

Process Development for the Production of Natural Dye Concentrates for the Textile Industry

Master Thesis of
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Aim of the work

GENERAL

Based on a patented extraction route at laboratory scale (Bechtold et al, 2010), the aim of this work was:

- to create a basic engineering concept and,
- to generate a calculation model for mass flow and energy requirements for the production of 1600 kg/year of NDC in powder form.

Aim of the work

SPECIFIC

Identification of process parameters which influence the quality and output of the NDC

→ strategies to approach the standardization problematic

The design of a semi-industrial production line for different plant materials.

Advantages and disadvantages of the implementation of different equipments.

Ensure low ecological impact.

Fundamentals

Plant material

R1

Onion peelings
(*Allium cepa* L.)



R2

Canada goldenrod
(*Solidago canadensis*)



R3

Walnut shells
(*Juglans regia* L.)



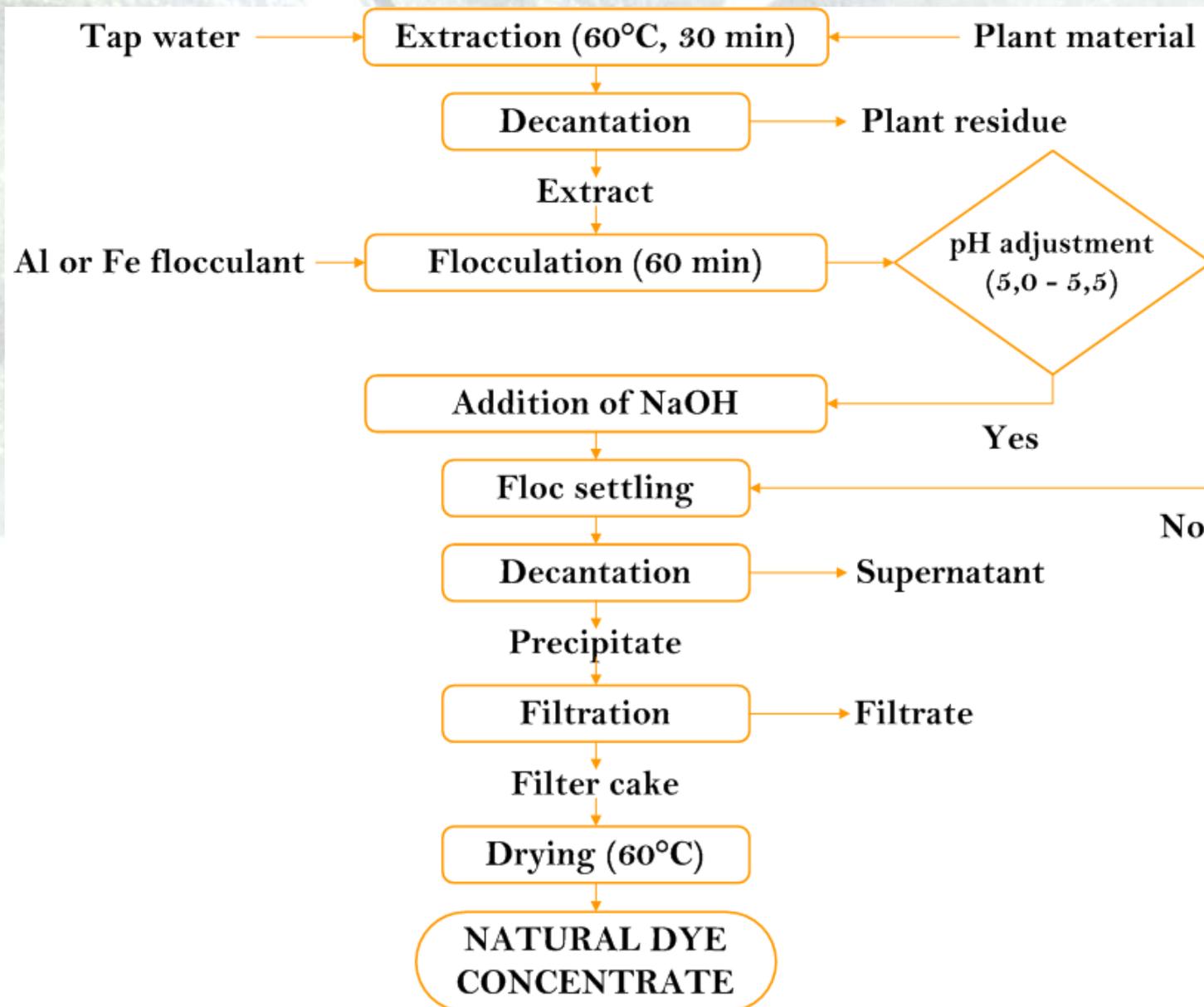
R4

Ash tree barks
(*Fraxinus excelsion* L.)



