

AEROBIC DEGRADATION OF A NON-IONIC SURFACTANT IN A MEMBRANE BIOREACTOR(MBR)

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Abstract : A membrane bioreactor (MBR) was used to investigate the aerobic degradation of foam active substance - non-ionic surfactant, APG 2000 UP. The surface aeration using the propeller loop reactor (PLR) guaranteed sufficient O₂ for substrate removal and bacteria growth and avoided foam development. Moreover, the cross-flow membrane filtration enabled the separation of the bacteria still loaded with surfactant in the collecting container. The biological degradation of the surfactant with varying hydraulic retention time (HRT) and influent concentration c_{S0} showed high substrate removal of nearly 95 % at high volumetric loading rates up to 7.4 kgCOD m⁻³d⁻¹ and at sludge loading rates up to 1.8 kgCOD (kgVSS d)⁻¹ for biomass concentration $c_B \approx$ constant. The increasing c_B from 3.4 to 14.5 gL⁻¹ TSS respectively sludge retention time (SRT) from 5.1 to 442 d under complete biomass retention by the membrane filtration resulted in high removal of substrate $\alpha > 90$ % with reducing excess sludge production.

Key Words : Aerobic degradation, Surfactant, MBR, Propeller loop reactor, Membrane filtration

INTRODUCTION

The valuable fresh water is widely becoming a scarce resource in many populated areas of the world due to expanding water pollution by human domestic and industrial activity. To keep the natural water state the water used for domestic purposes and industrial processes has to be reused, recycled and mostly cleaned by appropriated treatment concept.¹⁻³⁾

Surfactants are produced in large variation in continuously operating plants according to their application purposes.⁴⁾ The use of surfactants in

household and industrial products is almost universal, as emulsifiers, detergents, flotation aids, anti-foaming agents and wetting agents, due to their ability to influence interfacial tension at low concentrations.⁴⁾ The wide variety of surfactants used in many technical areas leads to their production in small quantities and therefore in batch processes. Owing to the frequent product changes all components of production equipment must often be rinsed, causing product and water loss.^{5,6)} Rinsing waters containing high surfactant concentrations must be treated to reduce the volume and the chemical oxygen demand of the wastewater prior to disposal in municipal sewer systems in order to avoid operational disturbances. The on-site aerobic treatment of rinsing water is

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a conceivable way.⁶⁾

Membrane technology such as reverse osmosis, micro- and ultrafiltration has developed into one of the most used environmental technologies, not only for drinking water treatment but also for wastewater treatment.⁷⁻⁹⁾ Membrane technology coupled with a biological reactor (MBR) allows the total retention of the biomass within the bioreactor so as to reach higher removal efficiency of pollutants.¹⁰⁾ Membrane bioreactors have found application in various areas of municipal wastewater treatment, landfill leachate treatment and industrial wastewater treatment,^{9,11-14)} The characteristic of nearly complete retention of the biomass by membrane filtration makes reactor operation at high biomass concentrations possible as well as allows the separation of sludge loaded with foaming active substances like surfactants.¹⁵⁾

In this study, the biological degradation of non-ionic surfactant (APG 2000 UP) in a propeller loop reactor (PLR) combined with cross-flow membrane filtration is investigated. The PLR with surface aeration is to guarantee sufficient supply of oxygen for bacteria growth for aerobic degradation of surfactant.

The biological degradation of surfactant is determined with varying hydraulic retention time (HRT) and influent concentration of substrate. A higher

biomass concentration is readily reached by using membrane filtration in order to determine the effect of various biomass concentrations, c_B , and solid retention times (SRT) on the biological degradation of surfactant in this membrane process. The long term biological efficiency and stability of a lab scale MBR system are guaranteed.

MATERIALS AND METHODS

Experimental Systems

The propeller stirrer with 3 blades (Fa. Fluid Mischtechnik (EKATO), Typ PR 100) produced a strong axial flow. Dependent on the stirrer speed, two different typical flows could be observed due to special geometries of the reactor. The typical recycle flow for the loop reactor was developed for the stirrer speed up to $n = 600$ rpm (Figure 1(a)). For $n > 750$ rpm (Figure 1(b)) a strong overflow was developed at the upper edge of the inner conduit, which provided for the inclusion of air into the inner conduit. The air was further dispersed by the propeller stirrer. The foam bubbles rose up through the outside of the inner conduit. As soon as they reached the surface of the liquid, they were pulled into the inner conduit and pushed down through the inner conduit. The foam bubbles were completely

