BUCHI
SWITZERLAND

Your Evaporation Guide – 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.
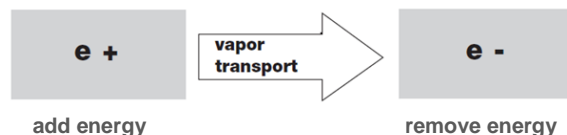

Figure 1: *Schematic representation of the evaporation-condensation process. Heating → evaporation; cooling → condensation.*

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.
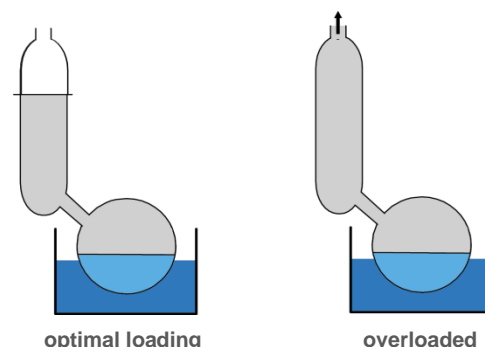

Figure 2: *Illustration of the optimal utilization of condenser's capacity (left); condenser is overloaded → loss of solvent (right).*

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 – Temperature difference

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

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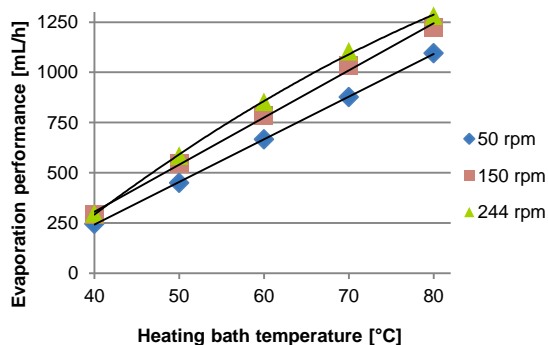
Graphic 1: Illustration of the influence of the bath temperature on the evaporation output.

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].
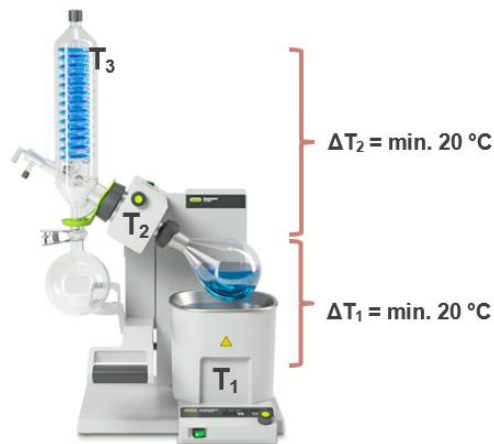

Figure 1: Representation of the "Delta 20 Rule", the parameters 10/30/50 applied.

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