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Adjustment of the wastewater matrix for optimization of membrane systems applied for water reuse in breweries

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Abstract

The objective of this study was to optimize membrane systems applied for wastewater reuse in breweries in consideration of economic efficiency and safety of operation by defining and adjusting the wastewater matrix. With the help of extensive quantitative and qualitative analyses of relevant process water flows from several breweries with similar product ranges and different wastewater treatment concepts, the following process water flows were identified as critical regarding to the membrane filtration: Polyvinylpolypyrrolidone regeneration lye, cleaning and rinsing lyes from cleaning in place systems as well as effluents from the bottle washing machine and conveyor belts. Their negative effects on the filtration performance of low pressure membranes were shown in laboratory-scale tests. In addition, treatment concepts of recycling the alkaline process water flows (Polyvinylpolypyrrolidone regeneration lye, cleaning in places lyes) were developed and investigated regarding to technical, ecological and economic aspects. As secondary objectives, the reduction of inert organic substances, the recovery of valuables (polyphenols, sodium hydroxide solution) and the substitution of critical substances (conveyor belts) were pursued.

Key words: brewery wastewater; membrane filtration; polyphenols; water reuse; fouling.

Nomenclature

BOD	Biological oxygen demand
BWW	Brewery wastewater
CIP	Cleaning in place
COD	Chemical oxygen demand
DOC	Dissolved organic carbon
LC-OCD	Liquid chromatography - organic carbon detection
MWCO	Molecular weight cut off
PVPP	Polyvinylpolypyrrolidone
SB	Sold beer
SEC	Size exclusion chromatography
TMP	Transmembrane pressure
TNb	Total nitrogen bound
TOC	Total organic carbon

1 Introduction

Not only against the background of rising water and wastewater costs, but also due to the limited supply and quality of fresh water, the stewardship of the resource "water" is becoming increasingly important for the brewing industry. In addition to a cleverly chosen water management system, the treatment of the total effluent (end-of-pipe) and the treatment of process water flows are already the focus of several research projects and technical applications [1,2,3,4,5,6,7].

Thereby, the motivations for wastewater reuse may be different. One good reason for it is to reduce production costs or to keep them on a constant level. The sharpening of conditions for direct and indirect discharge of wastewater can be another one. Furthermore, if fresh water supply is limited, a carefully selected reuse with an advanced wastewater treatment can lead to additional production capacity and therefore economic benefits.

Especially in countries with comparatively high water costs, wastewater recycling can have monetary advantages as it is proven by the following model calculation. The specific water consumption of a modern brewery (without water recycling plant) amounts to 4 L/L sold beer (SB) depending on the individual production conditions (Fig. 1); the resulting wastewater amount averages 2.2 L/L SB although the range of variations is very large [8]. Therefore, a medium-scale brewery with an annual beer output of 1 million L requires 400,000 m³/year fresh water and produces 220,000 m³/year wastewater. Calculating with a fresh water price of 2.20 €/m³ and a wastewater price of 2.50 €/m³ for Berlin region, even modern breweries have to bear annual water costs of 1.4 million €. Considering that up to 30% of fresh water used for processes without product contact (e.g. external rinsing and cleaning processes, cooling towers or boiler feeding) can be substituted by recycled water [9], significant cost savings in breweries are possible by wastewater recycling.

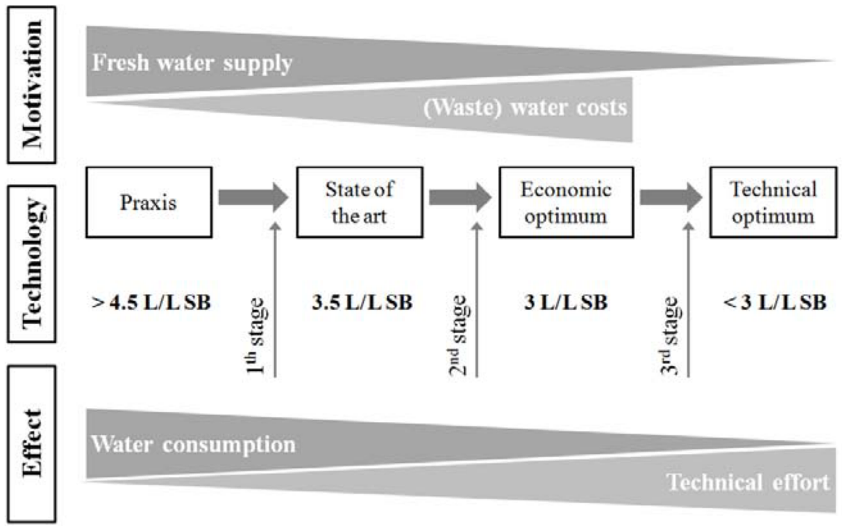

Fig. 1: Developement stages of water management in breweries [10]

On the other hand, this is accompanied by the costs for wastewater treatment which are a function of plant structure and scale. The operating costs (exclusive of personnel) of a membrane bioreactor followed by nanofiltration can be calculated with 1.35 €/m³ wastewater [11]. It can be followed that the annual treatment costs amount to 337,000 € related to the model brewery mentioned in the previous paragraph. Assuming investment costs of 200,000 to 300,000 € [2], the wastewater recycling plant would have been amortized in respectively two or three years in consideration of the reduced water costs.

For these reasons, the advanced treatment of biologically cleaned brewery wastewater by membrane processes to close internal water cycles and to reuse the recycled wastewater for processes without product contact was the objective of several research projects [1,2]. Former investigations related to the recycling of process water flows are focussed on the reduction of fresh water consumption for bottle washing with preferably low technical effort [6], because of the huge amounts of needed fresh water and the high potential for water reuse. Furthermore, investigations were carried out to treat the regeneration and rinsing waters from weakly acidic cation exchangers [12] as well as to recycle the Polyvinylpolypyrrolidone (PVPP) regeneration lye [7].

In the last decade, some worldwide operating breweries have already applied large-scale end-of-pipe plants for wastewater recycling [4,5] (Table 1). Central components are two-staged membrane systems, which can be operated stably, but there is still potential for improvements with a positive economic impact. This is resulting from the low scientific knowledge about the effects of brewery-specific wastewater ingredients on the filtration performance of membrane processes. Furthermore, there is a broad variety of the wastewater composition within a brewery (Table 2), but also between different breweries so that membrane technology has to meet different technical demands.

During the membrane filtration, several ingredients of brewery wastewater can cause blockings and cover layers (fouling, scaling). According to Cornel [13], the following substances are regarded as potential impurities: Long-stranded, filamentous, highly abrasive or sharp-edged ingredients (e.g. kieselguhr), scaling-causing ingredients (e.g. calcium, phosphate, sulphate, barium, strontium and silicates), fouling-causing ingredients (e.g. persistent colloids, defoamers, surfactants) as well as wastewater flows with high product or co-product load (e.g. high yeast concentration, out-pressed liquid from spent grain, water flows with concentrated protein, polyphenol and polysaccharide fractions from filtration processes). To keep the permeate flux on an economically acceptable level, frequent and partly extensive membrane cleanings which causes a significant chemical and energy consumption have to be conducted. Moreover, every cleaning process reduces the durability of membranes and the spent cleaning solutions have to be disposed and/or treated. Due to the complexity of the wastewater matrix (Fig. 2), it is difficult to assess which substances cause blockings on and/or within membranes and which do not decrease the filtration performance.

