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Treatment of soy bean process water using hybrid processes

Research Article

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Abstract: The soy bean process water that is a by-product of soy processing technology can be utilized with a hybrid separation system recommended and investigated in this work. The aims of the soy bean water processing are to i.) concentrate the valuable components of the soy process water and ii.) reuse its water content. Two hybrid separation systems are considered and investigated: ultrafiltration followed by nanofiltration and centrifugal separation followed by nanofiltration. These hybrid separation systems are new in the area of their current application. Experiments verify that centrifugal separation is a more appropriate pre-treatment method for the removal of suspended solids and for the preservation of the sucrose content of the soy bean process water than ultrafiltration. Total sucrose can be rejected by nanofiltration membrane forming a sugar-rich retentate that contains other valuable components, too. Both tested hybrid processes result in clear and reusable permeates with low chemical oxygen demand that can be recycled to the production process reducing its water consumption and improving its sustainability. The recommended new hybrid separation system, centrifugal separation followed by nanofiltration, proves to be successful in this area of the biochemical industry.

Keywords: *Soy process water • Hybrid system • Centrifugal separation • Ultrafiltration • Nanofiltration*

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1. Introduction

Soy based food products have been spreading in wide range of food culture due to the rich variety of constituents of soy beans. Among valuable food components are proteins, lipids, carbohydrates, and saponins [1], mapped about a century ago [2], soy beans also contain anti-nutritional components, such as stachyose. Stachyose is not completely digestible by humans so its removal is recommended before use. The stachyose removal process from soy beans, patented by Hungarian researchers [3], provides the production of stachyose-free soy products that otherwise still contain valuable components, like isoflavons. Food products derived from soybeans are studied and the potential role of isoflavon containing soy products in the prevention of cancer is also investigated. Medical use of saponin as a valuable component is also known but its concentration in soybeans is higher than that of edible amount by human [4], however, saponin is out of the focus of our work.

The focus of our research is to study the treatment options of soy process water formed during the aqueous extraction of soy beans, which have been pretreated by a patented process [3]. Due to this production process the soy product does not contain anti-nutritional components, since these are either decomposed during the pretreatment or extracted by water. The foaming of process water verifies the presence of saponin, a non-edible component. The treatment options are as follows: i.) to increase the concentration of valuable components of process water and ii.) to reduce its water content aiming to recycle this water to the production process. In the course of the treatment alternatives environmental benign operations are considered, like membrane operations.

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The membrane processes have been applied in the food industry in the last decades, however, the main application of membranes is in the dairy industry about 40% of the total applications. Considering beverages such as beer or fruit juices, concentrates and waste streams are also treated by membrane processes [5]. Clarification of liquid sugar containing beverages by microfiltration and ultrafiltration are widely applied in industrial scale and it shows also a rapid development [6]. Not only discoloration of clarified liquid juices can be carried out by nanofiltration but salts can be also separated from the juices. In this way carbohydrate syrups can be produced and use as food ingredients for soft drinks, jams or as carbon sources for fermentation [7]. Hybrid processes (membrane filtrations connected with other conventional processes) are favorable in the food and beverage industries. When concentrating juices or other squeezes, fermentation can often take place resulting in a reduced carbohydrate containing juices. Microfiltration and ultrafiltration are capable to filter bacterial germs causing fermentation, and this pretreatment allows concentrating the residual organics (including carbohydrates) by nanofiltration. Not only membrane filtrations (micro-, ultra-, nanofiltration and reverse osmosis) but other different processes (membrane bioreactor [8], UV disinfection [9], evaporation [10]) can be integrated in a hybrid process, too. Two-stage nanofiltration integrated in a hybrid process is also frequent implementation [11]. Hybrid processes have two main advantages: i.) concentrated syrups or juices can be produced, ii.) reuse of process water can be executed.

Protein containing process water from soy protein production was treated by ultrafiltration to enhance protein yield in the product and reduce the waste protein [12]. Soy protein concentrates are produced by combining electro-acidification and ultrafiltration in a hybrid process [13]. The effect of pH on the permeate flux of ultrafiltration was tested on a similar hybrid process. It was found that the decrease of pH resulted in a decrease of permeate flux [14]. Purification and refinement of raw soy sauce using microfiltration is tested in order to stabilize the flavor and color of soy sauce and to stop microbial activity [15]. Others published microfiltration and ultrafiltration of soy sauce lees to recover refined soy sauce [16]. Desalination of soy sauce by nanofiltration was successfully carried out using nanofiltration membranes. Among desalination the recovery of nutritional components of soy sauce could be achieved [17]. Ultrafiltration of aqueous extract of soy flour was investigated from several aspects including studying the constituents of foulant deposit and optimizing the cleaning process of membranes. The foulant deposit was consisted of soy lipids and protein-polysaccharide matrix [18]. After the successful experiments, the modeling of ultrafiltration flux was performed. Modell was able to describe shear stress on the membrane surface that swept away the gel layer formed on the membrane surface [19]. Ultrafiltration was proven to be effective in separating fractions of soy bean flour extracts. For example: soy protein concentrates were produced from extracts of defatted soybean flour using different ultrafiltration modules [20].