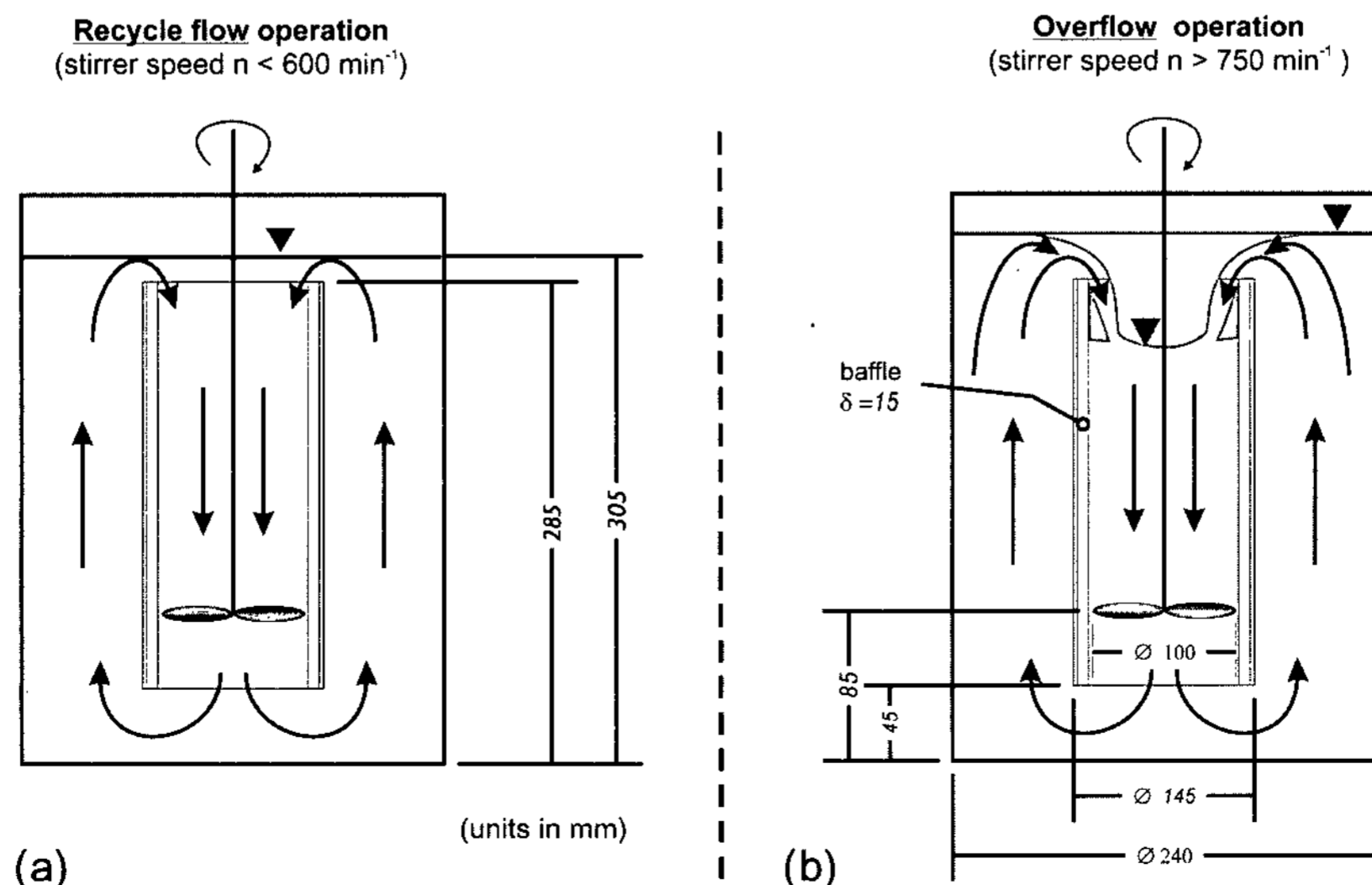


Figure 1. Propeller loop reactor at recycle flow (a) and overflow (b) operation.⁵⁾

destroyed. This function enabled the reactor to aerate successfully without anti foaming agents and to improve the operating safety.⁵⁾

The reactor was equipped with a double jacket to control the temperature in the reactor and with a baffle to avoid the building of a water-pout inside the inner conduit. The reactor was made from acrylic glass and covered and floored with high quality steel. The construction of the propeller loop reactor used for this study is shown in Figure 1.

Figure 2 shows the experimental set-up for the aerobic degradation of the non-ionic surfactant, APG 2000 UP. It consisted of 4 main parts: the propeller loop reactor, an unit for wastewater synthesis and nutrient dosage, a cross-flow membrane filtration system and an unit for gas analysis of oxygen and carbon dioxide. The unit for gas analysis of O₂ and CO₂ stands for the online monitoring and control of the biological degradation of surfactant,¹⁶⁾ but its results are not presented in this paper.

The reactor volume ranged from 13 to 14 L, depending on the stirrer speed. The liquid volume of the membrane filtration system, which

consists of a collecting container (≈ 7 L), modules and connecting hoses, was about 15 L. The reactor was assumed to be a completely mixed continuous-flow stirred tank reactor (CSTR). The synthetic wastewater was continuously produced by mixing surfactant and tap water. In order to avoid bacterial growth in the mixing flask, the nutrients were added to the influent after the mixing flask. The pumping rates were measured by hand and used to calculate a theoretical influent surfactant concentration. To minimize the influence of the temperature on the mass balance, the reactor was heated and cooled by using a double water jacket connected to a thermostat.

The non-ionic surfactant APG 2000 UP used is presented in Figure 3.

APG 2000 UP (Henkel KGaA) is a technical

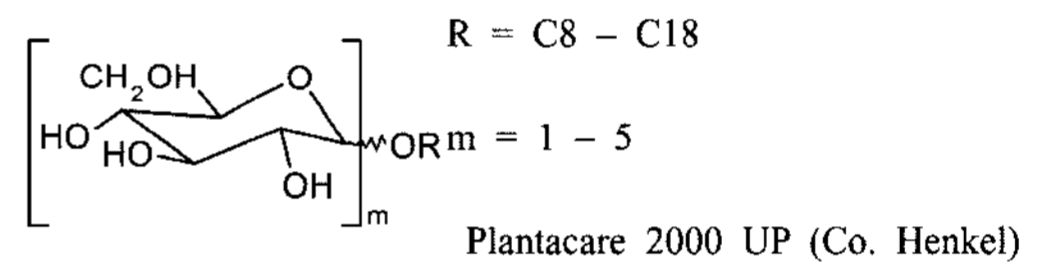


Figure 3 Structure of the non-ionic surfactant of APG 2000 UP.