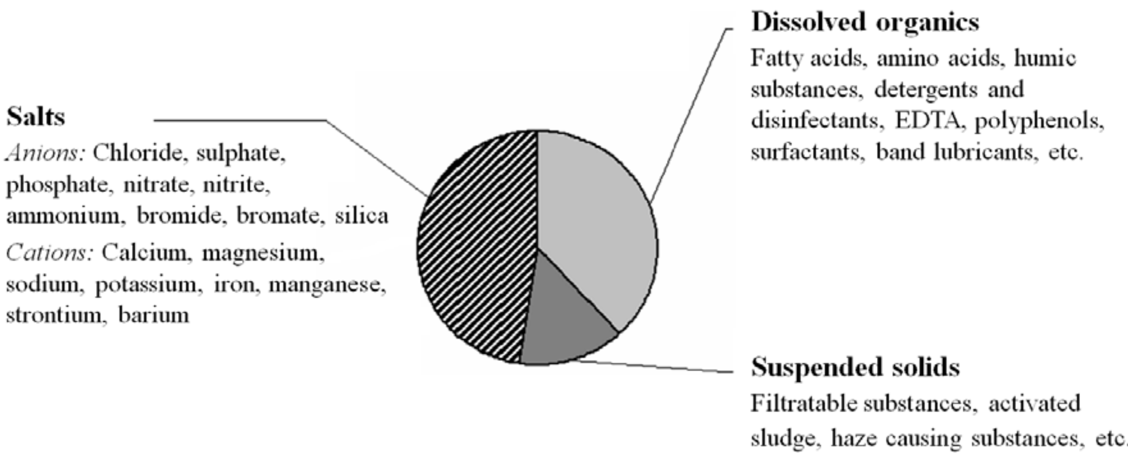

Fig. 2: Composition of brewery wastewater after biological treatment

The innovative approach of this study was to optimize membrane systems applied for wastewater recycling in breweries in consideration of economic efficiency and safety of operation by defining and adjusting the wastewater matrix. These investigations have been carried out for the first time. In past and present, research projects and technical applications mainly focus on the optimization of membrane systems by adjusting operating conditions and module configurations (end-of-pipe approach). In this study, a precautionary approach is pursued for a sustainable, safe and economical application of water reuse technologies. Critical wastewater ingredients and process water flows will be substituted or treated separately *before* feeding into the total wastewater stream and *before* they can have a negative effect on the filtration performance of membranes. This approach provides completely new opportunities to optimize membrane systems applied for wastewater reuse and can also be easily transferred to other sectors of industries.

Furthermore, the characterization of brewery wastewater is also of high interest, especially against the background that comprehensive analyses date back several years [14,15] and meanwhile, the wastewater composition is expected to be modified due to changed production, product ranges and/or wastewater treatment concepts.

2 Materials and methods

2.1 Sampling

To identify process water flows critical for membrane filtration, seven breweries with similar product ranges and different wastewater treatment plants were selected. Their annual beer output and the output distribution are shown in Fig. 3. Samples of process water flows with high organic and/or salt loads were taken for quantitative and qualitative analysis. Therefore, alkaline cleaning and regeneration waters from the sections brew house, filtration cellar, fermenting cellar and filling were sampled. The grab sampling of the breweries was conducted within the time period 02/2010 to 11/2011. The amount of samples was based upon the process-specific availability on the respective day of sampling. Table 3 gives an overview on the sampling of process water flows from the breweries A to F.

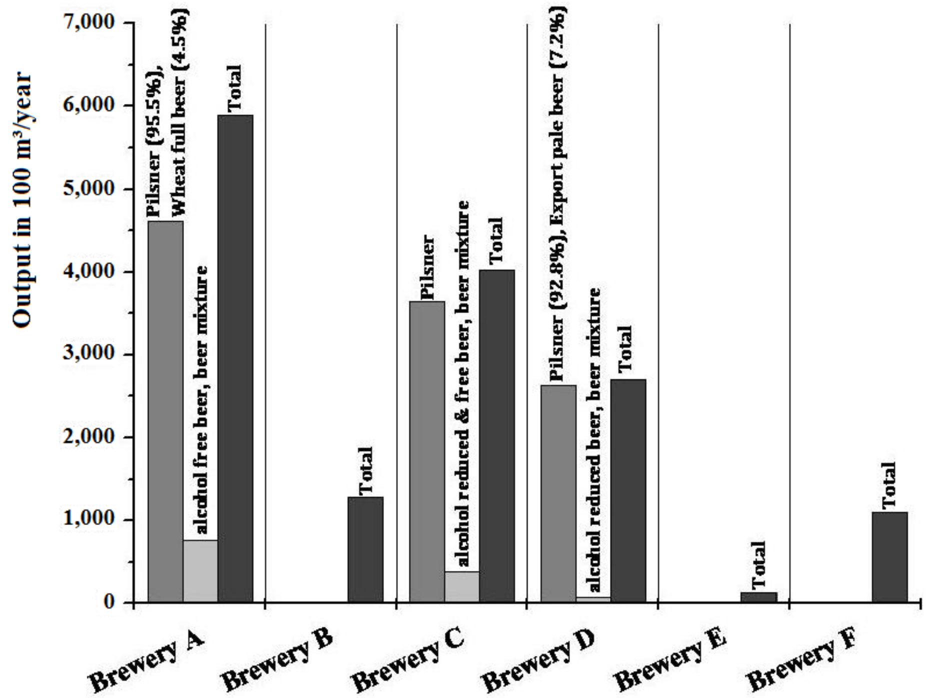

Fig. 3: Annual total beer output and output distribution of the sampled breweries A-F in 2011 according to INSIDE [16]

2.2 Analytical methods

The following parameters were analysed to characterize process water flows from breweries and/or feed, permeate and concentrate samples during laboratory-scale experiments: pH, conductivity, chemical oxygen demand (COD), biological oxygen demand (BOD), total and dissolved organic carbon (TOC and DOC), total nitrogen bound (TNb), particle size, polyphenols as well as fractions of dissolved organic carbon.

Conductivity and pH were measured by using Knick Portamess 913 Cond (Berlin, Germany) and WTW microprocessor pH meter pH 537 (Weinheim, Germany), respectively. COD measurements were achieved by using cuvette tests LCK 314, LCK 514 and LCK 014, a thermostat LT 200 and a UV-VIS spectrophotometer DR 5000 (all HACH LANGE, Düsseldorf, Germany) according to German Standard Methods [17]. BOD was determined by pressure measurements using a WTW Oxitop Control 12 System (Weinheim, Germany). Analysis of TOC, DOC and TNb were performed using Analytik Jena TOC analyzer multi N/C 3100 (Jena, Germany) where DOC samples were pre-treated by filtration through Whatman 0.45 µm membrane filters (Kent, UK). Determination of the particle size distribution was achieved by HORIBA laser scattering analyzer LA-950 (Kyoto, Japan) with a measuring range from 10 nm to 3 mm.

