MODELING AND EXPERIMENTAL VALIDATION OF SUBLIMATION-CONDENSATION PROCESS IN SOLID-VAPOR SYSTEM

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Abstract

The sublimation condensation technique is one of the most convenient separation methods used commercially. The parameters that affect the deposition layer thickness in desublimation heat exchanger tubes was investigated and determined in this paper. Experimental setup was fabricated, assembled and performed at Standard Temperature and Pressure, PIEAS, Pakistan. Condenser was fabricated of stainless steel having diameter (18 mm) and total length (340 mm) at PIEAS. Four Pyrex capillary tubes were used with different thicknesses and diameters. Laboratory grade Naphthalene and Ammonium chloride was selected as sublime materials. Direct heating method was employed to separate condensable sublime material from non condensing carrier gas (Air). Concentric type single pass desublimation heat exchanger was made by placing capillary tube inside the condenser. The deposited layer thickness versus time and tube length as function of the tube diameter, thermal conductivity of sublime material, coolant temperature and type of coolant was determined experimentally. To measure thickness of desublimed material which was deposited inside very small capillary tubes is difficult to measure so the qualitative deposited weight method was employed.

Keywords: *Sublimation, desublimation heat exchanger, naphthalene, ammonium chloride , coolant, deposited layer thickness*

INTRODUCTION

Sublimation/desublimation is the inter conversion of matter of solid phase and vapor phase without the appearance of liquid phase and vice versa. For separation and purification of Solid-vapor systems, freeze drying is a viable commercial technique for the deposition of polymers, metals, and drugs (Marc *et al*.,1997).

The quantity of the deposited solid during desublimation is frequently expressed as solid material changed to vapor per unit time, per unit area of solid during sublimation. Rate of transformation from solid to vapor embark upon the fate of various materials into the ecosystem and sublime at different rates. The material transform from solid to vapor state is an important parameter to address deposition rates are widely used in the chemical industries to determine the fate of various materials in the environment and to evaluate the risk associated during inhalation of toxic or carcinogenic compounds by factory workers. To evaluate the vapor pressure from the rate of conversion of materials is an important parameter that can be analytically studied during simultaneous heat and mass transfer processes (Marc *et al*.,1997).

Carbon dioxide a chemical compound that sublimes at atmospheric pressure, a block of solid CO_2 (dry ice) at a room temperature and atmospheric pressure turns in to vapors without the appearance of the liquid phase. Iodine produce fumes when heated gently. In contract to CO_2, through possible to obtain the liquid iodine by controlling the melting point temperature.

Some solids can easily change into vapors (volatilization); these vapors (instead of condensing into a liquid and the liquid freezing back to the solid) can sublime, that is, the vapors form a solid without going through the liquid state. Even ice can be volatilized and sublimed (reduced pressure, 1/100th of the usual atmospheric pressure). Volatilization / sublimation are useful to purify some solids as menthol, naphthalene, iodine, etc. Heat sensitive medicines and enzymes are dehydrated by means of cooling them down until water freezes and ice formed can be sublimed. Sublimation is an endothermic phase transition that occurs at temperatures and pressures below the triple point.

Vacuum sublimation under a temperature gradient as purification method is known to be most effective for separation of impurities from a solid provided that these impurities show a vapor pressure which is sufficiently different from the desired product. Actually, the sublimation technique can be more than just evaporation under vacuum conditions. Depending on detailed process control, the previously mentioned temperature gradient vacuum sublimation technique may already combine basically two separate purification steps within one process: fractionate evaporation and re-crystallization close to equilibrium (Drechsel, 2006).

Uranium Hexaflouride (UF_6) is one of the most important components used in the nuclear industry. UF_6 is used for fuel fabrication and for the production of weapon grade uranium . UF_6 is formed by the chemical reaction of UF_4 and F_2 at 250 ^oC in copper reactors (Benedict and Pigford, 1957). The gas stream leaving the reactor is cooled to low temperature and UF_6 is deposited as solid in a batch sublimation heat exchanger, solid gas is melted and purified by fractional distillation to get highly purified UF_6 (Dewitt, 1960) .

Objectives

A theoretical model was developed earlier to design uranium hexafluoride cold traps has been verified using gamma spectroscopy analysis of a pilot plant scale cold trap operation. The usefulness of the model is to predict plugging and loading in the trap requires accurate frost density and frost thermal conductivity data (Robert, 1979).

