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OPTIMIZATION OF FREEZE DRYING SYSTEM

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MASTER'S THESIS

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ACCEPTANCE AND APPROVAL

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LIST OF ABBREVIATIONS

CFD : Computational Fluid Dynamics

L/D : Length to width ratio

N₂ : Nitrogen Gas

T_g : Glass Transition Temperature

H₂O : Water Molecule

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ÖZET

DONDURARAK KURUTMA SİSTEMİNİN OPTİMİZASYONU

Abbakar M, Aydın Adnan Menderes Üniversitesi, Fen Bilimleri Enstitüsü, Makine Mühendisliği, Yüksek Lisans Tezi, Aydın, 2022.

Dondurarak kurutma, süblimasyonu kurutma aracı olarak kullanan bir kurutma tekniğidir. Bu işlem, malzemeleri kurutmanın en etkili yollarından biridir. İşlem ürünü etkili bir şekilde kurutabilse de, işlem genellikle pahalı ve uzundur. Süreci daha verimli hale getirmek için yapılmış birçok çalışma bulunmaktadır. Bu çalışmada kullanılan yöntem, su buharı kütle akışını arttırmak üzerinedir. Bu tez, işlemin verimliliğini artırmak için çoklu kanal kullanımının nasıl etkili bir yol olabileceğini tartışmaktadır. CFD analizinden elde edilen veriler, kanalların yerleşiminin ve kanal sayısının dondurarak kurutucunun performansı üzerinde önemli bir etkisi olduğunu göstermiştir.

Anahtar Kelimeler: liyofilizasyon; ANSYS; süblimasyon; CFD, Fluent.

ABSTRACT

OPTIMIZATION OF FREEZE DRYING SYSTEM

Abbakar M, Aydın Adnan Menderes University, Graduate School of Natural and Applied Sciences, Mechanical Engineering, Master Thesis, Aydın, 2022

Freeze drying is a drying technique that uses the sublimation as a means of drying. This process is one most effective ways of drying materials. Though the process might dry the product effectively, the process is often expensive and long. There are many studies that have been done to make the process more efficient. One method that was performed is by increasing the water vapor mass flow. This thesis discusses how the use of multiple ducts might be an effective way for to increase the efficiency of the of the process. The data from the CFD analysis showed that the placement of the ducts and the number of the ducts had a significant effect on the performance of the freeze dryer.

Key Words: Lyophilization; ANSYS; Sublimation; CFD, Fluent.

1. INTRODUCTION

1.1. Background

Lyophilization or Freeze drying is a drying method used for heat sensitive materials. Lyophilization utilizes sublimation to dry the product. This method of drying is very effective in removing the water molecules from the product. Approximately 95%-99% of water molecules are removed during the process. Freeze-dryers are mainly used in pharmaceutical and food industry as there are many products there that are damaged if not preserved well. The lyophilization process consists of three phases:

- I. Freezing phase,
- II. primary drying,
- III. and secondary drying.

The freezing approach, freezing temperature and freezing time are very important factors that are needed to take into consideration before the drying process. During the pre-treatment, along with freezing, the freezing technique modifications the cellular shape which in turn impacts the rate of freezing. The approach wherein the material is frozen performs an essential role inside the whole freeze-drying manner.

Usually, the quicker the freezing rate, the smaller the ice crystals which ends up in longer drying times, consequently giving a better very last product great. On the other hand, slow freezing rates create large ice crystals which may damage the inner cellular walls of the material which may affect the final dried product. Due to this, the freezing phase is one of main crucial factors that should always be considered before a product is dried.

The primary drying phase is where most of the water molecules are removed. During the operation the pressure is dropped down to almost vacuum levels and the temperature is dropped sub-zero levels (approximately -20C). The products are placed on top shelves which mostly having heating plates beneath them. Through this the ice molecules go through sublimation.

The water vapor will then travel from the main chamber via a duct to the condenser. The goal of secondary drying is to get rid of unfrozen water molecules, due to the fact not all the ice was removed during the primary drying segment. This is done through the method of desorption. To accomplish the best conditions for desorption, the bottom feasible pressure in addition to a better shelf temperature is needed. Product stability ought to be taken into consideration whilst selecting the shelf temperature. on the end of the process, the moisture content material inside the product must be within the variety of 1-9%.

A freeze dryer mainly consists of:

- The main drying chamber (Vacuum chamber)
- Heating plates
- Condenser chamber
- A duct connecting the vacuum chamber and condenser chamber

Within the product chamber is wherein the shelves are stored. There is generally an inlet duct that is located at the top of the chamber that carries the refrigerant that may be used to control the pressure inside the chamber.

Connecting the product chamber and the condenser chamber is a duct. The duct performs a crucial function within the freeze-drying operation. A duct that isn't always well designed may be the primary purpose as to why the product is not dried properly.



Figure 1.1. Commercial freeze dryer (courtesy Allied Rental Company)

1.2. Drying process

Drying is a mass transfer operation where a liquid is removed from a material. This process often requires a source of heat to provide energy for the mass transfer to occur. There are 3 main methods of drying:

I. Direct (Conductive Drying)

This drying process includes direct touch among product and heating medium.

II. Indirect (Convective Drying)

In this method, drying occurs from a heated surface in touch with the product. The heating medium and the product are separated via a wall.

III. Radiation drying

This method of drying doesn't require an intermediate such as water or air. Infrared rays are directed towards the material to heat up the water molecules via radiation energy.

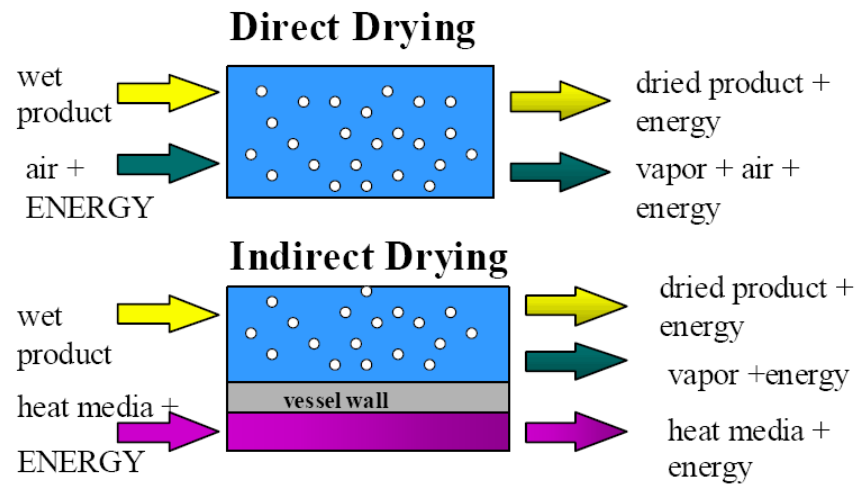


Figure 1.1. Drying and indirect drying

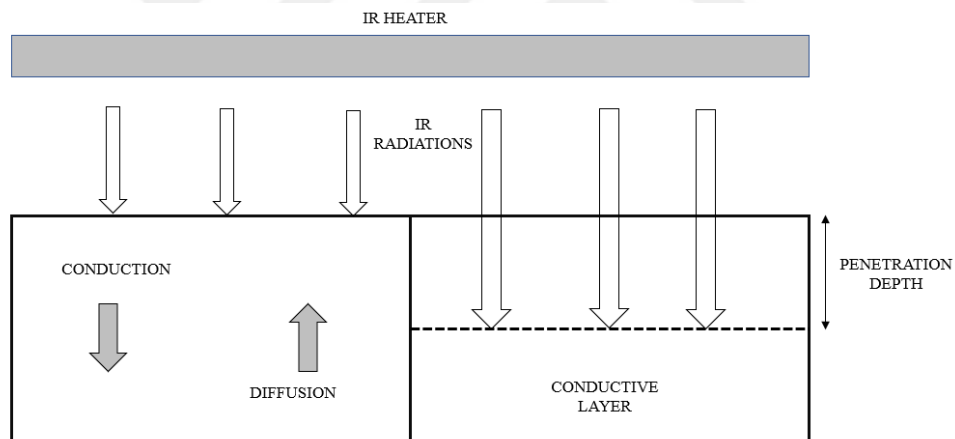


Figure 1.2. Radiation drying (courtesy Sanjay B. Pawar)

1.3. Sublimation drying process

Sublimation is a process where in a substance changes its state from solid-to-gas without going through the liquid state. This can be achieved by reaching the triple point of the material. The triple point is the state where the molecule can exist in all phases (solid, liquid and gas).

The triple point can be reached by precise control of the pressure and temperature of the material. The vapor pressure charts can be utilised to determine the trip point of water as shown in figure 1.5.

