HERBAL EXTRACTS

Turbidity reduction of selected extracts

NOTC
Product names and filter sheet grades may have changed since the application note was created.

Author: Bettina Ledergerber

FILTROX AG, Moosmühlestrasse 6, 9001 St.Gallen, Switzerland

E-Mail: applications@filtrox.com

In collaboration with: Lokal365 AG, St.Gallen

Date: March 2022

Keywords: extracts, herbal extracts, filtration, depth filtration, alluvial filtration, turbidity reduction, lab scale, purification

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Abstract

Plant extracts consist of valuable ingredients and are thus used both in the food industry as supplements and in the cosmetics industry. Depending on the plant from which the extract is obtained, secondary nutrients (e. g. flavonoids, terpenes, alkaloids) or the fragrance of the plant should be retained in the extracts. In this application note, five different herbal extracts from the company Lokal365 AG in St.Gallen were purified using depth filtration. The aim is to retain the flavor and fragrance of the valuable herbs. Filtration tests on a laboratory scale were carried out to evaluate the optimum production process. In order to filter out all solid particles, which have a strong particle size distribution independent of the herb, an alluvial filtration had to be carried out in each case. Based on the generated results regarding filtration capacity, a scale-up to a batch size of 100 L was calculated for each herbal extract.

1 The Challenge

Extracts were produced from the five herbs ysop, sage, chili, thai basil and basil from the company Lokal365 AG in St.Gallen. The extracts are to be purified as far as possible so that they can then be diluted with water, resulting in a flavored water as the end product. The purification is to be carried out by filtration, whereby on the one hand the turbidity is to be reduced to a maximum and on the other hand the extracts are to be preserved. The latter, however, shall be carried out by using a filter membrane with absolute retention rate of 0.2 µm to guarantee the removal of any microorganisms. The removal of solid particles will be carried out in laboratory scale filtration experiments to develop a suitable filtration process. Depth filter sheets will be used for turbidity reduction.

The extraction was first carried out by mechanical comminution and then by ultrasonic treatment. Due to this procedure, particles of different sizes are produced, which poses a major challenge during the purification process. Depending on the type of herb, the addition of filter aid and thus alluvial filtration is necessary to enable sufficient purification.

Figure 1: Overview of herbs whose extracts are to be filtered. After preparation of the extracts, they are to be purified by means of depth filtration and a filter membrane with an absolute retention rate of 0.2 µm.

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2 Material and Methods

All the materials used for the filtration experiments of the herbal extracts on a laboratory scale are listed below. The procedure for pre-rinsing the filter sheets used is described in subchapter 2.2.

2.1 Material

The setup and thus the materials used are shown in figure 2. The corresponding FILTROX FIBRAFIX® depth filter sheet was inserted into a FILTRODISC™ BIO SD 2" capsule (FILTROX), which consists of two individual parts (figure 2b and 2c). In this process, a filter cake can be built up in the upper part of the BIO SD 2" capsule. Subsequently, the FILTRODISC™ capsule with the filter sheet was each clamped into the synthetic filter holder (FILTROX, see figure 2d) and this was attached to a laboratory stand. The inlet of the capsule was then connected to a pressure gauge (TRI-MATRIX AG) via a #17 silicone hose (Shenchen Precision Pump Co. Ltd.). A peristaltic pump (Baoding Shenchen Precision Pump Co. Ltd. with the YZ1515x pump head) was used to deliver the extracts. The maximum differential pressure of 2.5 bar should not be exceeded. Since the peristaltic pump used cannot exceed a maximum pressure of 2 bar, the experiments were stopped prematurely in each case. The turbidity of the unfiltered and filtered samples was measured using the Eutech TN-100 turbidity meter (Thermo Scientific™).

