

Unlock extraction efficiency with a Rotavapor®

1. Introduction

Soxhlet extraction is a simple and cost-effective method of extracting a desired compound with a limited solubility in a solvent, whereas the impurity is insoluble in that solvent. The benefit is to allow unmonitored and unmanaged operation while efficiently recycling a small amount of solvent to dissolve a larger amount of material. [1]





This type of extraction is widely used for valuable bioactive compounds from natural sources. Its applications can be found in natural products, forensics, environmental and pharmaceutical segments to name a few.

2. Scale up

The benefit of using a Rotavapor[®] instead of a static glassware apparatus is to have multiple instruments in one. Meaning, one can perform the extraction process at a higher distillation rate (greater heat transfer due to rotating evaporating flask and vacuum) and then remove the solvent from the extract without having to change the instrument.

Normally one is limited with in sample volume which can be put into the extraction chamber. Consequently, the maximum amount of final extract is already predefined, and the only way to increase the amount of extract is to increase the number of Soxhlet units. Or is it?

Once the target substance and suitable parameters have been indentifed on a laboratory Rotavapor[®], one can increase the sample volume from around 500 mL to 2.5 L using an industrial Rotavapor[®].

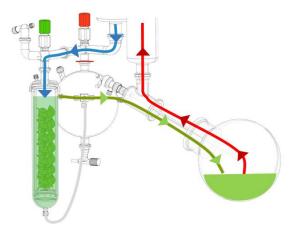


Fig. 2: How does the extraction process on an industrial scale look like?

3. Experiment

The aim of this experiment was to show differences and similarities of an extraction process carried out on a laboratory and an industrial Rotavapor[®]. The focus was on the amount of fat extracted from the sample and the required time.

Sample	Cookies from a local supermarket with 10% fat content
Solvent	Heptane

Table 1: Sample and chemical used

The experiment on a laboratory scale was carried out on a Rotavapor[®] R-300 equipped with a Heating Bath B-305, Recirculating Chiller F-314, Interface I-300 Pro and Vacuum Pump V-300. The glassware consisted of a 1 L evaporating flask, reflux (S) condenser and Soxhlet accessory.

The experiment on an industrial scale was performed on a Rotavapor[®] R-220 Pro Extraction with Recirculating Chiller F-325, Interface I-300 Pro and Vacuum Pump V-600. Glassware consisted of a 20 L evaporating flask and extraction glass assembly.

Parameter	R-300 System	R-220 Pro System
Rotation speed [rpm]	280	150
Heating bath T [°C]	60	60
Cooling T [°C]	10	10
Pressure [mbar]	170	170

Table 2: Experiment parameters



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The experiment was conducted by first homogenizing the sample and weighing individual components. After placing the sample into the extraction chamber, distillation was started. The process was terminated, when the yellow color of the distillate after passing the extraction chamber completely disappeared (lack of fat). After the extraction step, the solvent was evaporated from the evaporating flask yielding the extracted fat.

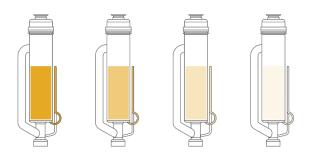


Fig. 3: Extraction progression on a laboratory Rotavapol[®] with a Soxhlet accessory. Ligher color corresponds to reduction of fat.

4. Results

	Laboratory scale	Industrial scale
Initial sample weight [g]	Ca 100	Ca 800
Extracted fat in the evaporting flask (%)	9.63	9.17
Fat loss of the sample (g)	9.69	80.1
Average distillation rate [mL/min]	58.3	141
Process time [min]	45	75

Table 3: Result comparison

The experiments show that one can extract close to (if not) all the fat from the initial sample with a laboratory or industrial Rotavapor[®] extraction system. Small deviations in the results could come from too short extraction time, instrument deviations and sample losses during the process.

5. Advantages

An industrial Rotavapor[®] is able, due to the larger surface area of the flask, to distill 2.4x more heptane and extract almost 5x more fat during the same time in comparison to the laboratory Rotavapor[®] using the same extraction conditions. This means that the R-220 Pro Extraction can successfully be used to scale up the extraction process, previously evaluated on a laboratory Rotavapor[®].

6. Limitation

In contrast to a laboratory version, where cycles can be easily monitored and calculated, there is no automatic cycle changing on an industrial Rotavapor[®]. The whole process is based on diluting the sample in the extraction chamber with fresh solvent. However, if required, one can still manually induce a draining step at a chosen time.

7. References

[1] <u>https://en.wikipedia.org/wiki/Soxhlet_extractor</u>