Your Evaporation Guide
Operation – Immersion angle

Achieve higher distillation efficiency when using a rotary evaporator – Impact of immersion angle

Summary

The evaporation rate of a rotary evaporator depends to a large extend on the immersion angle of the evaporating flask. The flatter the immersion angle, the higher the evaporation rate. This is, among other reasons, due to the fact that a flat immersion angle increases turbulence inside the evaporating flask, hence accelerating the evaporation. However, with a flatter immersion angle the evaporating flask cannot be as deeply immersed in the heating bath medium compared with a steep immersion angle. This can be an issue especially working with smaller evaporating flasks. Generally speaking, a 40 degrees immersion angle leads to a maximum efficiency rate without restriction in handling or risking losing parts of the sample due to over spilling and bumping into the receiving flask.

Introduction

Only a matter of decades ago, the rotary evaporators' immersion angles were rather flat. Furthermore, only a few sizes of evaporating flasks were available, features were limited and the heating bath a separate unit, hence manually movable. Nowadays the rotary evaporator and heating bath are most often a single unit. They are designed to operate with a wide range of evaporating flask sizes and several different glass assemblies are available so that the rotary evaporators are adjustable for various applications.

Figure 1: *First generation of rotary evaporators with rather flat immersion angles (BUCHI Rotavapor, 1957).*

With modern rotary evaporators, the immersion angle is adaptable to different circumstances. The main impacts of different immersion angle are:

- If the axis is vertical, there is almost no mixture inside the evaporating flask and heat transmission is limited to the immersion area of the flask. Moreover, there is only little turbulence in the heating bath. As a consequence, only limited heat transfer occurs within the heating bath.
- Flattening the immersion angle, leads to better mixing of the content. A larger percentage of the rotating evaporating flask is wetted by the heating bath medium. This generates a more effective heat transfer for faster evaporation and prevents local overheating.
- In the case of a very flat immersion angle, an even larger percentage of the flask is covered by the heating bath medium. Through the rotation, a thin film of the water/oil of the heating bath is distributed almost over the whole surface of the rotating evaporating flask. Moreover, the heating medium is more agitated, which leads to more even heat distribution, resulting to more efficient heat transfer. However, a flatter angle creates a risk of the solvent that is being held in the flask, of pouring over into the receiving flask.

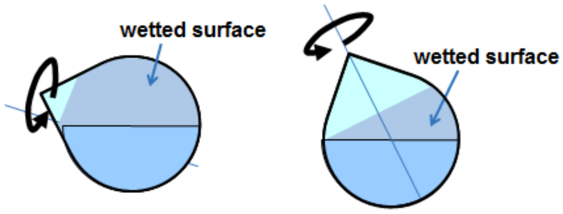


Figure 2: *Representation of the wetted surface of an evaporating flask. A flatter immersion angle results in a larger wetted surface area inside the flask (left); a steeper immersion angle results in a smaller wetted surface area inside the flask (right).*

A disadvantage of a flatter immersion angle is that the evaporating flask cannot be inserted as deeply in the heating bath. This is especially an issue with smaller flasks. Moreover, the sample solution is, due to less volume of the flask, more likely to bump and foam over.

Experiment

The factor of different angles is, in comparison with other variables of the evaporation process, relatively significant. For that reason is it worthwhile to find out the evaporation's effectiveness by applying different immersion angles.

The aim of the following experiment was to analyze the influence of different angles on the evaporation rate of a solvent single-stage distillation. The experiment was executed with a BUCHI Rotavapor®.

For the experiment the evaporation output of acetone was measured using immersion angles of 10, 25 and 35 degrees, each at a rotation speed of 50, 150 and 250 rpm.

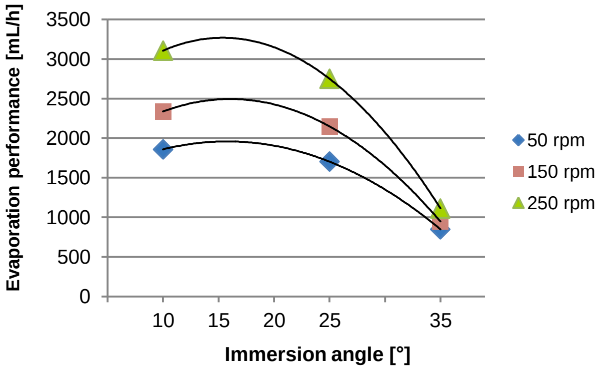
Parameter

Solvent	acetone
Heating bath temperature	60 °C
Cooling temperature	10 °C
Pressure	556 mbar
Flask size	1 L
Content	500 mL
Immersion depth	fill level