2.2.1 Quantification of polyphenols by HPLC-DAD

Gallic acid ≥ 97.5%, protocatechuic acid = 97%, catechin = 98%, vanillic acid = 98%, p-cumaric acid = 98% and ferulic acid = 98% were purchased from Roth (Karlsruhe, Germany), Merck (Whitehouse Station, New Jersey, United States) and Sigma-Aldrich (St. Louis, Missouri, United States), respectively. Quantification of the selected polyphenols in the PVPP regeneration lye was performed using an Agilent 1200 HPLC system with a VWD detector (Santa Clara, California, United States) monitoring at 280 nm. Separation and quantification was achieved by a gradient elution applying a flow rate of 1 ml/min and an injection volume of 10 µL using a Shimadzu Shim-pack ODS-2 column (Kyoto, Japan, 250 x 4.6 mm, 5 µm).

A mixture of 500:1 deionised water/acetic acid (v/v) and pure methanol (v) were used as solvent A and B, respectively. A four-step linear gradient analysis adapted from Whittle et al. [18] for a total run time of 90 min was used as follows: Starting from 100% solvent A and 0% solvent B, increasing to 30% solvent B over 60 min, increasing to 80% solvent B over 20 min, decreasing to 0% solvent B over 5 min, and finally isocratic for 5 min. The system was equilibrated between runs for 20 min using the starting mobile phase composition. The samples were prepared by acidification with hydrochloric acid (HCl) whereas the pH value was reduced from average 12.5 to 2.5 followed by filtration with Whatman 0.45 µm membrane filters to separate precipitations. Concentrate samples were additionally diluted 1 in 20. Each sample was analyzed at least twice.

2.2.2 Fractionation of dissolved organic carbon by LC-OCD

Based on the molecular size, dissolved organic carbon is separated by LC-OCD into the following fractions: biopolymers (polysaccharides, proteins and other organic colloids such as components of bacterial cell walls), humic substances, building blocks (substances similar to humic substances, but with a smaller molecular size), organic acids as well as neutral and amiphiphile substances (e.g. lyes, surfactants). Fractionation was achieved by using a DOC-LABOR Dr. Huber LC-OCD system (Karlsruhe, Germany) equipped with a Gräntzel thin film reactor, a KNAUER WellChrom fixed wave detector K-200 (Berlin, Germany) and a Siemens non-dispersive infrared detector ULTRAMAT 6 (Berlin, Germany). Hence, an elution flow rate of 1 mL/min was applied and a volume of 1 mL was injected with an Alltech-Grom SEC separating column (Rottenburg-Hailfingen, Germany, 250 x 20 mm, 20 to 40 µm) including the Tosoh Bioscience separating resin TOYOPEARL HW 50S (Tokio, Japan). The mobile phase is pre-cleaned by UV-oxidation prior to LC-OCD analysis. LC-OCD data were analyzed using DOC-LABOR Dr. Huber FIFFIKUS software. The samples were prepared by dilution according to the measurement range of 2 to 5 mg DOC/L and analyzed once with respect to a total run time of 180 min each.

2.3 Membrane test plant

A SIMA-tec (41366 Schwalmtal, Germany) laboratory membrane test plant LSta80 represented in Fig. 4 was applied in this study. The plant is suited for investigations in the field of microfiltration, ultrafiltration, nanofiltration and reverse osmosis up to an operating pressure of 80 bar and a feed flow rate of 90 L/h. Organic flat sheet membranes with an active membrane area of 85.6 cm^2 were tested.

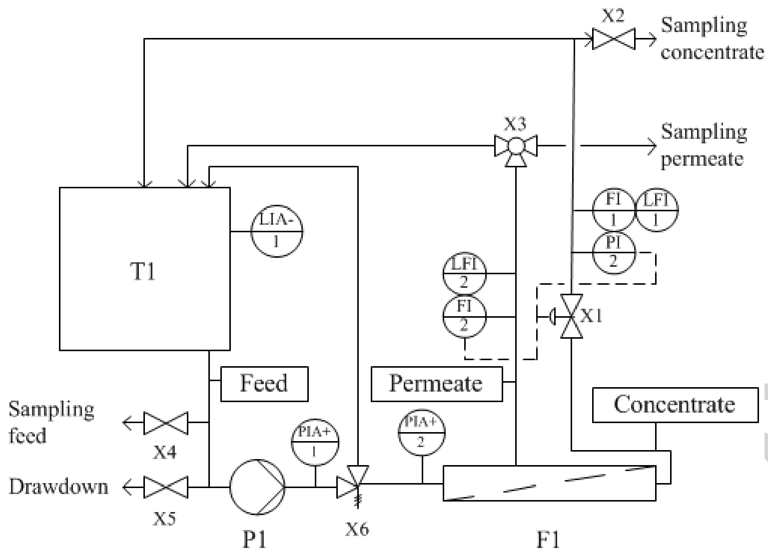

Fig. 4: Process flow sheet of SIMA-tec laboratory membrane test plant LSta80

The double walled feed tank (T1) with a capacity of 7.5 L was maintained at 22±1°C by means of a temperature controller. The plant also consists of a high pressure pump with adjustable speed motor (P1), a regulated pressure control valve (X1) and a cross flow test cell (F1). Manual valves (X2 to X4) allow to take samples of feed, concentrate and permeate stream during operation.

For measurement of the transmembrane pressure (TMP), two pressure sensors (0 to 100 bar) are installed before and after the membrane. A magnetic-inductive flow meter with a digital indicator downstream the pressure release was used to control the cross-flow velocity. Dry-running protection was achieved by a level sensor in the feed tank which actuated a switchable socket to facilitate the re-filling of test media. Two controllers enabled the operation under constant pressure or constant permeate flow. The measured values were recorded continuously at a data rate of 3 sec.

2.4 Membrane materials

Ultrafiltration was achieved by using Microdyn-Nadir (Wuppertal, Germany) flat hydrophilic polyethersulphone membrane type PM UH050; nominal MWCO 50 kDa; water flux > 250 L/(m^2·h) at 3 bar, 20°C, stirred cell (700 Rev/min). Nanofiltration was investigated by using Koch Membrane Systems (Stafford, UK) flat polysulphone membrane type MPS-34; pH-stable from 0 to 14; temperature-stable < 70°C; nominal MWCO 200 Da; rejection glucose/sucrose 95/97% and sodium chloride (NaCl) 35% at 30 bar, 30°C, feed solution 3% glucose/3% sucrose or 5% NaCl. A new membrane was used for each experiment and pre-treated for surface hydrophilization as follows: After solving in 25% isopropanol for 1 hour, the membrane was rinsed with and stored in deionized water for 1 hour. Then, the deionized water was renewed in order to keep the membrane for minimum 3 more hours.