This paper demonstrates laboratory scale experimental setup for verification of mathematical model of sublimation-condensation process in solid-vapor system.

The core objective is to evaluate and observe the desublimation of naphthalene from non-condensable carrier gas such as hydrogen and fluorine experimentally and mathematically. Naphthalene and Ammonium Chloride is used instead of UF_6 and the model is described at various temperature, mole fraction of condensable component, solid-gas interface temperature and the thickness of the desublimed solid layer as a function of temperature and the geometry of the heat exchanger.

The prime objectives are; fabrication and erection of experimental apparatus, theoretical model is compared with the experimental results for the validation of sublimation-condensation process, Coding of the mathematical model in MATLAB, comparison of theoretical and experimental models.

Experimental set up

Experimental setup for the study of sublimation-condensation process was fabricated and composed of ignition tubes, stirrer heater, thermocouples, digital thermometer, Pyrex and borosilicate capillary tubes, ring stand, electronic balance, fuel pump (PD pump), oven, condenser, holders and stopper. The pilot scale experimental set up as shown in Figure 1 *(Before Acknowledgment)*.

Theoretical Models used for the study of Vapor

Solid System

Desublimation is a process in which condensable component from a non-condensable carrier gas stream is separated by condensing it out as solid. Special design of desublimation heat exchanger is used during process. It differs from a liquid condenser primarily in that condensed material is not drained continuously but is collected within the desublimation heat exchanger, finally building up enough solid layers to stop the flow of gas through the unit. The deposited solid also affect the heat transfer as the desublimed solid causing the insulation effect as the conductivity of solid layer is considerably low. For this reason, a desublimation heat exchanger is usually designed to operate batch-wise.

Generally, when a gas stream enters a desublimation heat exchanger, the gaseous influent is superheated. Gas coming in contact with the refrigerated desublimation heat exchanger walls however, are cooled to saturation, causing solid to deposit on the walls and setting up a driving force for mass transfer between the bulk gas and the wall surface. At the same time, the gas stream is cooled by normal heat transfer.

As the bulk gas tends to approach saturation, since in traveling down the desublimation heat exchanger, normal mass transfer to the wall does not remove condensates fast enough to maintain the stream below the saturation concentration, which decreases rapidly as the temperature drops. After saturation is reached, this situation still exists.

As the gas stream is cooled, it theoretically must release enough condensable to prevent its becoming supersaturated. Material in excess of the amount traveling to the wall by normal mass transfer thus freezes out in the gas stream itself as a mist or snow and must travel to the surface by falling or other mechanical means. Thus, it would be anticipated that the solid deposit thickness would vary along the gas flow path and that increase in deposit thick-would be expected at the point where the bulk gas becomes saturated (Robert, 1979).

At any point within a desublimation heat exchanger, the base rate of mass transfer to the cooling walls may be expressed as (Eby, 1978).

$$\left(\partial m/\partial\theta\right)_z^o = M_l K(p - p_w) \equiv M_l K_p(1-\beta)$$

Where

$\left(\partial m/\partial\theta\right)_z^o$ = normal mass transfer rate, without including the saturation effect

Detailed 1-D Model:

Thickness of Desublimed Layer

The point thickness of the solid deposit due to mass transfer to the walls of desublimation heat exchanger can be determined from the following equation:

$$\left(\partial b/\partial\theta\right)_z^o = \frac{M_l K_p}{\rho_S}(1-\beta)$$

The mass transfer coefficient, Kp, is not as easily obtainable for diverse systems as is the heat transfer coefficient; however, this value may be estimated from heat transfer data acceptable for many engineering applications by the use of Chilton-Colburn analogy (Chiltan and Colburn, 1934).

This analogy states

$$J_m = \frac{KP_{gf}}{G/M_m} Sc^{\frac{2}{3}} = \frac{h}{C_m G} Pr^{\frac{2}{3}} = J_h$$

This equation allows the use of correlations or experimental data for heat transfer to estimate mass transfer. In our case, the heat transfer coefficients must have the same basis. For a mass transfer coefficient from a warm gas stream to a chilled wall, the required heat transfer coefficient is that for a warm gas to a chilled wall and not an overall coefficient or one which considers the transfer of latent heat.