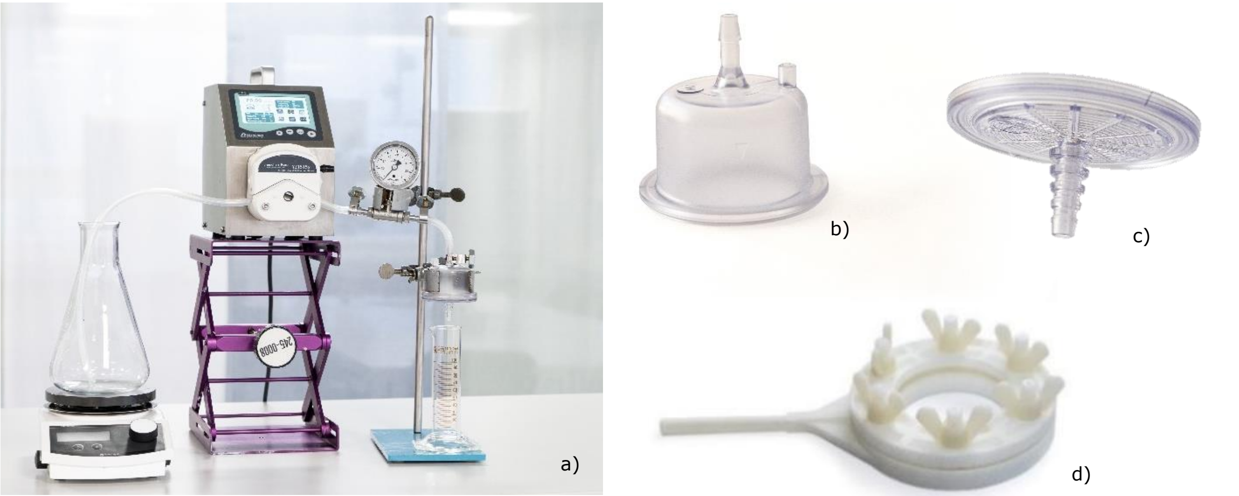

Figure 2: Overview of the experimental setup and the individual parts of the filter capsule used. The test setup (a) consisted of a peristaltic pump, a pressure gauge and the filter capsule. Any filter sheet can be placed between the two individual parts of the FILTRODISC™ BIO SD 2" filter capsule (b and c) and then sealed with the synthetic holder (d). Since the unfiltered extracts had high turbidity values, each was mixed using a magnetic stirrer. To measure the filtration flux over time, the filtrate was collected in a graduated cylinder.

2.2 Preflushing

In order to rinse out all loose particles from the depth filter sheet, it is recommended to preflush each filter sheet with a process-compatible solution. During the filtration of the herbal extracts, the sheets were rinsed with 50 L/m² of tap water before each filtration test. Since the filter area of a FILTRODISC™ BIO SD 2" capsule is 0.0021 m², this corresponds to a rinse volume of about 100 mL. As an alternative, the product can be filtered directly, but the first 50 L/m² should be recirculated. After preflushing, the individual extracts were filtered. In each case, the filter layer type, the filter aid used and the amount of the corresponding filter aid were varied.

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3 Results and Discussion

In this chapter, all filtration experiments carried out are explained. In order to achieve sufficient purification, a filter aid had to be added to each extract - with the exception of the chili extract. With the aid of this filter aid, a so-called alluvial or precoat filtration can be carried out, which usually results in a higher filter capacity. Celpure® diatomaceous earth was used for this purpose, which is mainly used in the pharmaceutical industry, but also in the cosmetics industry, due to its high quality. In order to compare the individual tests, the filtration flux, the differential pressure and the turbidity were measured over time.

3.1 Ysop extract

The purification of the ysop extract was determined in three trials. In the first experiment, no filter aid was added, which led to a rapid increase in pressure. As can be seen in table 1, 20 g/L on the one hand and 10 g/L on the other hand of the filter aid Celpure® C100 were added to the unfiltered extract in the following two experiments. Due to the available cake volume in the FILTRODISC™ BIO SD 2" capsule, which is the limiting factor, a significantly higher volume could be filtered with only 10 g/L diatomaceous earth.

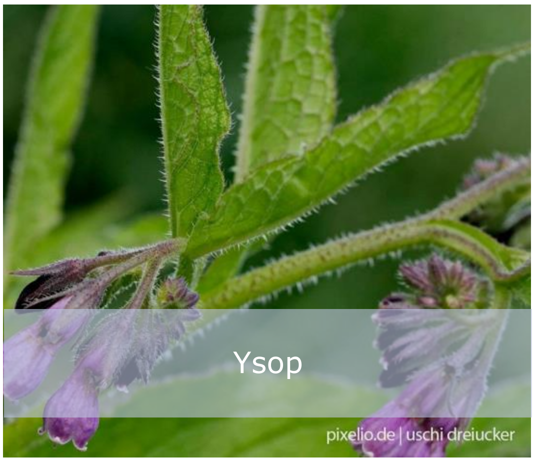

Table 1: Overview of the filtrations of the ysop extract. Compared to the first experiment, where no diatomaceous earth was added, a significantly larger volume could be filtered when filter aid was added.

Test No.	Filter sheet	Type & Amount FA*	Volume Filtrate	Filter cake height	Turbidity
Unfiltrate	N/A	N/A	N/A	N/A	789 ± 0.041 NTU
1	FIBRAFIX® AF 11H	N/A	125 mL	0.2 cm	7.76 ± 1.185 NTU
2	FIBRAFIX® AF 11H	Celpure® C100, 20 g/L	520 mL	3.0 cm	0.72 ± 0.645 NTU
3	FIBRAFIX® AF 11H	Celpure® C100, 10 g/L	675 mL	1.5 cm	4.52 ± 0.458 NTU

* FA = Filter aid

The chronological sequence of the filtration flux, the differential pressure as well as the turbidity of all three experiments is shown in the following graph 1. In the third and last experiment, about 680 mL of the ysop extract could have been filtered for 38 minutes. A final turbidity value of 4.52 ± 0.458 NTU (n = 7) was achieved, indicating that subsequent sterile filtration can be realized. Filtration had to be stopped because, on the one hand, no unfiltered extract was left and, on the other hand, an already high differential pressure of 1.5 bar was reached.

The following figure 3 shows the visual comparison of the unfiltered ysop extract with the filtrate from tests 2 and 3. The filter cakes generated differ clearly in height due to the different amounts of diatomaceous earth added.