1600 kg/year of NDC

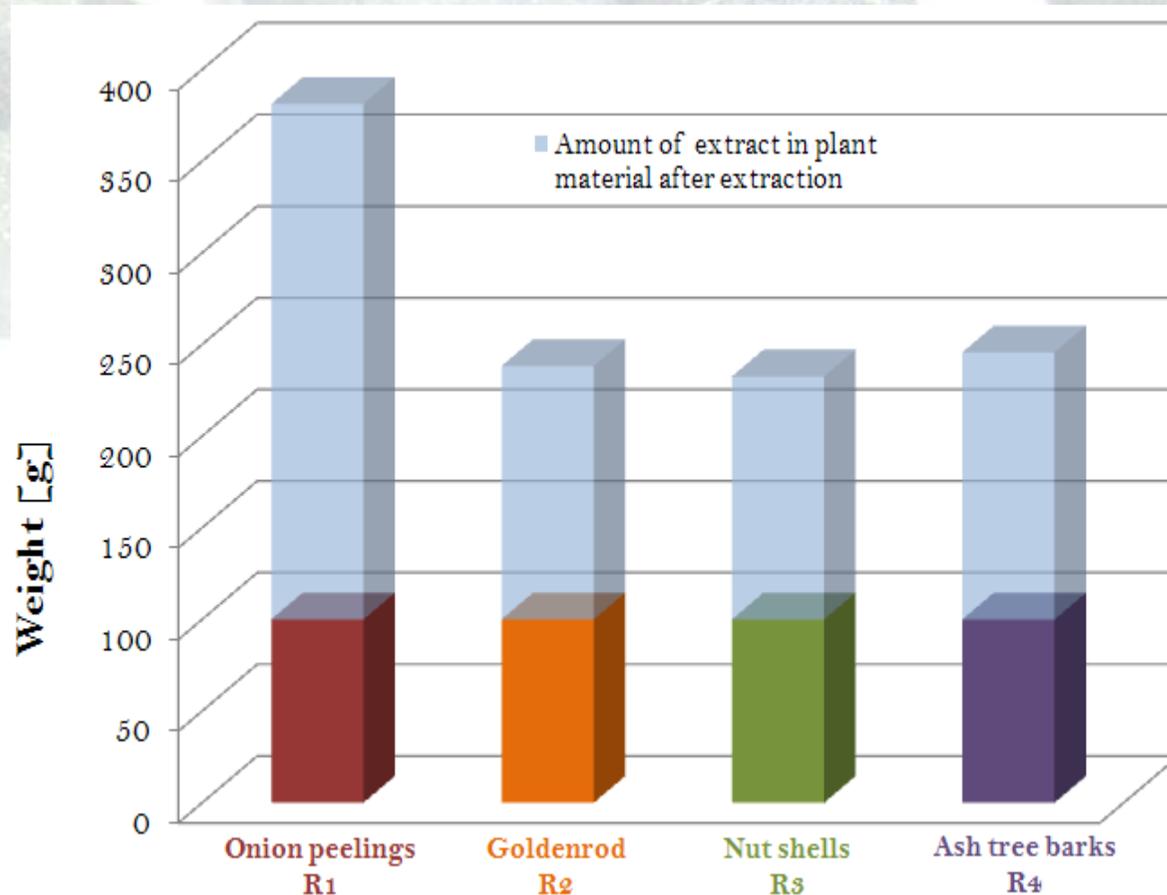
Fundamentals



Process parameters

1. Water absorption after extraction

- Pressing → 10 % increase of NDC output
- Equipment design → 5-10 % volume increase



Pressing 

Process parameters

2. Influence of the pH adjustment on the NDC output

- Aluminum sulphate = $\text{Al}_2(\text{SO}_4)_3 \cdot 16\text{H}_2\text{O}$
- Iron (II) sulphate = $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$