Your Evaporation Guide
Operation – Immersion angle

Achieve higher distillation efficiency when using a rotary evaporator – Impact of immersion angle

Result



Graphic 1: *Illustration of the influence of the immersion angle on the evaporation output.*

As shown in the graphic above, an immersion angle of 10 degrees, compared to an immersion angle of 35 degrees, led to a significant higher output of the evaporation rate, from 119 % (at 50 rpm) up to 180 % (at 250 rpm). An immersion angle of 10 degrees, compared to an immersion angle of 25 degrees, led to a 13 % higher evaporation output (at 250 rpm). The main increase in performance led the modification of an immersion angle from 35 to 25 degrees. In all three position, the disparity was always more significant at higher rotation speeds.

Interpretation

The experiment shows that the tests with a 10 and 25 degrees immersion angles (flat and standard position) obtained significantly more evaporation output compared to the tests with a 35 degrees immersion angle (steep position). This is for the simple reason that due to a flatter immersion angle, the active surface area inside the evaporating flask is enlarged, thus, accelerating the evaporation rate significantly. Though, the impact between the settings of 10 and 25 degrees was less noteworthy.

Recommendation

The immersion angle selected should be as flat as possible to produce maximum turbulence inside the evaporating flask. Moreover, in order to perform best, the flask's immersion depth should be set at least as deep that the level of the solvent is equal to the level of the heating bath medium.

The BUCHI Rotavapor® R-300 is manually adjustable to seven different immersion angles within 40 degrees and the lift movable 220 mm in the vertical axis. [1] Furthermore, the Heating Bath B-305 is movable horizontally to enable 50 mL up to 5 L evaporating flasks to be completely inserted into the heating bath and, thus, to be adjusted to each individual distillation situation.

Figure 3: *Rotavapor® R-300 pictured in 3 different immersion angle positions.*

The take home message is using an immersion angle at 25 degrees guarantees most efficient evaporation rate without jeopardize losing parts of the sample.

References

[1] Technical Datasheet, Rotavapor® R-300

Your Evaporation Guide
Operation – Evaporating flask size

Achieve higher distillation efficiency when using a rotary evaporator – Impact of flask size

Summary

Generally, the larger the evaporating flask the greater is the evaporation rate. This is due to the greater turbulence inside a larger flask, enlarging the active surface and leading to a higher evaporation output. On the other hand, smaller flasks are advantageous when the residue needs to be collected for further quantitative analyses. Moreover, larger flasks generally allow easier handling.

Introduction

After decades of building rotary evaporators, there is still potential to optimize the evaporation process. The choice of the adequate glass accessories is therefore an important aspect in increasing the evaporation output. For that reason it is interesting to examine the influence of the evaporating flask size on the evaporation output.

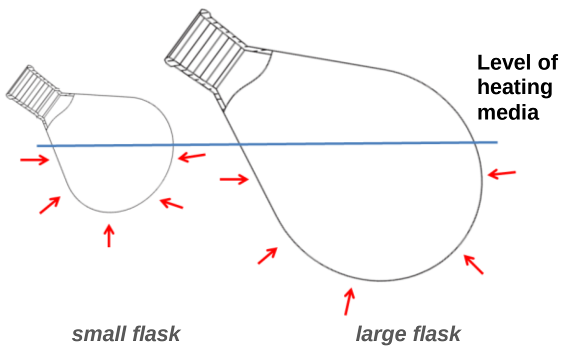


***Figure 1:** Schematic representation of two different sized evaporating flasks immersed in the heating medium. There is more heat transfer within a larger flask due to a larger surface area (right picture).*

The main impacts of the flask's size are:

- Due to increased velocity at the perimeter of larger flasks, the solvent inside the flask is more agitated, enlarging the active surface area of the solvent.
- Due to a larger glass wall surface, more heat energy transfers from the heating bath, through the evaporation flask, to the solvent.
- Larger flasks are more likely to prevent foaming over and the negative consequences of boiling delays and bumping.
- Smaller flask are more suitable for quantitative collection, for example, if the user is interested in the residue that remains after the evaporation
- Smaller flasks offer more flexibility when handling the rotary evaporator in the vertical axis.