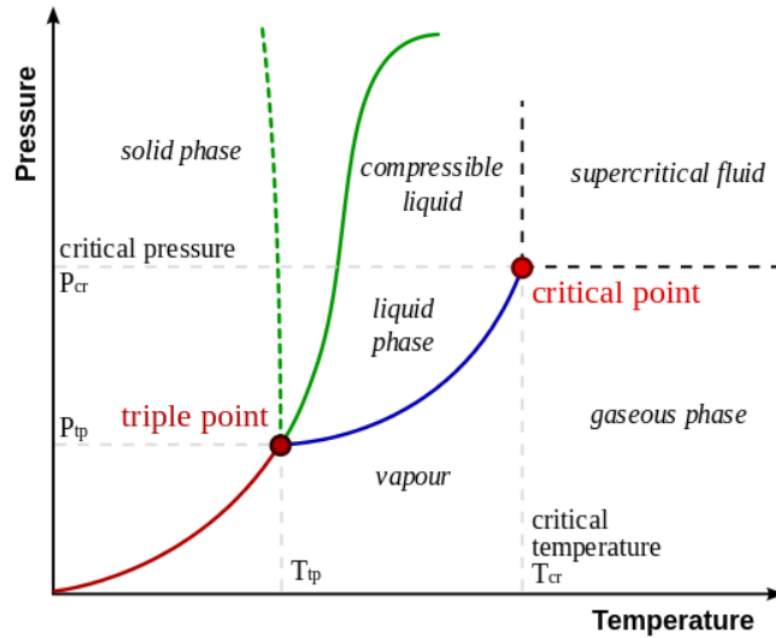


Figure 1.3. Triple point of water graph (courtesy Wikipedia)

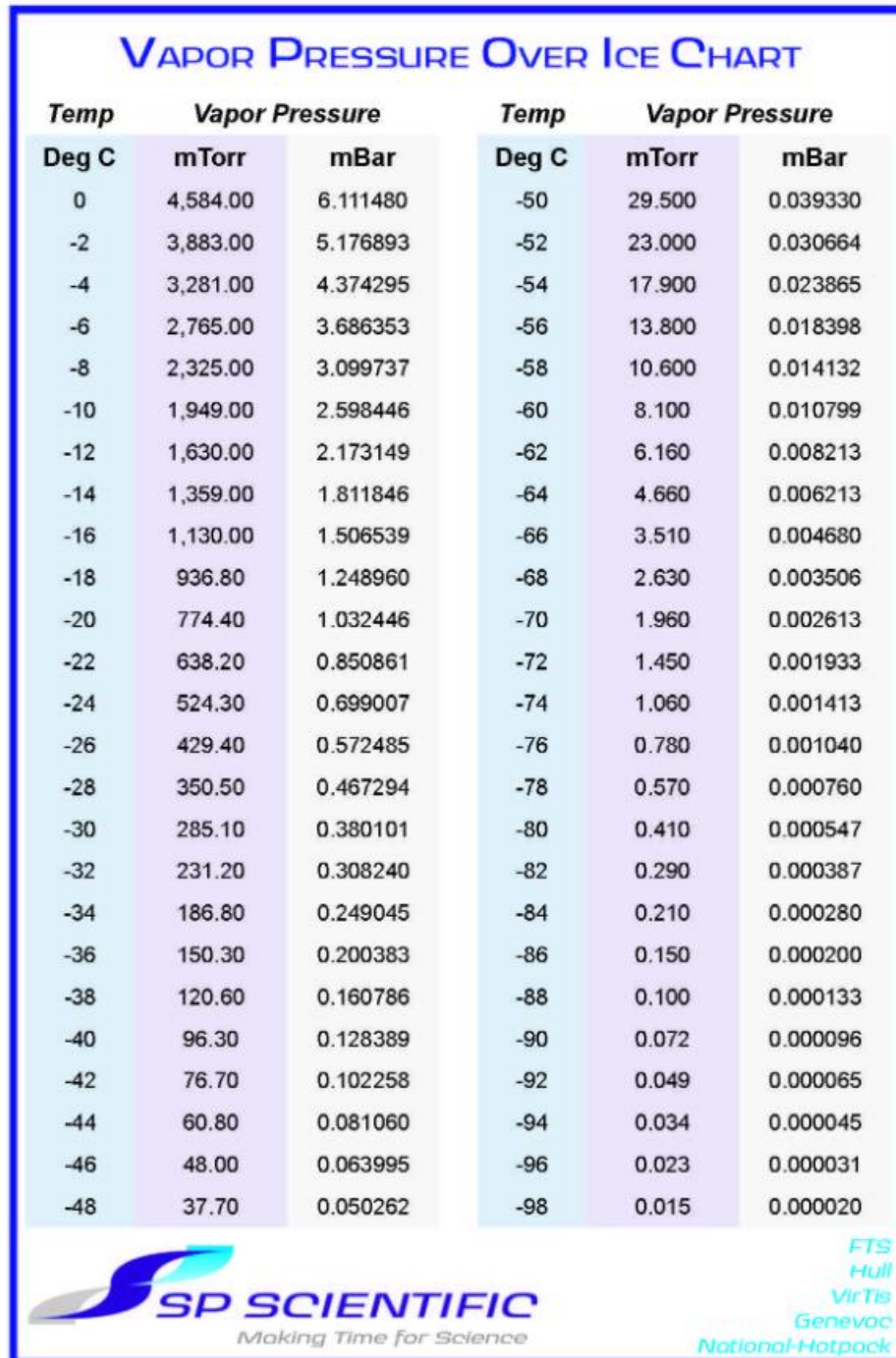


Figure 1.4. Vapor pressure over ice chart

1.4. Glass transition temperature (T_g)

This is the temperature where the material is between a glassy and rubbery state. This is an extremely crucial factor in freeze drying. During the primary phase, the shelves are heated

to a specific temperature. If the material is heated beyond the glass transition temperature, the product will have a rubbery structure which can stick to the heating plate making it hard to remove thus damaging the final dried product.

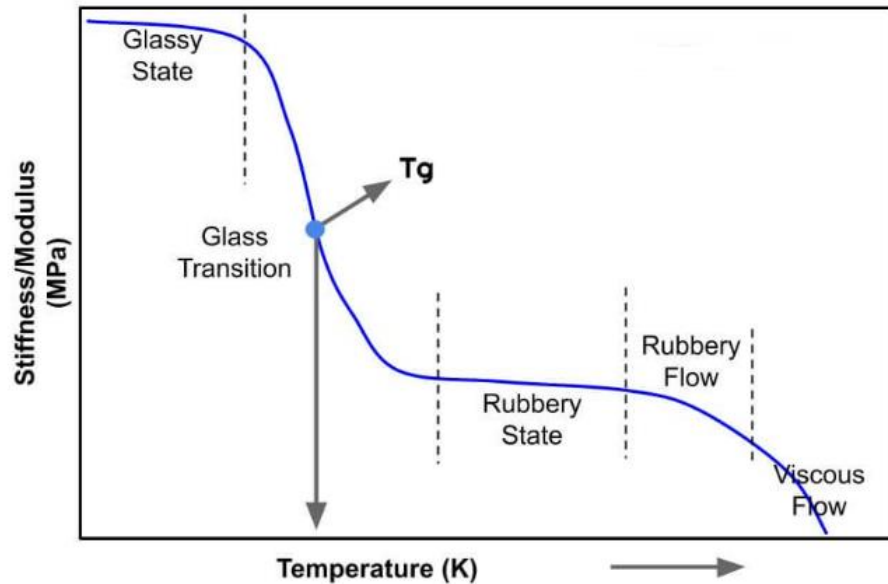


Figure 1.5. Glass transition temperature graph vs Temperature (Courtesy Corrosionpedia)

1.5. Mass transfer

During the drying phase, the water vapor needs to be transported otherwise the pressure inside the chamber may increase. This will then in turn increase the temperature inside the chamber the ice molecules will melting will occur instead of sublimation. To avoid this a vacuum pump will be used to transport the vapor from the drying chamber via a duct to a condensing chamber. Another method to improve the transport of the water vapor would be the freezing method. (Carvalho, Teresa de Melo, 2018)

1.6. Freezing methods

The molecular structure of a material can easily be changed by the type of freezing used. When a product is frozen, the water molecules inside change their size and structure depending on the freezing time. There are two main methods of freezing: fast freezing and slow freezing.

Slow freezing creates disruptive large ice crystals which damage the cell walls of the product. Since the ice crystals are larger, it would take more time and heat to sublime them. However, it's a much cheaper option than fast freezing. In fast freezing the ice crystals formed are much smaller and more uniform in size. The smaller ice crystals do not damage the cell walls of material. This works well for the freeze dryer as the final dried product would have a longer shelf-life since very little damage was done during the freeze-drying process (Mohammad Shafiur Rahman and Conrad O. Perera). However, there are downsides to this type of freezing. The main disadvantages are.

- The process is expensive,
- The extreme low temperature may affect the colour and flavour of the material.

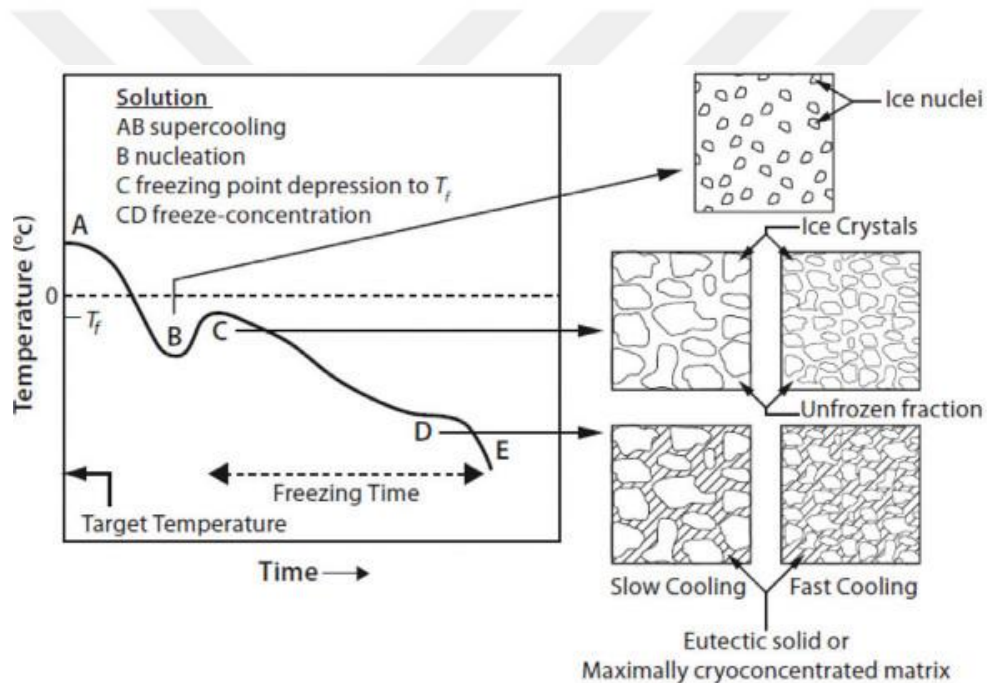


Figure 1.6. Slow freezing and fast freezing of a water molecule (courtesy American Pharmaceutical Review)

2. LITERATURE REVIEW

2.1. Application of CFD in freeze drying

The process of freeze dryers has always been expensive and time consuming compared to other drying processes. However due to advancements in technology, the use of computational Fluid dynamics (CFD) has made analysis and development of freeze dryers easier and cheaper. Using CFD, it is possible to analyse different sections of the freeze dryer to optimize the final design.