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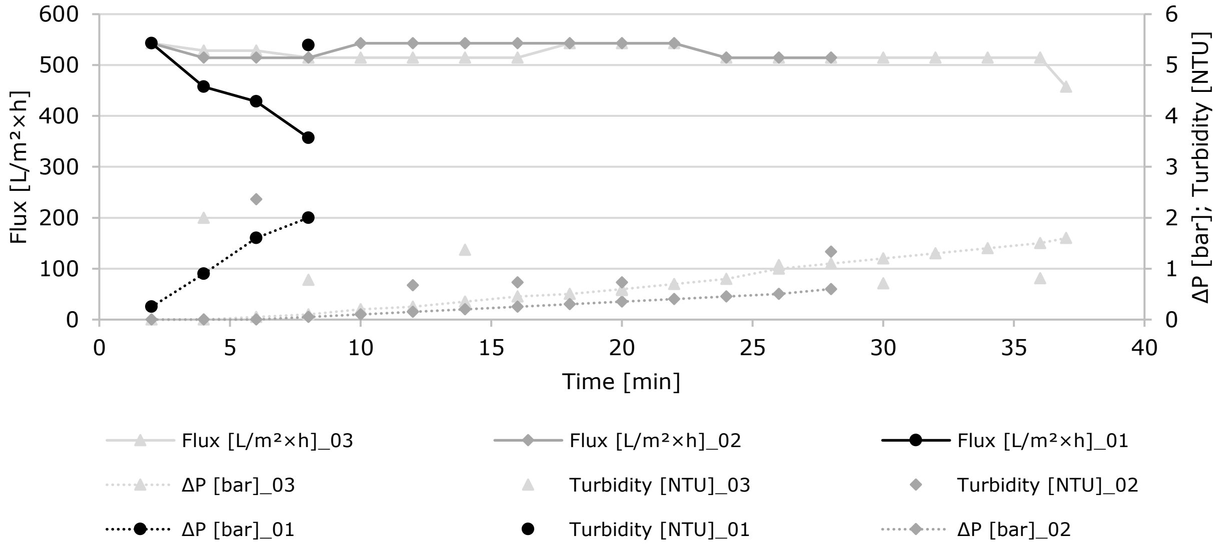

Graph 1: Ysop extract – filtration experiments 1, 2 and 3. The chronological sequence of filtration flux, differential pressure and turbidity is shown. An initial turbidity of 789 NTU was measured.

Figure 3: Ysop extract – unfiltered and filtered extract from experiments 2 (a) and 3 (b). The unfiltered extract is compared with the filtrate and the filter cake generated. Since twice the amount of diatomaceous earth was used in the second experiment, a significantly higher filter cake resulted - compared to experiment 3.

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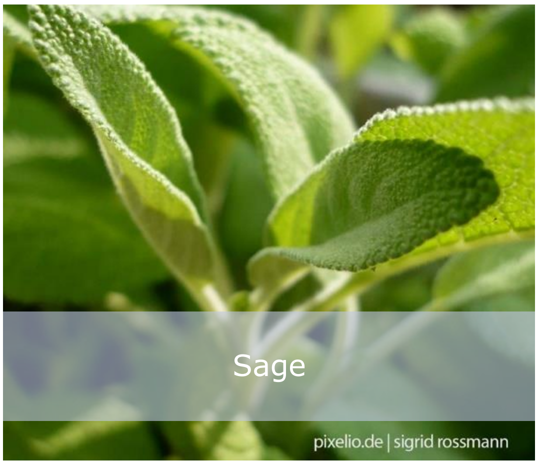
3.2 Sage extract



The sage extract was also filtered first without the addition of a filter aid (see table 2 below). Here, a strong pressure increase was already observed shortly after the start of filtration too, which is why a corresponding amount of Celpure® C100 was added in the subsequent experiments 2 and 3. As a comparison, a different Celpure® type was added to the unfiltrate in the fourth experiment and 880 mL of the extract was filtered within 44 minutes. This filtration test had to be stopped because the BIO SD 2" capsule was completely filled with filter cake. As mentioned above, the available cake volume inside the capsule is the limiting factor in precoat filtration.

Table 2: Overview of the filtrations of the sage extract. After a first test without filter aid, two different diatomaceous earth types and different amounts were added to the unfiltered extract in the subsequent tests.

Test No.	Filter sheet	Type & Amount FA*	Volume Filtrate	Filter cake height	Turbidity
Unfiltrate	N/A	N/A	N/A	N/A	1640 ± 0.172 NTU
1	FIBRAFIX® AF 11H	N/A	95 mL	0.2 cm	4.45 ± 0.047 NTU
2	FIBRAFIX® AF 11H	Celpure® C100, 10 g/L	250 mL	0.7 cm	1.91 ± 0.422 NTU
3	FIBRAFIX® AF 11H	Celpure® C100, 20 g/L	340 mL	2.2 cm	2.63 ± 0.278 NTU
4	FIBRAFIX® AF 11H	Celpure® C1000, 20 g/L	880 mL	3.5 cm	0.67 ± 0.471 NTU
5	FIBRAFIX® AF 11H	Celpure® C1000, 15 g/L	615 mL	2.9 cm	0.99 ± 0.292 NTU

* FA = Filter aid

The difference between the two Celpure® grades used is permeability, with the C100 being more finely porous and therefore having a lower permeability compared to the C1000. It turned out that the coarse-pored Celpure® C1000 created the better filtration conditions for this extract – with the same dosage. Therefore, it can be assumed that there are more larger particles in the sage extract. Turbidity values of less than 1 NTU were achieved. The coarse-pored filter sheet FIBRAFIX® AF 11H was used in all tests.