Apart from this, larger conventional evaporating flasks automatically have thicker glass walls which, in turn, lowers the efficiency of the evaporation performance as there is obviously less heat transfer through thicker glass walls [1].

Experiment

The flask size certainly affects the evaporation process. For that reason it is interesting to examine the influence of the evaporating flask size on the evaporation output.

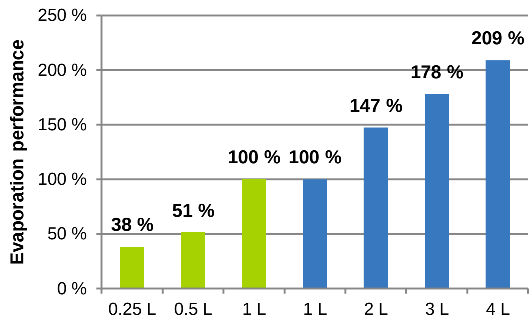
The aim of the following experiment was to analyze the influence of the size of the evaporating flask on the evaporation rate of a solvent single-stage distillation. The experiment was executed with a BUCHI Rotavapor®.

For the realization of the experiment, the evaporation process was performed using different sized evaporating flasks (from 250 mL up to 4 L). 200 mL solvent were used for the flasks up to 1 L, 750 mL for the flasks of 1 L and larger.

Parameter	
Solvent	acetone
Heating bath temperature	60 °C
Cooling temperature	10 °C
Pressure	556 mbar
Flask size	250 mL - 4 L
Content	200 mL, 750 mL
Immersion depth	fill level

Results

To evaluate the outcomes of the evaporation performance, the evaporation outputs of the two 1 L flasks were set to 100 % and the outcomes were divided into two groups, according to the amount of the solvent, and put in relation to the 1 L flasks.



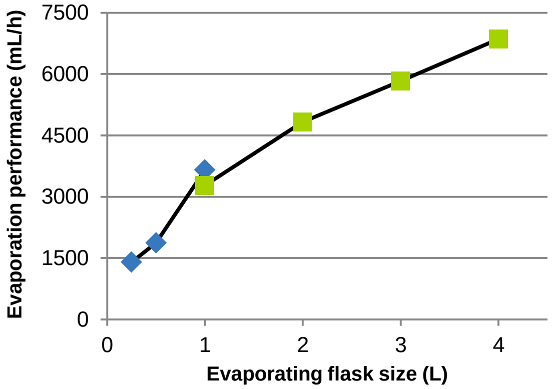
***Graphic 1:** Illustration of the influence of the flask size on the evaporation output.*

As illustrated in the graphic above, the evaporation rate increases with larger flask size. If the evaporation output of the 1 L evaporating flask is put at 100 %, the 2 L flask achieved 147 % and the 4 L flask 209 % output. The 250 mL flask obtained only 38 % of the output compared to the 1 L flask.

Interpretation

As seen from the experiment, the larger the evaporating flask, the higher the evaporation output. For instance, the evaporation rate using a 4 L flask is significantly higher compared to a 1 L flask.

Achieve higher distillation efficiency when using a rotary evaporator – Impact of flask size



Graphic 2: *Illustration that with an increased evaporating flask size, the evaporation output increases.*

This is due to the correlation between the surface area inside the evaporating flask with the flask's volume. Therefore, with increasing flask volume, the active surface area is increased, raising the evaporation output.

Recommendation

The evaporating flask size should be selected to be as large as possible while still being suitable for the application. Basically, a larger evaporating flask provides sufficient greater interior surface area for increased evaporation performance. However, in the case the residual needs to be quantitatively collect for further analyses, the flask should be small enough not to risk losing the sample, as it will be more difficult to transfer the residue out of a larger evaporating flask. Therefore, the optimum flask size has to be adjusted to the respective sample and application. In general, it is appropriate to select a flask that accommodates at least twice the starting sample volume.

Figure 2: *Optimum filling quantity is 1/3 to 1/2 of the evaporating flask' volume.*

BUCHI's high quality glassware assortment offers evaporating flask sizes from 50 mL up to 5 L and a broad variety of accessories and glass assemblies which allow optimized operation. Moreover, the lift of the Rotavapor® R-300 is movable 220 mm in the vertical axis and the heating bath can be moved horizontally. [2] This enables evaporating flasks up to 5 L (Heating Bath B-305) to be deeply inserted into the heating bath and adjusted to each individual distillation situation. Nevertheless, BUCHI also offers customized glassware to meet special customer requirements.