Many papers have been written regarding the different components of the freeze dryer and how different design aspects can affect the quality of the final dried product. There are many factors that affect the final quality of the product. Some include:

- Gap between shelves,
- position of duct between primary drying chamber and secondary drying chamber,
- length to Diameter (L/D) ratio of duct,
- the use of a butterfly valve,
- the efficiency of condenser,
- thickness of sample.

2.2. Flow regime

It is very important to observe the flow patterns that occur inside the drying chamber. In vacuum studies, there are three main flows that occur: molecular flow, viscous flow, and Knudsen flow. Since the pressure inside the freeze dryer is extremely low, we can assume that the viscous flow is not applicable for the modelling of the CFD setup.

The Knudsen number (Kn) is a dimensionless number that is defined as the ratio of the molecular mean free path length to a representative physical length scale. This length scale could be, for example, the radius of a body in a fluid (Carvalho, Teresa de Melo, 2018). The flow inside the drying chamber is almost all laminar so the Knudsen number range can be between $0.01 < Kn < 0.1$.

$$kn = \frac{\lambda}{r}$$

Where λ is the mean free path

r is the physical length scale

2.3. Modelling to Equipment Scale

Whilst designing a freeze-dryer, crucial properties that are dependent on water vapor flow and system design ought to be addressed. The general design parameters that influence the overall performance of a freeze-dryer are the geometry of the condenser and, for freeze-dryers with condensers separated from the freeze-drying chamber, the scale and geometry of the duct and the isolation valve. The experimental examination of these topics is expensive and time consuming, and consequently it is simpler to observe them through a computational method like Computational Fluid Dynamics (CFD).

2.4. Duct to condenser

Research has been performed with the purpose of understanding the resistances to vapor flow from the product chamber to the condenser course while the condenser is positioned outside the drying chamber (S. M. Patel, S. Chaudhuri, and M. J. Pikal, 2010). The main loss of pressure control was the water vapor “choking” within the duct. The cause of choking took place because of volatile working situations in which the mass flow couldn't be maintained, and the condenser couldn't be maintained on the managed strain. The flow rate of the water vapor increases as the compressor pressure is reduced until the velocity of the water reaches the

velocity of sound at the end of the exit of the duct. After that moment, if the water vapor flow rate will increase any further, then the pressure in the product chamber will increase. This problem doesn't regularly arise in gradual freeze-drying cycles. but, because the need for need for a great deal higher ability freeze-dryers has increased, it is important to discover situations under which choked flows occur. studies were done to observe the important parameters responsible for the discrepancy while scaling up from laboratory to industrial freeze-dryers (A. Alexeenko et al, 2009).

2.5. Condenser

Many different designs had been made with the intention to enhance the performance and efficiency of the condenser. The designs defined the fluid dynamics, the method and patterns of the ice deposition inside the condenser. The studies showed that the duct connecting the product chamber and condenser increased the non-uniformity of on ice increase and that the presence of non-condensable gases increased the powerful resistance within the condenser, stopping the vapor from condensing on the coils far from the duct go out (A. Ganguly et al, 2012). The region and topology of the condensing coils extensively influence the ice uniformity and growth. The layout and hardware used inside the condenser significantly influence the vapor route and flow behaviour (M. Petitti, A. A. et al2013).

2.6. Shelf Plate Temperature

Shelf temperature is a crucial element especially at the end of the drying (secondary drying section) has ended. a few tips and equations have been written in an effort to determine ideal shelf temperature as a characteristic of primary drying situations (Charlie Tang,Michael J Pikal, 2004). This was done to preserve the product chamber throughout sublimation always under its collapse temperature. but other papers have observed that even though shelf temperature has an effect on the product temperature for the duration of sublimation, the outcomes become a lot smaller as compared to the chamber pressure effects on the rate of

sublimation. for the duration of primary drying the sublimation rate is also significantly affected by shelf temperature (Hong-Ping Cheng et al 2014).



3. MATERIAL AND METHOD

3.1. Computational Model

The analysis will be setup using ANSYS using the FLUENT module. The placement of the ducts and different duct diameter would be analysed to see how they affect the pressure and mass flow within the chamber.

As mentioned previously, since the Knudsen number for flow inside a vacuum is below 0.1, then the mechanical flow can be modelled using the Navier-Stokes equations. In addition to that, our model will also utilize the species transport equations and energy equations. The 3D model that will be used will have the following dimensions 0.6m x 0.6m x 0.6m. The model will consist of 4 shelves with a 100mm gap between them. On the top of the chamber there is a small inlet duct of 50mm diameter. This inlet is where nitrogen (N₂) gas will be released. This is one method in which the pressure inside the chamber is controlled (Varma, Nikhil P, 2014).

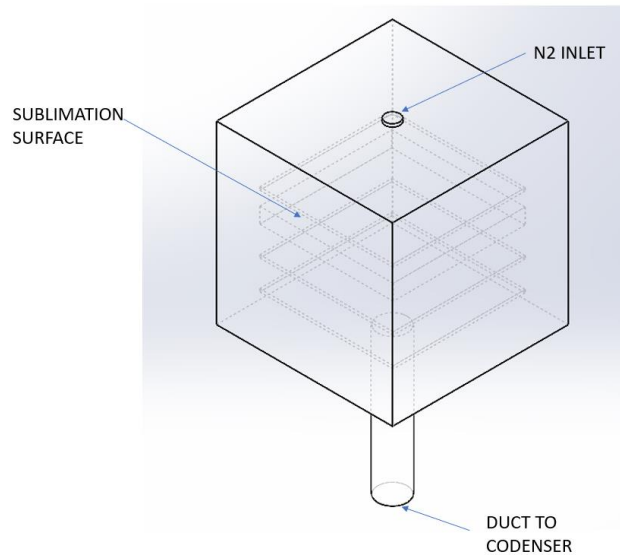


Figure 3.1. 3D model of freeze dryer

3.2. CFD setup

There are 5 different models that were analysed, each with different duct positions. The 3D models were first meshed using Ansys meshing module. The mesh had approximately 2,400,000 elements and 2,500,000 nodes between all 5 3D models. All the 3D models have a standard shelf gap of 100mm, an inlet duct diameter of 50mm and an outlet duct surface area of $0.08m^2$.

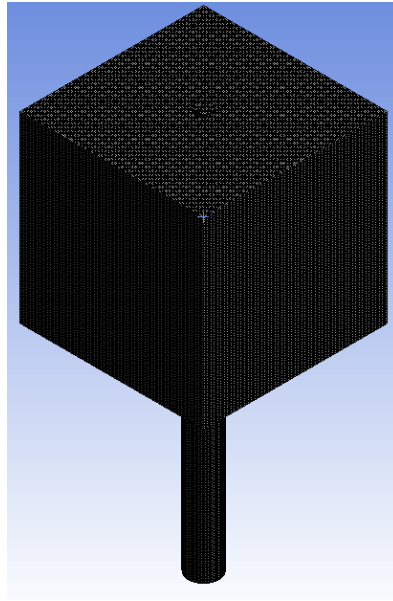


Figure 3.2. General mesh of freeze dryer

3.3. Boundary conditions

Table 3.1. Boundary condition

Model	N2 inlet pressure (Pa)	N2 inlet temperature (K)	Sublimation surface mass flux range (kg/m²-s)	Sublimation surface temperature (K)	Outlet to condenser pressure (Pa)	Outlet to condenser temperature (K)
1	20, 15, 10	300	0.25, 0.5, 1	273	5, 2	190
2	20, 15, 10	300	0.25, 0.5, 1	273	5, 2	190
3	20, 15, 10	300	0.25, 0.5, 1	273	5, 2	190
4	20, 15, 10	300	0.25, 0.5, 1	273	5, 2	190
5	20, 15, 10	300	0.25, 0.5, 1	273	5, 2	190