2.5 Experimental procedure

2.5.1 Influence of process water flows on ultrafiltration

The critical process water flows were added in realistic concentrations to biologically treated total effluent from brewery D which was chosen as model wastewater based on its low pollution and good filtration characteristics (Table 4). The mixture was treated by ultrafiltration using the laboratory membrane test plant to demonstrate the negative effect of the critical process water flows on the performance of low pressure membranes. Hereby, the filtration of biologically treated wastewater from brewery D (without addition of a process water flow) was used as reference. The alkaline process water flows were prepared by acidification with HCl whereas the pH value was reduced from average 13.2 to 7. The process water flows were not pre-treated biologically, because the results demonstrated that all of them are poor to extremely poor biodegradable and contain a high amount of refractory COD

(Table 4). The mixture was pre-treated by filtration with Whatman 4-7 µm membrane filters. Each experiment was performed in duplicate, for large deviations in triplicate, using differently loaded process water flows from the breweries C and D.

During ultrafiltration, permeate was continuously removed from the membrane system so that a reduction in feed volume and an increase of feed concentration were achieved. The experiments were carried out under the following test conditions: i) constant permeate flow of 0.6 L/h, ii) cross-flow velocity of 17 cm/s, iii) 44 mil spacer with diamond structure, iv) average temperature of 22±1°C. If the pressure limit of 1.5 bar or a permeate yield of 90% were reached, the experiment was terminated. At the beginning of the experiments, a feed sample was taken for measurement of COD.

2.5.2 Treatment of process water flows by nanofiltration

The PVPP regeneration lye and the CIP lyes were treated separately by nanofiltration using the laboratory membrane test plant (see chapter 2.3). Hereby, the objective was to proof technical, ecological and economic aspects of the separate treatment. As filtration medium, the PVPP regeneration lye from the brewery F and the CIP lyes from the breweries D and F were used. The CIP lyes from brew house, filtration cellar and fermenting cellar were mixed in a 2.1:2.2:3.1 ratio based on the real composition. Each experiment was performed in quadruplicate (PVPP regeneration lye) or triplicate (CIP lyes) testing differently loaded process water flows.

During nanofiltration, permeate was continuously removed from the membrane system. The separate treatment of PVPP regeneration lye and the separate treatment of the CIP lyes were carried out under the following test conditions: i) constant TMP of 32 and 20 bar, respectively, ii) cross-flow velocity of 31 cm/s, iii) 44 mil spacer with diamond structure, iv) average temperature of 22±1°C. The experiments were terminated after 42 and 46 hours, respectively. At the beginning of the experiments, feed and permeate flow were sampled. Concentrate and permeate samples were taken after 24 hours and at the end of the experiments. In all samples of the two test series, TOC and TNb were analyzed. Additionally, the concentrations of the selected polyphenols were determined in feed, permeate and concentrate samples during nanofiltration of the PVPP regeneration lye. COD and DOC measurements of feed, permeate and concentrate samples were carried out during nanofiltration of the CIP lyes.

3 Results and discussions

3.1 Characterization of selected process water flows

The following process water flows were identified to have a negative effect on the filtration performance of low pressure membranes due to its chemical-physical characteristics shown in Table 4 and discussed in the following: PVPP regeneration lye, cleaning and rinsing lyes from cleaning-in-place (CIP) systems as well as effluents from bottle washing machine and conveyor belts.

PVPP regeneration lye

To improve the colloidal stability of beer, haze causing polyphenols are removed by PVPP which is subjected afterwards to a regeneration process with heated (up to 85°C) 1-2% sodium hydroxide solution. The super alkaline PVPP regeneration lye (pH = 12.5) is characterized by a high organic load (COD = 6.2 g/L) which varies in the range from 3.5 to 14.2 g/L depending on the pollutional index at sampling time. The COD/BOD_5-ratio averages

4.3 indicating a poor biodegradability. Furthermore, the waste lye contains total polyphenol concentrations of a few hundred mg/L (see also chapter 0) as well as fine particles with equivalent diameters of 0.3 to 1.4 µm causing steric pore blockings of low pressure membranes.

CIP lyes

CIP systems are predominantly applied in the sections brew house, fermenting and filtration cellar to remove product residues and mineral deposits as well as to inhibit biological contamination in tanks and pipelines [19]. To avoid spreading of possible contamination the state of the art implies separation of the mentioned production sections whereby in general the additionally listed CIP cycles brew house, cold wort, unfiltered beer, yeasts and filtered beer result. For the elimination of organic compounds, heated (up to 80°C) 1-2% sodium hydroxide solution is used and rejected after several cleanings. CIP lyes are characterized by a high pH (13.2-13.3). COD averages out 3.4 g O_2/L (brew house), 0.4 g O_2/L (filtration cellar) and 1.4 g O_2/L (fermenting cellar). The COD of the CIP lye from the filtration cellar varies in a narrow range (0.2-0.7 g O_2/L) indicating a low pollutional index at sampling time. The COD variations of the CIP lyes from the section brew house and fermenting cellar are distinctively higher (0.9-8.9 g O_2/L and 0.3-5.2 g O_2/L, resp.) so that the average COD is distorted by sampling of low polluted lyes and it can be assumed that the organic load of the total effluent is even higher due to the disposal of high polluted CIP lyes. However, the average COD/BOD_5-ratios of 2.6 (brew house/fermenting cellar) and 5.3 (filtration cellar) imply that the organic substances of the CIP lyes are only partly biodegradable. The contained proteins and polysaccharides belong to the group of organic foulants and can cause pore blockings if they have a similar or smaller size than the membrane pores [20,21,22,23].

Effluents from bottle washing machine

Cleaning, disinfection and rinsing processes in the bottle washing machine produce 16% of the wastewater from filling [24] which is considered to be one of the main sources of polluting loads in brewery wastewater [7]. For bottle washing, 1.5-2% sodium hydroxide solution is mixed with additives (e.g. defoamers, complexing agents, surface active substances). By the cleaning process, soluble, insoluble or colloidally soluble ingredients like paper fibers of defibered labels, pigments, adhered dirt to bottles, label glue, etc. end up in the super alkaline (average pH = 12.8) cleaning lye from the bottle washing machine [19]. The COD averages out 7.9 g O_2/L and its distinctive variations in the range of 2.3 to 13.5 g O_2/L reflect the hebdomadal variations depending on intermediate treatment measures as per description in literature [7]. The maximum values also confirm investigations on the physical-chemical characterization of bottle washing lyes carried out at FIWAT [25]. The average COD/BOD_5-ratio of 3.7 indicates a poor biodegradability. In comparison to other process water flows from breweries, the main lye from the bottle washing machine contains high concentrations of biopolymers (18 to 23% of total DOC) which belong to the significant factors for membrane fouling in municipal wastewater [26]. A high fouling potential is also accredited to surface active additives [27] accounting for up to 17% of the COD in the cleaning lyes for bottle washing.