Graph 2 shows the chronological courses of filtration flow, differential pressure and turbidity of tests 1, 2 and 3. The courses of tests 4 and 5 are shown in graph 3 below. The visual comparison of the unfiltered sage extract in each case with the filtrates can be seen in the figure 4.

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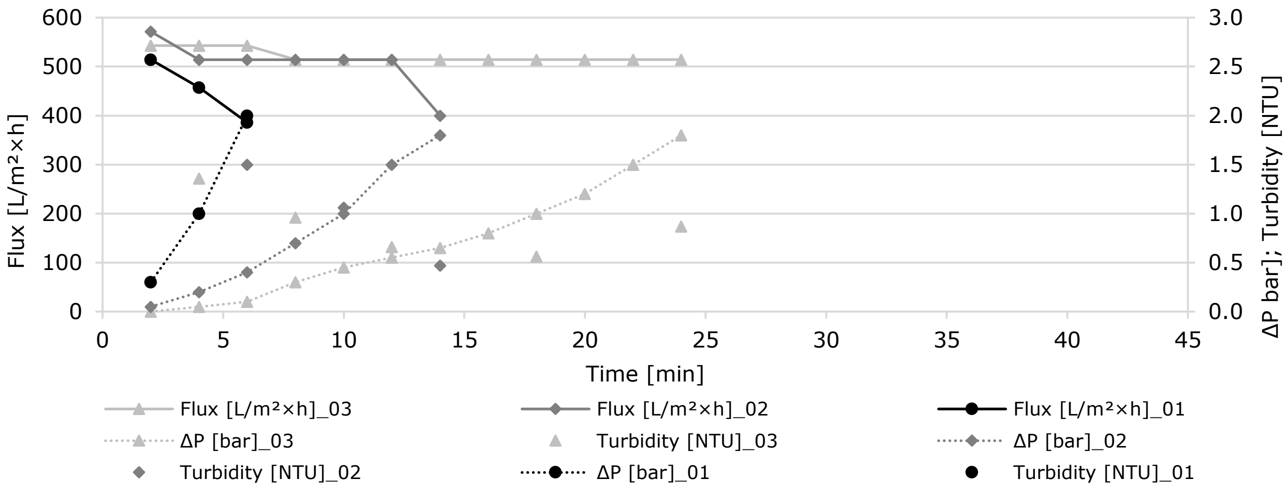
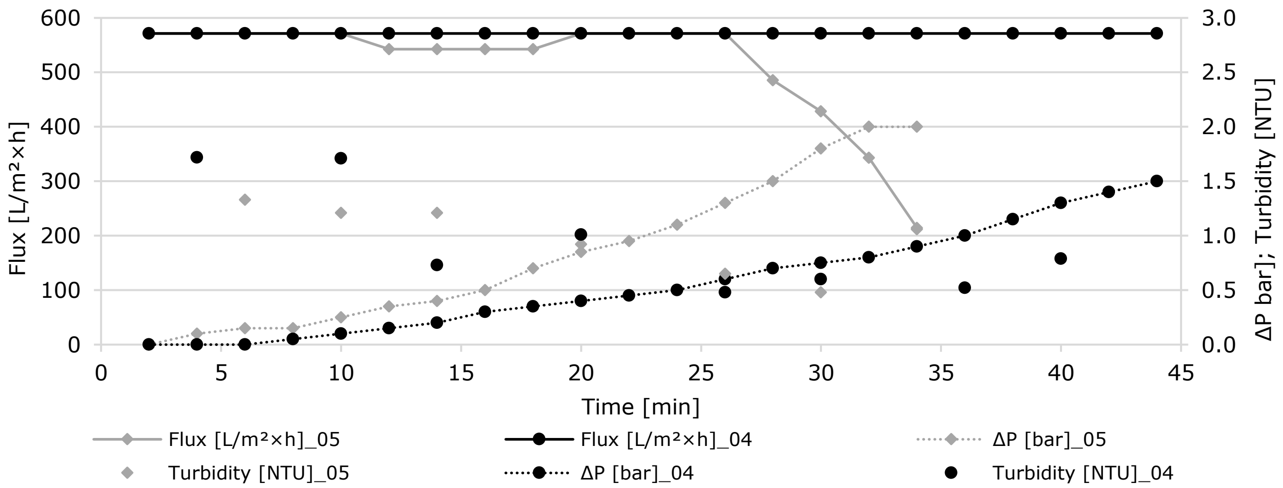


Graph 2: Sage extract – filtration experiments 1, 2 and 3. The chronological sequence of filtration flux, differential pressure and turbidity is shown. An initial turbidity of 1640 NTU was measured.



Graph 3: Sage extract – filtration experiments 4 and 5. The chronological sequence of filtration flux, differential pressure and turbidity is shown. An initial turbidity of 1640 NTU was measured.