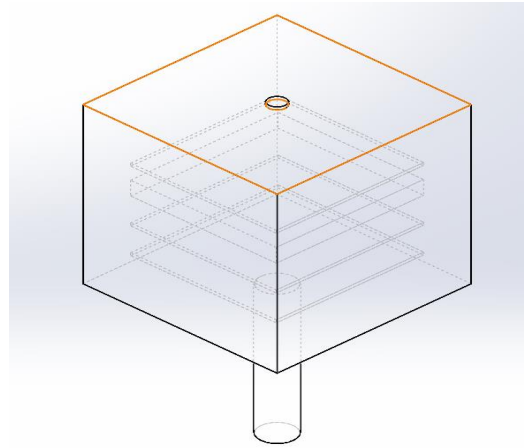


Figure 3.3. Model 1

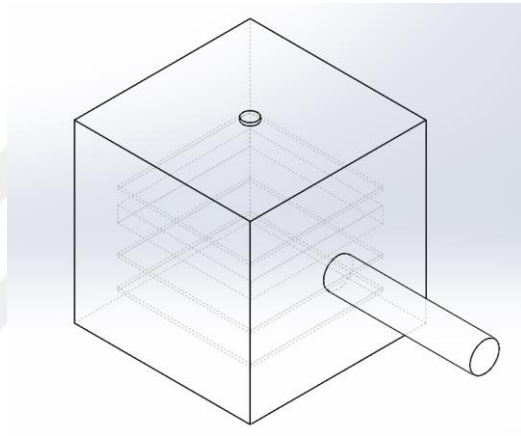


Figure 3.4. Model 2

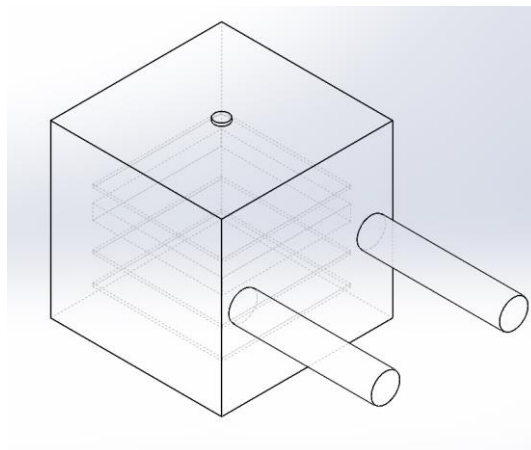


Figure 3.5. Model 3

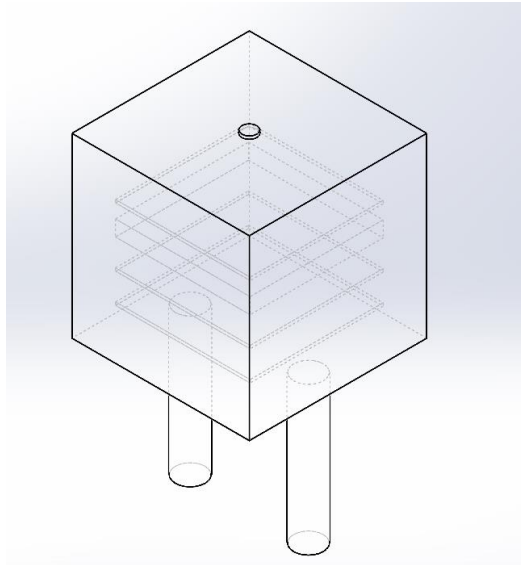


Figure 3.6. Model 4

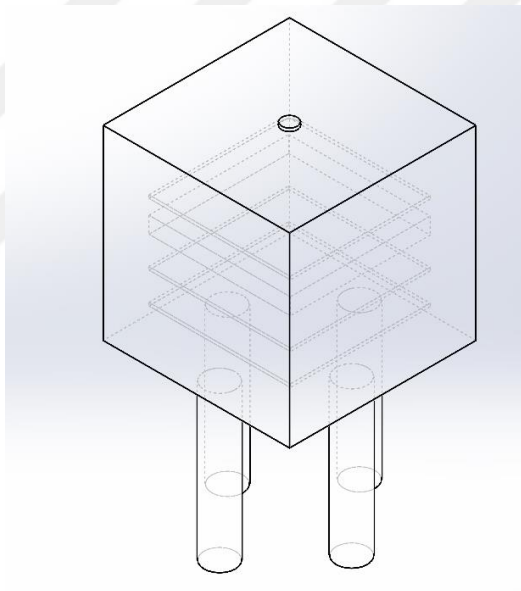


Figure 3.7. Model 5

4. RESULTS AND DISCUSION

One of the main methods of making a freeze dryer more efficient is by increasing the rate of sublimation. That means we are mainly focused on the mass flow exiting the duct. The results below will show how the different duct positions affect the flow rate and how the other different boundary conditions affect the flow rate and flow distribution.

4.1. Flow distribution

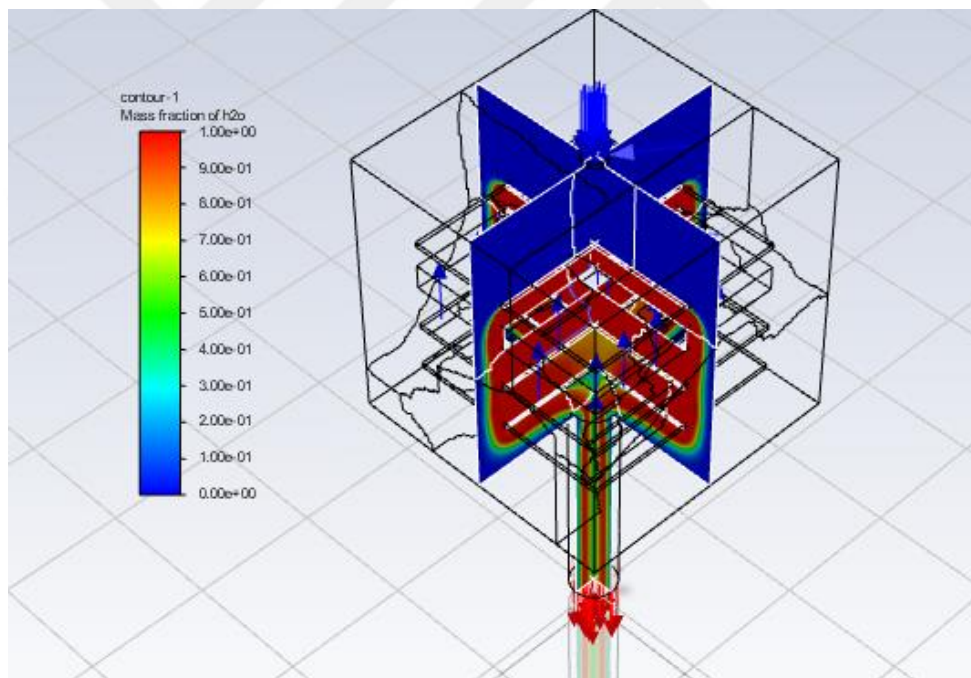


Figure 4.1. H₂O mass fraction of model 1

Model 1 of the analysis has a symmetrical body and a duct that is placed vertically under the heating plates. Figure 4.1 shows the flow distribution of the water vapor inside the drying

chamber (Hong-Ping Cheng et al, 2014). The results shows that the flow within the chamber is directed towards the duct and the flow is distributed evenly.

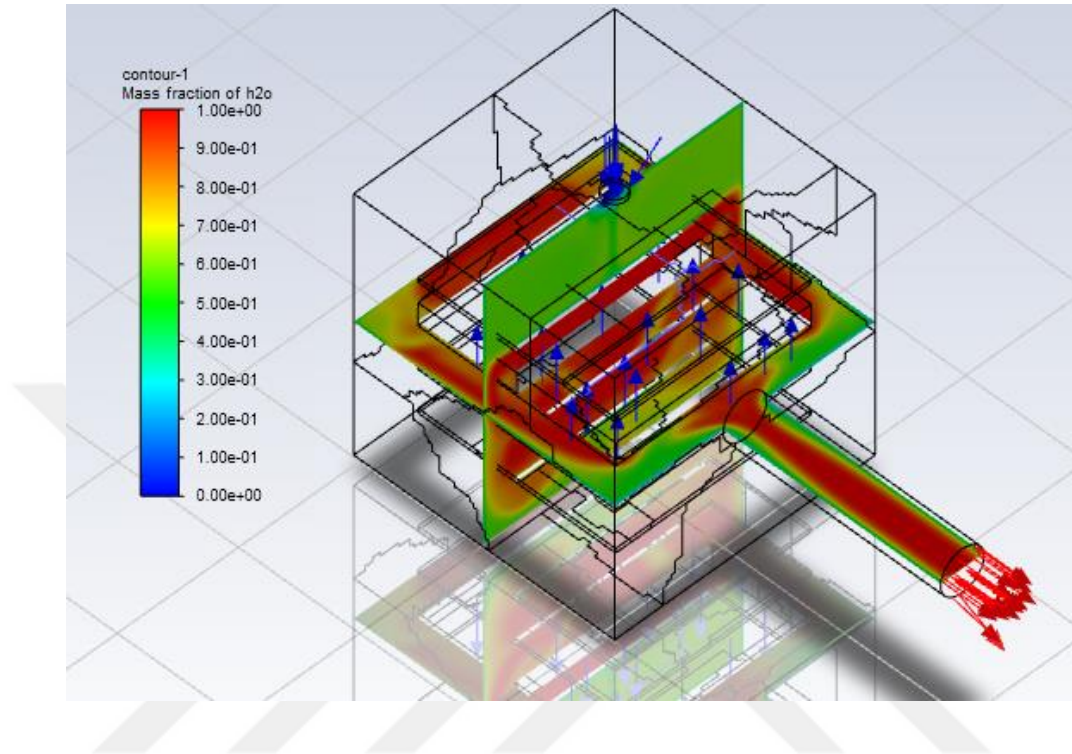


Figure 4.2. H₂O mass fraction of model 2

In figure 4.2 the duct is placed horizontally on the sides of the freeze dryer. The flow distribution of the water vapor is more unstable and uneven as compared with figure 4.1. Some of the water vapor is also seen to move near the inlet duct and the distribution of the of the flow near the outlet duct is also uneven.

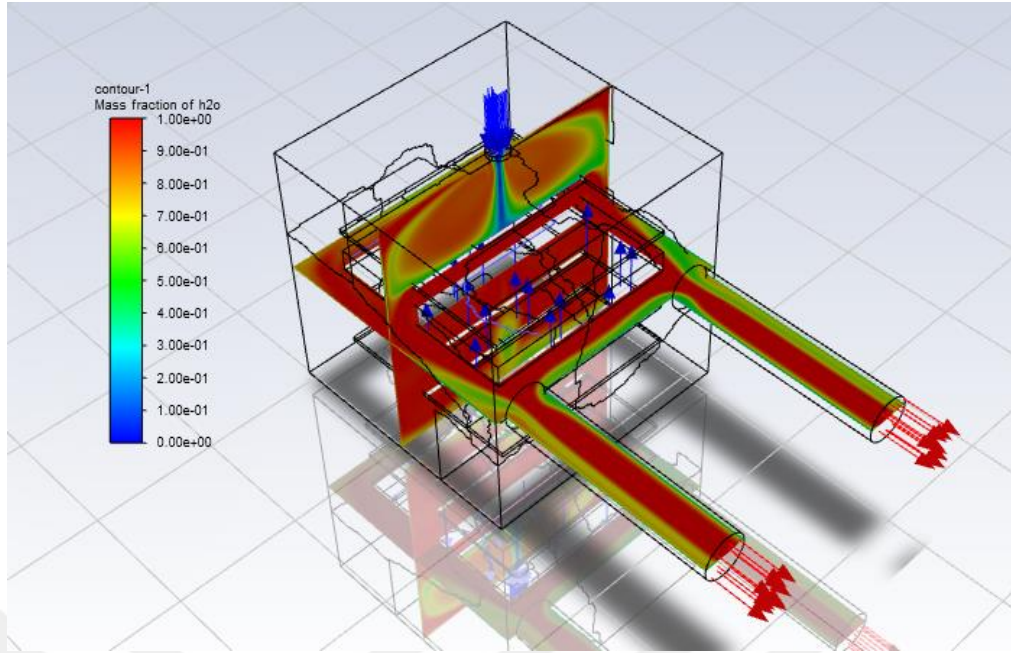


Figure 4.3. H₂O mass fraction of model 3

The outlet ducts of model 3 shown in figure 4.3 are also placed vertically similar to the ducts in figure 4.2. However, an extra duct was placed with similar dimensions with the figure 4.2. The flow distribution is very similar to that of figure 4.2 but a lot more water vapor is spread around the chamber. The water vapor inside the outlet ducts are spread evenly but near the nitrogen gas inlet there is a lot more disturbance of flow than the previous models mentioned.