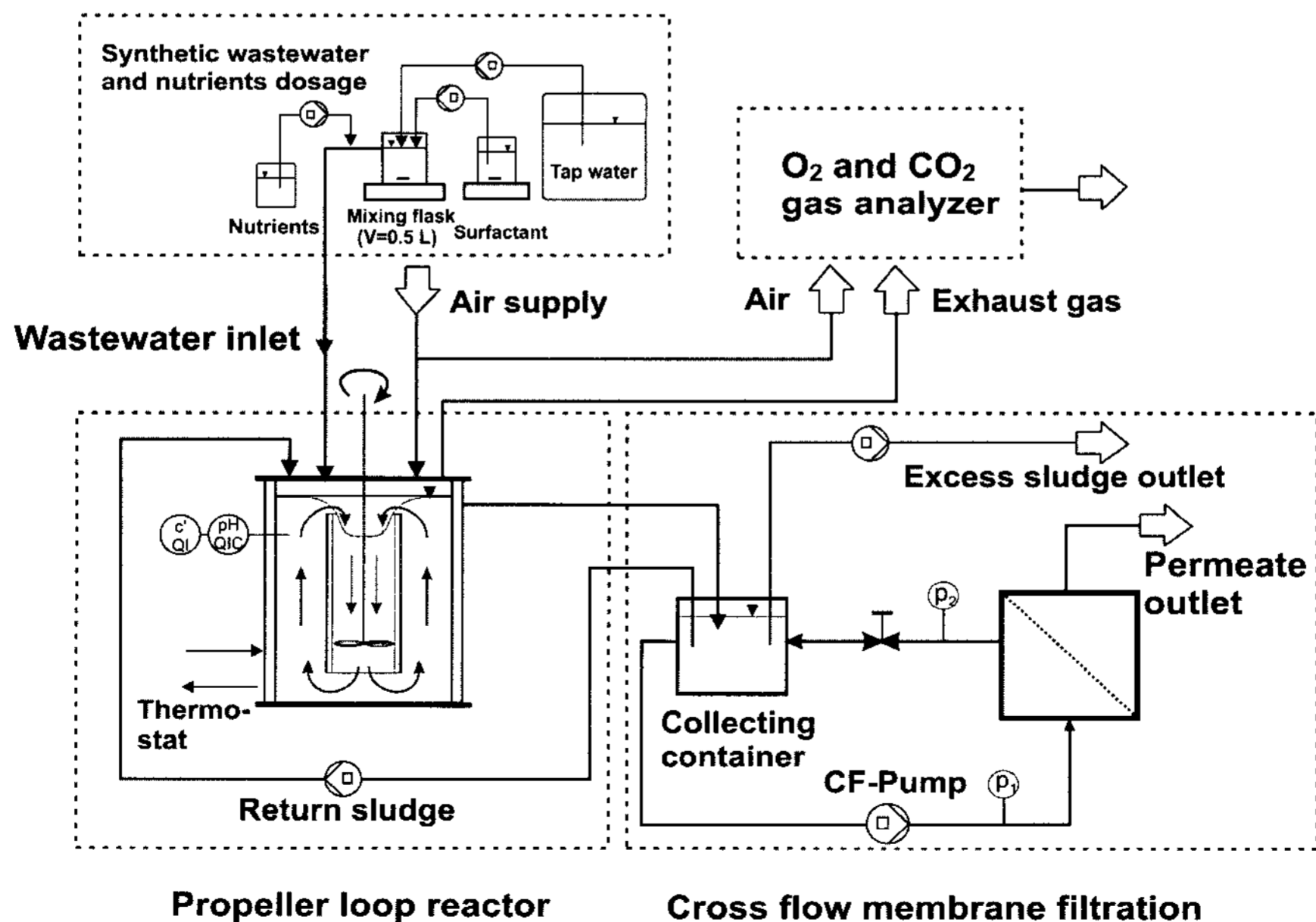


Figure 2. Continuously operated lab-scale plant for the aerobic degradation of the surfactant, APG 2000 UP.

surfactant consisting of a mixture of oligomeren. Billian¹⁷⁾ investigated more details about the characteristics and analytical methods for APGs. In order to supply the growing bacteria with nutrients, inorganic substances were added to the synthetic wastewater. Their compositions are given in Table 1. The surfactant solution was prepared by mixing with tap water. The operation of the bioreactor was started by supplying bacteria from a wastewater treatment plant in Berlin.

Table 1. Compositions of inorganic substances

	Components	Concentration
Inorganic salts	(NH ₄) ₂ SO ₄	55 gL ⁻¹
	K ₂ HPO ₄	16 gL ⁻¹
	KH ₂ PO ₄	8 gL ⁻¹
Mineral elements	Na ₂ MO ₄ ·2H ₂ O	195 µgL ⁻¹
	H ₃ BO ₄	1430 µgL ⁻¹
	MnCl ₂ ·4H ₂ O	905 µgL ⁻¹
	ZnSO ₄	110 µgL ⁻¹
	CuSO ₄ ·5H ₂ O	40 µgL ⁻¹
	Co(NO ₃) ₂ ·6H ₂ O	25 µgL ⁻¹

For the cross-flow membrane filtration system the membrane material was composed of polyvinylidene-fluoride (PVDF). The hydrophilic tubular PVDF membrane was cast on a composite polyester/polyolefin carrier (Co. X-Flow). The membrane had a mean pore size of 40 nm (Molecular Weight Cut off, MWCO of 50,000 D). The module was in a form of a pipe and could be operated over a pressure ranging from 1-3 bar, pH 2-10 at 25 °C and temperature 1-45 °C at pH 7 and 100 kPa. A module was 1 m long and had 7 membranes in it that had an 8 mm diameter. Total surface area of a module was 0.15 m².

Analytical Methods

Samples were withdrawn from the reactor and filtered immediately by air pressure with cellulose acetate filter having a 0.2 µm pore size (Sartorius, Göttingen, Germany), except for samples for biomass concentration. The samples were kept at the 4°C until analysis. Thus possible changes in the sample components was avoided.

DOC was determined by a DIMA-TOC 100 (Dimatec, Essen, Germany). The biomass concen-

tration was evaluated by measuring total suspended solids (TSS) and volatile suspended solids (VSS). Its measurement follows DIN 38414 (part 2 and 3). The sample taken was clarified by centrifugation for 20 min at 4000 rpm. After centrifugation the solid part found near the bottom of the centrifugation glass was carefully put into a porcelain cup and dried for 24 h at 105 °C for TSS. The dried matter was cooled in a desiccator for 2 h, and weighed. Further the sample in the porcelain cup was burnt for 2 h at 550°C in order to oxidize all organic matter completely for measuring VSS.

Continuous measurement of the dissolved oxygen concentration in the reactor was carried out with an Oxi 340 meter with an O₂ electrode CelloX[®] 325 (respectively, WTW, Weinheim, Germany). The measurement of the pH was performed online by a pH electrode InLab[®] (Mettler-Toledo) and a pH instrument Dulcometer[®] (Prominent, Heidelberg, Germany) which enabled the pH value to control by a dosage of soda solution (NaOH) automatically. The pH fluctuation ranged ± 0.02.

Operating Conditions

In order to test the influence of the volumetric loading rate on degradation rate of surfactant, the influent flow rate Q_0 and the substrate influent concentration were varied. With varying Q_0 the hydraulic retention time (HRT) varied resulting in changing volumetric B_V and sludge loading rate B_X .