References

[1] Your Evaporation Guide, Operation – Impact on glass thickness
[2] Technical Datasheet, Rotavapor® R-300

Your Evaporation Guide
Operation – Rotation speed

Achieve higher distillation efficiency when using a rotary evaporator – Impact of rotation speed

Summary

The evaporation rate of a rotary evaporator is greater than that of static distillation apparatus. Moreover, the evaporation rate increases with higher rotation speed. This is, among other factors, due to the greater surface area inside the evaporating flask at one given time. Especially significant is the dissipation at lower rotation speeds. For instance an increase in speed above 200 rpm has a relatively low influence on the evaporation output. A rotation speed above 300 rpm can result in mechanical problems, vibrations and spillage from the heating bath. Thus, considering the advantages and disadvantages of different rotation speeds, the optimum rotation speed is around 250 to 280 rpm.

Introduction

Even 50 years after the launch of the first rotary evaporator, there is much ambiguity concerning evaporation in the rotating evaporating flasks.

Figure 1: *First generation rotary evaporator (BUCHI Rotavapor, Model 1957).*

Even nowadays, evaporation and distillation are still one of the most frequently used separation methods. In fact, the evaporation output of a rotary evaporator during single-stage distillation is around four times greater than that of conventional, static distillation apparatus [1]. There are several reasons why the rotation influences the evaporation output:

- Through the rotation, the liquid inside the heating bath is agitated, causing additional turbulence, which leads to a massive improvement of the heat transfer coefficient and, therefore, to an increase of heat transfer from the heating bath to the evaporating flask as well as from the flask to the solvent.
- Rotation greatly enlarges the active surface area inside the evaporating flask, thus accelerating the evaporation. As the solvent is heated via the heating bath, it is distributed as a thin film over the warm, inner surface of the rotating evaporating flask. This film partially vaporizes, resulting in a higher evaporation rate. With the liquid remaining at any one point of the flask wall for a short time only, rotation homogenous mixture of the sample is ensured and overheating, thus incrustation inside the flask is reduced.
- The frictional force and the centrifugal force between the wall of the rotating evaporating flask and the liquid inside the flask, result in the formation of a thin film of warm solvent spread over a larger surface.

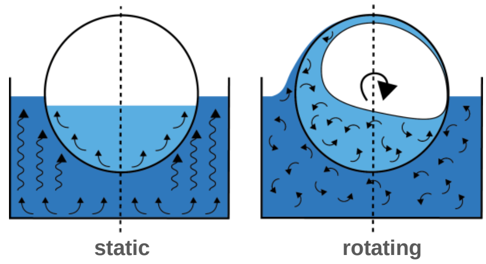


Figure 2: *Representation of heat transfer of a static and rotating evaporating flask. Convection → free convection, slow equalization of temperature (left); Turbulence → forced convection, improves heat transfer, thus a faster evaporation by keeping the liquid in motion (right).*

Additional advantages are that the forces created by the rotation suppress bumping, foaming and boiling delays.

However, the higher the rotation speed, the greater is the chance that mechanical problems with the rotary evaporator occur and the resulting vibration is more likely to cause deterioration of the instrument.

Experiment

Rotation certainly speeds up the evaporation. It is of great importance to scrutinize to what extent the speed of the rotation influences the evaporation output. For this reason, the dependence of the rotation speed on the evaporation rate should be determined.

The aim of the following experiment was to analyze the impact of the rotation speed on the evaporation rate of a single-stage solvent distillation. The experiment was executed with a modified BUCHI Rotavapor® where the rotation speed was adjustable to 500 rpm.

The experiment was executed in two steps. Firstly, in order to get a broad overview, the evaporation output of acetone was measured at nine different rotation speeds (from 50 up to 500 rpm). Secondly, the evaporation output of water was measured at stillstand and six different rotation speeds, within the actual operating range of up to 280 rpm.