Effluents from conveyor belts

The primary function of lubricants for conveyor belts consists in the wear reduction of chains, mat chains, slide rails, etc. in filling due to rubbing. Thereby, energy consumption for transport, generation of noise and the risk of mechanical damage of bottles, kegs, etc. is minimized [19]. The COD of the neutral to slightly alkaline effluent from conveyor belts averages out 566 mg O_2/L and shows low variations (350-700 mg O_2/L). The average

COD/BOD_5-ratio of 17 (1.7-44) and the resulting partly extremely poor biodegradability confirm the literature data related to the negative effect of lubricants for conveyor belts on wastewater treatment processes [28]. Comparable to additives of cleaning lyes from the bottle washing machine, lubricants generates a specific fouling potential in effluents of wastewater treatment plants due to a high content of refractory COD.

3.2 Influence of process water flows on the performance of low pressure membranes

The critical process water flows were added to biologically treated brewery wastewater to investigate their influence on the performance of ultrafiltration membranes. The time curves of TMP during laboratory scale experiments are shown in Fig. 5. COD in feed, permeate and concentrate as well as COD rejection can be taken from Table 5. The best filtration result was achieved by ultrafiltration of the most widely biologically treated brewery wastewater (brewery D) without addition of a process water flow. If a process water flow was added, a faster and higher increase of TMP was observed depending on the organic load of the feed stream. Therefore, the filtration performance correlates with the feed COD.

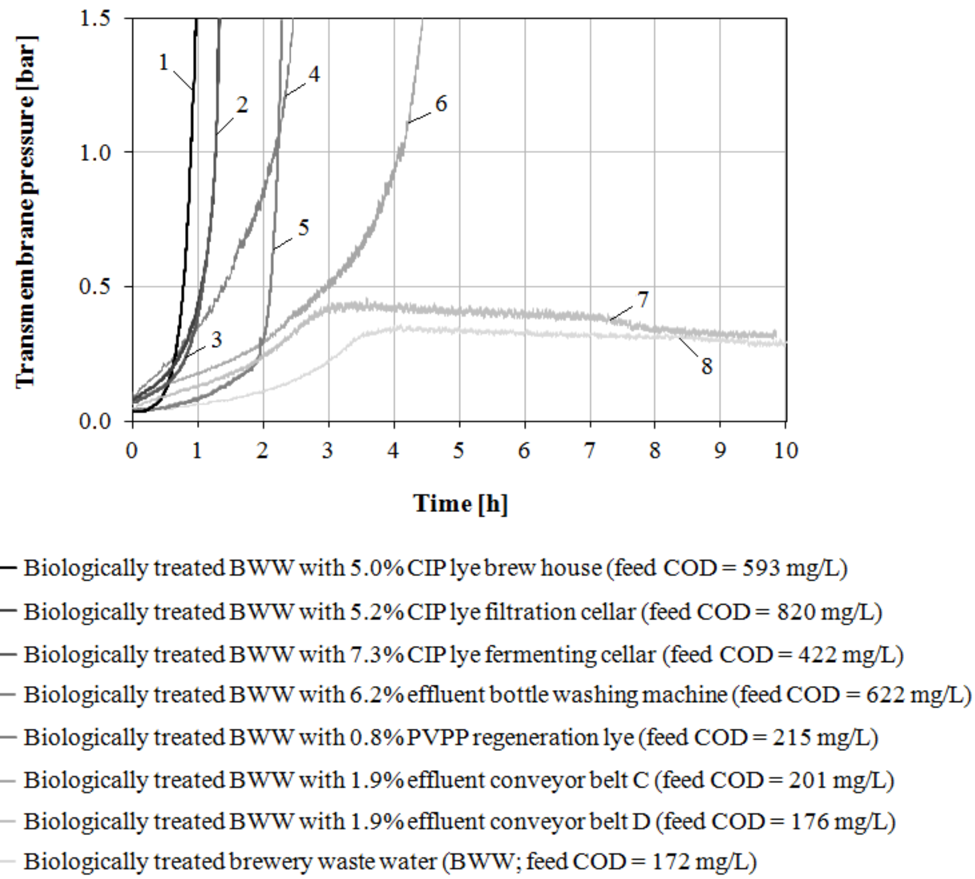

Fig. 5: Times curves of TMP during ultrafiltration of biologically treated brewery wastewater (BWW) mixed with critical process water flows in practically relevant concentrations in laboratory-scale (Microdyn-Nadir membrane type PM UH050 (hydrophilic polyethersulphone, 50 kDa), concentration of feed solution until a permeate yield of 90% or achieving a pressure limit of 1.5 bar, constant flux = 70 L/(m²·h), temperature = 22±1°C, cross-flow velocity = 17 cm/s, average of two to three experiments)

Besides the fouling causing ingredients mentioned in chapter 3.1, the fouling behavior of the tested ultrafiltration membrane was influenced negatively by the availability of the strong electrolyte NaCl. In the course of the biological wastewater treatment, the sodium hydroxide solution (NaOH) for cleaning or regeneration processes is neutralized with HCl. The resulting NaCl dissociates in aqueous solution in positive sodium cations and negative chloride anions which are almost steric separated and electrically charged. Although ultrafiltration membranes do not feature salt rejection and consequently scaling processes cannot occur, electrolytes in the feed stream can influence the fouling potential of other substances significantly. High ionic strengths correlate with a compression of electric charge layers around solids. Thus, the electrostatic repulsion between negatively charged particles and the negatively charged membrane surface is reduced so that the fouling behavior of the membrane is increased. [29]

The influence of the effluent from conveyor belts on the filtration performance depends on the kind of lubricant used by the brewery. Adding the effluent of the brewery C with an extremely poor biodegradable lubricant (COD/BOD_5-ratio = 44), a negative effect on the filtration performance of the low pressure membrane could be observed. Lubricant D with a good biodegradability (COD/BOD_5-ratio = 1.7) caused only a slightly increase of TMP. Anyway, it will be degraded during biological treatment. Hence, the choice of the lubricant also affects the recyclability of the total wastewater flow.

3.3 Treatment concepts

3.3.1 PVPP regeneration lye

The PVPP regeneration lye is currently supplied to the total wastewater of breweries. For discharge into treatment plants, it has to be neutralized. Therefore, considerable salt loads are produced depending on the used acid. In a model calculation, Meyer and Kiefer [30] elaborated that the annual lye consumption of a medium-scale brewery with an annual beer output of 100,000 m^3 amounts 840 m^3 2% sodium hydroxide solution. In the case of neutralisation with HCl, it results in 25 t NaCl which load wastewater treatment plants and receiving water bodies. By reusing the PVPP regeneration lye in the regeneration process, the effort for neutralisation, the salt load of the wastewater as well as the required amount of soda lye and heat can be reduced distinctly. Also, the recyclability of the total effluent can be improved by the separate treatment as shown in chapter 3.2.

For the recycling of the PVPP regeneration lye, nanofiltration has proven itself as an effective and economic technology. The separate treatment was feasible until a permeate yield up to 60% without an appreciable decrease of flux (Fig. 6). Linehan and Grennan [31] as well as Chmiel et al. [7] reported permeate yields of 90%. But as a result, investment and operating costs for the membrane system increase (larger membrane surfaces, more frequent membrane cleanings, reduction of membrane durability, treatment and/or disposal of cleaning solutions).