R1, $\text{Al}_2(\text{SO}_4)_3$
pH not adjusted ~ 3

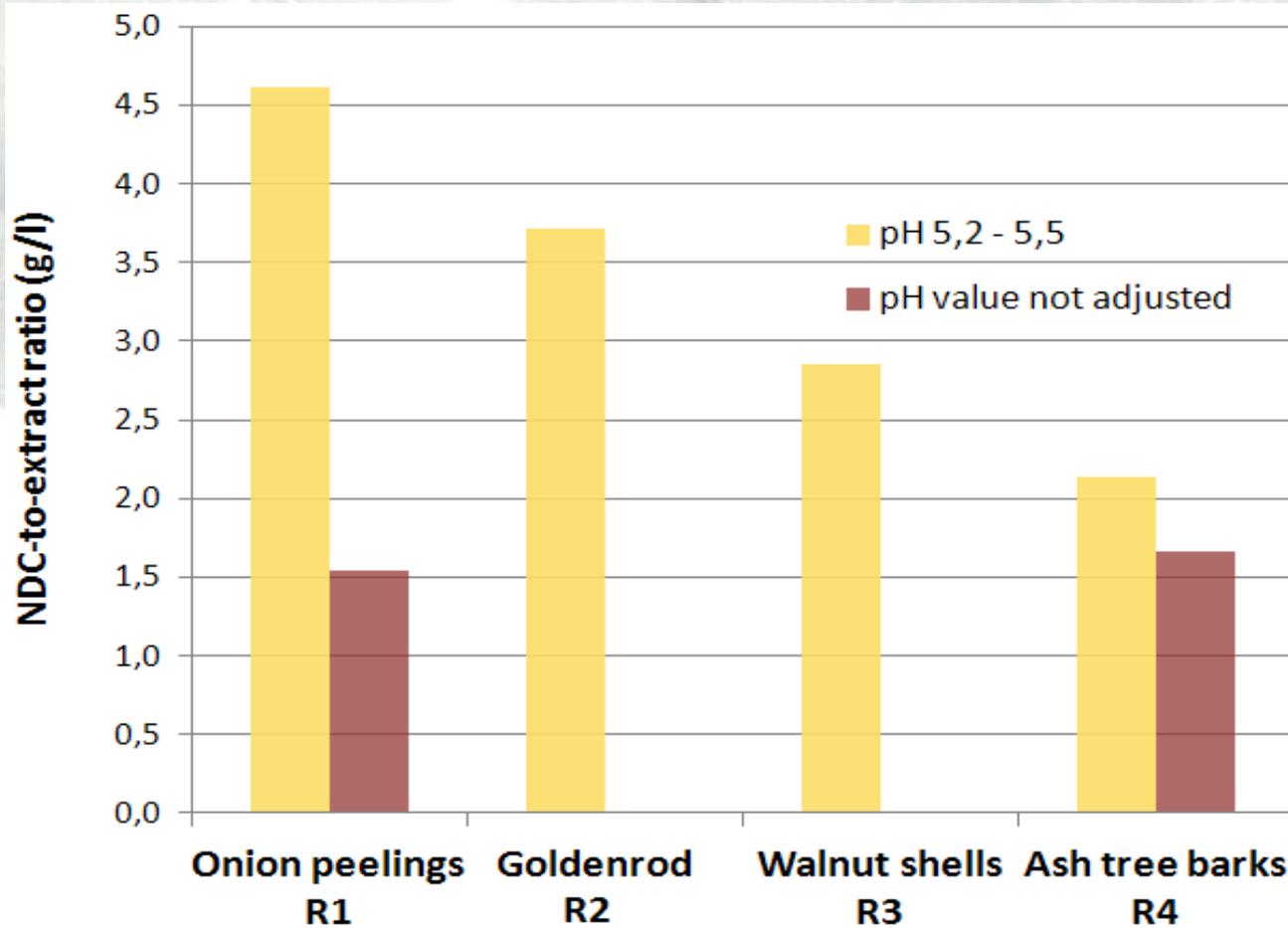


R1, $\text{Al}_2(\text{SO}_4)_3$
pH adjusted between 5,0 and 5,5



Process parameters

2. Influence of the pH adjustment on the NDC output

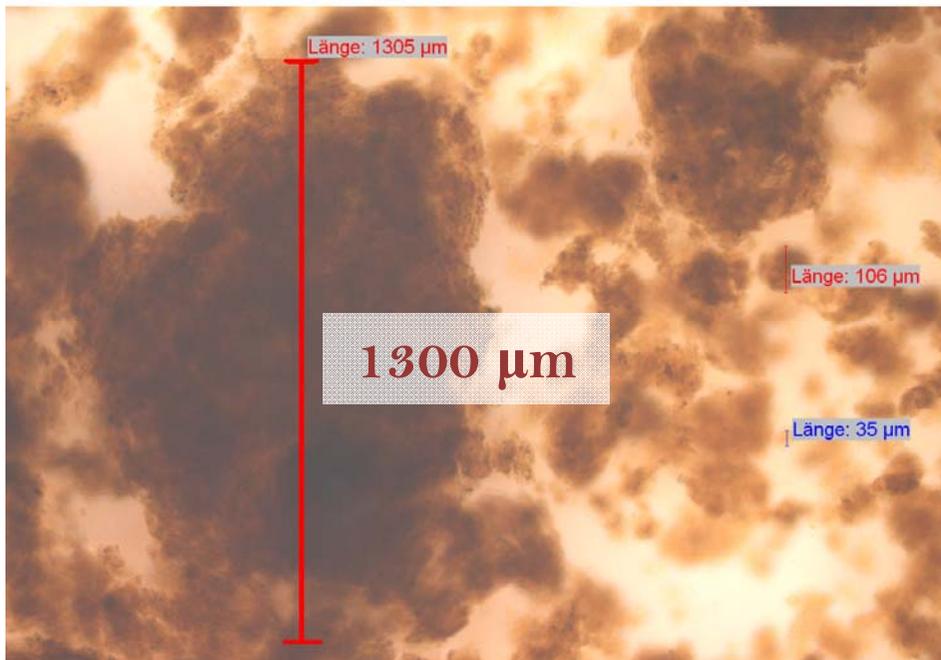


pH adjustment 

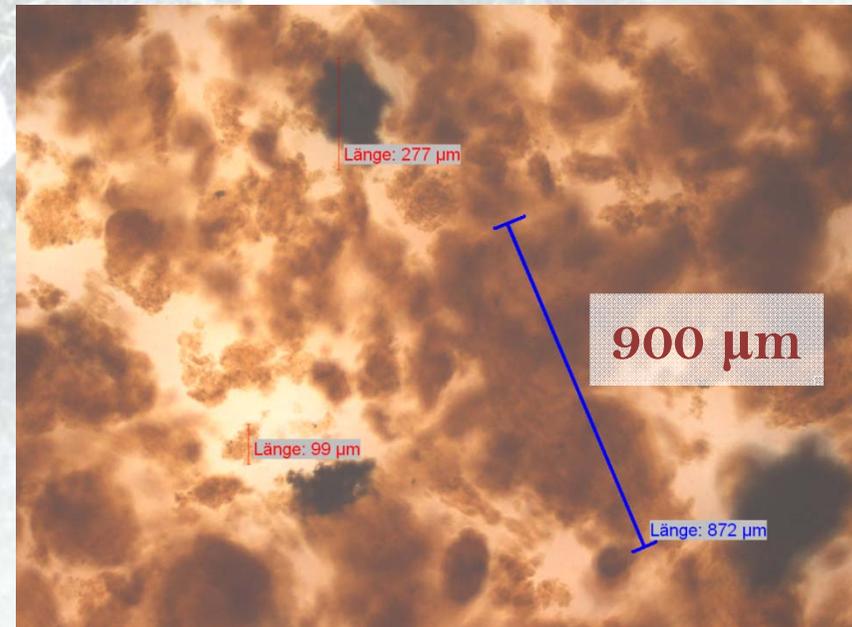
Process parameters

3. Influence of the flocculation time