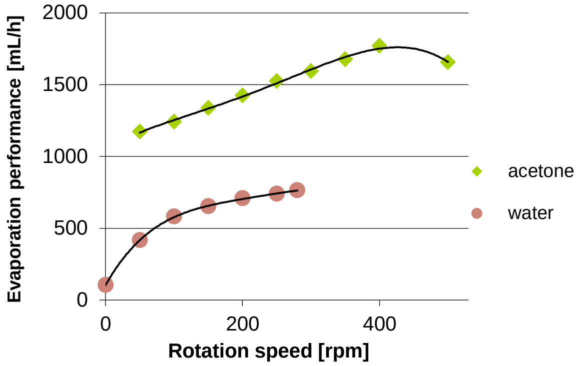
Parameter

Solvent	acetone water
Heating bath temperature	60 °C
Cooling temperature	10 °C
Pressure	556 mbar (acetone) 72 mbar (water)
Flask size	1 L
Content	500 mL
Immersion depth	fill level

Your Evaporation Guide
Operation – Rotation speed

Achieve higher distillation efficiency when using a rotary evaporator – Impact of rotation speed

Results



Graphic 1: *Illustration of influence of the rotation speed on the evaporation output.*

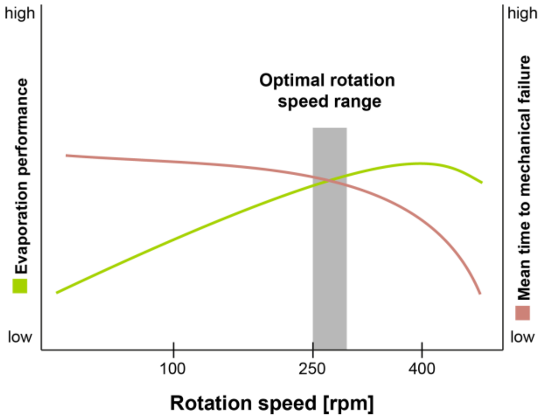
The graphic above shows that an increase of the rotation speed up to 400 rpm raised the evaporation output. Though, the evaporation rate decreased again at speeds above 400 rpm (acetone). The test series with water clearly showed that the dissipation was especially significant at lower rotation speeds and relatively less above 200 rpm.

Interpretation

The experiment shows that the maximum evaporation output is achieved at a rotation speed around 400 rpm. This is because the rotation speed can be raised to a point where the content will be pressed against the wall by centrifugal force and corotate with the flask. In this case the turbulence will decrease again, which consequently results in a lower evaporation rate. The optimal turbulence depends on the flask size, the consistency and texture of the solvent/substance and the filling quantity.

Recommendation

The rotation speed should be selected to produce maximum turbulence inside the flask as well as in the heating bath, in order to reach maximum evaporation output and still guarantee a long lasting life of the rotary evaporator.



Graphic 2: *Representation of the optimal rotation speed by the intersection of the two curves.*

The graphic illustrates the ideal rotation speed range, where the problems, such as spillage, vibration, mechanical problems, are still low.

Considering the factors of maximum output and long lifetime of the instruments, the optimum rotation speed of a BUCHI Rotavapor® is from 250 to 280 rpm. For this reason the Rotavapors are limited to a rotation speed of 280 rpm.

References

[1] BUCHI Training Paper, “Distillation with a Rotary Evaporator”

Your Evaporation Guide
Operation – Temperature difference

Achieve higher distillation efficiency when using a rotary evaporator – Impact of temperature differences

Summary

There is a direct relationship between the heating bath temperature and the evaporation rate. The more energy applied to the evaporation side, and at the same time removed from the condensation side, the more efficient is the distillation. Furthermore, sufficient cooling as well as an appropriate and stable under pressure are crucial for efficient distillation. On the other hand, the consumption of electrical energy is comparatively greater at higher temperatures. Moreover, some samples are thermo-sensitive, thus exacerbating the circumstances. Therefore the respective parameters have to be fine-tuned to the individual sample and application. The “Delta 20 Rule” is a guideline to compromise between high evaporation output and energy usage. For instance, using the 10/30/50 parameters is appropriate for the evaporation process in order to bring in and to carry off the accumulated energy efficiently.

Introduction

The performance of a rotary evaporator is limited by the input, the amount of heat that can be added to the evaporation side, and the output, the amount of heat that can be removed on the condensation side. Basically, energy is imparted to the solvent in order to transform it to the vaporous state; during the condensation cycle this energy has to be removed again within the same length of time.