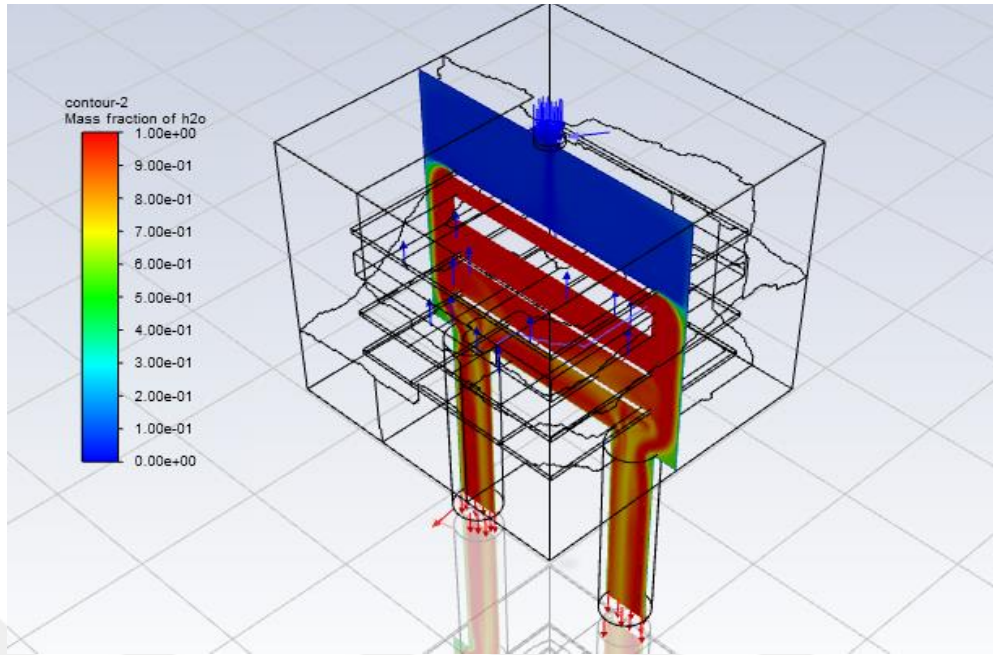


Figure 4.4. H₂O mass fraction of model 4

In figure 4.4 the model has 2 ducts with the same dimensions, but the ducts are placed vertically underneath the heating plates. The flow distribution of this model is very similar to that of model 1 shown in figure 4.1. There is a lot more water vapor inside the outlet ducts than compared to figure 4.1.

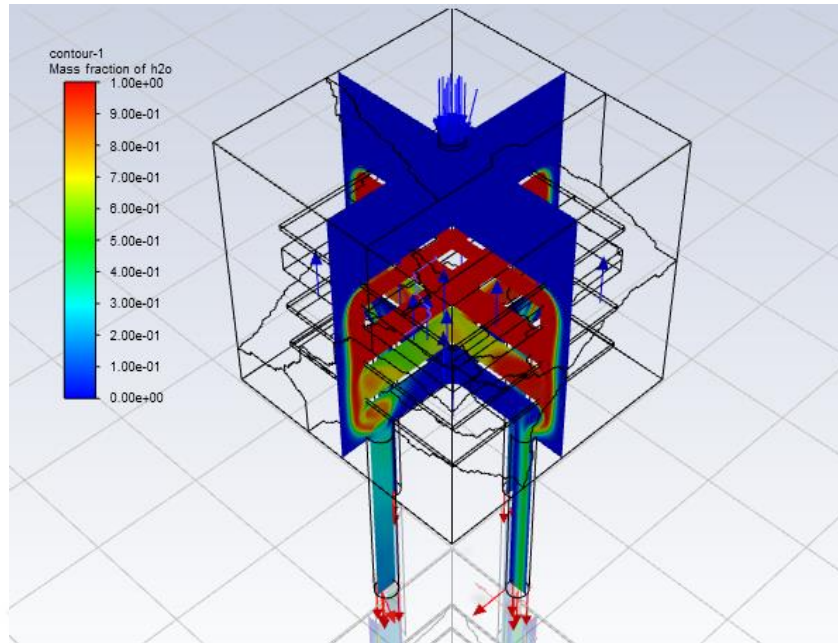


Figure 4.5. H₂O mass fraction of model 5

Finally model 5 has 4 outlet ducts placed at the bottom underneath the heating plates. The flow distribution is near the outlet duct entrance is more unstable and even than that of figure 4.1 and 4.4.

4.2. Temperature distribution on drying plate

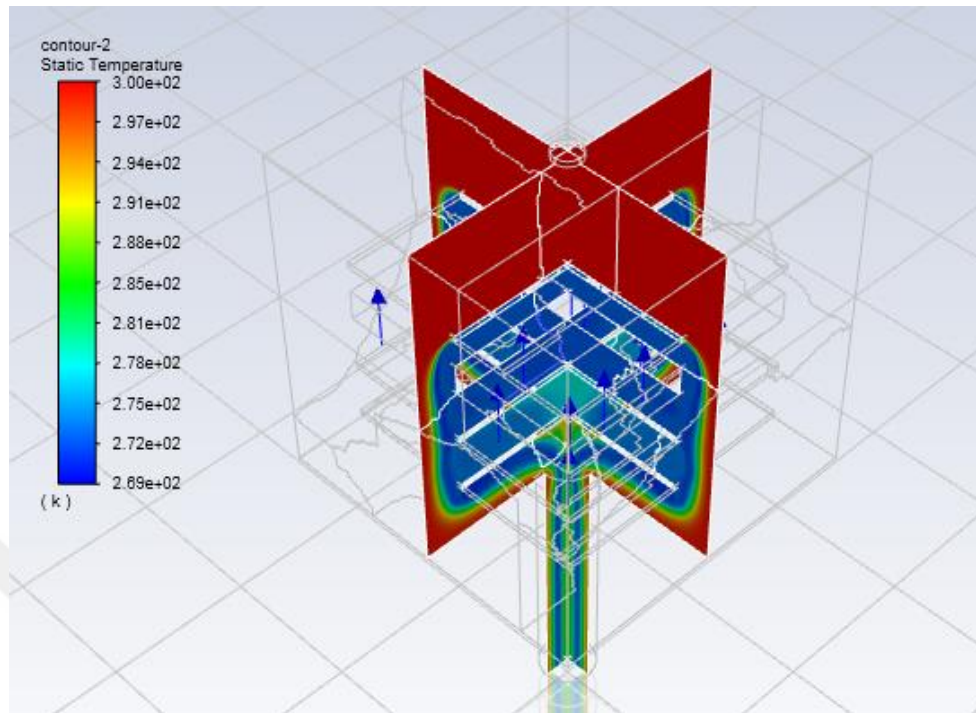


Figure 4.6. Temperature distribution on drying plate of model 1

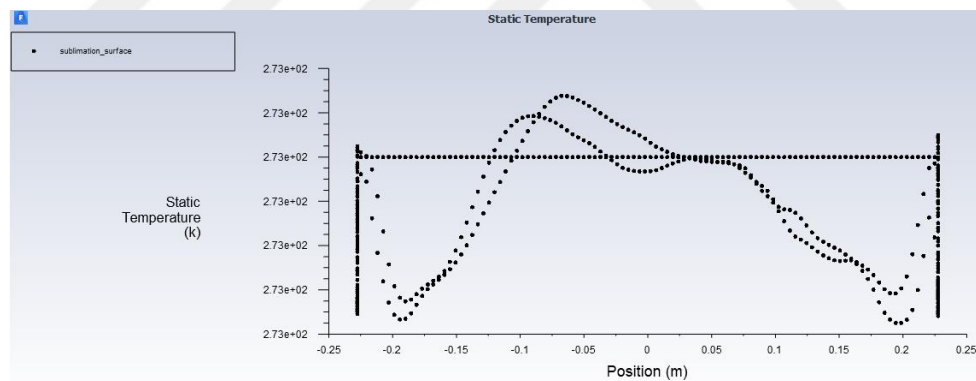


Figure 4.7. Temperature distribution graph on drying plate of model 1

The temperature distribution is another important factor to take note of. Fig 4.6 shows the distribution of temperature within the chamber. The temperature within the chamber is shown to be spread evenly. Fig 4.7 represents the how the temperature is distributed on the heating plate. The graph shows some symmetrical patterns across the plate.