According to the literature detailed above, membrane separations were successfully implemented in the treatment of different kinds of waste streams of soy products. Therefore, membrane filtrations seem to be efficient in the treatment of aqueous extract of soy beans, too. In order to recover the valuable nutritional components of the aqueous extract and to recycle the main volume of the applied process water, a hybrid process has been designed to achieving our aims. Treatment of soy extract has been performed by centrifugal separation and ultrafiltration. Both pre-treatment processes are followed by nanofiltration, chosen for the concentration of carbohydrate of soy process water.

2. Experimental procedure

2.1. Process water

During the production of Yaso® soy product, soy beans are washed and pretreated according to the patented process. After the pretreatment soy beans are cooked in salt water then ground and finally packed after cooling down. The cooking water used for aqueous extraction is actually the process water to be treated. The process water samples originate from different charges, so the measured parameters representing the compositions vary in a given range. The variation of composition of soy process water is shown in Table 1.

Concerning the physicochemical properties of process water, it is a yellowish, brownish colored liquid containing visible suspended solids such as denatured proteins. Therefore its viscosity is about three- or four-times higher than that of distilled water at 20°C [21]. The dry matter content varies between 7.3-7.5 wt% that represents the protein and carbohydrate content. The dry matter is nearly twice as high as aqueous soy extract of soy flour [18]. Out of the analyzed carbohydrates, sucrose concentration is significant, about 21 gL^{-1}. The ash content of process water is 1.7-1.8 wt% on wet basis that contains traces of minerals summarized in Table 2, on the other hand salting process during the aqueous extraction contributes to this high value. In

Table 1. Average composition of soy process water.

Parameter	Measured values
Density at 20°C	1.0341 – 1.1171 kg L^{-1}
Viscosity at 20°C	2.92 – 4.26 mPa s
Dry Matter Content	7.3-7.5 wt% (wet basis)
Fructose	0.20 – 0.24 g L^{-1}
Glucose	0.14 – 0.28 g L^{-1}
Sucrose	21.04 – 21.63 g L^{-1}
Protein	0.57 – 0.63 wt% (wet basis)
Ash Content	1.7-1.8 wt% (wet basis)
Chemical Oxygen Demand	70,000 – 85,000 mgO_2 L^{-1}

Table 2. Mineral content of soy process water measured on dry basis (analysed by ICP-MS method).

Elements	Concentration (mg kg^{-1} dry matter)
Ca	140
Fe	292
K	49.9
Mg	38.5
Na	137
P	45.5
S	151
Zn	51.7
Ca	140

order to determine the total organic content chemical oxygen demand (COD) is measured which varies in a wide range of 70.000 - 85.000 mgO_2 L^{-1}. Due to the foaming phenomena caused by the high velocity flow during the preliminary experiments of filtration, it can be concluded that process water may contain saponin, although its concentration has never been measured.

2.2. Pre-treatment processes

Two pre-treatment processes are selected to clarify the soy process water and remove floating and suspended solids before nanofiltration.

2.2.1. Centrifugal separation

The centrifugal separation experiments are carried out on a Rotina 380 (Andreas Hettich GmbH & Co. KG) lab-scale centrifugal separator at 5000 rpm using centrifuging time of 10, 15, 30, 60, 300 minutes. All experiments are performed with unforced damping with the exception of 15 and 30 minute long centrifuging since in these cases medium forced damping are studied as well. The clarified liquor is free of floating denatured proteins and other suspended solids, but the pre-treatment process preserves the carbohydrate content of the process water.

2.2.2. Ultrafiltration

Ultrafiltration is chosen as an alternative pre-treatment method for the removal of floating and suspended solids. Ultrafiltration experiments are carried out on 3DTA (Uwatech Ltd.) lab-scale membrane apparatus. The schematic drawing of the applied apparatus is shown in Fig. 1. Two different UF membranes (JW and GH) are tested at optimal trans-membrane pressures (TMP) (5 and 11 bar, respectively), controlled by a pump. The effective area of the membrane is 150 cm^2. During the experiments, inner recirculation ensures the shear force on the membrane surface to reduce the effect of fouling and gel formation. Permeate is taken continuously away and retentate is recycled back into the feed tank.