Onion peelings - R1
30 min



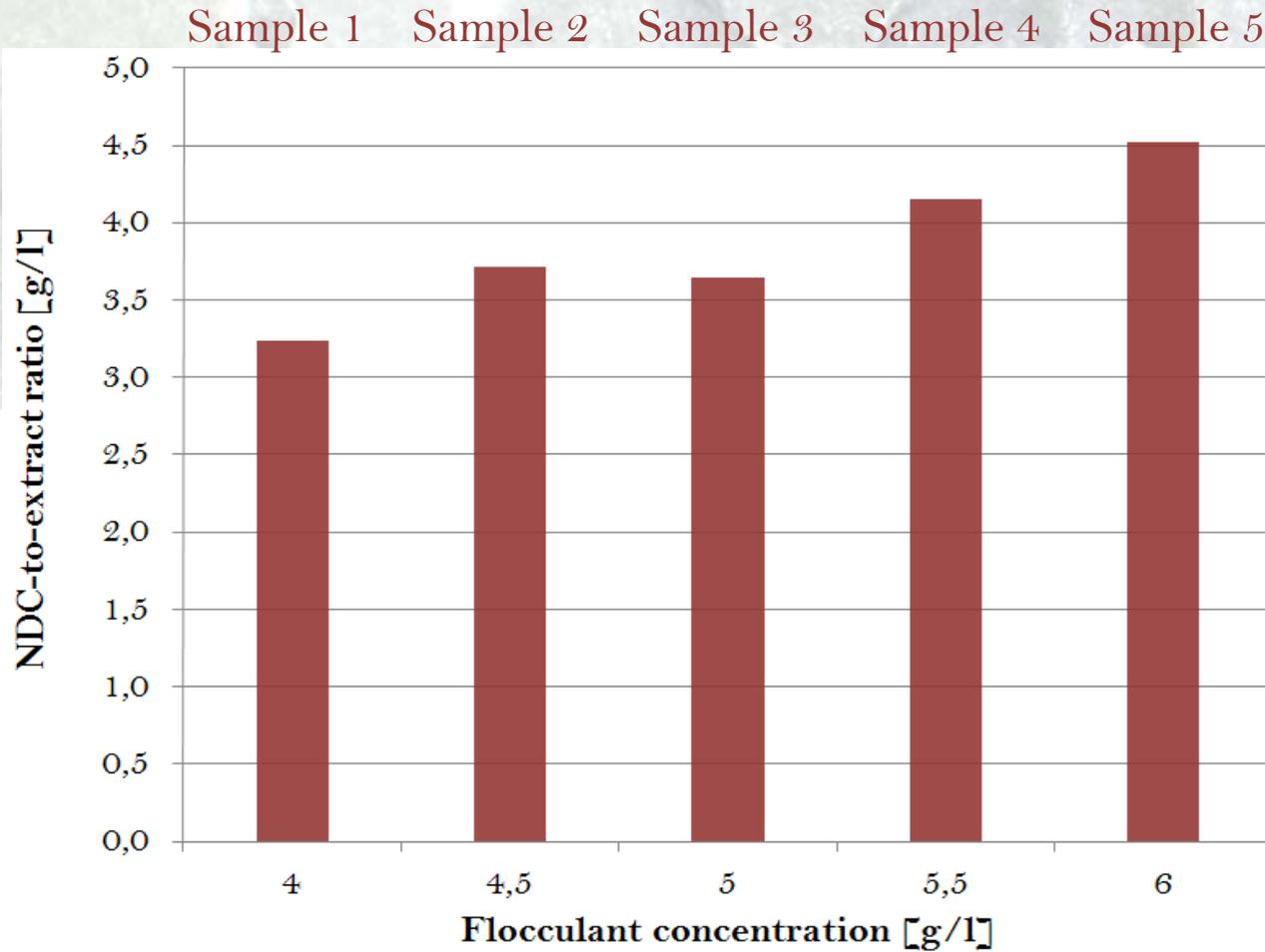
Onion peelings - R1
60 min



Flocculation time: 30 min ✓

Process parameters

4. Influence of the flocculant concentration on the NDC output



Pressing ✓

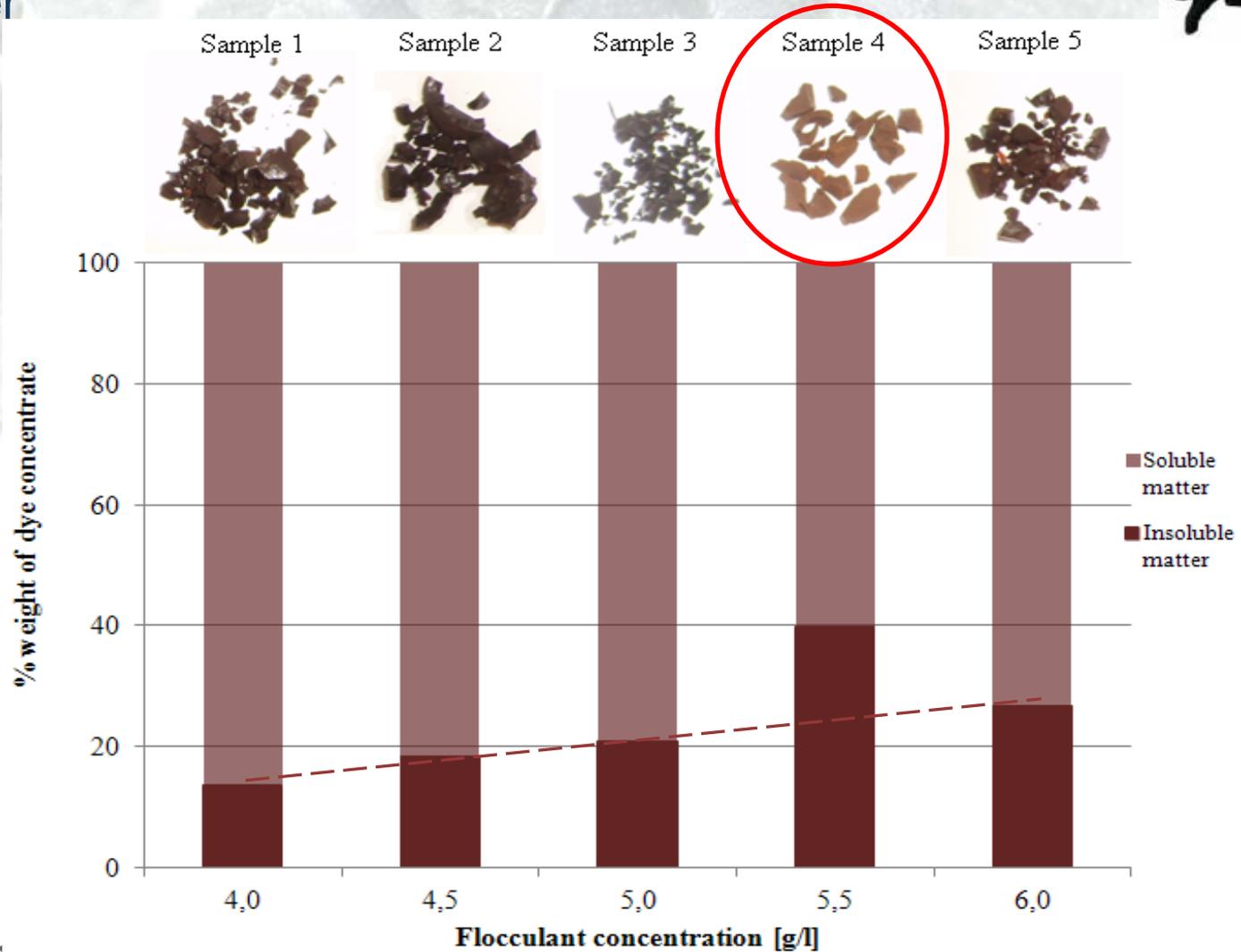
pH adjustment ✓

Flocculation
time: 30 min ✓

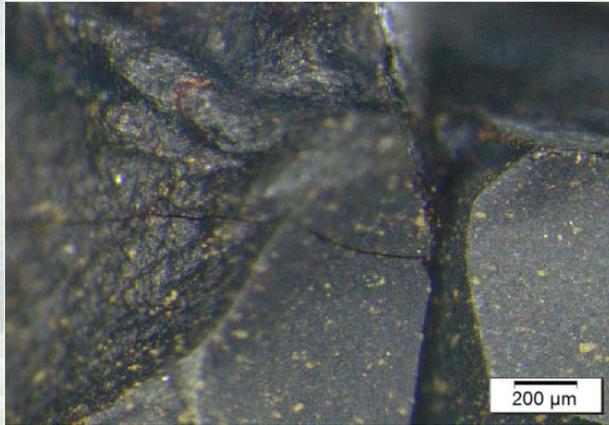
Process parameters