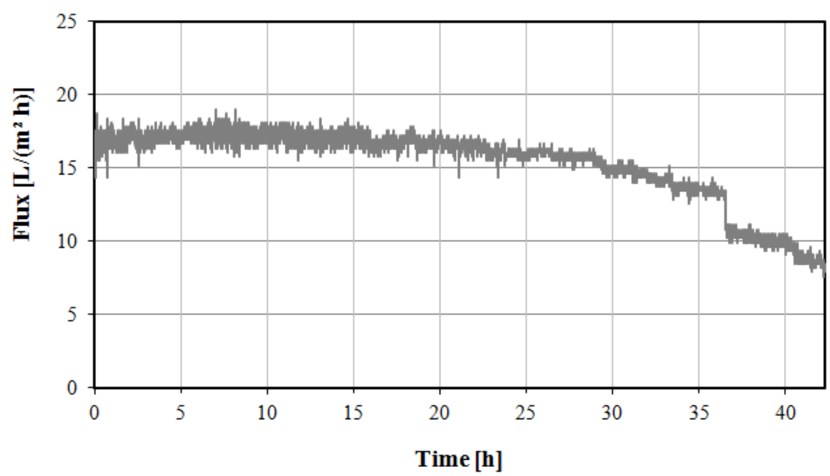

Fig. 6: Time curve of flux during nanofiltration of PVPP regeneration lye in laboratory-scale (Koch Membrane systems membrane type MPS-34 (polysulphone, 200 Da), concentration of feed solution up to a permeate yield of 85%, constant pressure = 20 bar, temperature = 22±1°C, cross-flow velocity = 32 cm/s, duration of experiment = 42 h, average values of four experiments)

The results of the analysis of feed, permeate and concentrate are listed in Table 6. In the permeate samples, nitrogen was unverifiable and a COD elimination rate of 88% was realized reaching a permeate yield of 48%. A positive aspect is that the permeate is almost free of the six selected reference polyphenols (Fig. 7) so that the reuse of the PVPP regeneration lye is suitable combined with a resharpen of the recycled lye. Due to the achieved increase of the polyphenol concentrations, the selective uptake of the polyphenols out of the nanofiltration concentrate is conceivable.

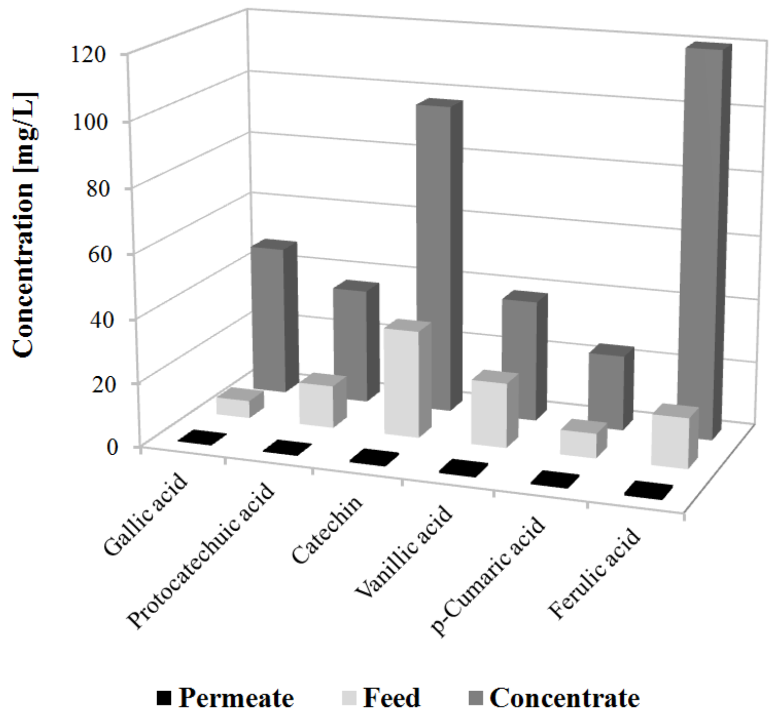

Fig. 7: Concentrations of selected polyphenols in feed (t = 0 h, permeate yield = 0%), permeate (t = 42 h, permeate yield = 85%) and concentrate (t = 42 h, permeate yield = 85%) during nanofiltration of PVPP regeneration lye in laboratory-scale (average values over four measurements)

The example of an Irish brewery demonstrates that the separate treatment of the PVPP regeneration lye is also of economic benefit. The treatment plant built within the framework of the "Cleaner Greener Production Program" enables a potential for savings of 34,000 €/year. Assuming investment costs of 100,000 €, the payback period of the plant would not last for more than ca. 3 years [32,33]. Besides, procurement costs for membranes were notably decreased in comparison to further investigations of Linehan and Grennan [31] and Chmiel et al. [7].

Additional profits could be taken by the recovery of polyphenols up for sale. Due to the complexity of the background matrix, the selective uptake of polyphenols out of nanofiltration concentrate poses a technical challenge, but can be an additional advantage if the PVPP regeneration lye is treated separately. In combination with the recycled alkalinity, heat energy (treatment at 70°C) and the resulting benefits for the wastewater treatment/reuse, it can be stated that the separate treatment of the PVPP regeneration lye by nanofiltration for reuse in the regeneration process is recommended with respect to technical, ecological and economical aspects.

3.3.2 CIP lyes

Also the cleaning and rinsing lyes from CIP systems are currently supplied to the total wastewater of breweries and neutralized for discharge. The fresh water consumption for the CIP lyes amounts to about 11% [9]. Related to a medium-scale brewery with an annual beer output of 100,000 m³, a lye consumption of 44.000 m³ 1-2% sodium hydroxide solution/year is required for the CIP systems. In the case of neutralization with HCl, around 970 t NaCl were produced yearly. By reuse of CIP lyes, the effort for neutralisation, the salt load of the wastewater as well as the required amount of soda lye and heat can be reduced significantly. Moreover, the recyclability of the total effluent can be improved by a separate treatment as shown in chapter 3.2.

For the recycling of the CIP lyes, nanofiltration has proven itself to be an effective and economic technology. The separate treatment was feasible until permeate yields up to 50% with an acceptable decrease of flux (Fig. 8). The results of the analysis of feed, permeate and concentrate are listed in Table 7. In the permeate samples, nitrogen and COD elimination rates of 75% and 93%, resp. were realized at a permeate yield of 45% so that the reuse of the CIP lyes is suitable combined with a resharpen of the recycled lye.