To concentrate the valuable dissolved solids of the pre-treated process water, nanofiltration is selected. Since the carbohydrate content of process water is preserved during the pre-treatments, DL type nanofiltration membrane recommended for sugar concentration is applied. Experiments are carried out on a lab-scale Celfa P-28 (Membrantrenntechnik AG) test membrane apparatus. The effective area of the membrane is 28 cm^2. The schematic drawing of the nanofiltration equipment is shown in Fig. 2. The efficiency of nanofiltration is studied at three operating pressures (20, 30 and 35 bar) and two different temperatures (20 and 40°C). Over pressure in the tank is adjusted through a reducer by using inert nitrogen gas. Similarly to the UF module, cross-flow mode and inner circulation with a flow rate of 0.032 dm^3 s^{-1} is maintained during the batch experiments. Avoiding foaming in the feed tank, different anti-foaming agents are added to the pre-treated process water.

2.3. Experimental plan

In case of centrifuging the effect of centrifugal separation time and damping are studied. In case of ultrafiltration membranes temperature, trans-membrane pressure and the type of anti-foaming agent are altered and they are studied. The different combinations of conditions are shown in Table 3.

2.4. Analytical methods

Chemical oxygen demand and sucrose concentration are determined for the evaluation of the efficiency of each step of the hybrid process. Dichromatic chemical oxygen demand is measured according to the Hungarian Standard that fully corresponds to the International Standard ISO 6060:1989. Sucrose analysis is done by Megazyme® enzymatic assay procedure.

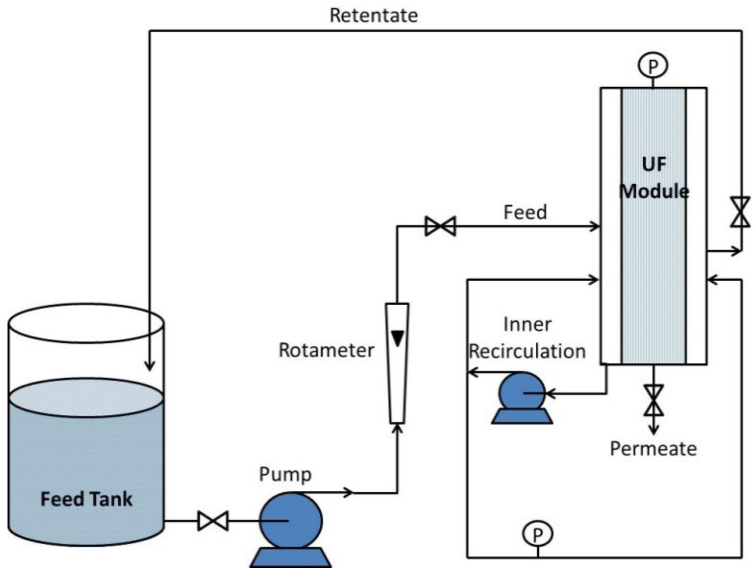

Figure 1. Schematic drawing of the 3DTA test membrane apparatus.

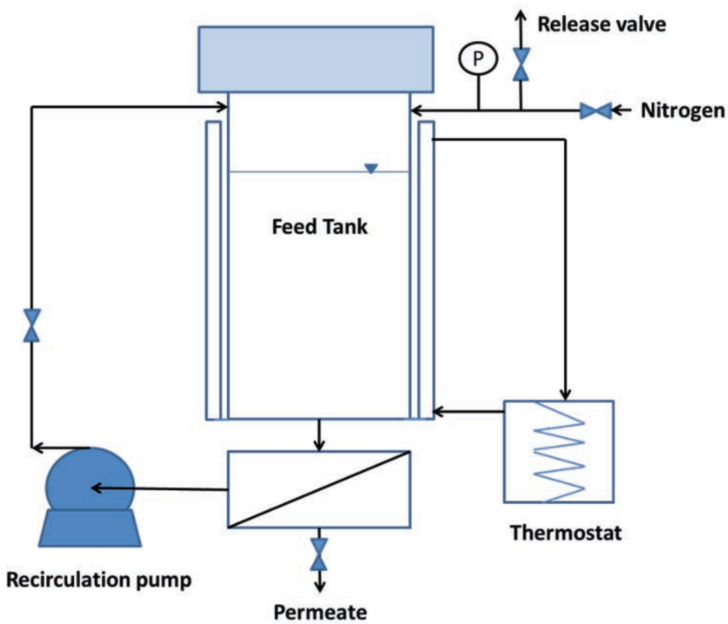

Figure 2. Schematic drawing of Celfa P-28 test membrane apparatus.

2.5. Parameters of evaluation

Specified parameters of permeate flux and rejection are calculated for the evaluation of experiments. Permeate flux is the volumetric rate passing through a membrane area of 1 m^2. The rejection of sucrose and the reduction of COD are calculated according to Eq. 1:

$$R = \left(1 - \frac{c_p}{c_f}\right) \cdot 100\% \quad (1)$$

where R is the rejection (%), c_p and c_f are the sucrose or COD values of the permeate and the feed, respectively.

