Application Note
No. 816/2023

Unlock extraction efficiency with a Rotavapor®

Rotavapor® R-300 system, Rotavapor® R-220 Pro Extraction system
Efficiency comparison of extraction process between lab and industrial Rotavapor®

1. Introduction

The following experiment will show differences and parallels of an extraction process carried out on a laboratory and industrial Rotavapor®. For this purpose, gravimetric determination of fat in the extracted sample (residue) and in the evaporating flask will be compared. The results will focus on the overall % of fat extracted and the time needed.

2. Equipment

- Rotavapor® R-300 System with Soxhlet accessory (including Vacuum Pump V-300, Recirculating Chiller F-314 and 1 L evaporating flask)
- Rotavapor® R-220 Pro Extraction System (including Vacuum Pump V-600, Recirculating Chiller F-325 and 20 L evaporating flask)
- Laboratory balance 1 (max weight 3200 g, accuracy ± 0.01 g)
- Laboratory balance 2 (max weight 7000 g, accuracy ± 1 g)
- Laboratory drying cabinet
- Laboratory mortar and pestle
- Extraction thimble 48 x 200 (for laboratory Rotavapor®)
- Washing bags 2x (for industrial Rotavapor®)
- Cotton wool

3. Chemicals and Materials

Chemicals:

- Heptane, technical grade

For a safe handling please pay attention to all corresponding MSDS!

Samples:

- Petit Beurre Guezli (10g fat / 100 g sample according to the package)

The samples were purchased at a local Coop supermarket.

4. Procedure

The determination of fat included the following steps:

- Homogenization of sample
- Extraction of fat
- Drying the evaporating flask
- Weighing the extract

4.1 Homogenization of sample

1. Sample was placed in small batches into the mortar and then crushed until uniform particles were obtained. The homogenized content of several packages was then transferred into one vessel and then mixed to get a uniform fat content.
2. Weights of empty evaporating flasks, extraction thimble, washing bags and cotton wool were measured before the actual extraction.

4.2 Extraction of fat

1. Sample was transferred into extraction thimble or a washing bag, placed inside the extraction chamber and sealed with cotton.
2. Parameter settings

Table 1: Set parameters for both laboratory and industrial versions

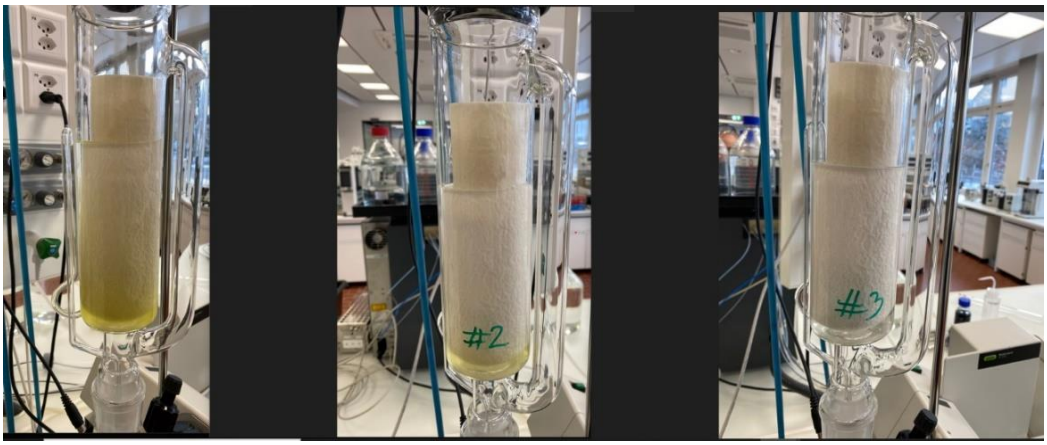
Parameter	R-300 System	R-220 Pro System
Rotation speed [rpm]	280	150
Heating bath T [°C]	60	60
Cooling T [°C]	10	10
Pressure [mbar]	170	170

Picture 1: Parameters on two different Rotavapor® systems

3. Once the parameters were reached, the evaporating flask with the solvent was submerged into the heating bath medium.
4. The duration of an extraction process was visibly determined by the lack of yellow coloring (lack of fat) in the distillate. Once the distillate passing through the extraction chamber was totally transparent, the process was terminated.

Visual observation and experiment stop:

- Although official guidelines require 20 cycles, in a case of a laboratory Rotavapor®, the experiment was terminated after 9 cycles (45 min). This included app 4 min per cycle and soaking up period of initial sample.
- In case of an industrial Rotavapor®, the experiment was terminated after 1 h and 15 min (did not include the soaking up period, which lasted additional 15 min). This included 2 manual cycles (solvent draining) at 30 min and 1h. The goal of both was to completely drain the extraction chamber and increase the efficiency of the process.



Picture 2: Extraction progression on a laboratory Rotavapor®. 1, 2 and 3 cycle

Picture 3: Extraction progression on an industrial Rotavapor® at time 0 and after 6 min

4.3 Drying the evaporating flask

Once the experiment was terminated, the solvent had to be removed from the evaporating flask.