Figure 4: Sage extract – filtration experiment 4 (a) and 5 (b). The unfiltrate is compared with the filtrate in each case. The filter cakes generated in the process are also shown and differ in height – depending on the volume filtered in each case as well as the amount of diatomaceous earth added. In the fourth experiment, 880 mL of extract was filtered, resulting in a filter cake of 3.5 cm (a).

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3.3 Thai basil extract

The optimal purification of the thai basil extract was elicited in four filtration experiments, which are listed in table 3. Again, no filter aid was added in the first experiment, and 20 g/L Celpure® C100 was added to the unfiltered extract in the subsequent experiment. Since the FILTRODISC™ BIO SD 2" capsule was not yet completely filled with filter cake, twice the amount of diatomaceous earth was subsequently added in both experiments 3 and 4. The aim was to be able to filter a larger volume. This worked well, so in a further step a filtration test was carried out with the same amount of diatomaceous earth, but a different type (Celpure® C1000).

Table 3: Overview of the filtrations of the Thai basil extract. The first experiment served as an orientation as to how the extract behaves during filtration. Therefore, no filter aid was added here. In the other trials, the addition of a filter aid was necessary.

Test No.	Filter sheet	Type & Amount FA*	Volume Filtrate	Filter cake height	Turbidity
Unfiltrat	N/A	N/A	N/A	N/A	1460 ± 0.104 NTU
1	FIBRAFIX® AF 11H	N/A	10 mL	N/A	N/A
2	FIBRAFIX® AF 11H	Celpure® C100, 20 g/L	60 mL	0.5 cm	2.08 ± 0.005 NTU
3	FIBRAFIX® AF 11H	Celpure® C100, 40 g/L	210 mL	2.5 cm	1.54 ± 0.035 NTU
4	FIBRAFIX® AF 11H	Celpure® C1000, 40 g/L	450 mL	3.4 cm	1.36 ± 0.459 NTU

* FA = Filter aid

As already described in chapter 3.2, the two diatomaceous earth types differ in their permeability. The thai basil extract also seems to contain predominantly larger particles, which is why the Celpure® C1000 performed significantly better. As can be seen in the following graph 4, in the fourth experiment 450 mL could be filtered for 24 minutes. Subsequently, the filtration had to be stopped because a differential pressure of 1.8 bar was reached. In addition, a filter cake height of 3.4 cm and a final turbidity of 1.5 NTU were measured. As can be seen in figure 5b, the filter cake was already growing slightly into the silicone tubing. In the same figure, the visual comparison of the unfiltered Thai basil extract with the generated filtrates of experiments 2, 3 and 4 is shown.

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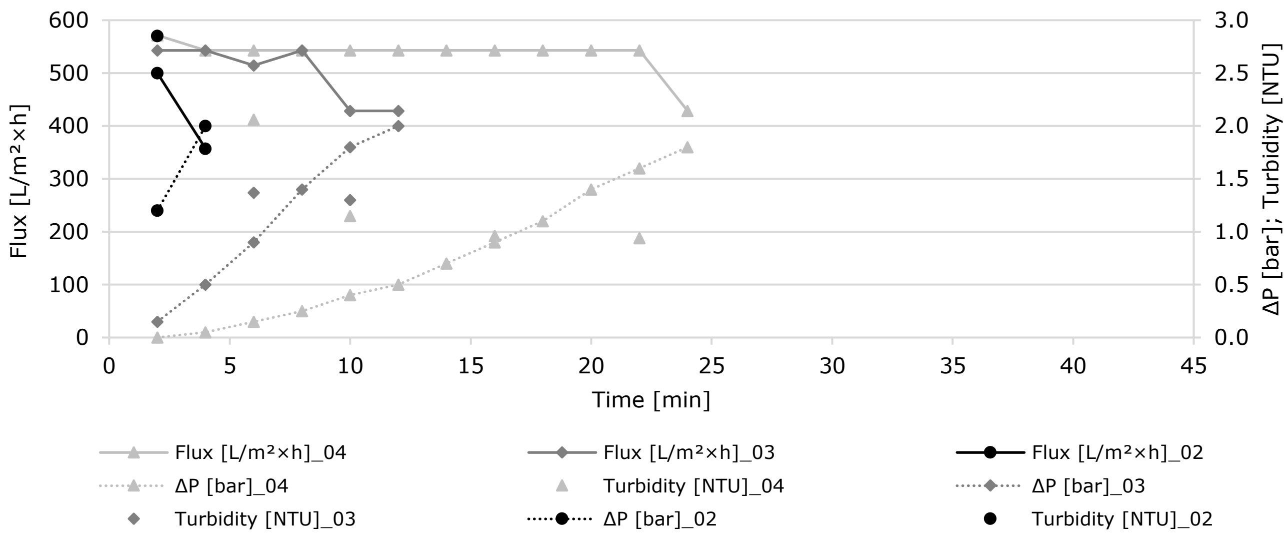

Graph 4: Thai basil extract – filtration experiments 2, 3 and 4. The chronological sequence of the filtration flux, differential pressure as well as turbidity is shown. An initial turbidity of 1231 NTU was measured.