3. Results and Discussion

3.1. Centrifugal separation

In the centrifugal separation pre-treatment, different time intervals and the effect of damping are studied. Fig. 3 shows the comparison of clarified liquid and residue ratio in the clarification experiments. In case of unforced damping, the amount of residue increases with increasing centrifuging time. Considering the forced damping experiments however, no clear connection between centrifuging time and the amount

Table 3. Summary of experimental plan.

Type of pretreatment	CENTRIFUGAL SEPARATION							ULTRAFILTRATION				
Memb.[1]											JW	GH
MWCO[2]**, kDa**											30	1
Time, min	10	10	15	30	30	30	60	60	300	300	-	-
Damping	no	no	yes	yes	no	no	no	no	no	no	-	-
	NANOFILTRATION											
Temp[3]**,°C**	40	40	20	20	40	40	40	40	40	40	20	20
TPM[4]**, bar**	20	35	30	30	20	35	20	35	20	35	30	30
AFA[5]	no	buthanol	no	no	DC 1510	buthanol	DC 1510	DC 1510	DC 1510	DC 1510	no	no

[1] *Memb.: Types of applied ultrafiltration membranes*
[2] *MWCO: Molecular Weight Cut-off given in kilo-Dalton*
[3] *Temp.: Temperature applied during nanofiltration*
[4] *TMP:Trans-membrane pressure applied during nanofiltration*
[5] *AFA: Type of anti-foaming agent applied during nanofiltration*

of residue can be observed. It can be concluded that the centrifuging time has insignificant effect on the ratio of clarified liqueur and residue. All centrifugal separation experiments result in clear, suspended solid-free liquids therefore centrifuging is a feasible and successful way of pre-treatment. According to Table 1, the composition of the soya process water varies between a minimum and a maximum value. Considering the density range of 1.0341 – 1.1171 kg L^{-1}, the efficiency of centrifugal separation depends first of all on the density difference. The lower density process water assumes a lower concentration of suspended solid content that worsens the efficiency of separation. Increasing the centrifugal separation time can help avoid the decrease in separation efficiency. It seems like the process time of 15 min is enough to obtain good results, due to practical considerations, longer centrifugal separation time (30 min) is recommended.

Sucrose and COD concentrations of clarified liquid are presented in Fig. 4. The dark bars on Fig. 4 represent how much percent of the initial COD value of process water is removed by centrifugal separation. Sugar content of the clarified liquid is measured in case of the forced damping experiments, in order to verify efficiency of sugar preservation by centrifugal separation. Since the sugar content is dissolved in the liquid phase, it can be kept in the liquor, and cannot be removed by a simple sedimentation process. This advantage leads to a relatively small loss of the valuable components in the treated phase, *i.e.*, 99.5% of the sugar remains in the clarified phase. Considering the COD values, ratios of the clarified liquid COD and average COD of the initial soy process water are calculated to observe the effect of centrifuging on COD. As it can be seen in Fig. 4, no tendency in centrifuging time and COD reduction can be observed. COD reduction varies from 1 to 25 percent, depending on the actual initial feed composition originating from different charges of production. The average COD removal by clarification is 12%, and 88% of the COD that containing all the sucrose can be further processed.

According to data analysis, the optimal length of centrifuging time for a future process design should be 30 min. Centrifugal separation experiments resulted in suspended solid free clarified liquids containing the sucrose content of the process water allowing the further concentration by nanofiltration.

3.2. Ultrafiltration

According to the process water analysis, the effluent contains proteins that can be concentrated by ultrafiltration [5,22]. Considering the effective removal of proteins and suspended solids by ultrafiltration [23], ultrafiltration is also chosen as an alternative pre-treatment method of centrifuging. In our research two ultrafiltration membranes with different molecular weight cut-off (MWCO) values are tested, in a two-stage filtration experiments. In the second stage, the retentate of the first stage is re-filtered to enhance the yield of permeate.

The measured fluxes of the UF are shown on Fig. 5. Data clearly show the different characteristics of the two UF membranes: Although lower TMP is applied during ultrafiltration with JW membrane (5 bar) than GH membrane (11 bar), higher permeate flux is recorded due to the higher MWCO. In the case of the GH membrane

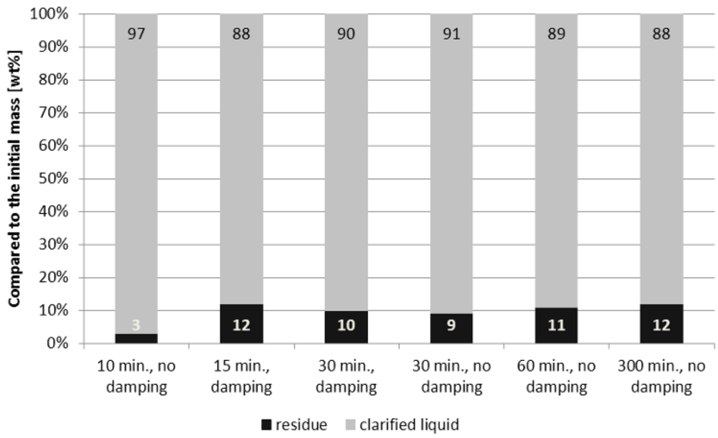

Figure 3. Comparison of clarified liquid and residue ratio in the centrifugal separation experiments.