Brown Fin-Tube Company recommends the following semi-empirical relationship [Brown Fin Tube Company]

$$\frac{h}{C_m G} = (h_{con})(Pr)^{-\frac{2}{3}} f(Re)$$

A least squares fit to data given by the finned tube manufacturer indicated the following Reynolds number dependency:

$$f(Re) = exp\left[-1.2587 - 0.62828\, ln\left(\frac{D_e G}{\mu}\right)\right]$$

The heat exchangers having finned cooling surfaces, the semi-empirical relation has been found to predict the heat transfer quite well and is given. (McAdams, 1954).

$$\frac{h}{C_m G} = 0.67\left(\frac{LG}{\mu}\right)^{-1/2} Pr^{-2/3}$$

The term, Pgf, in the above relationship may be expanded:

$$P_{gf} = \frac{P_i - P_{iw}}{\ln\left(\frac{P_i}{P_{iw}}\right)}$$

Using standard assumptions,

$$p_i = p_T - p = p_T - xp_T$$

$$P_{gf} = \frac{P_w - P}{\ln\left(\frac{P_T - xP_T}{P_T - P_w}\right)} = \frac{p(1-\beta)}{\ln\left(\frac{1-\frac{P_w}{P_T}}{1-x}\right)}$$

Combining the above equations to the form;

$$\left(\frac{\partial b}{\partial \theta}\right)_z^o = \frac{M_l h}{\rho_S M_M C_m}\left(\frac{Pr}{Sc}\right)^{2/3} \ln\left(\frac{1-\frac{P_w}{P_T}}{1-x}\right)$$

The above equation is applied only for a superheated gas. In the case of a saturated gas, though, it also represents the buildup due to the diffusive mass transfer phenomena. The relationship between the actual buildup$(\partial b/\partial \theta)z$ and $(\partial b/\partial \theta)0$ can be defined as:

$$\left(\frac{\partial b}{\partial \theta}\right)_z = \frac{1}{(1-\alpha)}\left(\frac{\partial b}{\partial \theta}\right)_z^o$$

$$\left(\frac{\partial b}{\partial \theta}\right)_z = \frac{M_l h}{\rho_S M_M C_m (1-\alpha)}\left(\frac{Pr}{Sc}\right)^{2/3} \ln\left(\frac{1-\frac{P_w}{P_T}}{1-x}\right)$$

To determine the point thickness distribution throughout the length of the heat exchanger and total mass deposited in heat exchanger, the deposited thickness should be multiplied by the area of the heat exchanger and density of the solid. Where α is defined as the mist fraction. Under superheated conditions, α is zero, and the equation reduces to the previous form, representing only normal mass transfer. Under saturated conditions, however, the mist fraction will represent that portion of condensable which reaches the wall by means other than diffusive mass transfer (Robert, 1979).

Concentration of Condensable Component

Using the above rate equation for solids build up when a differential material balance for a section of desublimation heat exchanger is considered in terms of bulk average concentration, assuming plug flow and neglecting the gas concentration change with time at a point (Eby, 1978).

$$\frac{1}{v}\left(\frac{\partial X}{\partial \theta}\right)_z + \left(\frac{\partial X}{\partial Z}\right)_\theta + \frac{(1-X)^2}{V}\frac{\rho_S P}{M_l}\left(\frac{\partial b}{\partial \theta}\right)_z = 0$$

In general, for desublimation heat exchangers as for similar gas treatment units, the gas concentration at a point does not vary rapidly with time

$$\frac{1}{v}\left(\frac{\partial X}{\partial \theta}\right)_z \ll \left(\frac{\partial X}{\partial Z}\right)_\theta$$

$$\frac{dX}{dZ} + \frac{(1-X)^2}{V}\frac{\rho_S P}{M_l}\left(\frac{\partial b}{\partial \theta}\right)_z = 0$$

The following expression for the concentration profile along the trap is obtained by using equation;

$$\frac{dX}{dZ} = \frac{-(1-x)^2}{V}\,\frac{hP\left(\frac{Pr}{Sc}\right)^{2/3}}{M_M C_m (1-\alpha)}\ln\left(\frac{1-\frac{P_W}{P_T}}{1-x}\right)$$

Variation of Gas Stream Temperature

The expression for the variation of gas stream temperature along the length of desublimation heat exchanger can be determined by applying an energy balance over the desublimation heat exchanger section (Eby, 1978).

$$\frac{a}{1-e^{-a}}h(T-T_W) = \lambda L\alpha - \frac{V}{P(1-X)}M_M C_m \frac{dT}{dz} + h(T-T_W)a$$

Where

$$a = \frac{M_l C_l}{M_M C_m}\left(\frac{Pr}{Sc}\right)^{2/3} \ln\left(\frac{1-\frac{P_W}{P_T}}{1-x}\right)$$

Where the term, α is commonly called the Ackerman correction factor and include the heat transfer to the wall through mass flow.