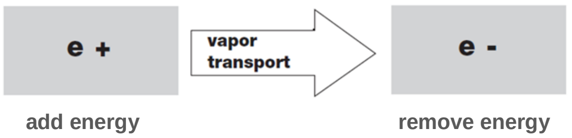


Figure 1: Schematic representation of the evaporation-condensation process. Heating → evaporation; cooling → condensation.

Formerly, only the energy supply was easily controllable. The cooling temperature was rather inflexible as mainly tap water was used as the cooling source. Moreover, the vacuum was only roughly controllable.

Nowadays, the vacuum can be adjusted very precisely and kept stable. Furthermore, with the possibility of the modern “recirculating chiller”, the energy supplied for cooling the condenser can be selected accurately, typically to produce temperatures as low as -5 to 10 °C. Therefore recirculating chillers are very effectively in cooling and the distillation can be kept at low temperatures.

The heating bath temperature, the vacuum as well as the cooling temperature need to be adjusted to the condenser’s capacity. A condenser is working at its optimum capacity if two-thirds of its height is covered with condensate, hence the top third acts as a safety barrier for "entrained" low-boiling solvent plus for pressure fluctuations. A condenser is overloaded if condensate is seen to form downstream from the condenser or if the vacuum pump sucks continually in order to maintain a specific pressure. The speed of evaporation and condensation should be attuned to maintain a balanced dynamic pressure.

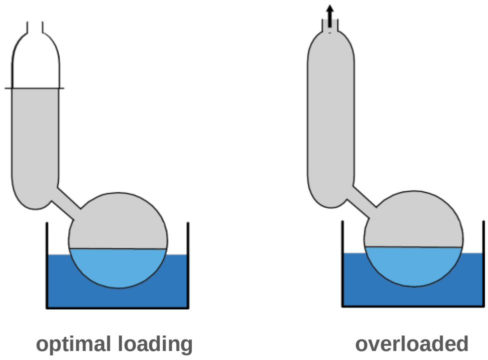


***Figure 2:** Illustration of the optimal utilization of condenser’s capacity (left); condenser is overloaded → loss of solvent (right).*

When working with a thermo-sensitive sample, a mild heating bath temperature needs to be selected in order not to harm the compounds. In addition, a heating bath at lower temperature is more convenient to work with. For instance, with a heating bath temperature of 50 °C, the evaporating flask can be changed without any risk of scalding. With higher temperatures, the vaporizing rate of the heating bath medium (e.g. water) increases, and it thus has to be refilled more frequently. This results in additional consumption of energy.

Experiment

The aspects of heating and cooling are very important and determine the evaporation rate.

It is interesting to examine to what extend different heating bath temperatures influence the evaporation output. The aim of the following experiment was to analyze the impact of the amount of energy, in form of heat, applied to the system on evaporation rate of a solvent single-stage distillation. The experiment was executed with a BUCHI Rotavapor®.

For the experiment the evaporation process was executed using five different heating bath temperatures (from 40 to 80 °C).

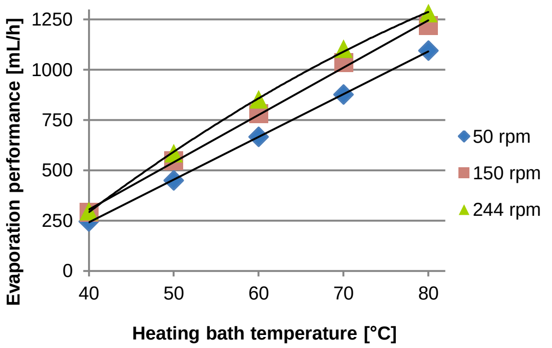
Parameter

Solvent	acetone
Pressure	556 mbar
Vapor temperature	30 °C
Cooling temperature	7 °C
Flask size	1 L
Content	500 mL
Immersion depth	fill level

Your Evaporation Guide
Operation – Temperature difference

Achieve higher distillation efficiency when using a rotary evaporator – Impact of temperature differences

Result



Graphic 1: *Illustration of the influence of the bath temperature on the evaporation output.*

As illustrated in the above graphic, the higher the heating bath temperature, the higher is the evaporation rate. The differences of the evaporation output increased more or less linearly with the temperature rise. For instance, with a heating bath of 80 °C, the distillation output was about four times greater compared to a heating bath temperature of 40 °C.