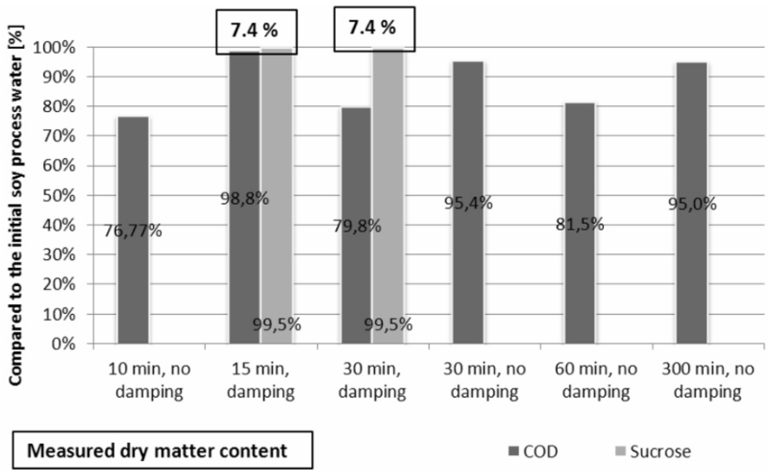

Figure 4. COD and sucrose reduction caused by centrifugal separation.

the lower MWCO resulted in lower flux. Comparing the fluxes of JW stages 1 and 2, a significant difference can be observed between them, that might be caused by the higher viscosity and dry matter content of the feed of stage 2. On the contrary JW fluxes, no difference can be observed between the two stages of GH fluxes allowing the conclusion that the flux of GH is independent on the feed concentration in the cases studied.

The results of the analytical analysis are shown in Fig. 6. Calculating the sucrose and COD rejection, JW membrane has very low sucrose rejection and significant COD retention is detected second stage only. On the contrary the GH membrane shows much higher rejection in both parameters. Rejection of about 80%, and over 99% can be reached in the first and second stage, respectively. From the point of view of nanofiltration pre-treatment, the application of the GH membrane is not favorable due to the observed high sucrose rejection.

Fig. 6 shows that only the JW membrane is suitable to provide a proper pre-treatment for the nanofiltration since efficient removal of suspended solids coupled with the preservation of the original sucrose content. The GH membrane is not applicable for reaching the primary goal (concentrating the sucrose content of process water). Considering the second approach of treatment however, the reduction of COD of the process water is feasible using GH membrane.

3.3. Nanofiltration

Nanofiltration experiments are carried out using a DL (Sterlitech Co.) thin-film type membrane in order to concentrate the sucrose and valuable components of pre-treated soy process water. Due to the cross-flow filtration and the inner circulation of the retentate intensive foaming occurred in the feed tank. To avoid foaming two different anti-foaming agents are applied, butanol and DC 1510 silica oil are added in 0.1 V/V% to the feed of nanofiltration. The measured fluxes of nanofiltration versus yield are shown in Figs. 7-9. Yield is defined as a ratio of permeate volume and feed volume.

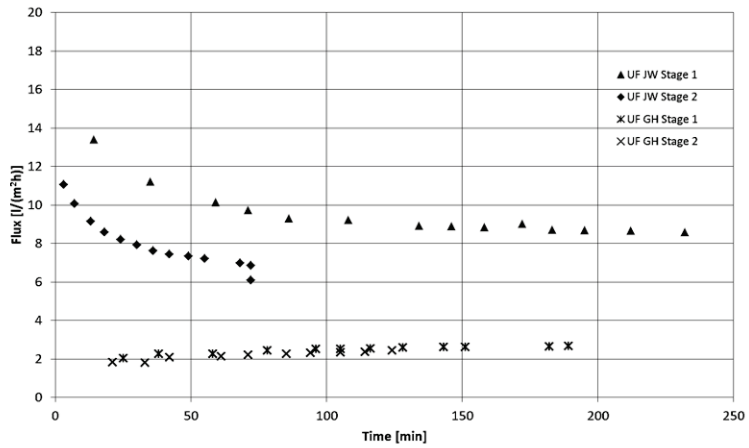

Figure 5. Permeate fluxes of ultrafiltration pre-treatment.