Besides changing HRT, the solid retention times (SRT) was stepwise increased by using membrane filtration in order to determine the effect of various biomass concentrations, c_B , on the biological degradation of surfactant in the membrane process.

Evaluation Methods

Direct comparison of effluent substrate concentrations should only be made for the same influent concentrations. Therefore, the substrate removal, α , is introduced by (1) as followed

$$\alpha = \frac{c_{S0} - c_{S1}}{c_{S0}} \quad \text{in \%} \quad (1)$$

where: c_{S0} substrate influent concentration
 c_{S1} substrate effluent concentration

The substrate concentration can be described by TOC, COD, N or P-components in wastewater.

The substrate degradation rate, r_S , can be determined for the CSTR systems used by an integral mass balance for steady state operation:

$$0 = Q_0(c_{S0} - c_{S1}) - r_S \cdot V_R \quad (2)$$

where: Q_0 influent flow rate
 V_R reactor volume

Introducing a hydraulic retention time (HRT), t_H , of Eq. (3) in Eq. (2)

$$t_H = \frac{V_R}{Q_0} \quad (3)$$

results in Eq. (4) as the substrate degradation rate

$$r_S = \frac{c_{S0} - c_{S1}}{t_H} \quad (4)$$

Sludge retention time (SRT), t_S , is controlled by sludge wastage from the whole biological system defined as

$$t_S = \frac{c_B \cdot V_R}{c_{B,1} \cdot Q_{ES}} \quad (5)$$

where: c_B biomass concentration of the reactor
 $c_{B,1}$ bacteria concentration of the excess sludge
 Q_{ES} volumetric flow rate of the excess sludge

The volumetric loading rate is defined as

$$B_V = \frac{c_{S0} \cdot Q_0}{V_R} = \frac{c_{S0}}{t_H} \quad (6)$$

The amount of an influent mass per unit volume and per unit time, B_V , is calculated at a position before a possible recycle stream. The volumetric loading rate is dependent on the substrate influent concentration and hydraulic retention time and cannot be increased by an internal recycle in the plant. The sludge loading rate, B_X , describes the amount of organic matter applied to the sludge per day and is defined as follows:

$$B_X = \frac{c_{S0} \cdot Q_0}{c_B \cdot V_R} = \frac{c_{S0}}{c_B \cdot t_H} \quad (7)$$

RESULTS AND DISCUSSION

The synthetic wastewater with the foaming active surfactant (alkylpolyglycoside - APG 2000 UP) was successfully treated in an aerobic bioreactor with surface aeration for different hydraulic retention times, t_H (Figure 4). The surface aeration by a propeller loop stirrer guaranteed sufficient aeration for the degradation of substrate and bacteria growth. The foam development in the reactor was avoided totally.

Hydraulic retention time (HRT) ranges from $t_H = 11$ to 6.2 h and c_{S0} from 510 to 980 mgL⁻¹ DOC. The experiment consisted of 4 experimental periods. The conditions of each period are summarized in Table 2.

In Figure 4 the HRT decreases stepwise from 10.3 to 6.1 h until the end of the 3rd period, resulting in increase in the volumetric loading rate B_V . The substrate influent concentrations c_{S0} vary from 510 to 580 mgL⁻¹ DOC (aver. 540 mgL⁻¹ DOC). The substrate effluent concentrations c_{S2} range from 19 to 35 mgL⁻¹ DOC (aver.

Table 2. Operating conditions of the continuously operated MBR (pH = 7±0.02 and Temp. = 22.5°C) (for Fig. 4).

Period	c'_{O_2} [mgL ⁻¹]	c_B [gL ⁻¹ TSS]	c_{BV} [gL ⁻¹ VSS]
I	3.2 - 5.0	5.1	4.6
II	2.5 - 4.6	5.0	4.5
III	2.0 - 4.9	5.4	4.9
IV	3.6 - 4.8	4.8	4.4