Rearranging the above equation yields the following expression for the temperature profile along the length of the desublimation heat exchanger.

$$\frac{dT}{dz} = \frac{ha(1-X)P}{VM_M C_m}\left[\frac{\lambda\alpha}{M_l C_l (1-\alpha)} - \frac{(T-T_W)}{(e^a-1)}\right]$$

The above equation can be solved to determine the bulk stream temperature profile along the length of exchanger.

Saturated Region Calculation

After the time when saturated conditions are attained, however, α may no longer be assumed zero, since not all condensing material will be carried to the wall by normal mass transfer. By assuming the condensable desubliming from the vapor stream settles out at the point of formation and the bulk concentrating and temperature of the saturated gas stream follow the Clausius Clapeyron equation that condensable desubliming within the gas stream immediately travels to the wall. The bulk concentration and temperature of the gas stream are assumed to follow the vapor pressure equation for the condensable, which is, in the differential Clausius-Clapeyron form (Robert, 1979)

$$\frac{dX}{dT} = \frac{x\lambda}{RT^2}$$

The factor may be obtained by combining the equations

$$\alpha = 1 - \frac{\frac{\lambda}{M_M C_m} + \frac{RT^2(1-X)}{x\lambda}}{\frac{\lambda}{M_M C_m} + \frac{M_l C_l (T - T_W)}{M_M C_m (e^a - 1)}}$$

Solid-Gas Interface Temperature

The above methodology does not predict any effects of solid deposit on the desublimation heat exchanger walls other than the effects of narrowing the flow channel. The most important effect of this solid deposition, however, is to raise the gas-solid interface temperature, TW. At the beginning this temperature usually may be assumed nearly equal to that of the coolant being used, making allowance for cooling fin efficiencies, since most of the resistance will be experienced in transferring heat from the gas stream to the wall, with much less resistance through the desublimation heat exchanger wall and the system refrigerant. Solid desubliming on the desublimation heat exchanger walls, however, usually has a low thermal conductivity and conduction of heat through this layer must be considered to be determined by using the TW in the above equation. TW is determined by equating the heat flux through the deposited to the heat flux at the deposit surface and latent heat from solids condensed on the surface (Eby, 1978).

$$\frac{K_S}{b}(T_W - T_c) = \frac{ha}{1 - e^{-a}}(T - T_W) + \frac{\lambda ha}{M_l C_l}$$

As long as the gas stream remains superheated then α will remain zero; and equations is solved numerically to obtain the temperature profile and the concentration profile. Solid buildup rates are obtained by using equation, and the results of this calculation may, in turn, be used to determine new profiles, repeating the numerical solution cycle in order to obtain a time variation according to the quasi-steady state approximation (Robert, 1979).

Experimental Setup to study sublimation Condensation Process

Experimental determination of sublimation rate at standard temperature (298 K) and pressure (101.325 kPa; STP) involves measurement of either the weight loss of the solid or the mass of sublimed vapor as a function of time. This experiment was performed at standard temperature (298 K) and pressure (101.3 kPa; STP). Experimental setup was arranged in such a manner that do not violet the safety and proposed procedure:

i. Preparation of samples by means of electronic balance in grams of sublime material and injecting the sample into the Ignition tube.
ii. Placing the Ignition tube on the stirrer heater and rearranged the experimental setup.
iii. Maintaining the temperature inside the condenser with constant flow rate of water by using Fuel pump (PD pump) before the start of the experiment.
iv. Adjust temperature of stirrer heater at 85oC in case of naphthalene because sublimation temperature of naphthalene is 80.26°C.
v. Adjusting the temperature of stirrer heater at 340°C in case of ammonium chloride.

Air and superheated vapors of sublime material passes from capillary tube. When these vapors pass through the condenser, solid buildup or thickness deposition is started inside the capillary tube due to desublimation.

As this process is considered as transient, so that temperature is measured by thermocouples which are adjusted inside capillary tube.