Soluble and insoluble matter

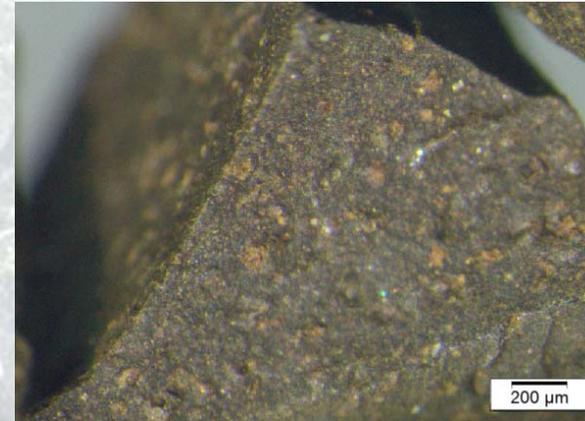
Increasing the flocculant dosage resulted in a higher content of insoluble matter



Process parameters



Sample 3 – R1



Sample 4 – R1



Insoluble matter of sample 4 – R1



Goldenrod - R2



Process development (semi-industrial scale)

Selection criteria

CRITERION 1: Output of NDC

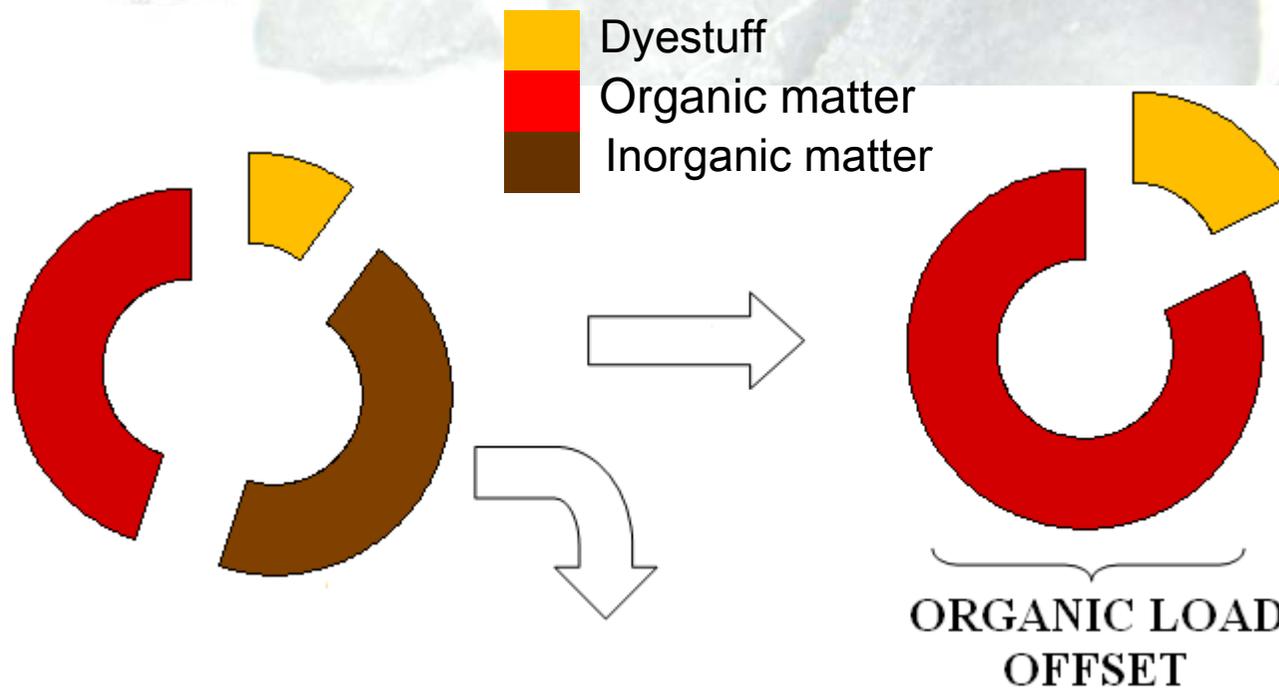
Up to 10 % more extract was obtained by pressing

→ PRESSING DEVICE

CRITERION 2: Standardization of NDC

Removal of inorganic matter: soil and dirt load (~ 3 %)

→ SLUDGE SEDIMENTATION TANK



Stoke's law:

$$v_s = \frac{g \cdot d^2 \cdot (\rho_P - \rho_F)}{18 \cdot \mu_F}$$

$$t = 15,6 \text{ min}$$

Selection criteria

CRITERION 3: Operation time

Separation efficiency:

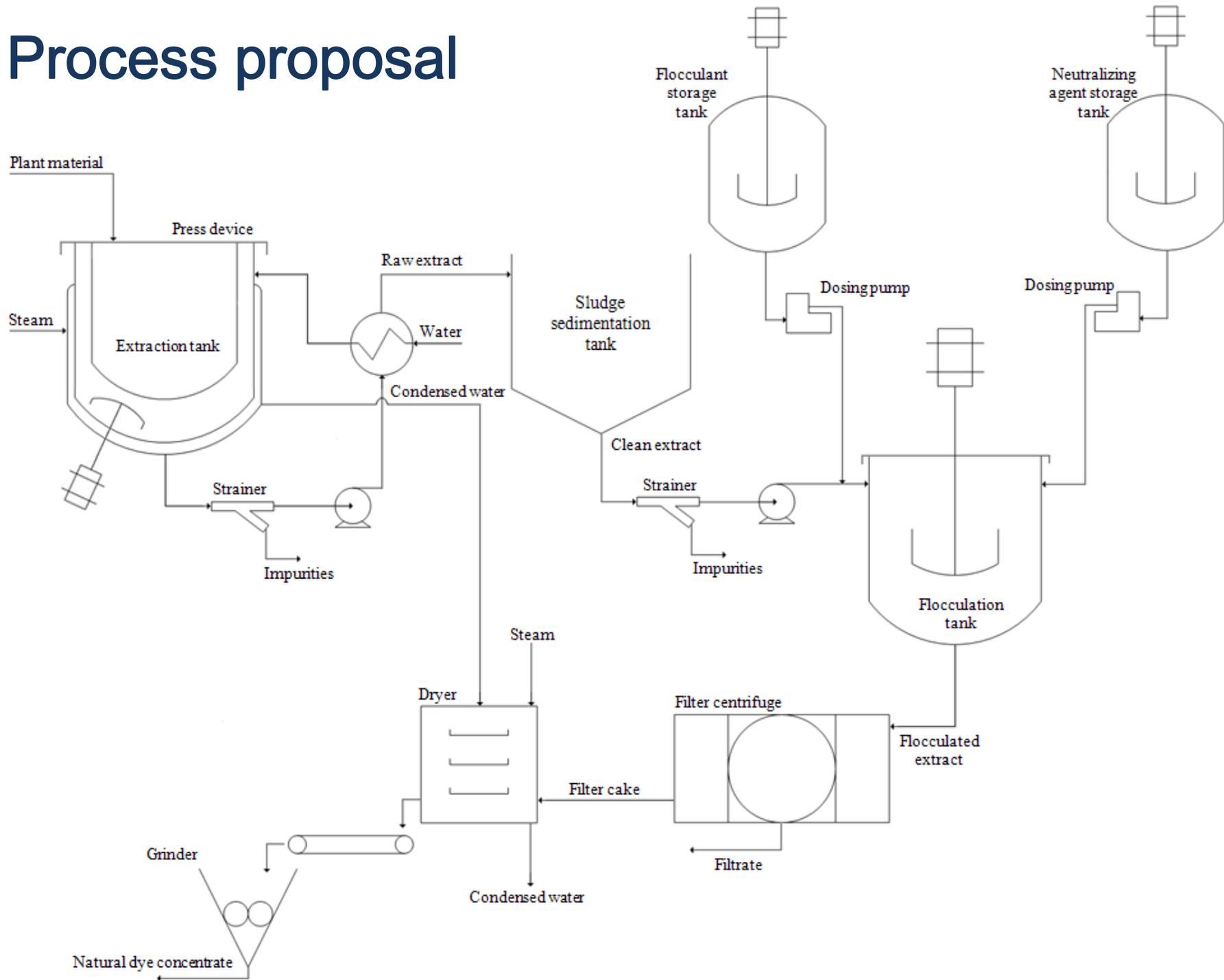
$$\eta = \frac{m_{extract} - m_{solids}}{m_{extract}} \cdot 100$$

→ The predefined step of floc settling could be avoided!!!

	PREDEFINED		PROPOSED
	Settling	Filtration	Centrifugation
Time [h]	> 2	1	0,25
Water content [%]	>98	95	86
η [%]	60	90	96



Process proposal



Mass and Energy Requirement

Extraction tank capacity

Assumptions: 1 Extraction tank
4 Charges/day
250 Working days/year
Output: 1600 kg/year