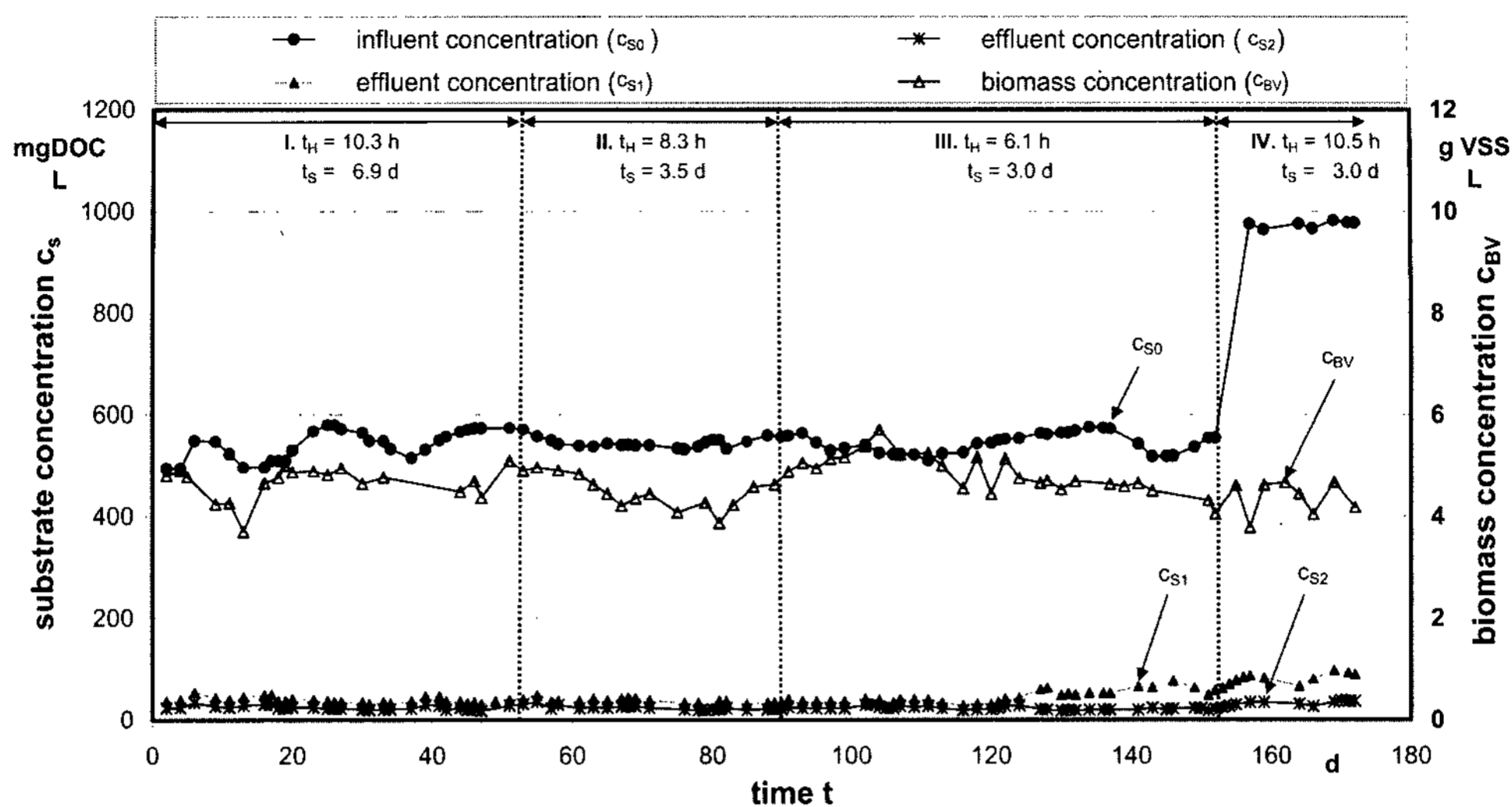


Figure 4. Dependence of the biological degradation of surfactant (APG 2000 UP) in the continuously operated MBR on the HRT and SRT.

24.4 mgL⁻¹ DOC) in the permeate. The degree of substrate removal is > 95%, over the whole experiment (Figure 5).

The substrate effluent concentration of the permeate c_{S1} is lower than that of the reactor c_{S2} , about 1.8 to 3.8 % (aver. 2.6%) until the 155th experimental day and thereafter 5 to 8 % (aver. 6.1%). The difference ranges from 14.1 to 59.4 mgL⁻¹ DOC. This additional removal of substrate results from a physical separation of rest substrate by the membrane filtration. The rest substrate is difficult to biodegrade and has a larger molecular size than the membrane pore size of 40 nm used in this work. The absolute bacteria free permeate can be used for the purpose of water reuse as i.e. rinse water.

During long term filtration runs over 180 days, a rapid decrease in permeate flux by layering and clogging of the membrane was observed. 2 membrane modules were in operation at the same time and the cross-flow rate was kept at $Q_f = 3 \text{ m}^3\text{h}^{-1}$. For both modules the starting permeate flux J_v was high, about $120 \text{ Lm}^{-2}\text{h}^{-1}$, which rapidly decreased to 1/3 of the starting value after a few days. In order to avoid the rapid decrease in permeate flux, one of the both membrane modules was separated and cleaned chemically. The chemical cleaning consisted of alkaline, chlo-

ric, acidic and enzymatic steps. Each step took about 20-30 minutes. The tightly attached bacteria on the membrane surface and the clogged holes affecting J_v could be removed by this chemical cleaning method. This chemical cleaning of the membranes was effective to recover the permeability significantly.

Although the volumetric loading rate B_v increased (Figure 5) with decreasing HRT, the biomass concentration c_B did not increase because the SRT decreased too. The increase in removal rate of excess sludge in order to keep the biomass concentration at constant values resulted in decreased SRT. The average biomass concentration c_B was kept at about 4.9 gL^{-1} VSS and 5.4 gL^{-1} TSS. At the end of the experiment (the 4th period) the B_v increased due to an increase in the substrate influent concentration c_{S1} . Despite similar B_v in the 3rd and 4th period, c_{S2} increased slightly from 22.7 to 34.5 mgL⁻¹ DOC. Compared to that, the increase in c_{S1} is more significant (see Figure 4).

The amount of inert substrate increases with increasing c_{S0} and can be a reason for this increase in c_{S1} . The increase in lysis products from the biomass caused by some operational disturbances in the membrane filtration unit at the end of the 3rd period can be an additional