Experiment is performed with different diameter of capillary tubes, sublime materials and time periods. Some important parameters are observed during the experiment is as follows:

- Stream temperature Vs length of capillary tube.
- Variation of bulk stream temperature with time
- Deposited thickness by weight as a function of time and length.
- Relation of weighted value of thickness Vs length of capillary tube.
- For qualitative analysis, 30 cm Capillary tube was taken for each experiment. After desublimation, tube was cut into equal parts of 4 cm. Weighing electronic balance were employed to measure Deposited weight of sublime material inside tube.
- Four holes were drilled at surface of tube for placement of K-type thermocouples to measure transient temperature inside tube and capturing the profiles of temperature during desublimation.

RESULTS AND DISCUSSION

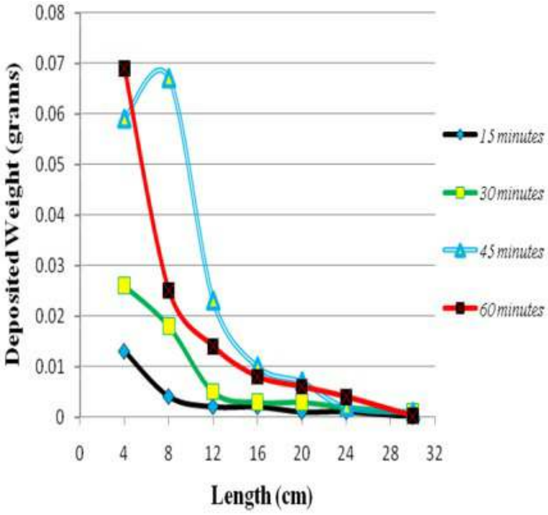
The most important motivation behind devising a experimental model to evaluate the parameters which involved was to enhance the performance of desublimation process.

Results of diameter of capillary tubes against different time period

The variation of deposit weight of Naphthalene along the length of same diameter of capillary tubes is plotted for different time period in order to study cooling load variation by the formation of solid layer by desublimation process.

The Naphthalene solid layer is deposited in capillary tube by different time period for same diameter of tube is shown in Figure 2. The thickness of layer at the beginning of capillary tube for 60 minutes is more as compared to other time periods. The reason for this is when superheated gas stream comes in contact with the cold surface it deposit the solid layer of Naphthalene on the capillary walls. This effect only continues for a small period of time as the deposit layer will form a film which will resist heat transfer rate due to less thermal conductivity. The Figure 2 also shows that solid is deposited only up to 30 cm of the length of capillary tube for same diameter.

Figure 2: Same Diameter of Caplliary Tube in Different Time period



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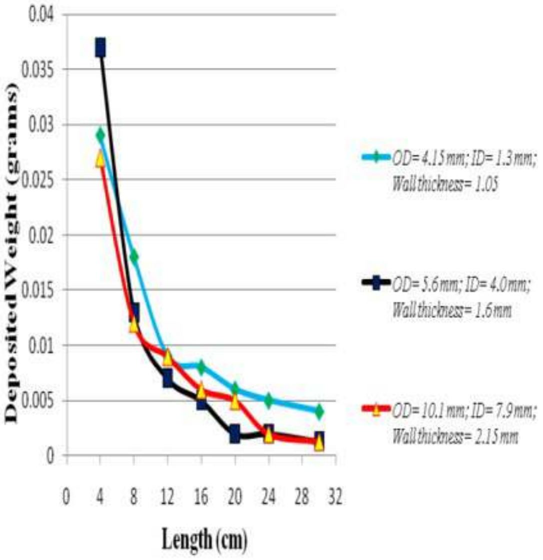
Results of different diameter of capillary tubes with same time period

The Naphthalene solid layer is deposited in capillary tube by different diameter of tube for same time period is shown in Figure 2. The thickness of layer at the beginning of capillary tube (OD= 5.6 mm; ID= 4.0 mm) is more as compared to other two different diameter of tubes. The reason for this is when superheated gas stream comes in contact with the cold surface it deposit the solid layer of Naphthalene on the capillary walls as the thickness of wall is less so that more heat is transferred to the coolant. As tube is made of Borax so that thermal conductivity of Borax is more then Pyrex. Where as other two tubes are made of Pyrex.