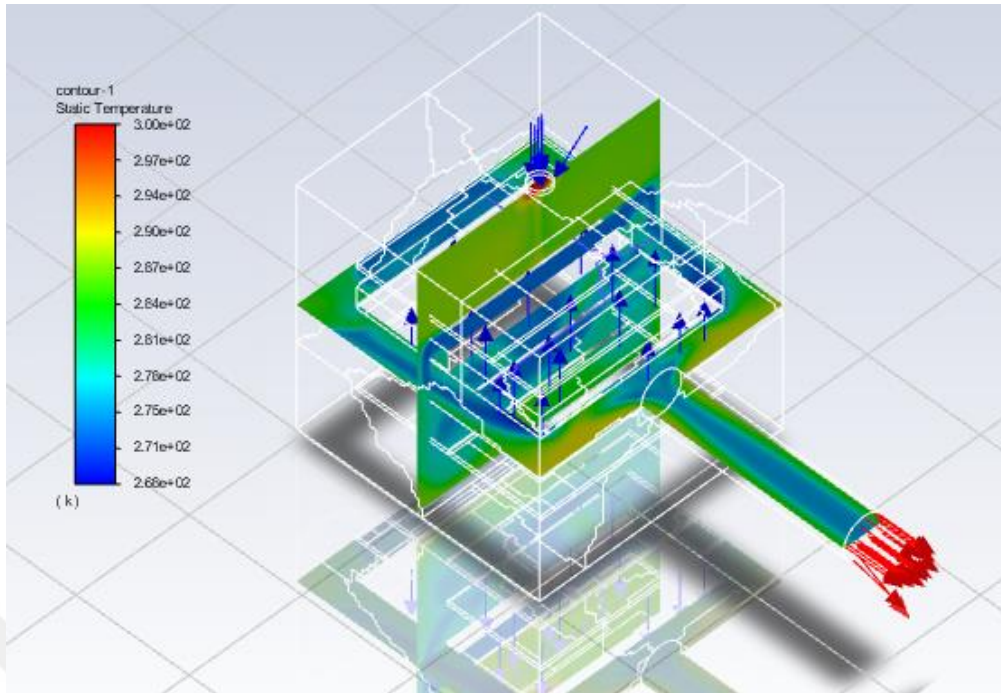


Figure 4.8. Temperature distribution on drying plate of model 2

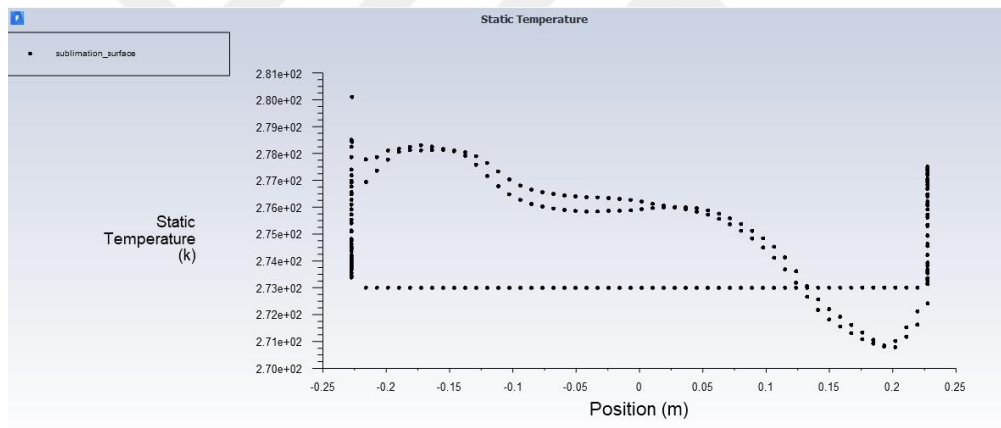


Figure 4.9. Temperature distribution graph on drying plate of model 2

In model 2 we can see that the temperature has been spread across the chamber. The overall temperature across the chamber has dropped. The graph shown in figure 4.9 shows the unsymmetrical pattern of the temperature.

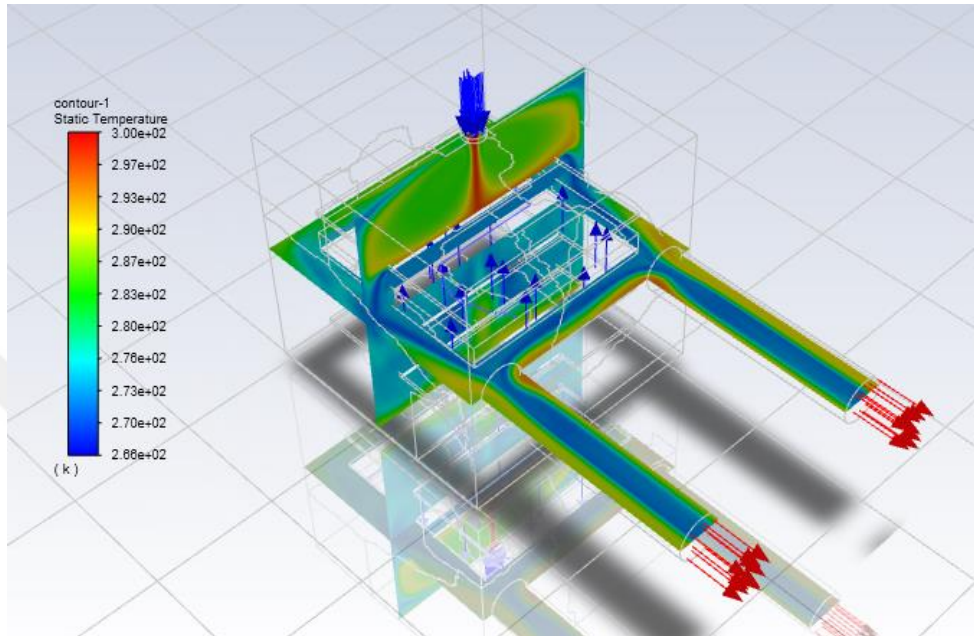


Figure 4.10. Temperature distribution on drying plate of model 3

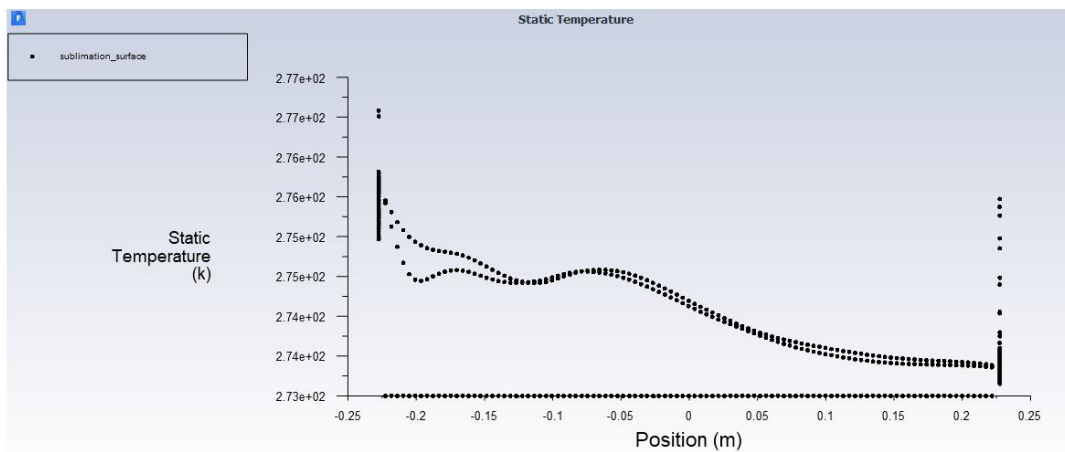


Figure 4.11. Temperature distribution graph on drying plate of model 3

Similarly in figure 4.10 the temperature has also been spread across the chamber. However, model 3 shows a lot more uneven areas of temperature as compared to model 2 (figure 4.8).

The distribution of temperature across the sublimation surface also has an unsymmetric pattern as seen in figure 4.11.