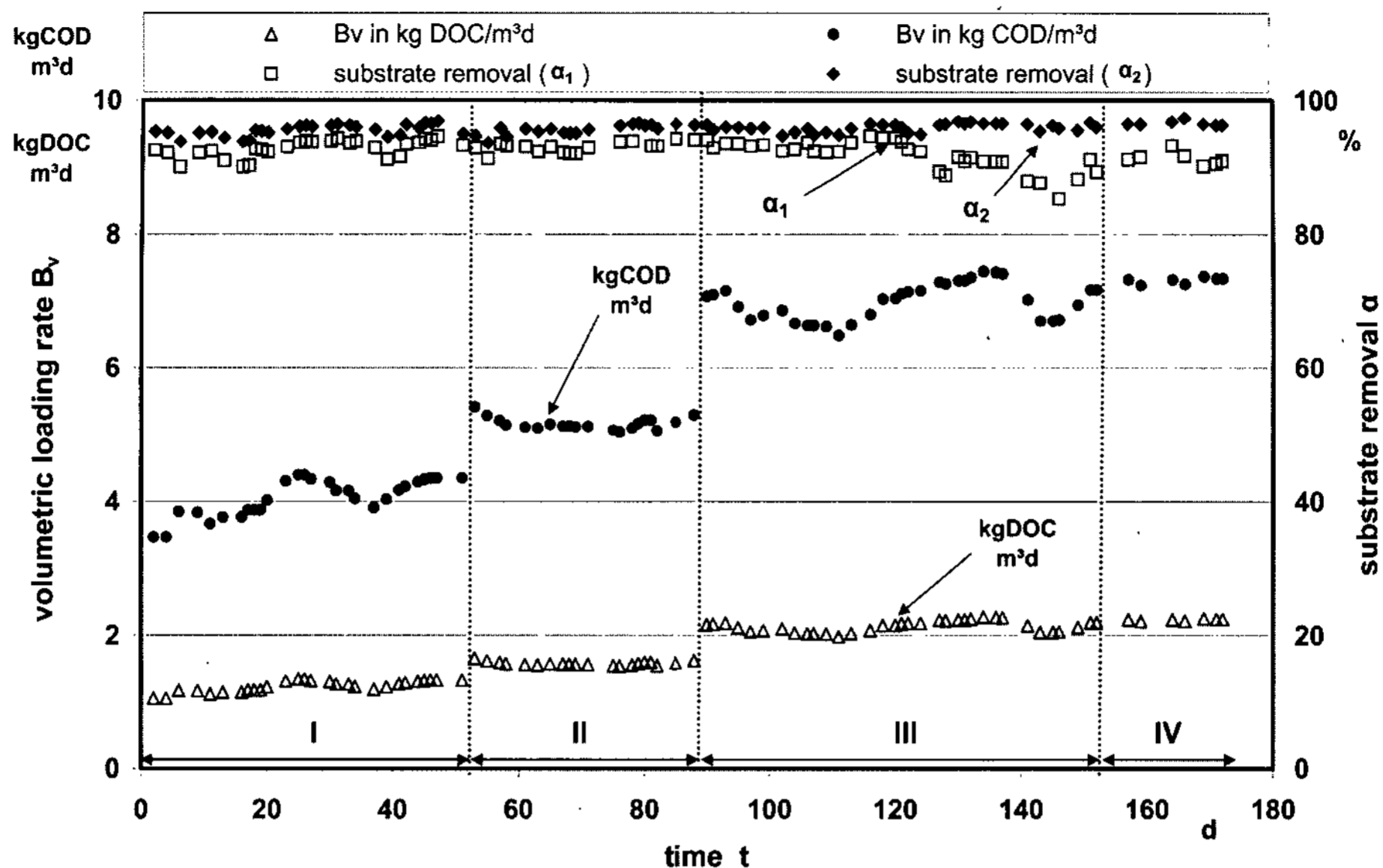


Figure 5. Volumetric loading rates and substrate removal.

reason. The c_{S2} of the permeate confirms the high removal of substrate α by membrane filtration compared to that of the reactor c_{S1} (see Figure 5).

The volumetric loading rates range from 1.1 to 2.3 $\text{kgDOC m}^{-3}\text{d}^{-1}$ (~ 3.5 to $7.4 \text{ kgCOD m}^{-3} \text{d}^{-1}$) calculated with a measured factor of $\text{COD/DOC} = 3.27$. This factor is used as a conversion fac-

tor between COD and DOC for APG 2000 UP. With decreasing HRT, the B_v increases to $7.4 \text{ kgCOD m}^{-3}\text{d}^{-1}$ without serious foaming problem by the surfactant.

The removal of substrate is $\alpha_1 = 90\%$ and $\alpha_2 = 95\%$ (Figure 5). α_1 decreases down to 84% at the end of the 3rd period and in the 4th period due to some operational disturbances and increased

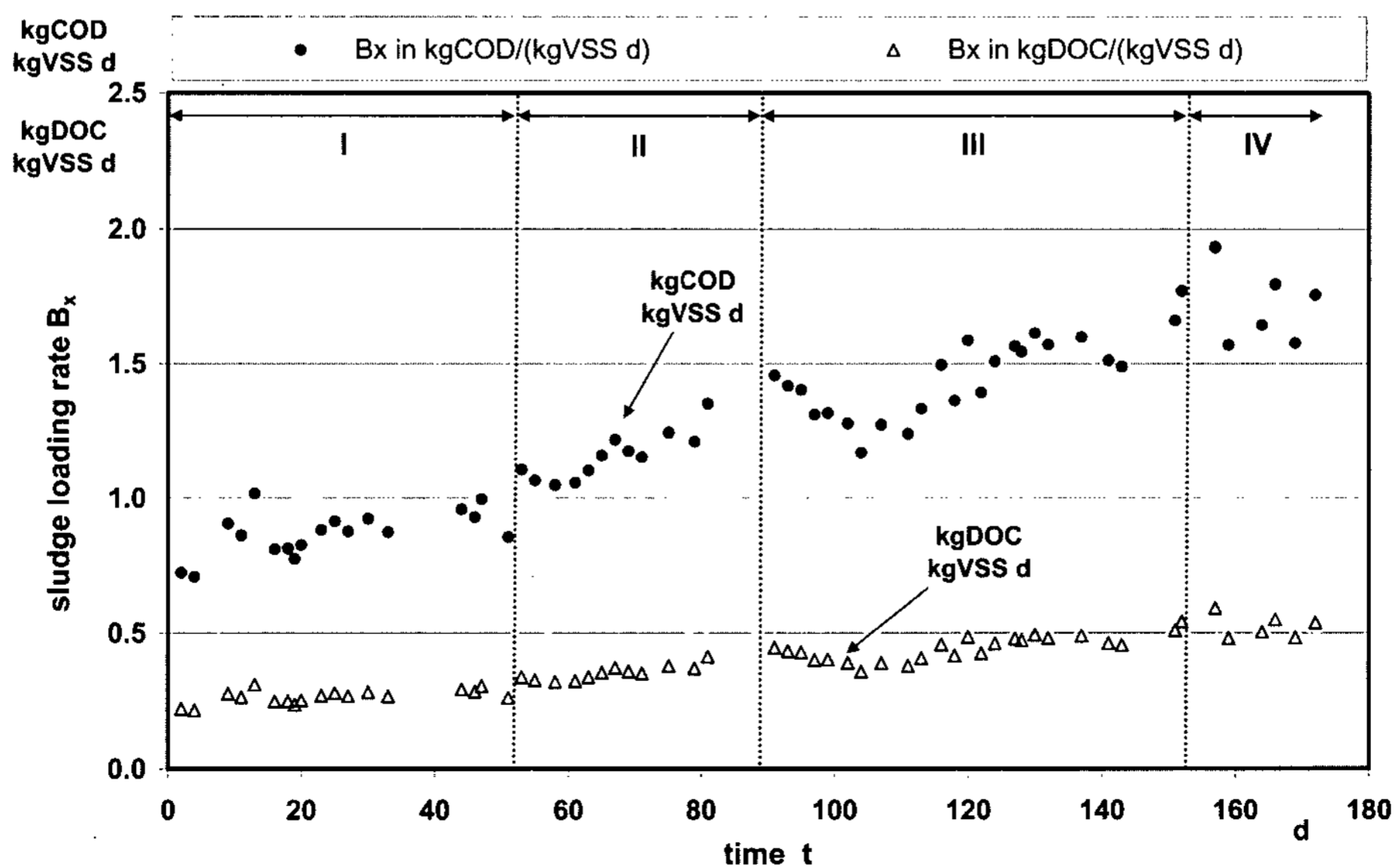


Figure 6. Sludge loading rate B_x in $\text{kgCOD (kg VSS d)}^{-1}$ and $\text{kgDOC (kgVSS d)}^{-1}$.