Figure 3: Different Diameter Caplliary Tube in Same Time Period



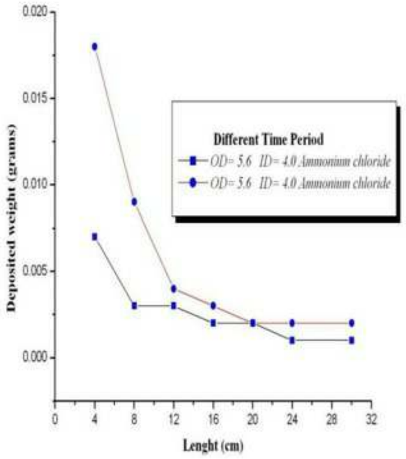
Results of Same Diameter of Capillary Tubes with same time Period but different Sublime Materials

The Naphthalene and Ammonium chloride solid layer is deposited in capillary tube by same diameter of tube for same time period is shown in Figure 3. The thickness of layer at the beginning of capillary tube (OD= 5.6 mm; ID= 4.0 mm) with Naphthalene is more as compared to Ammonium chloride. The reason for this is crystal size of Naphthalene is greater than Ammonium chloride and gravitational effect is more due to horizontal placement of tubes.

Results of Same Diameter of capillary tubes with same Sublime material but Different in Time periods

The Ammonium chloride solid layer is deposited in capillary tube by different time period for same time period is shown in Figure 4 . The thickness of layer at the beginning of capillary tube for 30 minutes is more as compared to 15 minutes. The variation in deposited thickness is much more for 30 minutes. The reason for this is when superheated gas stream comes in contact with the cold surface it deposit the solid layer of Ammonium chloride on the walls. This effect only continues for a small period of time as the deposit layer will form a film which will hinder the heat transfer rate due to less thermal conductivity.

Figure 4: Same Diameter of Capillary Tube with Same Sublime material but Different Time Period



Results of Temperature Variation with respect to time

The Naphthalene solid layer is deposited in capillary tube during temperature measurement shown in Figure 5 and 6. The variation during temperature measurement is not significant. It is observed during experiment that in absence of coolant temperature inside the capillary tube is increasing gradually with time as shown in Figure 5 but in presence of coolant the temperature inside the capillary tube approaches to the temperature of coolant as shown in Figure 6. The reason behind this is small diameter of tubes and high flow rate of coolant. By this temperature inside the tube become stead state after 15 minutes.

Figure 5: Time Period Vs Thermocouple Reading (Absence of Coolant)

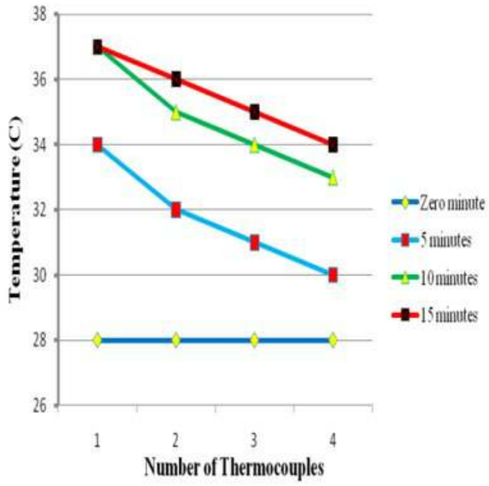
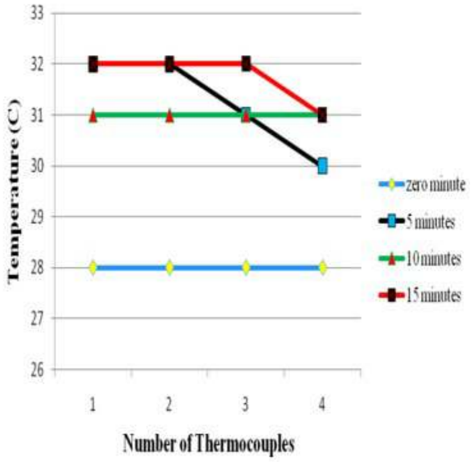


Figure 6: Time Period Vs Thermocouple Reading (Presence of Coolant)



Simplified 1-D Model with Constant Heat/Mass Transfer:

This model is based on constant mass diffusion and heat transfer coefficients. Various assumptions are incorporate before modeling of sublimation and condensation process;

- Heat transfer coefficient is constant
- Thickness affects are neglected
- Diffusion coefficient is constant
- The Fluid behaves as continuum
- The flow pattern has already developed at the entrance
- The gas properties are constant

Material Balance around Capillary Tube

Rate of Accumulation = Rate of sublime material IN – Rate of sublime material OUT – Rate of condensation

$$\frac{\partial(C_A A dx)}{\partial t} = C_A F - \left(C_A F + \frac{\partial(C_A F)dx}{\partial x}\right) - 2\pi r D_{AB} C_A dx$$