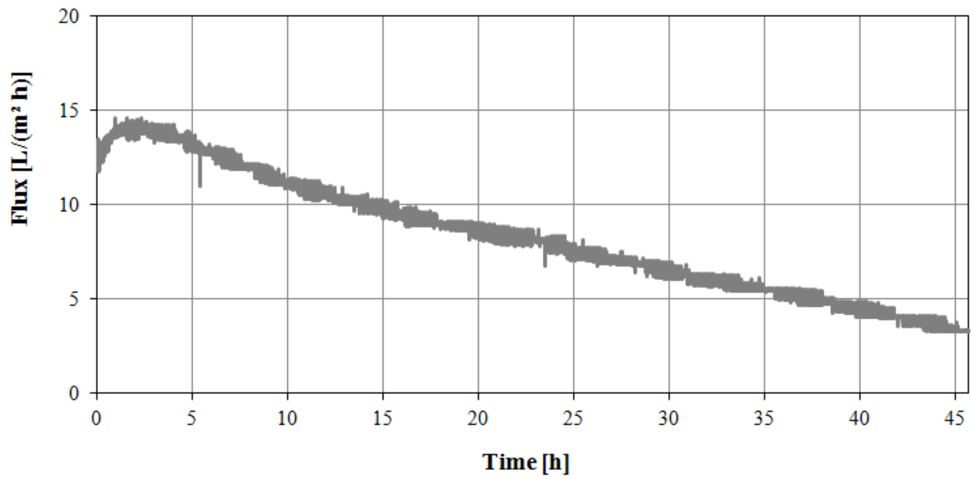

Fig. 8: Time curve of flux during nanofiltration of CIP lyes in laboratory scale (Koch Membrane systems membrane type MPS-34 (polysulphone, 200 Da), concentration of feed solution until a permeate yield of 85%, constant pressure = 20 bar, temperature = 22±1°C, cross-flow velocity = 32 cm/s, duration of experiment = 46 h, average values of three experiments)

The following model calculation shows that a separate treatment of the CIP lyes is also economically advantageous. Depending on the concept of the plant, the specific consumption of 50% sodium hydroxide solution of a brewery amounts 2 to 5 kg/m³ SB [34] which are used for CIP systems, but also in the sections filtration (regeneration of PVPP) and filling (bottle and keg washing). Based on the share of the fresh water consumption and a price of 180 €/t [34], the model brewery has to defray annual chemical costs in the amount of 45,000 € which can be cut to 22,500 € by the reuse of CIP lyes (assuming a permeate yield of 50%). Furthermore, costs can be reduced by lowering fresh water and wastewater costs. Calculating with a fresh water price of 2.20 €/m³ and a wastewater price of 2.50 €/m³ for Berlin region, water costs of about 100,000 € can be saved every year.

This is accompanied by the operating and investment costs for the applied membrane system. Assuming specific operating costs of 1.35 €/m³ [11], the annual treatment costs amount to 60,000 €. The investment sum is set at 100,000 €. In consideration of the saved water and chemical costs, the membrane system amortizes in about two years so that the recycling of the CIP lyes is also financially attractive.

Restrictively, it has to be considered that the CIP lyes are several small process water flows produced in different sections of the brewery (brew house, fermenting cellar, filtration cellar). Walter [1] already determined that the separate treatment and the reuse of the CIP lyes in the cleaning process are connected to high costs for additional pipe installations so that the cost effectiveness of the separate treatment is directly related to the infrastructure. Therefore, the application of nanofiltration for recycling of the CIP lyes is technical, ecological and economical favourable especially in case of retrofitting of a brewery.

3.3.3 Main lye from bottle washing machine

To eliminate the sludge from polluted cleaning lyes for bottle washing, several treatment processes are applied in praxis. The settling of sludge to separate contained particles (sedimentation) is most commonly used. An alternative consists in the application of a self-emptying separator in a partial flow of the bottle washing machine, which cleans the waste lye from trubs by centrifugation. The cleaning lye from the bottle washing machine can be also treated by membrane processes. A COD elimination of up to 80% can be achieved and the required amounts of water, energy and chemicals will be reduced significantly. The applied membrane system amortizes in many cases in two to three years [19]. Because the separate treatment of cleaning lyes from the bottle washing machine is considered to be state of the art, no further investigations were carried out concerning this matter within the scope of this study.

3.3.4 Effluent from conveyor belts

The conveyor belts discharges only a small quantity of wastewater (1.9% of the total effluent [24]). Furthermore, the possibilities for the collection of this process water flow are considered to be limited so that a separate treatment is not suitable. Because the influence on the performance of low pressure membranes is connected to the biodegradability of the lubricant used by the brewery for conveyor belts, the substitution by a biodegradable lubricant is recommended with respect to the improvement of the recyclability of brewery wastewater.

4 Conclusion

(1) Membrane processes are state of the art for an advanced treatment of biologically cleaned brewery wastewater. They can be operated stably, but there is still potential for improvements with a positive economic impact.

(2) Super alkaline process water flows with a high product or co-product load (e.g. PVPP regeneration lye, CIP lyes, main lye from the bottle washing machine) have a negative effect on the filtration performance of low pressure membranes and should be eliminated or substituted before the filtration process (Table 8).

(3) The separation and treatment of critical process water flows improves not only the recyclability of the total effluent and reduces the costs. It is also recommended to achieve a better effluent quality after biological treatment, to reduce the salt load, to close internal water cycles and to recover valuables.

(4) By defining and adjusting the wastewater matrix, membrane systems can be applied economically, efficiently and safely for water reuse in breweries.

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Tables

Table 1: Worldwide breweries applying membrane systems for water reuse

Year	Brewery	Production site	Membrane system
2010	Bluetongue Brewery	Warnervale, Australia	MBR + RO
2009	Lion Nathan‘s Castlemaine Perkins	Brisbane, Australia	MF + RO
2009	SAB Tanzania Breweries	Dar es Salaam, Tanzania	MBR + RO
2008	Cerveceria Polar	Caracas, Venezuela	MBR + RO
2007	Boon Rawd Brewery	Khon Kaen, Thailand	UF + RO
2007	Martens Brewery	Bocholt, Belgium	MBR + RO + UV
2006	König Brewery	Duisburg, Germany	MBR + RO
2004	Foster's CUB Yatala	Yatala, Australia	MF + RO

Table 2: Process water flows of a brewery during a week [35]

Section	Process water flow	Characterization	Amount [m³]	COD [g/L]
Brew house	Spent grain (out-pressed liquid)	Product residues	1.2	40
	Emptying lauter sieve	Product residues	0.45	17.7
	Separating water-gyle-mixture	Product residues	13.5	7.2-155
	CIP:			
	Separator	Detergents and disinfectants	5.8	0.5-1.7
	Wort kettle	Detergents and disinfectants	1.6	4.1-18
	Wort pipe	Detergents and disinfectants	3	0.9
	Wort cooler	Detergents and disinfectants	24	2.7
Fermenting/ storage cellar	Separating beer-yeast-mixture	Product residues	1.5	180
	Separating beer-water-mixture	Product residues	0.6	37
	CIP:			
	Cellar pipe	Product residues	9.2	22.5
	Cylindrical-conical brewing tank (pre-rinsing)	Product residues	9	5.2
	Cylindrical-conical brewing tank (cleaning)	Detergents and disinfectants	16.8	2.0-3.6
Filtration cellar	**Kieselguhr press:**			
	Hosing down	Product residues	0.8	4.4
	Out-pressed liquid	Product residues	2.2	34
	Sludge	Product residues	5	63
	CIP:			
	Filter	Product residue s/ detergents and disinfectants	5.6	7.6-22.4
	Pressure tank	Product residues / detergents and disinfectants	1	4.4
	Centrifugal high-performance filter	Detergents and disinfectants	12	1-1.2
Filling	**Filler:**			
	Emptying	Product residues	0.12	8
	Rinsing	Product residues	18	0.8
	Crate washer	Detergents and disinfectants	15	0.3-0.45
	Bottle washing machine	Detergents and disinfectants	16.4	0.1-6.4
	Full product shower	Product residues	6	21.4
	Conveyor belt	Detergents and disinfectants	6	4.5