c_{s0} , respectively. In contrast to α_1 , α_2 remains constant. By comparing α_1 and α_2 it can be noted that very stable effluent concentrations can be obtained by the membrane filtration. Excellent and consistent effluent quality is one of the main advantages of the MBR.

In Figure 6 the increasing B_X with decreasing t_M are presented. The B_X values follow a course very similar to those of the B_V . The biomass concentrations were kept nearly constant by continuous removal of excess sludge even though B_V increased with decreasing t_H . Usually, MBRs are operated with $1.2 < B_V < 3.2 \text{ kgCOD m}^{-3}\text{d}^{-1}$ and $0.02 < B_X < 0.4 \text{ kgCOD (kgVSS d)}^{-1}$. The hydraulic retention time in MBRs in muni-

cipal area is between 2 and 24 h, in industrial applications often over 24 h.¹⁸⁾ Compared to that, in this study B_X is higher as $B_X = 0.7 \text{ to } 1.8 \text{ kgCOD (kgVSS d)}^{-1}$ for $c_B \approx \text{constant}$. The relationship between gVSS and gTSS, c_{BV}/c_B reaches up to 90% to 92% for the whole period.

Besides, an experiment was carried out with increasing SRT. The c_B is increased stepwise from 3.4 to 14.5 gL^{-1} TSS under complete biomass retention by the membrane filtration (Figure 7). In Figure 7 four experimental periods are distinguished with different SRTs, resulting in different biomass concentration c_B . The operating conditions of each period are summarized in Table 3.

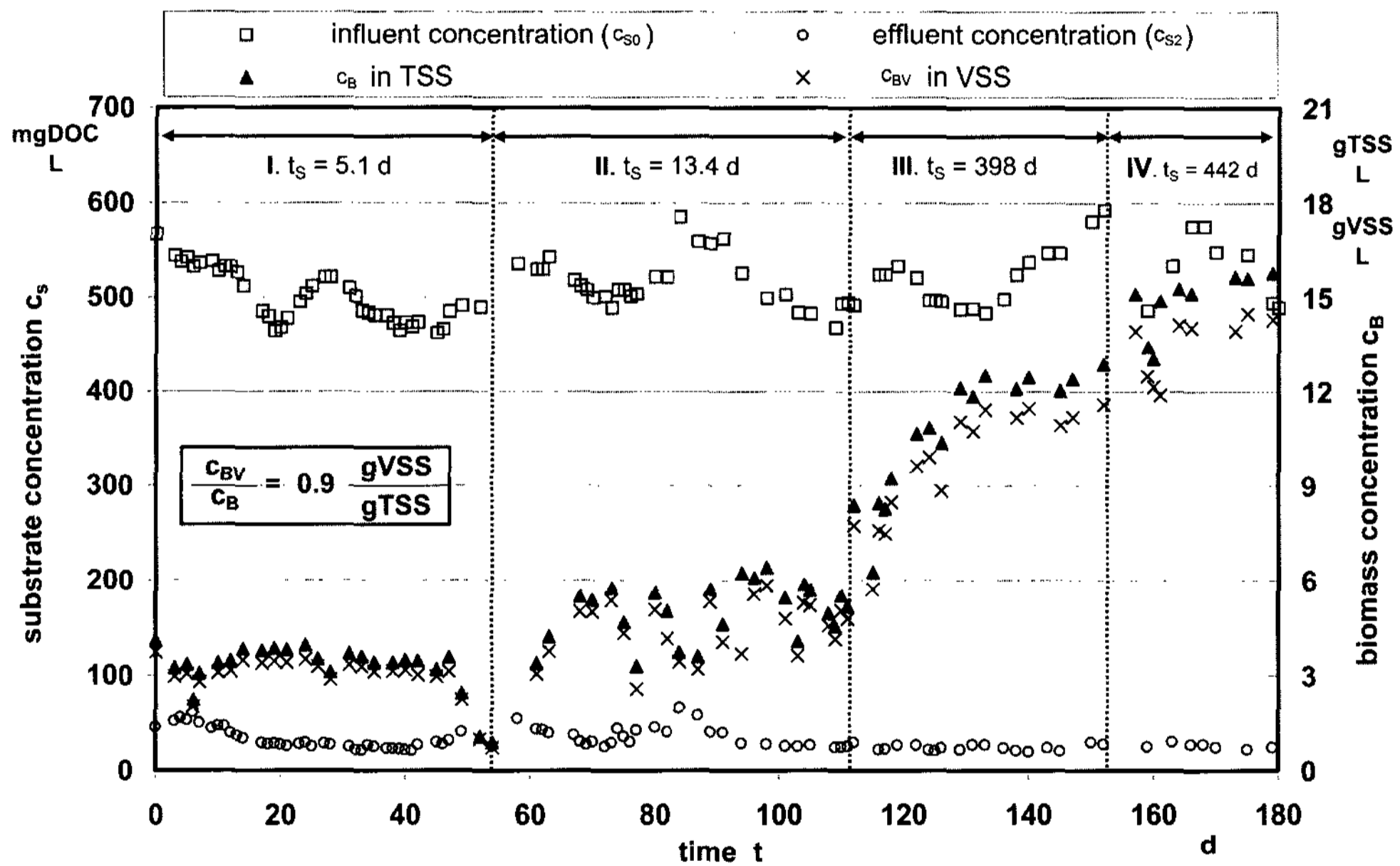


Figure 7. Biological degradation of surfactant (APG 2000 UP) in the continuously operated MBR with increasing biomass concentrations c_B .