CA = Concentration (mol/ cm3)

A = Heat transfer area (cm2)

F = Air and Sublime material flow rate (1200 cm3 / mint)

r = Inner radius of tube (cm)

L = Tube length (30 cm)

DAB = Diffusion coefficient

Simplifying the equation

$$\frac{\partial(C_A A)}{\partial t} = -\frac{\partial(C_A F)}{\partial x} - 2\pi r D_{AB} C_A$$

Applying Boundary conditions

$$C_A(0,t) = X(0)T(t) = C_{A0}$$

$$C_A(L,t) = X(L)T(t) = 0$$

Using separable method to solve equation

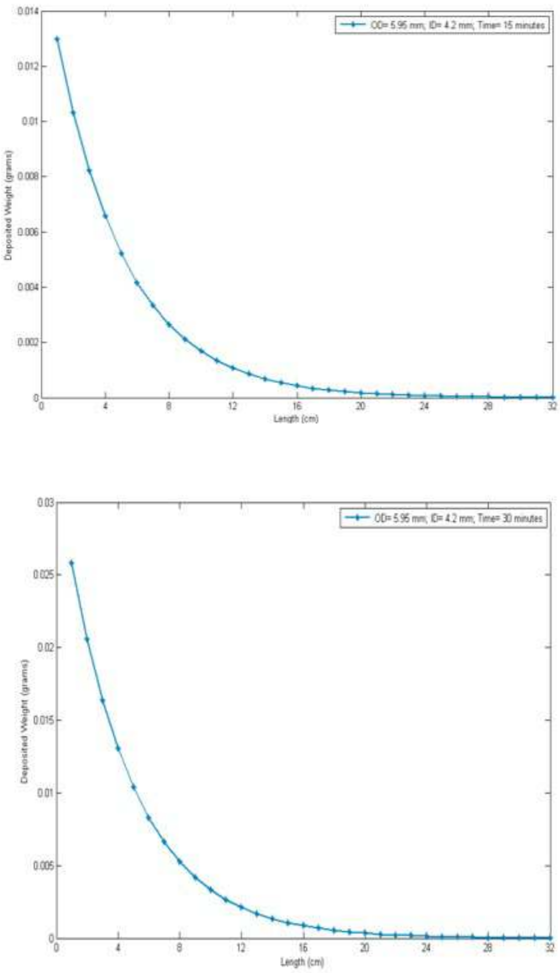
$$C_A = C_{A0}\left(e^{-4\pi r D_{AB} x}\right)\left(e^{\frac{2\pi r D_{AB} t}{a}}\right)$$

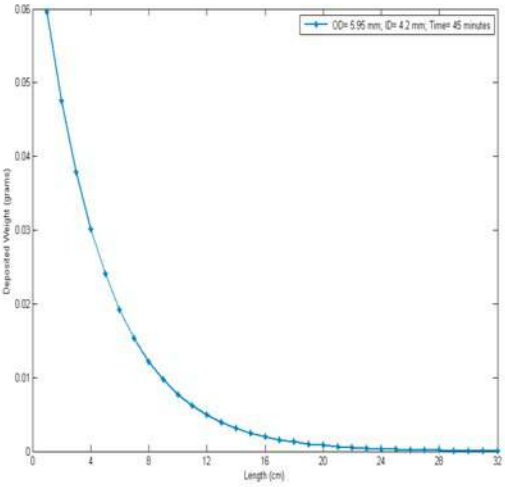
At this stage MATALB code was developed to solve equation and the computational results are based on assumptions which are incorporated in computational model.

The deposition rate variation

At the inlet of the capillary tube is much more then experimental results, but become constant at the exit. Computational results of same diameter of capillary tube with different time periods are shown in Figure 7.

