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The sequencing batch reactor as an excellent configuration to treat wastewater from the petrochemical industry

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The sequencing batch reactor as an excellent configuration to treat
 wastewater from the petrochemical industry

- 3
- Short title: The SBR as an excellent configuration to treat wastewater from
  the petrochemical industry
- 6
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#### 20 Abstract

21 In the present study, the influence of a changing feeding pattern from continuous to

22 pulse feeding on the characteristics of activated sludge was investigated with a

23 wastewater from the petrochemical industry from the harbour of Antwerp. Continuous

seed sludge, adapted to the industrial wastewater, was used to start up three lab-scale

sequencing batch reactors. After an adaptation period from the shift to pulse feeding, the

effect of an increasing organic loading rate (OLR) and volume exchange ratio (VER)
were investigated one after another. Remarkable changes of the specific oxygen uptake

rate (sOUR), microscopic structure, sludge volume index (SVI),  $SVI_{30}/SVI_5$  ratio, and

29 settling rate were observed during adaptation. sOUR increased 2 to 5 times and

30 treatment time decreased 43% in 15 days. Stabilization of the SVI occurred after a

31 period of 20 days and improved significantly from 300mL.g<sup>-1</sup> to 80mL.g<sup>-1</sup>. Triplication

32 of the OLR and VER had no negative influence on sludge settling and effluent quality.

Adaptation time of the microorganisms to a new feeding pattern, OLR and VER was

34 relatively short and sludge characteristics related to aerobic granular sludge were

35 obtained. This study indicates significant potential of the batch activated sludge system

36 for the treatment of this industrial petrochemical wastewater.

37 Keywords: activated sludge adaptation; carbon removal kinetics; sequencing

38 batch reactor; settling characteristics; specific oxygen uptake rate

### 39 Introduction

40 The continuous-flow conventional activated sludge system (CAS) is the main type of 41 biological wastewater treatment system for the removal of carbon, phosphorus and 42 nitrogen from domestic and industrial wastewater. CAS has been successful for the 43 treatment of various types of wastewater, although the system is well known for sludge

44 settlement limitations due to the presence of filamentous microorganisms (Martins et al.

- 45 2004).
- 46

47 The feeding pattern was mostly investigated to prevent filamentous bulking and to 48 obtain better settling properties (Chudoba et al. 1973). In earlier research, using 49 synthetic wastewater with acetate, bactopeptone or glucose as carbon source, it was 50 demonstrated that changing the feeding pattern significantly improved the settling 51 characteristics of AS (Ciggin et al. 2011; Cubas et al. 2011; Guo et al. 2014). All studies 52 described better settling properties when the feeding time was as short as possible (pulse 53 feeding) and the substrate gradient was as high as possible. More in detail, the research 54 of Martins et al. (2004) showed clearly the impact of the feeding time on the sludge 55 characteristics using acetate as substrate. Promoting a strong substrate gradient in the SBR with an aerobic fill time ratio (AFTR) lower than 5.4% resulted in good settling 56 sludge (SVI  $< 120 \text{ mL.g}^{-1}$ ). Whenever acetate was added in a limiting rate with an 57 58 AFRT > 6.2%, a condition in which acetate concentration in the reactor was always very low, the sludge settling deteriorated (SVI between 150 mL.g<sup>-1</sup> and 500 mL.g<sup>-1</sup>). To 59 conclude, sludge settling could be improved by changing the feeding strategy to a pulse 60 61 feed in this research. Ciğgin et al. (2011) reported a SVI of almost 500 mL.g<sup>-1</sup> during operation under continuous feeding. After a shift from continuous to pulse feeding, the 62 sludge settling property quickly improved to a SVI  $< 120 \text{ mL.g}^{-1}$ . 63 64

- 65 Since the 1970s, a real revolution happened in wastewater treatment processes.
- Advanced bubble diffusers were developed and more energy efficient material, like 66
- 67 pumps, valves and mixers were designed. Also PLC's and automation software were
- developed. Thanks to this development, integrated real time control of the sequencing 68
- 69 batch reactor (SBR) became possible (Wiese et al. 2005; Simon et al. 2006; Shaw et al.
- 70 2009; Dries 2016). Despite this industrial development, the real success of the SBR
- 71 system remains off (Artan et al. 2001).
- 72 Different reasons can be formulated for the absence of a general accepted use of the
- 73 SBR technology. Firstly, in the authors opinion, a "lack of knowledge" is still present in
- 74 literature about the treatment of industrial wastewater with SBRs. Secondly inhibition
- 75 of sludge due to toxic components in the influent is discussed as a reason to not use
- 76 SBRs (De Schepper et al., 2010; USEPA, 1991). Furthermore, the CAS configuration is
- 77 seen as a safe operation mode in industry. Although different disadvantages, like poor
- 78 settling characteristics, the absence of flexible process control and low performance for 79
- shock loading are known, the system is still widely used. Lastly, the role of the feeding 80
- pattern for the treatment of industrial wastewaters is a topic that is still largely
- 81 unexplored in literature which is in contrast to the amount of studies on synthetic
- 82 wastewater. However, this could deliver important information about the operation
- 83 mode of treatment systems.
- 84 The objective of the current study is formulated in function of the reasons mentioned
- 85 above. Firstly, adaptation of AS from continuous to batch feeding for industrial
- 86 wastewater was investigated. After the adaptation period, the total performance of the
- 87 system was investigated. The sludge selected, originated from a chemical company in
- 88 the harbour region of Antwerp (Belgium), and was characterized by poor settling 89 properties.
- 90 The most important research gaps that can be filled with this research are (i) Can AS
- 91 adapt to a changing feeding pattern of petrochemical industrial wastewater and how
- 92 long does it take to adapt? (ii) How will the microbial kinetics and the physical
- 93 variables changes during adaptation? (iii) Can the treatment efficiency be increased by
- 94 changing the organic loading rate (OLR) and the volume exchange ratio (VER)?