Figure 5: Thai basil extract – comparison of the filtrates from experiments V2, V3 and V4 (a) and direct comparison with the filtrate from experiment 4 (b), in each case with the unfiltered extract. Using the alluvial filtration technology, the initial turbidity of 1460 NTU could be reduced to around 2 NTU and lower (experiments 2, 3 and 4). Since very little filtrate was generated in the first experiment, it is not shown here.

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3.4 Basil extract

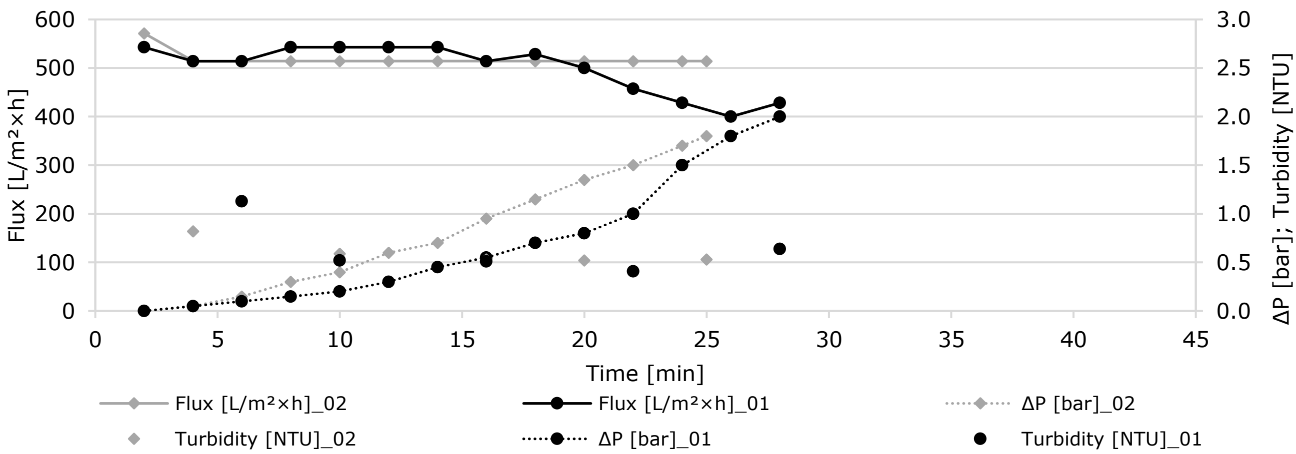
The individual filtrations of the basil extract are shown in table 4. In the first filtration, 20 g/L Celpure® C100 was added, and a pressure increase to 2 bar was observed within 28 minutes. The second test, in which only 10 g/L Celpure® was added, could be run at the same flux and a comparable pressure curve was recorded (see graph 5). Therefore, the amount of Celpure® was further reduced in trials 3 and 4 to 5 g/L and 8 g/L, respectively. In test 3, a too rapid pressure increase was observed, which is why 8 g/L filter aid seemed ideal. After 32 minutes was a pressure of 2.0 bar reached (see graph 6).

Table 4: Overview of the filtrations of the basil extract. An appropriate amount of filter aid was added for all tests. Since a relatively large volume could be filtered in the first test, the amount of filter aid was steadily reduced.

Test No.	Filter sheet	Type & Amount FA*	Volume Filtrate	Filter cake height	Turbidity
Unfiltrate	N/A	N/A	N/A	N/A	1148 ± 0.089 NTU
1	FIBRAFIX® AF 11H	Celpure® C100, 20 g/L	490 mL	3.8 cm	1.02 ± 0.255 NTU
2	FIBRAFIX® AF 11H	Celpure® C100, 10 g/L	455 mL	3.2 cm	0.74 ± 0.114 NTU
3	FIBRAFIX® AF 11H	Celpure® C100, 5 g/L	345 mL	2.5 cm	0.57 ± 0.146 NTU
4	FIBRAFIX® AF 11H	Celpure® C100, 8 g/L	600 mL	3.2 cm	0.40 ± 0.406 NTU

* FA = Filter aid

Compared to the thai basil extract, the Celpure® C100 was used for the filtration of the basil extract. In addition, a significantly lower amount of filter aid was required – a volume of 600 mL of extract could be filtered with only 8 g/L. A cake height of 3.2 cm and a turbidity of 0.40 NTU were achieved. All filtrates are are shown and compared with the unfiltered basil extract below in figure 6a.



Graph 5: Basil extract – filtration experiments 1 and 2. The chronological sequence of filtration flux, differential pressure and turbidity is shown. An initial turbidity of 1148 NTU was measured.