R-300 System:

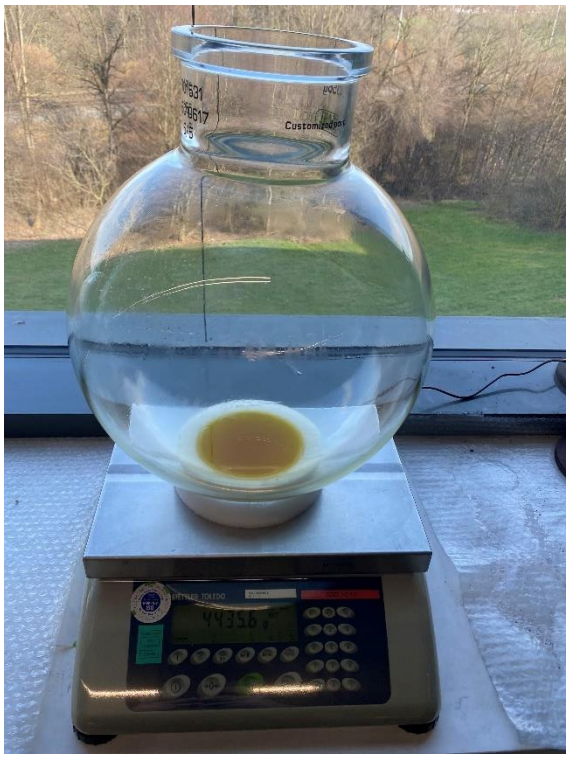
a) Soxhlet accessory was removed after the last drainage step and the shut off valve was opened to enable the removal of solvent at the set parameters.
b) After the removal of solvent, the shut off valve was closed again, and the pressure was set to 10 mbar to ensure no solvent was left in the evaporating flask.
c) Once the actual pressure setting was close the set value, it indicated that no solvent was left in the system and that the flask containing the extract was ready for weighing.

R-220 Pro Extraction system:

a) Extraction chamber was sealed off and drained. Simultaneously, the receiving flask valve was opened to enable the removal of the solvent.
b) After the removal of solvent, the shut off valve was closed again, and the pressure was set to 10 mbar to ensure no solvent was left in the evaporating flask.
c) Once the actual pressure setting was close the set value it indicated that no solvent was left in the system and that the flask containing the extract was ready for weighing.

4.4 Weighing the extract

1. Evaporating flasks containing the extract were weighed on a laboratory balance.
2. For obtaining results, the individual initial values were subtracted from the dried weights.



Picture 4: Extract in the evaporating flask

5. Results

The results are calculated as a percentage of fat in the initial sample.

Extract on R-300 system:

Table 2: Weight results for a laboratory Rotavapor®

	Initial weight [g]	final weight [g]]
m sample	100.64	/
m evap. flask	247.96	/
m evap. flask + extract	/	257.65

$$m\ extract = m\ (evap.\ flask + extract) - m\ evap.\ flask$$

$$m\ extract = 257.65 - 247.96$$

$$m\ extract = 9.69\ g$$

$$\%\ fat\ content = m\ extract / m\ initial\ sample$$

$$\%\ fat\ content = 9.63$$

Extract on R-220 Pro Extraction System:

Table 3: Weight results for an industrial Rotavapor®

	Initial weight [g]	final weight [g]]
m sample	873.86	/
m evap. flask	4355.5	/
m evap. flask + extract	/	4435.6

$$m\ extract = m\ (evap.\ flask + extract) - m\ evap.\ flask$$

$$m\ extract = 4435.6 - 4355.5$$

$$m\ extract = 80.1\ g$$

$$\%\ fat\ content = m\ extract / m\ initial\ sample$$

$$\%\ fat\ content = 9.17$$

Distillation rate:

During the experiment a distillation rate of heptane was also measured. This was done by measuring the volume of distillate in three separate intervals.

Table 4: Distillation rates for heptane on a laboratory Rotavapor®

t [min]	V [mL]	Φv [mL/min]
2	122	61
3	174	58
4	224	56
		Average: 58.3

Table 5: Distillation rates for heptane on an industrial Rotavapor®

V [L]	t [s]	Φv [mL/min]
1	450	133
2	890	134
3	1240	145
4	1600	150
		Average: 141

Table 6: Extraction rate on individual systems

	m extract [g]	t process [min]	Φ extract [g/min]
Laboratory Rotavapor®	9.69	45	0.22
Industrial Rotavapor®	80.1	75	1.068

6. 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.

An industrial Rotavapor® is able, due to the larger surface area of the flask, to distil 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. Additionally, the industrial version has 5x more volume in the extraction chamber, which means one can fill in more sample. This means that the R-220 Pro Extraction can successfully be used to scale up the extraction process, previously evaluated on a laboratory Rotavapor®.