Interpretation

As the temperature of the heating bath was raised, the evaporation output increased significantly. However, the energy consumption of the heating bath and recirculating chiller increased, too. For instance, when using an 80 °C heating bath, it should be remembered that much more energy has to be supplied and again removed from the system than is the case when working at lower temperatures.

Recommendation

The heating bath temperature and the vacuum needs to be coordinated for the condenser to work as closely as possible to optimum condenser's capacity without being overloaded. For a sufficient condensation of the vapor, the cooling temperature should be about 20 ºC lower than the vapor temperature.

BUCHI recommends that the "Delta 20 Rule" should be applied. This rule of thumb can be applied as following: set the bath temperature at 50 °C to yield a solvent vapor temperature of 30 °C, which is subsequently condensed at 10 ºC [1].

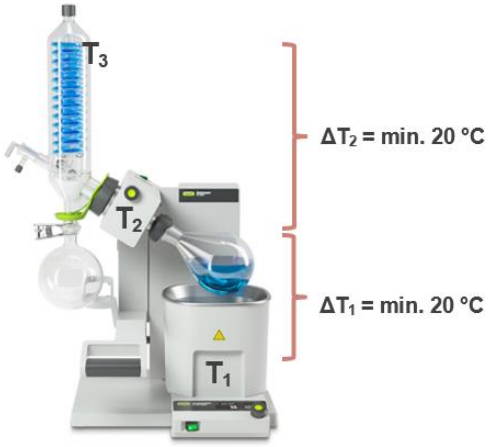


Figure 1: *Representation of the "Delta 20 Rule", the parameters 10/30/50 applied.*

The "Delta 20 Rule" can also be applied to lower heating bath temperatures for solvents with a low boiling point or thermo-sensitive products. For example: cooling media: 0 ºC; vapor: 20 ºC; heating bath: 40 °C and the pressure lowered in order to lower the solvents boiling temperature.

A heating bath temperature above 50 °C is less easy to handle, thus increasing the risk of accidents. Moreover, environmental and economical issues should also be taken into account. The "Delta 20 Rule" makes solvent removal simple and efficient. The vacuum is the only setting that has to be changed and the pressure for each solvent can be conveniently selected from BUCHI's "List of solvents".

The Heating Bath B-100 and B-305 have a heating power of 1300 watts and a standard condenser a cooling area of 1500 cm^2 [2], hence it can achieve high evaporation output.

To sum up, the "Delta 20 Rule" compromises evaporation output and energy consumption. The optimized settings of the heating and cooling temperatures are depending on the specific application and have to be fine-tuned for each individual sample.

References

[1] BUCHI - Training Paper, Distillation with a Rotary Evaporator

[2] Technical Datasheet, Rotavapor® R-300

Your Evaporation Guide
Operation – Thickness of evaporating flask

Achieve higher distillation efficiency when using a rotary evaporator – Impact of thickness of flask

Summary

The evaporation rate is noticeably greater if the evaporating flask's glass walls are thinner. The simple reason is that thinner glass allows better heat transmission from the heating bath through the glass of the evaporating flask to the solvent. Generally speaking, the glass wall thickness should be selected to be as thin as possible, while still being robust enough to prevent breakages of the evaporating flask even at very low pressures. Concerning both these issues, the optimum thickness for a 1 L evaporating flask is between 1.5 and 2.0 mm.

Introduction

Formerly, the glass walls of evaporating flasks were generally thicker. Thanks to years of research, modern technology and automated manufacturing processes, the design of the evaporating flask is now optimized to maximize efficiency. The demands and requirements are high as the glass has to be resistant to breakages and withstand conditions such as high temperatures as well as rapid temperature changes at very low pressure. It is therefore of great importance to be aware of the optimum glass thickness and of the safety issues when choosing an evaporating flask.

The evaporation efficiency depends primarily on the amount of heat energy which reaches the solvent inside the evaporating flask. It is obvious that a thicker (glass) barrier hinders heat transfer.