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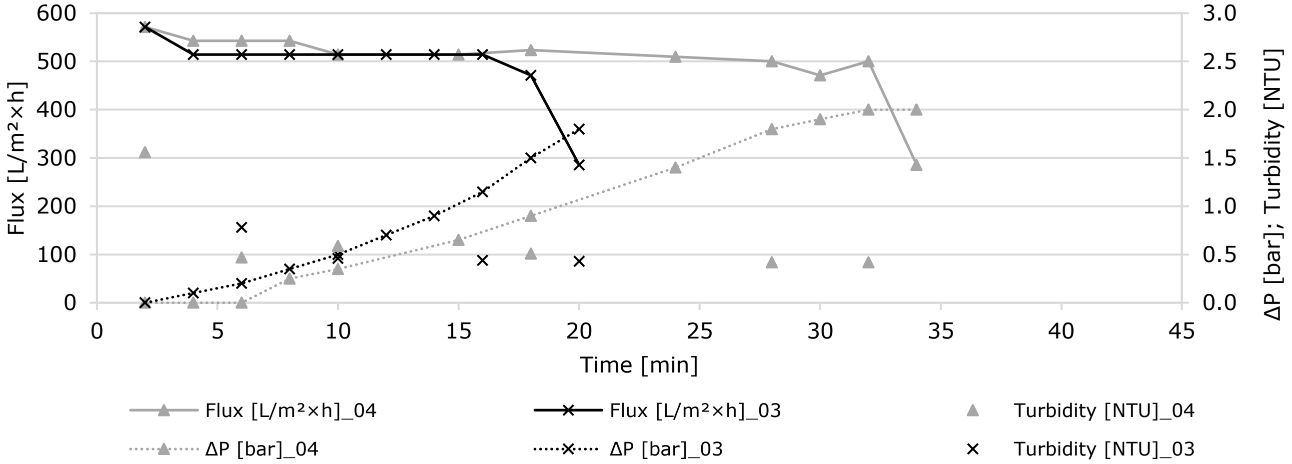

Graph 6: Basil extract – Filtration experiments 3 and 4. The chronological sequence of filtration flux, differential pressure and turbidity is shown. An initial turbidity of 1148 NTU was measured.

Figure 6: Basil extract – Overview of all filtrates (a) and comparison of the unfiltered extract with the filtrate from experiment 4 (b). The turbidity was reduced from over 1100 NTU to 0.4 NTU in experiment 4. A filter cake with a height of 3.2 cm was generated.

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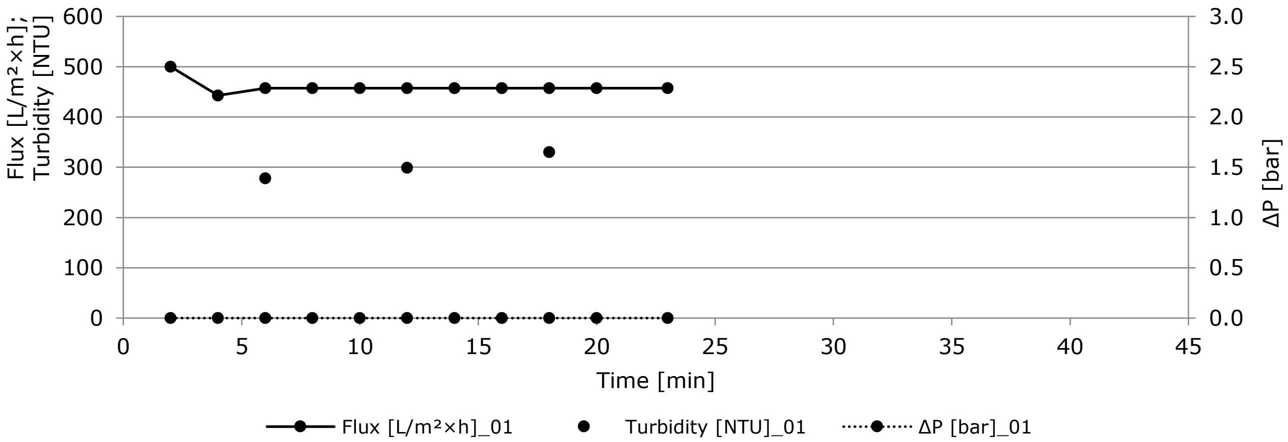
3.5 Chili extract

Due to the limited amount of chili extract, only two filtration experiments were performed. In both experiments, no filter aid was added and the trials differed only in the filter sheet used. In the first experiment FIBRAFIX® AF 11H was used – as in the filtrations of all other extracts. In order to achieve a higher turbidity reduction, the finer filter sheet FIBRAFIX® AF 101H was used in the subsequent experiment (see table 5). Graph 7 shows the chronological curves of the filtration flux, the differential pressure as well as the turbidity values.

Table 5: Overview of the filtrations of the chili extract. Two filtration experiments were carried out, whereby no filter aid was added in each case.

Test No.	Filter sheet	Volume Filtrate	Filter cake height	Turbidity
Unfiltrat	N/A	N/A	N/A	853 ± 15.121 NTU
1	FIBRAFIX® AF 11H	370 mL	1 cm	318 ± 21.359 NTU
2	FIBRAFIX® AF 101H	200 mL	0.8 cm	23.7 ± 2.457 NTU

Figure 7 shows the unfiltered chili extract with both filtrates. In these experiments, no filter aid was added to the unfiltered extract, but a filter cake was nevertheless built up. This consisted mainly of the chili seeds, but also of others coarse and fine solid particles. Due to the significantly finer filter sheet FIBRAFIX® AF 101HP, which was used in the second filtration test, the turbidity could be reduced to about 23 NTU. In order to reduce the turbidity even further, further filtration tests with a finer filter sheet and, if necessary, filter aids would have to be carried out in a further step.