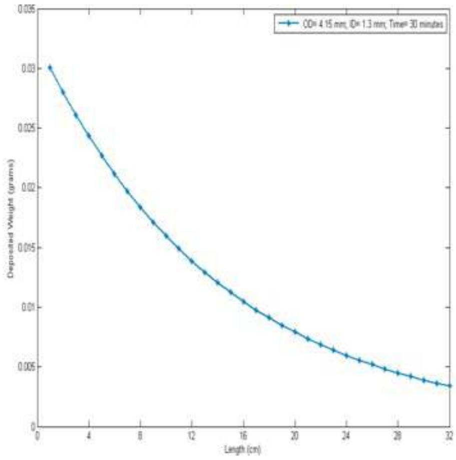
Figure 7. Computational Results of deposited weight vs Length for 15 mints, 30 mints and 45 mints



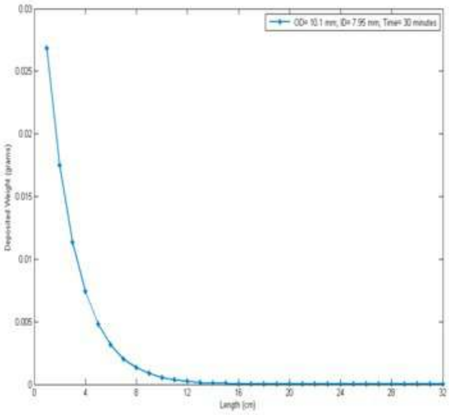


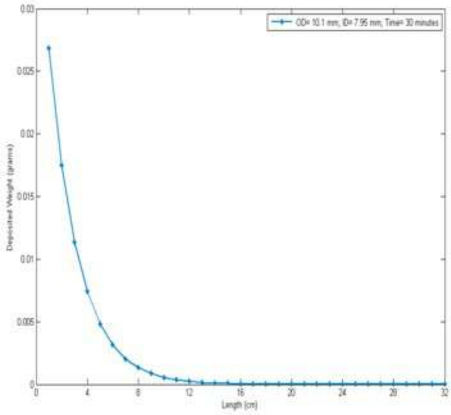
Computational results of different diameter of capillary tube with same time periods

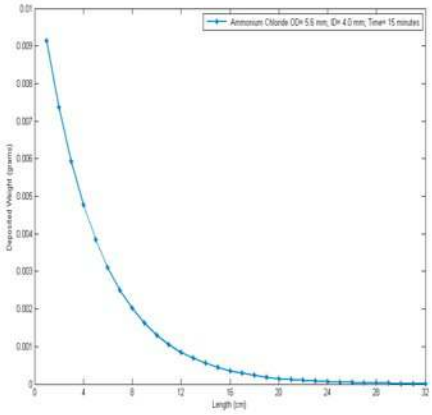
Figure 8: Computation Result of Deposited Weight Vs Length for 30 minutes with ID=1.3mm and ID=7.95 mm



Computational result of same diameter of capillary tube with same time periods but in this case sublime material is Ammonium chloride.







CONCULSION

The variation of deposit weight of Naphthalene and Ammonium chloride along the length of same and different diameter of capillary tubes are experimented for different and same time periods in order to study cooling load variation by the formation of solid layer by desublimation process. From this experimental study, various results are concluded:

- Deposited weight is dependent upon time, as time increases so that deposited weight increases but decreases with tube length.
- Tube Diameter is function of Deposited Weight, as tube diameter increases so that Deposited Weight decreases.
- Deposited Weight is also dependent upon thermal conductivity of tube, as in our case Borosilicate ((20°C) 1.14W/m°C) have high value of (k) then Pyrex(1.005W/m°C), as thermal conductivity of tube increases so that Deposited Weight increases.
- Deposited weight is dependent upon sublime material, as Naphthalene

deposition rate is greater than Ammonium chloride.

- Temperature of bulk material is not significantly vary with time due to small tube diameter.

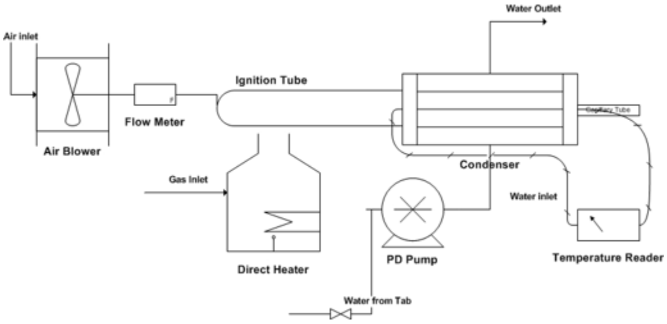


Figure 1 : Experimental setup

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RECOMMENDATIONS

- The parameters which effects on Deposition thickness can be analyzed in prototype system.
- Deposition thickness can be analyzed by varying Coolant flow rate, type of Coolant, Air flow rate and mole fraction of sublime material.
- The analysis can be done by using Simulation for 2D and 3D Geometry of tube.

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