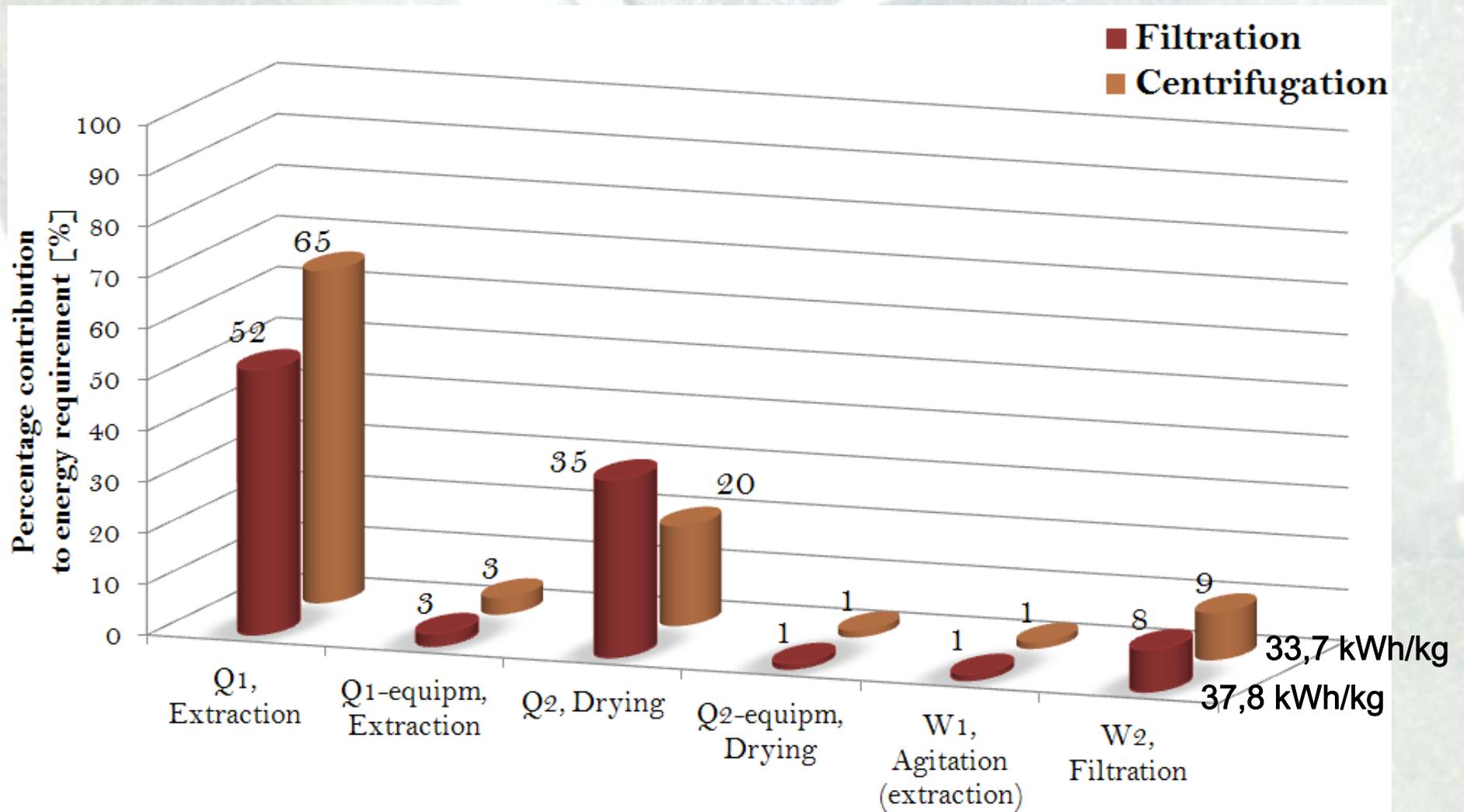
	R1	R2	R3	R4
Plant material	Onion peelings I119	Goldenrod AI42	Walnut shells I117	Ash tree barks I32B
Required capacity [m ³]	0,53	0,60	0,75	1,07
Safety factor %	25	25	25	25
Tank capacity [m ³]	0,67	0,75	0,94	1,34

Mass requirement (1 YEAR)

	R1	R2	R3	R4
Plant material [t]	24,1	27,4	35,3	49,3
Flocculant [t]	1,9	2,1	5,5	3,8
Neutralizing agent [t]	0,8	0,8	2,2	1,5

Mass and Energy Requirement

Energy requirement



Conclusions

GENERAL

Pressing: About 1/3 of the extract which is retained in the inner structure of the plant material after extraction was recovered by pressing → NDC output was increased in ~ 10 %.

pH adjustment: in the range 5,0 to 5,5 for $\text{Al}_2(\text{SO}_4)_3$, the NDC output was 3 times higher than for pH~3. Under the same conditions

Flocculation time: there was no observable floc growth after 30 min.

Flocculant dosage: Increasing NDC output with increasing flocculant dosage was observed, however:

the output of dyeing components (phenolics) was not significantly improved.

the filterability was affected.

due to the sweep flocculation mechanism, an extract with a high load of impurities can erroneously lead to a higher output of NDC.

Conclusions

PROCESS

Three items were determining on the adaptation of the predefined lab-scale extraction steps to the semi-industrial scale:

1. Output: a pressing device was added after the extraction step.
2. Standardization: an sludge sedimentation tank was included between the extraction and the flocculation tanks for removal of impurities.
3. Operation time: it was demonstrated that flocculated extracts could be directly centrifugated after flocculation, and thus, the predefined time-consuming step of floc settling was avoided.

The most energy-consuming steps are extraction and drying → 90 %

filtration was in third place → 5 % of the total required energy.

Centrifugation tests showed that a separation degree of up to 96 % could be achieved → 10 % less of the required energy.

6. Outlook

Following aspects should be considered for further investigation:

Plant material specific conditions are required:

T, t, repetitive extractions → higher NDC output!

An exhaustive investigation of flocculation should allow the standardization of NDC

The influence of transport phenomena as well as the energy losses was neglected due to the lack of sufficient data → need for a detailed study of the energy requirement for extraction and drying operations.

The potential of reutilization of waste heat from:

- extract: for preheating the extraction water
- condensed water: for drying of filter cake, predrying plant material