Graph 7: Chili extract – Filtration experiment 1. The chronological sequence of the filtration flux, differential pressure and turbidity is shown. An initial turbidity of 853 NTU was measured.

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Figure 7: Chili extract – Overview of the filtrates (a) and comparison of the unfiltered filtrate with the filtrate from filtration experiment 1 (b). Although no filter aid was added to the unfiltered chili extract, a filter cake was built up in each case. However, due to the very large chili seeds, this was formed very irregular.

4 Scale-up

The following calculations [1] and [2] are used to determine the minimum available cake volume and filter area required for each batch size on a pilot or production scale. In addition, the values for the herbal extracts, calculated to a final batch size of 100 L, are shown in table 6 below. Based on the filter capacity achieved in each case, the same filter system is recommended for each herbal extract, with the exception of the chili extract. The FILTRODISC™ BIO SD 12S system, which can be seen in figure 8, consists of a 12" diameter module. Due to the fact that the module is packed in a bag, easy handling as well as only minimal cleaning effort is guaranteed.

As can be seen in table 6, a significantly lower filter capacity was calculated for the chili extract filtration. Therefore, for the filtration of 100 L of chili extract, the FILTRODISC™ BIO SD 12D double module is recommended. As can be seen in figure 9, the difference between the two systems is the number of module elements and thus the number of lenses within a bag. This in turn results in a larger filter area as well as a larger volume available to build up a filter cake. Due to the pressure rise reached during filtration, the modules must each be installed in the appropriate stainless steel housing (DISCSTAR™ BIO SD).

$$A_{prod.}[m^2] = \frac{V_{prod.}[L] \times A_{test}[m^2]}{V_{test}[L]} \quad [1]$$

$$c_{prod.}[m^3] = \frac{V_{prod.}[L] \times c_{test}[m^3]}{V_{test}[L]} \quad [2]$$

$A_{prod.}[m^2]$	Minimum required filter area in production scale (for a given batch size)
$A_{test}[m^2]$	Filter area, which was used for the laboratory scale tests
$V_{prod.}[L]$	Volume on a production scale (batch size)
$V_{test}[L]$	Filtered volume during laboratory scale tests
$c_{prod.}[m^3]$	Minimum required cake volume on a production scale (for a given batch size)
$c_{test}[m^3]$	Achieved cake volume in laboratory scale (= cake height [m] × filter area [m²])

Table 6: Overview of scale-up parameters calculated for batch size of 100 L. For each extract it is indicated which system is recommended in production scale.

		Ysop	Sage	Thai basil	Basil	Chili
Laboratory	Filtered volume [L]	0.675	0.880	0.450	0.600	0.200
	Filter capacity [L/m²]	321.4	419.0	214.3	285.7	95.2
	Cake height [cm]	1.5	3.5	3.4	3.2	0.8
	Cake volume [L]	0.0315	0.0735	0.0714	0.0672	0.0168
Production (100 L Batch)	Required filter area [m²]	0.32	0.24	0.47	0.35	1.05
	Required cake volume [L]	4.7	8.4	7.2	6.8	8.4
	Recommended System	FILTRODISC™ BIO SD 12S*				FILTRODISC™ BIO SD 12D*
	Number of lenses	6 lenses / module				5 lenses / module
	Filter area [m²]	0.66				1.10
	Cake volume [L]	8.9				19.8

* The FILTRODISC™ BIO SD 12D module consists of two module elements within one bag. The 12S contains only one 12" module element.

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5 Conclusion

Filtration tests on a laboratory scale are essential. This minimizes potential problems and risks in the subsequent pilot or production scale. The filtration tests described have shown that purification by depth filtration is a suitable purification method and that turbidity can be significantly reduced as a result. After just one filtration step, the extracts are purified to such an extent that sterile filtration can be realized. However, the addition of filter aids was necessary to achieve a correspondingly high filter capacity – with the exception of the chili extract. Here, however, it was already apparent before filtration that the extract contained a high proportion of chili seeds. These helped to quickly build up a filter cake – without the addition of a filter aid. Due to the much finer filter sheet FIBRAFIX® AF 101HP, which was used in the second filtration test, the turbidity could be reduced to about 23 NTU. To reduce the turbidity even further, additional filtration tests would have to be carried out in a further step with a finer filter sheet and, if necessary, filter aids.

For further optimization, additional filtration tests with different combinations of Celpure® (grades and quantities) as well as FILTROX depth filter sheets could be carried out in a next step, if necessary. If parameters are determined that lead to an increased filter capacity, the scape-up would have to be calculated again.

Figure 8: FILTRODISC™ BIO SD 12S module and DISCSTAR™ BIO SD. The single module can consist of 4, 5 or 6 lenses – depending on the required filter area and the volume needed for precoating. It is packed in a bag.

Figure 9: FILTRODISC™ BIO SD 12D module and DISCSTAR™ BIO SD. The double module can consist of 4, 5 or 6 lenses – depending on the required filter area as well as the volume necessary for precoating. It is packed in a bag.