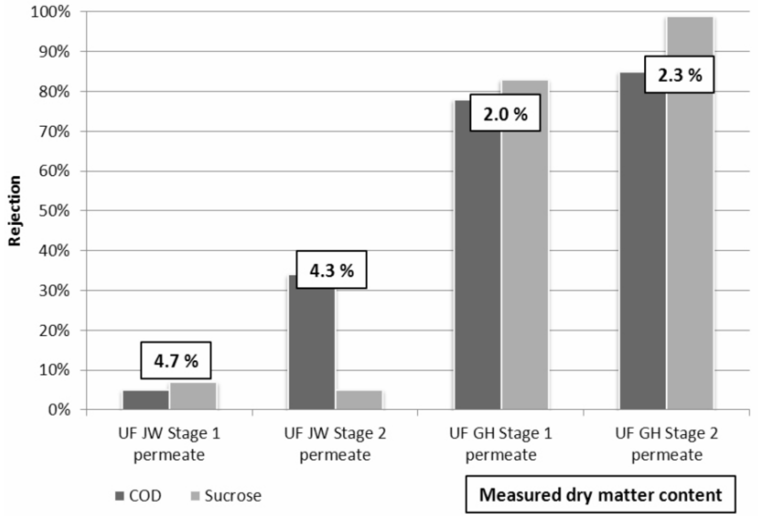

Figure 6. Chemical oxygen demand and sucrose rejection in cases of ultrafiltration.

Considering the experiments at 20 bar and 40°C (Fig. 7), the increasing centrifuging time results in a lower concentration of suspended solids in clarified liquid. When a clearer liquid is fed into the apparatus the effect of membrane fouling is reduced and the permeate flux is increased. Comparing the experiments without AFA (♦) and with AFA (×,+,*), it can be seen that addition of AFA decreases the flux.

Fig. 8 shows the comparison of nanofiltration fluxes after pre-treatment by ultrafiltration and centrifuging. Values of nanofiltration fluxes pre-treated by ultrafiltration are approximately the same independently of the types of the UF membrane applied. When comparing the nanofiltration fluxes pre-treated by centrifugal separation with the UF pre-treatment, lower fluxes can be reached that is in correlation with the measured dry matter content. The UF pre-treatment results in 50% higher flux than centrifuging pre-treatment. In the case of a 15 minute long centrifugal separation, the fraction of the residue is higher (Fig. 3) than in case of a 30 minute long centrifuging which explains the higher nanofiltration flux measured during the experiment of the 15 minute centrifugal separation pre-treatment.

Similarly to the observation at 20 bar and 40°C, the increasing centrifuging time results in higher flux at 35 bar and 40°C (Fig. 9) when using the same AFA. Butanol is also tested as an AFA and the results suggest that higher flux could be reached by using butanol instead of DC-1510, however butanol can be detected in the permeate. This theory is verified by the COD measurements of the permeate (Fig. 10).

Sucrose concentration and COD of permeates are analyzed in order to determine the efficiency of nanofiltration. The concentration of sucrose in the permeate is under the detection limit, so it can be regarded as zero. As a conclusion a nanofiltration

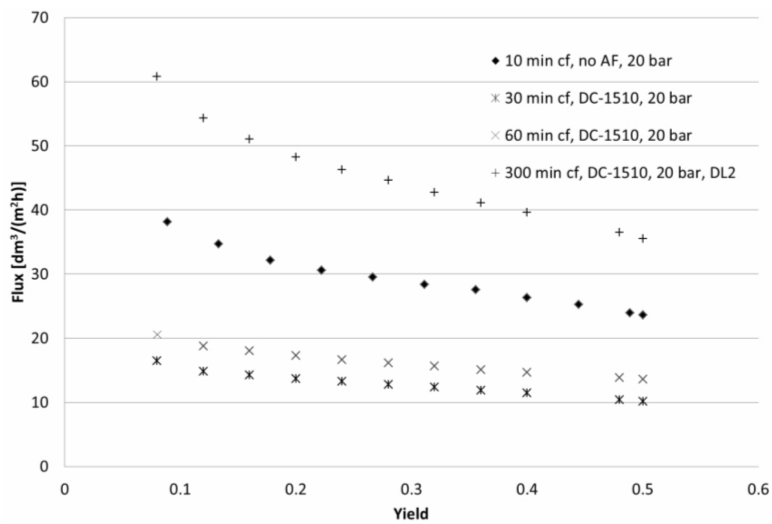

Figure 7. Measured fluxes of nanofiltration at 20 bar, at 40°C.