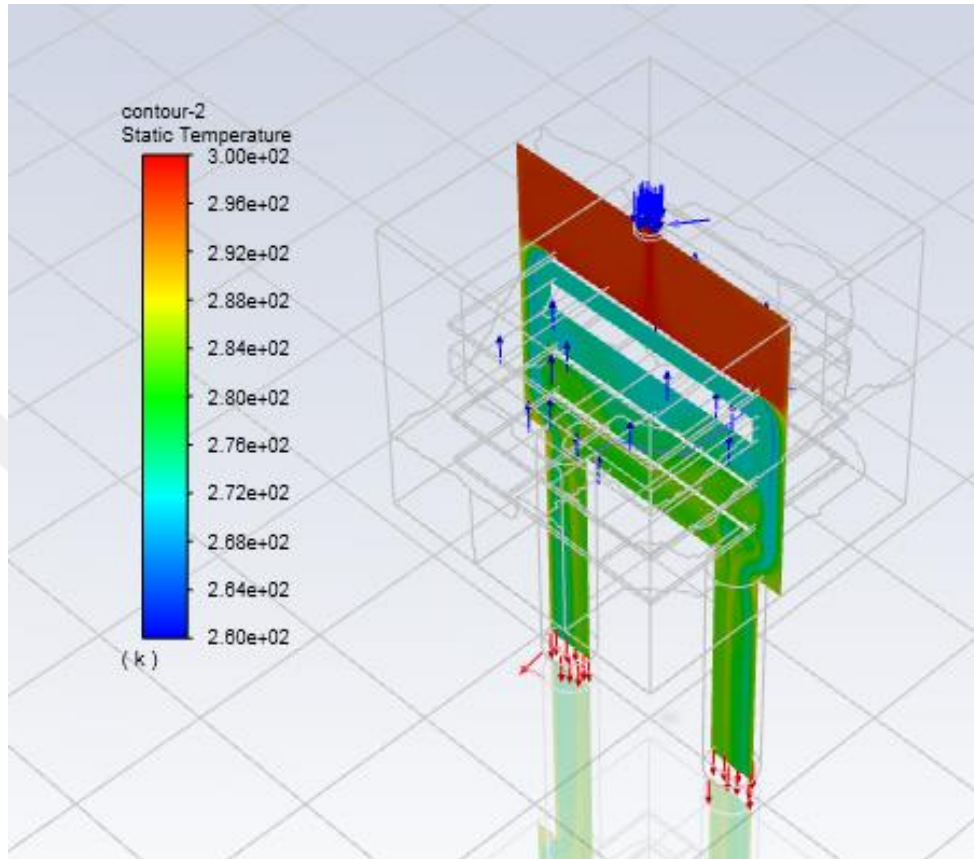


Figure 4.12. Temperature distribution on drying plate of model 4

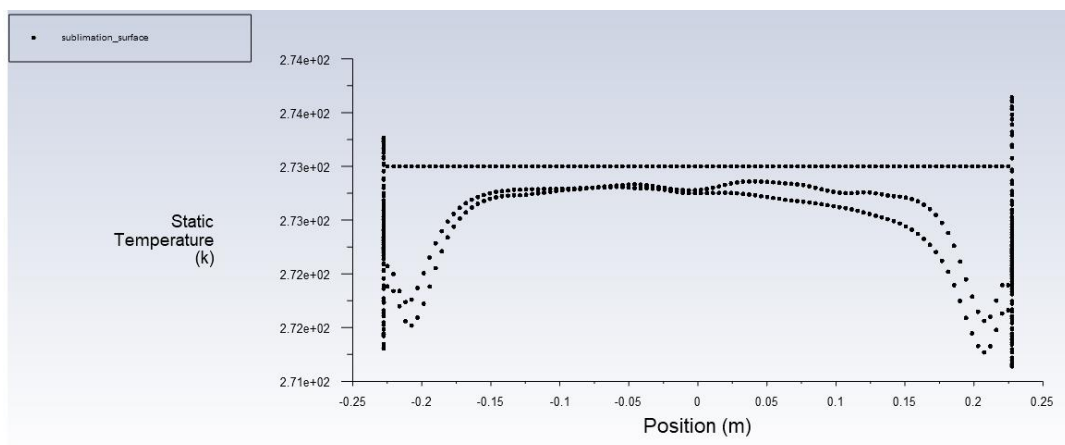


Figure 4.13. Temperature distribution graph on drying plate of model 4

In figure 4.12 we see that there is a difference in temperature. The temperature inside the chamber is seen to be higher starting from above the sublimation surface and is considerably a lot lower underneath the sublimation surface. The temperature distribution across the drying plates in figure 4.13 is shown to be very symmetrical and even.

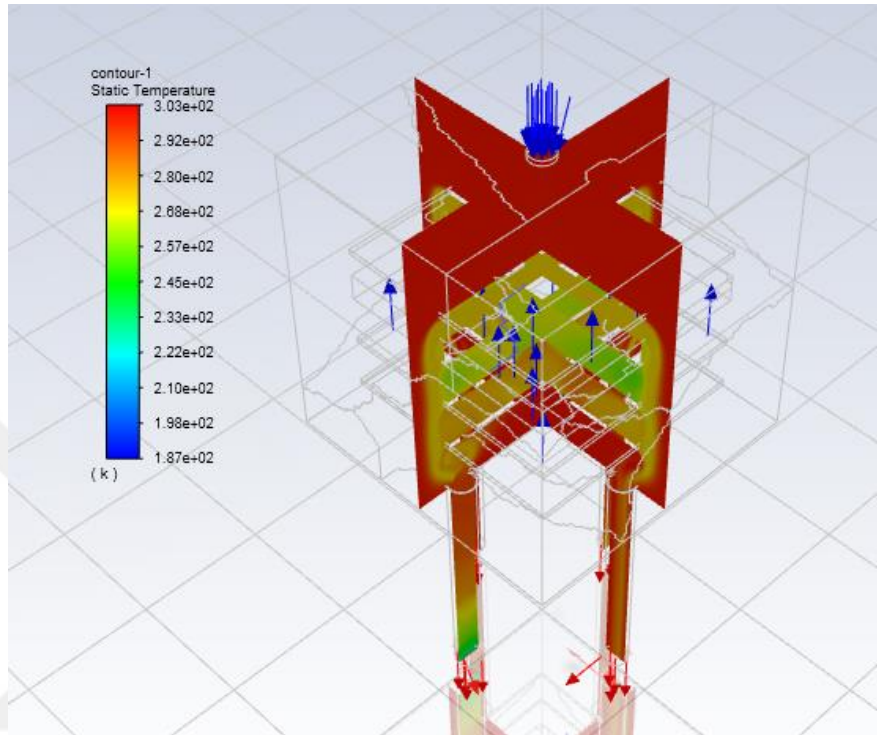


Figure 4.14. Temperature distribution on drying plate of mode

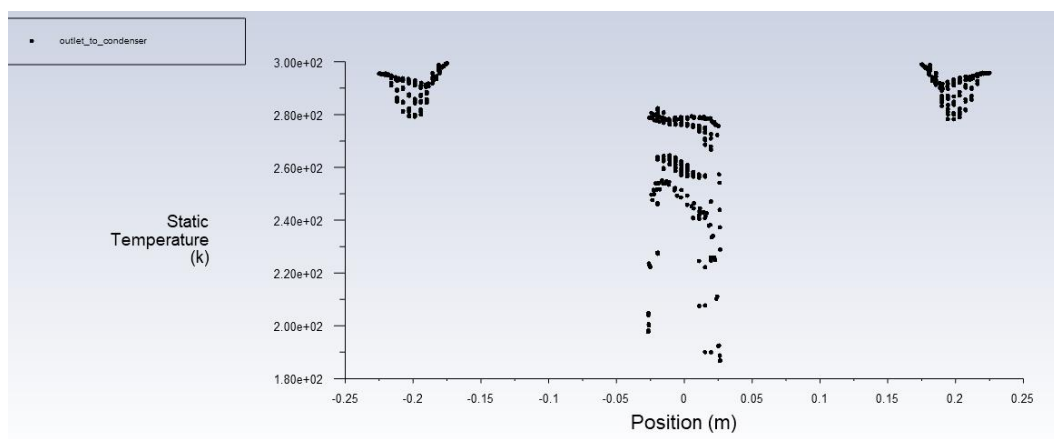


Figure 4. 15. Temperature distribution graph on drying plate of model 5

In model 5 the temperature distribution is similar to that of model 2 and 3. However, there is an overall temperature increase than compare with models 2 and 3.

4.3. Pressure distribution on drying plate

Another important factor that needs to be observed is the pressure, specifically the pressure distribution on the drying plate. Factors that affect the drying plates are very important as they have direct effects on the products. Therefore, is it also just as important to have almost even distribution of pressures and temperatures on the drying plates surface.

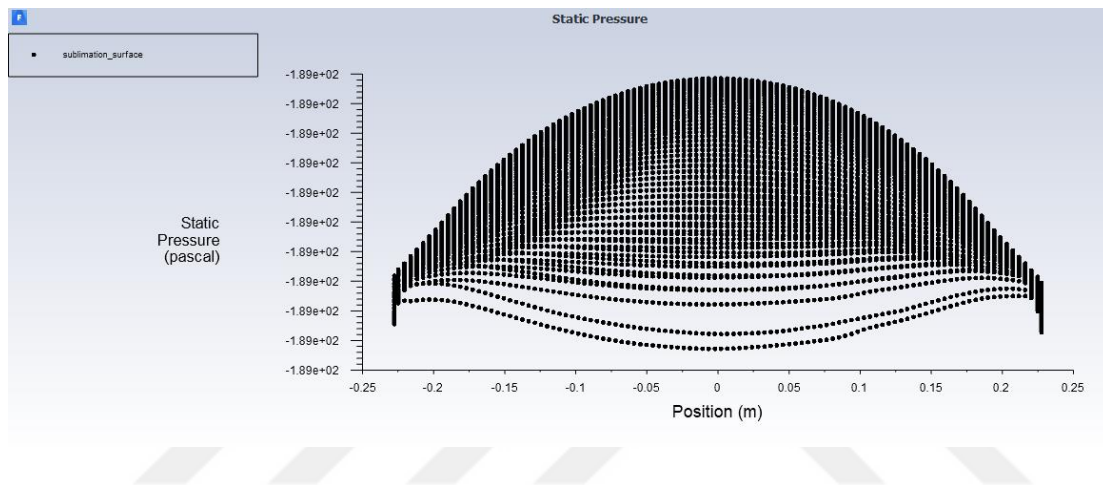


Figure 4.16. Pressure distribution graph on drying plate of model 1

The pressure distribution across the drying plates is another important factor to observe. This helps us understand if the final dried products are evenly dried or not. In figure 4.16 we can see that the pressure across the plate is symmetrical but not evenly distributed.

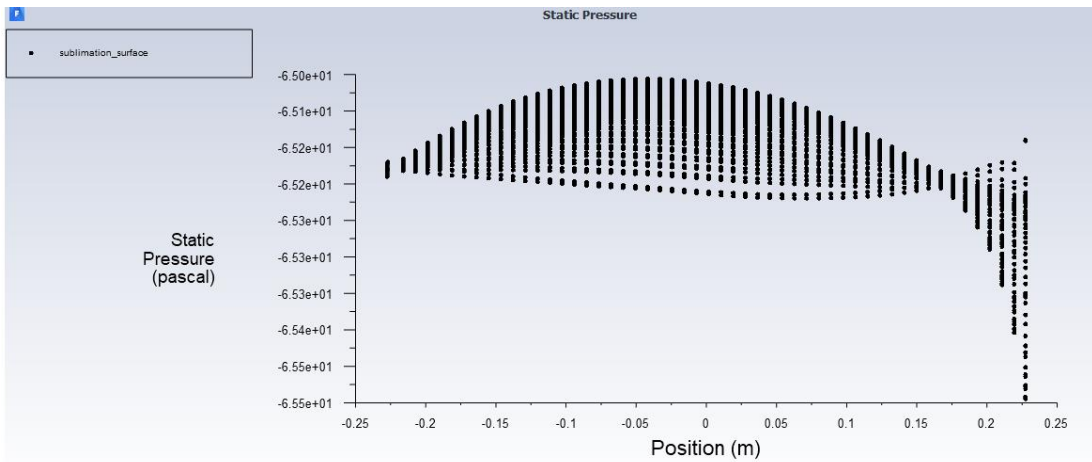


Figure 4.17. Pressure distribution graph on drying plate of model 2

In figure 4.17 the pressure across the plate is observed not be symmetrical. There is a noticeable pressure spike near the outlet duct.