Table 3: Sampling of process water flows from the breweries A-F (x = sampled, - = not sampled)

Section	Process water flow	Brewery A	Brewery B	Brewery C	Brewery D	Brewery E	Brewery F
Filtration cellar	PVPP regeneration lye	-	x	x	x	-	x
	CIP lye	-	-	-	x	x	x
Brew house	CIP lye	x	x	x	x	x	x
Fermenting cellar	CIP lye	x	-	x	x	x	x
Filling	Main lye from bottle washing machine	x	x	x	x	x	x
	Effluent from conveyor belts	-	-	x	x	-	x

Table 4: Physical-chemical characteristics of the biologically treated total effluent from brewery D and selected process water flows from the sampled breweries (average values, ranges in parentheses)

Wastewater flow	**pH** [-]	**Conductivity** [mS/cm]	**COD** [g/L]	**TOC** [g/L]	**DOC** [g/L]	**BOD_5** [g/L]	**BOD_{10}** [g/L]	**COD/BOD_5** [-]	**TNb** [mg/L]
Total effluent from brewery D	8.3	2.4	219	42	40	34	46	6.4	0.1
PVPP regeneration lye	12.5 (9.3-3.4)	68 (19-94)	6.2 (3.5-14.2)	2.3 (1.4-5.2)	2.2 (1.2-5.1)	1.9 (0.99-4.1)	2.4 (1.3-4.9)	4.3 (3.5-5.4)	102 (22-206)
CIP lye brew house	13.2 (12.9-13.9)	97 (52-126)	3.4 (0.92-8.9)	1.5 (0.33-3.7)	1.2 (0.29-3.5)	1.8 (0.42-2.9)	2.3 (0.52-5.7)	2.6 (2-3.7)	184 (53-250)
CIP lye filtration cellar	13.3 (12.9-13.8)	78 (40-114)	0.4 (0.2-0.7)	0.18 (0.1-0.24)	0.16 (0.06-0.23)	0.37	0.46	5.3 (1.8-11.9)	5.2 (2.3-9.2)
CIP lye fermenting cellar	13.2 (12.8-13.8)	88 (34-150)	1.4 (0.3-5.2)	0.46 (0.14-1)	0.25 (0.12-0.37)	0.28 (0.15-0.34)	0.34 (0.18-0.45)	2.6 (2-3.1)	42 (1-119)
Main lye from bottle washing machine	12.8 (9.7-13.7)	94 (83-105)	7.9 (2.3-13.5)	2.8 (0.62-4.3)	2.2 (0.29-4.3)	2.4 (0.53-3.8)	2,7 (0.85-4.1)	3.7 (1.6-4.8)	3.5 (0-6.6)
Effluent from conveyor belts	7.6 (6.8-8.2)	0.7 (0.2-1.1)	0.57 (0.35-0.71)	0.096 (0.058-0.16)	0.087 (0.046-0.15)	0.11 (0.014-0.21)	137 (14-253)	17 (1.7-44)	204 (15-388)

Table 5: COD in feed, permeate and concentrate as well as COD rejection during ultrafiltration of biologically treated brewery wastewater mixed with critical process water flows

Parameter	**COD feed**	**COD permeate**	**COD concentrate**	**COD rejection**
Unit	mg/L	mg/L	mg/L	%
Sampling time	beginning	end	end	end
Biologically treated BWW with 5.0% CIP lye brew house	593	539	639	15.6
Biologically treated BWW with 5.2% CIP lye filtration cellar	820	625	879	28.9
Biologically treated BWW with 7.3% CIP lye fermenting cellar	422	187	469	60.1
Biologically treated BWW with 6.2% effluent bottle washing machine	622	285	687	58.5
Biologically treated BWW with 0.8% PVPP regeneration lye	215	108	236	54.2
Biologically treated BWW with 1.9% effluent conveyor belt C	201	37	253	85.4
Biologically treated BWW with 1.9% effluent conveyor belt D	176	128	300	57.3
Biologically treated wastewater	172	190	283	32.9

Table 6: TOC and TNb concentrations in feed, permeate and concentrate as well as rejection during nanofiltration of PVPP regeneration lye in laboratory-scale (average values over four samples)

Parameter	**Unit**	**Feed**	**Permeate**			**Concentrate**		**Rejection** [%]		
Sampling time	h	0	0	24	42	24	42	0	24	42
Permeate yield	%	0	0	48	85	48	85	0	48	85
TOC	mg/L	2,850	261	341	1,119	7,900	17,850	90.8	95.7	93.7
TNb	mg/L	54	bld	bld	bld	553	662	<100	<100	<100

Table 7: COD, TOC, DOC and TNb concentrations in feed, permeate and concentrate as well was rejection during nanofiltration of CIP lyes in laboratory-scale (average values over three samples)

Parameter	**Unit**	**Feed**	**Permeate**			**Concentrate**		**Rejection** [%]		
Sampling time	h	0	0	24	46	24	46	0	24	46
Permeate yield	%	0	0	45	85	45	85	0	24	46
COD	mg/L	2,945	194	198	660	5,492	12,390	93.4	96.4	94.7
TOC	mg/L	2,973	77	151	579	5,624	11,921	97.4	97.3	95.1
DOC	mg/L	2,104	77	151	407	5,594	11,557	96.3	97.3	96.5
TNb	mg/L	131	25	33	35	235	472	80.9	86.0	92.6

Table 8: Characteristics of critical process water flows and suggestions for their elimination or substitution

Process water flow	Characteristics	Elimination/Substitution
PVPP regeneration lye	Super alkaline, very high organic and salt load, high concentrations of N and P, polyphenols, almost free of solids, poor biodegradability	Separate treatment by nanofiltration → Reuse in the regeneration process; selective uptake of polyphenols, if applicable
CIP lyes	Super alkaline, very high organic and salt load, high concentration of N, almost free of solids, moderate biodegradability	Separate treatment by nanofiltration → Reuse in the cleaning process
Effluent from bottle washing machine	Super alkaline, very high organic load, high concentrations of biopolymers and N, good biodegradability	Separate treatment (sedimentation, centrifugation or membrane filtration) → Reuse in the cleaning process
Effluent from conveyor belts	Neutral, high organic load, partly extremely poor biodegradability and many refractory substances	Substitution by a lubricant of good biodegradability

Research highlights

- Membrane systems for water reuse in breweries can be economically optimized.
- Highly loaded cleaning solutions decrease the recyclability of the total effluent.
- The separation and treatment of critical process water flows is recommended.
- Adjusting the waste water matrix is a new approach to optimize membrane systems.