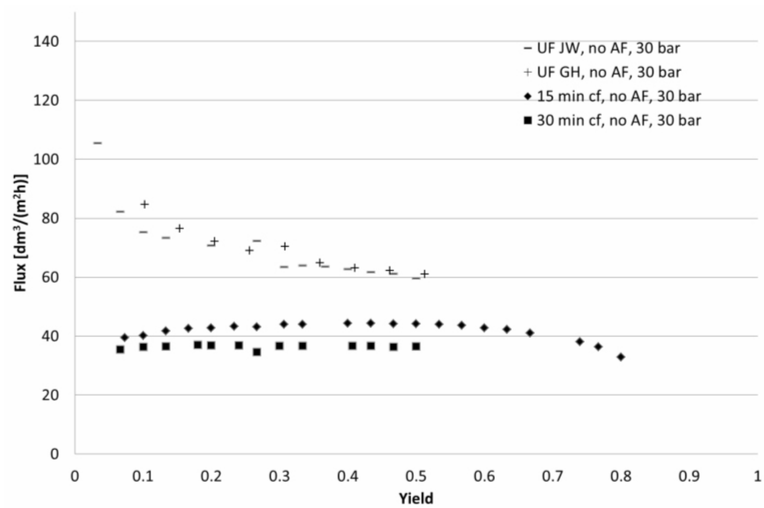

Figure 8. Measured fluxes of nanofiltration at 30 bar at 20°C.

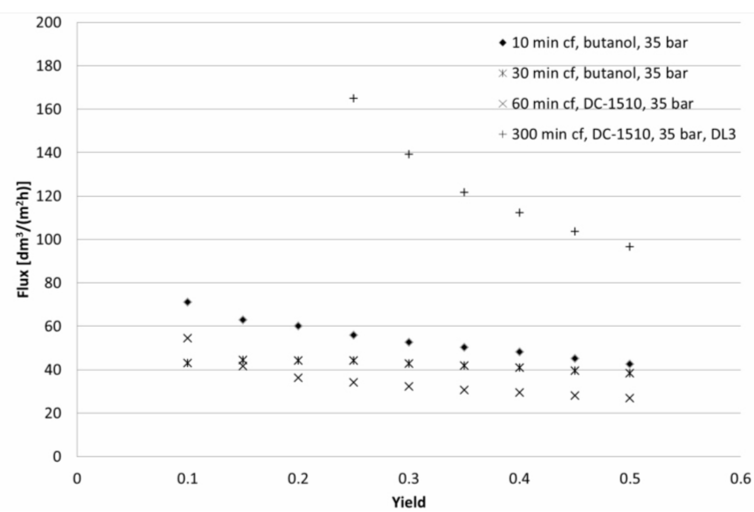

Figure 9. Measured fluxes of nanofiltration at 35 bar, at 40°C.