Table 3. Operating conditions of the continuously operated MBR varying SRT (pH = 7±0.02 and Temp. = 22.5°C) (for Figure 7).

Period	t_s [d]	c'_{O_2} [mgL^{-1}]	c_{BV} [gL^{-1} VSS]	c_B [gL^{-1} TSS]	k_{La} [h^{-1}]	r_{O_2} [$\text{mgL}^{-1}\text{h}^{-1}$ O_2]	r_{s^*} [$\text{mgL}^{-1}\text{h}^{-1}$ DOC]
I	5.1	2.5 - 7.0	3.0	3.4	52.1	0.081	47.0
II	13.4	2.0 - 6.0	4.3	4.9	36.6	0.086	46.9
III	398	2.0 - 6.5	9.7	10.7	30.0	0.120	48.0
IV	442	2.6 - 7.3	13.3	14.5	26.6	0.131	51.6

* regarding permeate as effluent

In order to reach the higher $t_s > 300$ days and $c_B > 12 \text{ gL}^{-1}$ TSS, the output of surplus sludge had to be stopped except for sampling, which also had to be reduced to the minimum possible.

The c'_{O_2} was controlled at $c'_{O_2} > 2 \text{ mgL}^{-1}$. The influent flow rate was constant with $Q_0 = 1.3 \text{ Lh}^{-1}$ and therefore the HRT was almost constant at $t_H = 10$ to 10.2 h . The small change in HRT was caused by changes in reactor volume V_R due to changing stirrer speed n caused by the surface aeration system.

During the entire experimental period, the c_{S0} of the synthetic wastewater was kept at a value of $c_{S0} \approx 500 \text{ mgL}^{-1}$ DOC. The non-ionic APG 2000 UP was biologically degraded, achieving a $> 90 \%$ where the c_{S2} were stable at $20 < c_{S2} < 50 \text{ mgL}^{-1}$ DOC. The high degree of biological degradation of the surfactant was obtained independent of the SRT. If operating with higher SRT over long terms, then the activity of single cell can be significantly decreased. But in this study the active organic portion of biomass as VSS seemed to not be affected by long SRT and higher c_{BV} up to 13.3 gL^{-1} VSS.

The mass transfer coefficient of oxygen k_{LA} in a stirred reactor is affected by many process parameters (e.g. energy input and flow rate) and the physical parameters (e.g. density, viscosity and surface tension).¹⁹⁾ In the MBR the biomass concentration c_B can significantly increase to high values, which decrease the k_{LA} and have negative influence on aeration of the reactor (see Table 3). Not only the higher c_B but also the stirrer speed can seriously influence k_{LA} .

The increase in SRT leads to a decrease in sludge loading rate, B_X , under constant B_V by constant c_{S0} and HRT. The B_X decreased from $B_X = 2.3$ to $0.5 \text{ kgCOD (kgVSS d)}^{-1}$ with increasing SRT from $t_s = 5.1$ to 442 days. An increasing SRT is combined with a simultaneous increasing c_B and decreasing B_X .¹⁰⁾

The increase in SRT led to two effects: a decrease in the mass transfer coefficient k_{LA} and an increase in the oxygen consumption rate (see Table 3). Thus, the air flow rate had to be

increased to supply enough oxygen. The high bacteria concentration reduces the oxygen mass transfer coefficient k_{LA} , which would result in lower c'_{O_2} in the reactor.¹⁶⁾ As t_s increased, so did the oxygen consumption rate r_{O_2} . The rate measured with the gas analyzer was considerably higher at high bacteria concentrations due to an increased lysis and endogenous respiration rate. If no increase in bacteria lysis and endogenous respiration occurred at the higher bacteria concentration, then the bacteria concentration in the reactor would have been 19 times higher. A biomass concentration of $c_B \approx 270 \text{ gL}^{-1}$ TSS would be expected using the yield coefficient found at low SRTs ($Y_{B/S} = 0.95$).

With increasing SRT and decreasing sludge loading rate B_X the biomass for endogenous respiration increases.¹⁰⁾ The increasing SRTs mean that the surplus sludge decreases, which is linked to saving disposal costs of surplus sludge, which may compensate for the added costs resulting from the low k_{LA} . This operation with high SRT is economically interesting for decentralized wastewater treatment plants, where the disposal costs for excess sludge would be too high, combined with inadequate infrastructure for the collecting, transfer and disposal of the sludge. Therefore, for the decentralized WWTP with small size of WWTP the operation of the MBR can be recommended.

SUMMARY AND CONCLUSIONS

A bioreactor coupled with membrane filtration has been used to investigate the aerobic biodegradation of the surfactant APG 2000 UP. The following results were obtained:

- An aerobic treatment of wastewater containing foam active substances (non-ionic surfactant - APG 2000 UP) was successfully carried out. The surface aeration using the PLR supplied sufficient O_2 for substrate removal and bacteria growth and avoided foam development during aeration resulting in significant increase in the operational safety of the treatment of wastewater containing foam active surfactant. The cross-

flow membrane filtration improved the separation of the bacteria still loaded with surfactant in the collecting container.

- High degrees of biodegradation of the surfactant of nearly 95% were obtained at high volumetric loading rates up to $7.4 \text{ kgCOD m}^{-3}\text{d}^{-1}$ and at sludge loading rates up to $1.8 \text{ kgCOD (kgVSS d)}^{-1}$ for $c_B \approx \text{constant}$.
- The special characteristic of membrane filtration, the complete retaining of bacteria, enables high biomass concentrations up to 14.5 gL^{-1} TSS to be obtained, resulting in very high SRT up to $t_S = 442$ days. The biological degradation of the non-ionic surfactant achieved a high degree of mineralization $\alpha > 90\%$. The excess sludge can be reduced to $Y_{B/S} \approx 0.05 \text{ gTSS (gDOC)}^{-1}$ for $t_S = 442$ d. This is only about 5% of the sludge obtained at low SRT. In contrast to these advantages due to the reduced excess sludge production, the disadvantages of increased energy consumption at higher c_B due to the extremely high O_2 requirement and the decreased k_{LA} have to be taken into account.

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