM water

Material loaded	1200 kg
Drying cycle time	55 Min
Vacuum cycle time	15 Min
Material temperature at the end of Heating Cycle	70.5
Material temperature at the end of vacuum cycle	72.3
Moisture content	0.02%

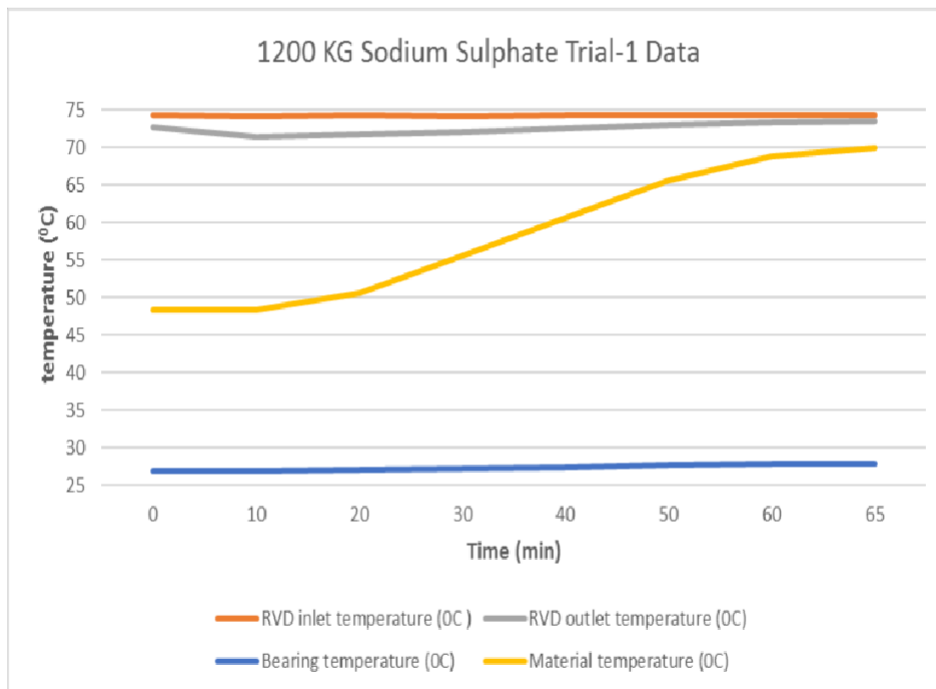


Figure 5.3.2 Sodium Perchlorate drying data curve Temp vs Time

5.3.3 RVD trial run with Ammonium Perchlorate using DM water as heating medium

Table 5.3.3 Ammonium perchlorate drying data

Material loaded	600 kg
Drying cycle time	50 Min
Vacuum cycle time	15 Min
Material temperature at the end of Heating Cycle	71.0
Material temperature at the end of vacuum cycle	72.0
Moisture content	0.01%

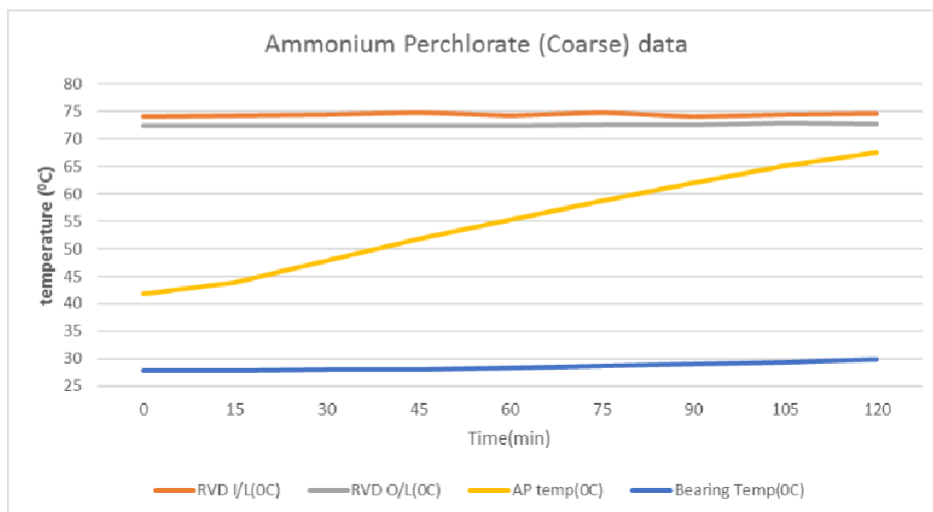


Figure 5.3.3 Ammonium Perchlorate drying data curve Temp vs Time

CHAPTER - 6

CONCLUSION

The required heat transfer for the material ,Ammonium Perchlorate was achieved in 2000 ltr RVD from 43 degree celcius to 72 degree celcius with the DM water instead of ordinary raw water and through which the usage of DM water for the better heat transfer efficiency was calculated. The design parameters are matched with available RVD. The heating and vacuum cycles were calculated for the given materials loaded and the experimental results were matched. The test was carried out with the different levels. The RVD initially in Heat cycle, where the material temperature was raised and controlled at temperature of 72, vaccum cycle was initiated.

The heat associated for raising and maintenance of material tempersture during two cycles was provided by hot water that was considered with the ordinary water and DM water supply and it was found out the total time consumed for the overall process was reduced to 15 to 20 minutes, based on thequantity of the material loaded to the RVD. This results in the miniumum time required for the batch process that is carried out in house process activites.

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