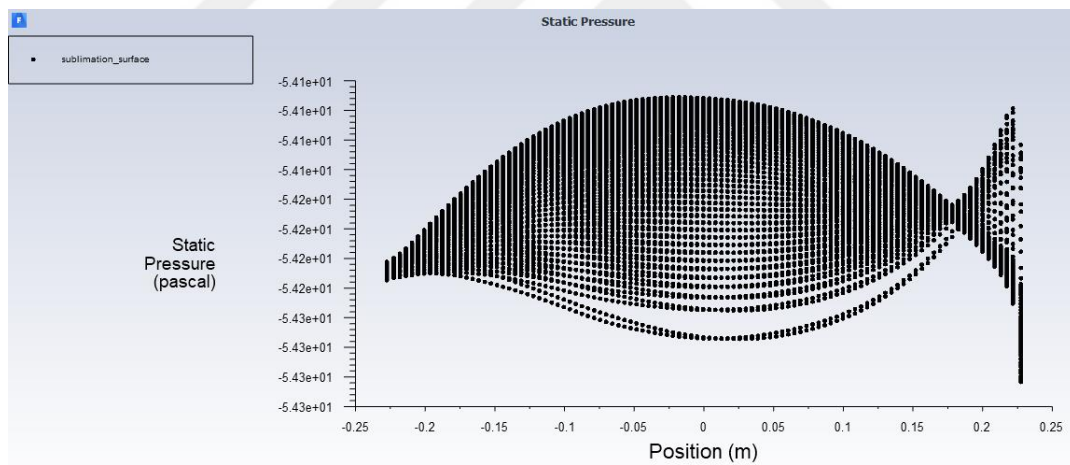


Figure 4.18. Pressure distribution graph on drying plate of model 3

Model 3 is shown to similar to model 2 in the pressure distribution. Both models show high pressure differences near the duct of the outlet ducts.

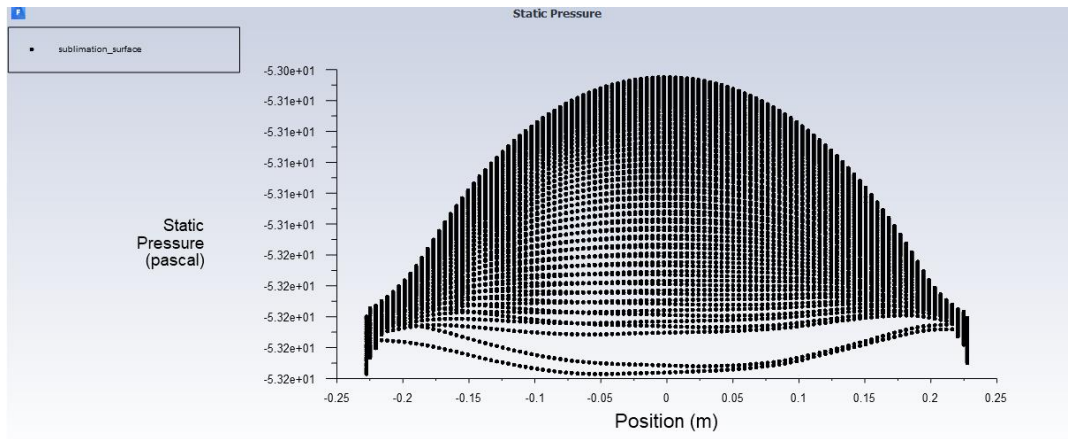


Figure 4.19. Pressure distribution graph on drying plate of model 4

In model 4 we can see that the distribution of pressure is similar to that of model 1 (figure 4.16) where the pattern is a symmetrical however the distribution is still uneven.

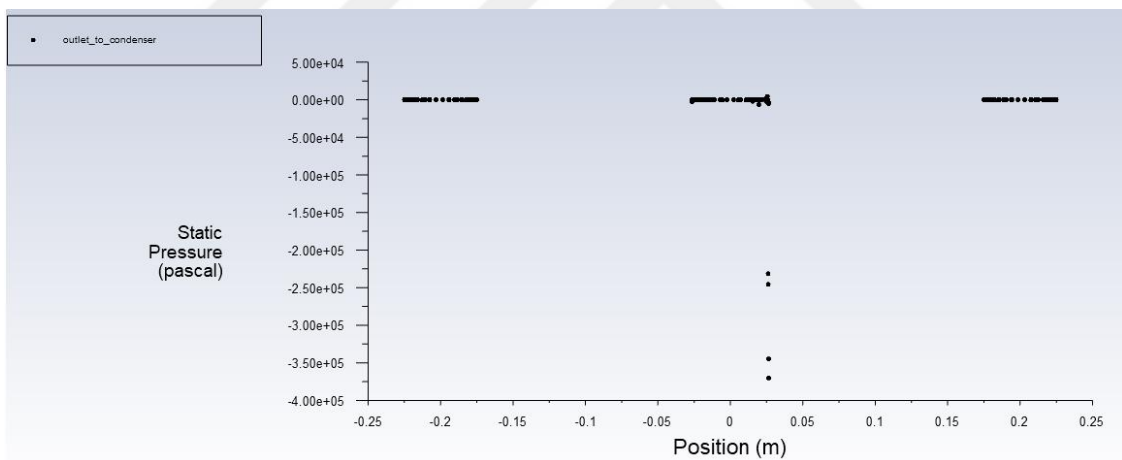


Figure 4.20. Pressure distribution graph on drying plate of model 5

Finally in figure 5.20 model 5 shows that the distribution of the pressure is both even and symmetrical.

4.4. Mas flow rate

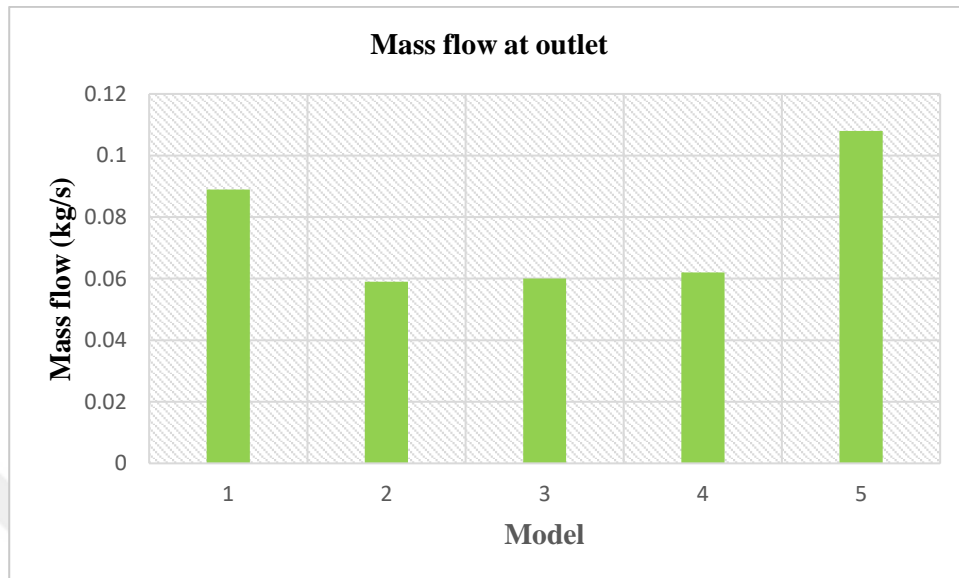


Figure 4.21. Mass flow at outlet

From the graph above we can see that the models 2, 3 and 4 have very similar mass flow rates. While model 5 is shown to have the highest flow rate and model 1 is slightly lower than model 5.

6. CONCLUSION

Flow distribution

The first group has ducts underneath the shelves, while the other group has the duct on the side of the shelves. From the results we can see that model 2 and model 3 have their ducts on the side on the shelves. The water vapor mass fraction shows that so some of the vapor moves up near the N₂ inlet. This is problematic because this could affect the pressure within the chamber. This could lead to poor dried products at the end of the freeze-drying process. Another negative effect from this is that some of the vapor may freeze inside the drying chamber walls which also reduces the quality of the final dried product.

The second group of freeze dryers (model 1, model 4, model 5 and model 6) show different flow patterns. As shown in the pictures above we can see that the vapor only travels down through the condenser duct. This is more preferred than the other group of freeze dryers.

Temperature distribution

High temperatures within the chamber may cause some of the vapor particles to condense. This may lead to varying pressure distributions and partially frozen products at the end of the drying process. The distribution of temperature on the sublimation should be even across the surface. Uneven temperature distributions may lead to some products being dried while others not completely dried.

Pressure distribution across the heating plates

The graph plot showing the pressure distribution shows high pressures around the centre of the plate and the pressures gradually decrease away from the plates (Model 1, 4, 5, and 6). This is due to the shelf gap between the plates. The reason for such a pattern is because as the vapor moves away from the centre there is more space for the vapor to exit through the sides of the plates. As mentioned previously, the shelf gap was set to 100mm for this. By adjusting the shelf gaps, we could get a more horizontal plotted graph, but this was not the aim of this study. However, we can still observe an even distribution of pressure on the surface of the drying plate.

Mass flow rate

Finally, the mass flow rate of the vapor is an important factor we need observe. As shown in the previous results, we can see clearly that one group of freeze dryers is more consistent than the other. The group with the duct on the side of the walls showed undesirable results than the other group. However, this doesn't mean that they are less efficient. The higher the mass flow, the better the final dried product.

From this we can say that the more ducts that are available the higher the flow rates. There is of course a limit to the number of ducts we can place, but the results support a freeze dryer with more ducts.



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SCIENTIFIC ETHICAL STATEMENT

I hereby declare that I composed all the information in my master's thesis Optimization of freeze dryer within the framework of ethical behavior and academic rules, and that due references were provided and for all kinds of statements and information that do not belong to me in this study in accordance with the guide for writing the thesis. I declare that I accept all kinds of legal consequences when the opposite of what I have stated is revealed.

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22/04/2022

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