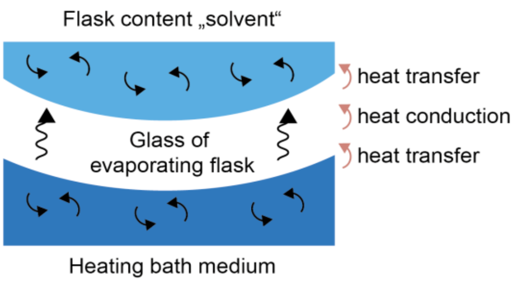


Figure 1: *Schematic representation of heat transfer from the heating bath to the solvent.*

Experiment

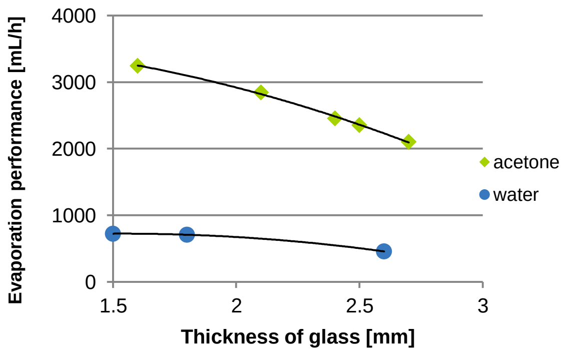
The aim of the following experiment was to analyze the influence of the evaporating flask glass thickness on the evaporation rate of a solvent single-stage distillation. The experiment was executed with a BUCHI Rotavapor®.

For the realization of the experiment, the evaporation process was first performed with acetone, using five 1 L evaporating flasks differing in the glass wall thickness (from 1.6 to 2.7 mm) and then with water, using three different flasks (from 1.5 to 2.6 mm thickness respectively).

Parameter

Solvent	acetone water
Heating bath temperature	60 °C
Cooling temperature	10 °C
Pressure	556 mbar (acetone) 72 mbar (water)
Flask size	1 L
Content	500 mL
Immersion depth	fill level
Rotation speed	280 rpm

Results



Graphic 1: *Illustration of the influence of the glass wall thickness of flask on the evaporation output.*

As illustrated in the above graphic, the thinner the evaporating flask' glass walls, the higher the evaporation output. The disparity was especially obvious in the experiment using acetone. For example, the evaporating flask, with a thickness of 1.6 mm, achieved 54 % higher evaporation output compared to the flask with 2.7 mm glass wall thickness.

Interpretation

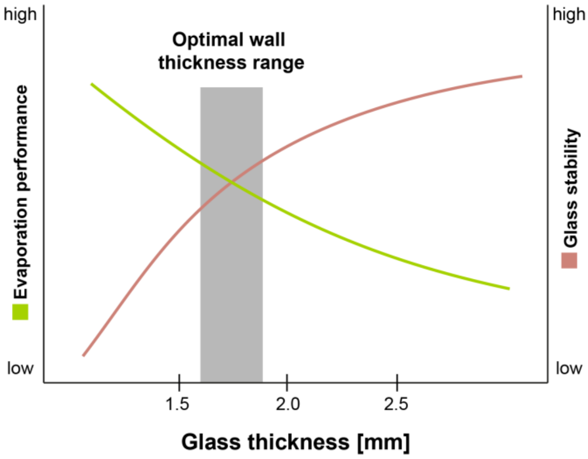
This experiment shows that glass wall thickness has a significant impact on the evaporation output. With thinner glass walls, the heat transfer from the heating bath medium to the solvent inside the evaporating flask is improved, speeding up the evaporation. On the other hand, the thicker the glass, the more prolonged the heat transfer becomes, thus reducing the evaporation output significantely.

Recommendation

There is a fine line between glass that is too thin and glass that is too thick. For instance, using thinner glass might put the glass at higher risk of breaking. On the other hand, thicker glass slows down the evaporating output. Generally speaking, the evaporating flask should be as thin as possible, but still withstand very low pressure and other exposures as high temperature and rapid temperature changes.

Achieve higher distillation efficiency when using a rotary evaporator – Impact of thickness of flask

The BUCHI evaporating flasks are designed for high evaporation output and to fulfill highest safety requirements. The standardized 1 L evaporating flask has a glass wall thickness around 1.8 mm. According to customer feedback and internal tests, this condition provides the optimum ratio of high heat transfer efficiency along with high safety.



Graphic 2: *Representation of optimal glass thickness (1 L flask) by the intersection of the two curves.*

The high-quality glass consists of inert industrial glass, borosilicate glass 3.3, resistant to chemicals high temperatures and rapid temperature changes. The quality and precision of BUCHI glassware also guards from breakages. In addition, if there are special requirements from customers, BUCHI modifies glass components in any ways or develops complex new designs for individual needs.