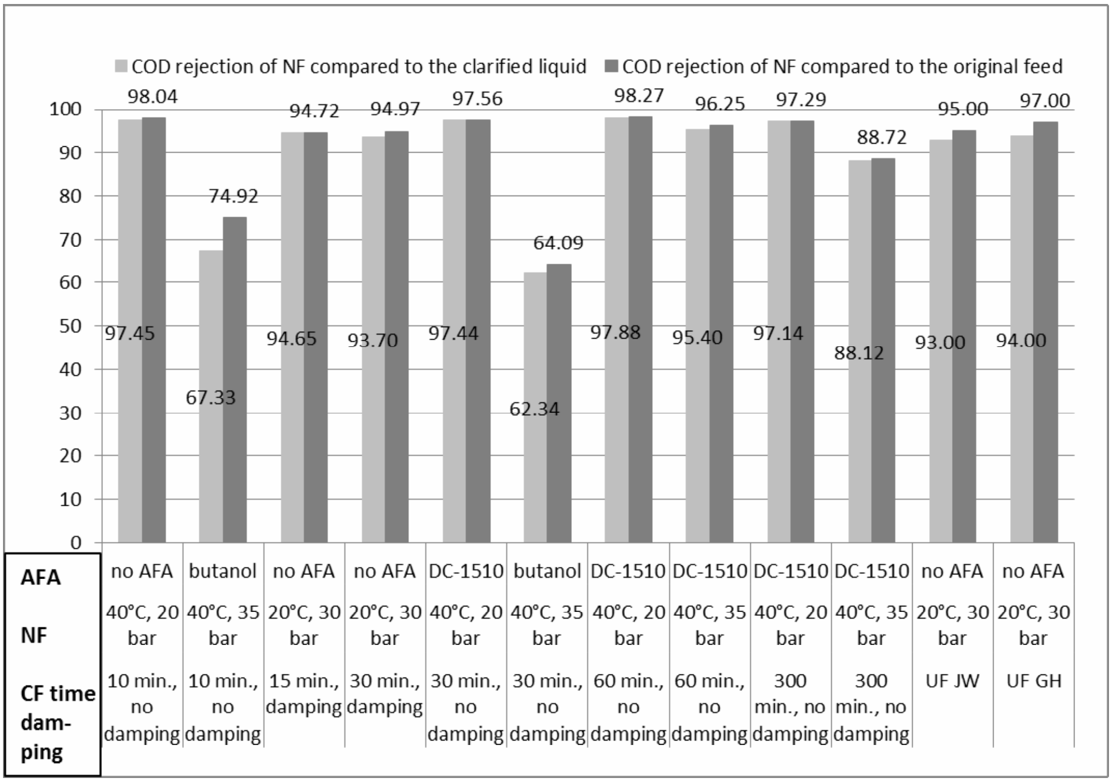

Figure 10. COD rejection in cases of nanofiltration (AFA: Type of anti-foaming agent applied during nanofiltration; NF: the conditions of nanofiltration temperature and trans-membrane pressure respectively; CF-time: the duration of centrifugal separation; damping: forced damping of no damping is indicated; UF JW: pre-treatment by ultrafiltration investigated by JW type membrane; UF GH: pre-treatment by ultrafiltration investigated by GH type membrane).

membrane can reject all the sucrose therefore the rejection of sucrose is as high as 100% in all cases, and the primary goal can be achieved successfully.

Fig. 10 shows a complex evaluation of nanofiltration and the complete hybrid separation process from the point of view of COD removal. The lights bars represent the percentage of COD removal only by nanofiltration calculated according to Eq. 1, *i.e.*, c_f is the COD value of the pre-treated liquor, which is the feed of nanofiltration, and c_p is the COD value of the permeate of nanofiltration. The dark bars stand for the percentage of COD removal by the whole hybrid process calculated according to Eq. 1, too, but c_f is the COD value of the initial/original process water and and c_p is the same, *i.e.*, the COD value of the permeate of nanofiltration. The differences between the light and dark bars vary in the range of 0.5 – 8% which stands for the efficiency of pre-treatment method, namely the centrifugal separation or the ultrafiltration.

The COD rejections are always over 88% except when butanol is added (see groups number 2 and 6) The addition of butanol largely increases the flux and the COD of the permeate, resulting in calculated rejection of about 65%. Using DC 1510 AFA, COD rejection remains high compared to experiments without AFA, so the membrane successfully rejects the anti-foaming agent, while the foaming phenomena during the filtration process can be eliminated.

Considering Entry 2 in Fig. 10, the temperature and trans-membrane pressure of nanofiltration has no significant influence on the rejection of COD, the significance of butanol added as an anti-foaming agent is much more conspicuous, since the application of butanol decreases the COD rejection drastically.

4. Conclusions

This study focuses on the utilization of soy process water formed during the aqueous extraction of soy beans. Treatment alternatives of soy process water with environmental benign processes are provided. All experiments are carried out in a hybrid system: clarification or ultrafiltration followed by nanofiltration.

Achieving the aim of increasing the concentration of valuable components, in accordance with the expectation, the longest centrifuging time proves to be the most efficient pre-treatment method since it results in a highly clarified liquid with low dry matter content and the highest nanofiltration flux following these pre-treatment options. Considering the sucrose concentration, it can be concluded that centrifugal separation is the more appropriate method for the removal of floating and suspended solids and for the preservation of sucrose than ultrafiltration. Total sucrose can be rejected by the nanofiltration membrane. An optional use of sugar-rich concentrate can be as an ingredient in the soft drink industry. COD rejections are over 90%, however, the type of anti-foaming agent influences its value. Out of the anti-foaming agents, DC-1510 proves to be a proper choice reducing the foaming phenomena and guaranteeing the high COD rejection.

Approaching the aim of recycling the process water to the production process, experimental results indicate that all the tested hybrid processes resulted in clear permeates with low COD values, so they can be reused as extracting agents in the soy bean extraction technology. This work verifies the successful applicability of centrifugal pre-treatment followed by nanofiltration hybrid system for treatment of aqueous soy extract. Finally, it can be concluded that this new hybrid process of the side product/waste of a new biotechnological process can offer to get the valuable biological compounds in concentrated form and the water that can be reused. Although the industrial application of the recommended hybrid technology is about to be applied, no data are available on cycles of reused water. Although the effluent of nanofiltration step needs to be adjusted by fresh water reaching the same rate of extraction, the total water consumption of the plant can be remarkably reduced. The application of our new hybrid separation process is needed by the industry to facilitate the reuse of the compounds mentioned in this study and to realize more sustainable processes.

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Nomenclature

COD: Chemical Oxygen Demand [mg O_2L^{-1}]
UF: Ultrafiltration
NF: Nanofiltration
Memb: Membrane
MWCO: Molecular Weight Cut-off [kDa]
Temp: Temperature [°C]
TMP: Trans-membrane pressure [bar=10^5Pa]
AFA: Anti-foaming agent
Min: minute
R: rejection [%]
c_p: concentration of given component in the permeate [mg L^{-1}]
c_f: concentration of given component in the permeate [mg L^{-1}]

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