#### **Methods** 95

#### 96 **Reactor set-up**

- 97 Experiments were conducted using three identical, completely automated lab-scale
- 98 SBRs. Respectively called SBR 1, 2 and 3. The working volume of the reactors was
- 99 13L with a VER of 30% to 60% and a fill time ratio lower than 1%. Every cycle, 4 to
- 100 8L of wastewater was added to each SBR with an internal diameter of 230 mm.
- 101 A Siemens (Germany) programmable logic controller (PLC), type CPU 319-3PN/DP,
- 102 controlled the reactor. Configuration of this PLC was done with Siemens Simatic Step7
- 103 software. LabViewTM (National Instruments, Austin - Texas, United States of
- 104 America) was used as user interface to change reactor settings and to visualise the
- 105 process. This software was also used for data capture and data transfer to MS Excel.
- 106 The dissolved oxygen (DO) concentration in the reactors was monitored with a Hach
- 107 Lange LDO sc sensor. pH and oxidation reduction potential (ORP) were measured in

- 108 the reactors with Jumo TecLine sensors. Sensor signals were transferred by a process
- 109 field bus (profibus) from a Hach Lange sc 1000 module to the PLC.

#### 110 *Reactor operation*

111 The reactors were operated in three different stages, described in Table 1. At the

beginning of stage I, reactors were inoculated with the industrial seed sludge. During

113 stage I, adaptation of the sludge to a new feeding pattern was investigated. Sludge was

fed once a day with 4L of wastewater. At the end of stage I, the sludge of the three

reactors was mixed and divided again in the reactors. In stage II the OLR was varied between the reactors. The operation of SBR 1 was not changed, compared to stage I.

The cycle time of SBR 2 and 3 was lowered to have 2 and 3 feedings a day of 4L each

118 with a corresponding number of 2 and 3 cycles a day respectively. This resulted in a

doubling and tripling of the organic loading rate for SBR 2 and 3. At the end of this

- 120 period, sludge of SBR 2 and 3 was mixed and divided again in SBR 2 and 3. After the
- 121 mixing, both reactors were subjected to a different VER. SBR 2 was operated with 2

122 cycles a day with each feeding 4L of wastewater. SBR 3 was operated with 1 cycle a

123 day with a feeding volume of 8L. Reactor 1 was not further used in stage III.

	Operation days	Reactor	Phase (duration (min))							
Stage			Pre-aeration	Feeding (with mixing)	Aerobic reaction	Excess sludge withdrawal	Settling	Effluent discharge	Total cycle time (h)	Feeding volume/ cycle
Stage I	154	SBR1 SBR2 SBR3	30	2	1283	3	120	2	24	4 L
Stage II	220	SBR1	30	2	1283	3	120	2	24	4 L
		SBR2	15	2	639	2	60	2	12	4 L
		SBR3	10	2	425	1	40	2	8	4 L
Stage	75	SBR2	15	2	639	2	60	2	12	4 L
ĪĪ		SBR3	30	4	1280	4	120	2	24	8 L

124 Table 1. Operating conditions of the lab-scale SBRs during stage I, II and III

125

126 Dissolved oxygen concentration in the reactors was controlled between a lower and

upper DO set point of 1.5 and 5.0 mg  $O_2$ .L<sup>-1</sup> respectively. In Labview<sup>TM</sup>, data pairs of

time and DO concentration were made during the air-off period to calculate the oxygen

129 uptake rate by performing a linear least-square regression analysis.

130 The pH in the reactors was kept between 7.7 and 8.8, using a sodium hydroxide solution

131 of 3 mol.L<sup>-1</sup>. The SRT was kept constant at 30 days, which is the same SRT as in the

132 full scale industrial plant. SRT was regulated by removing a volume of 430 mL excess

133 sludge every day. Temperature was kept at room temperature which was always

134 between  $18^{\circ}C$  and  $22^{\circ}C$ .

#### 135 Sludge & influent

- 136 The seed sludge and influent wastewater used in this study were obtained from a
- 137 petrochemical company located in the harbour region of Antwerp. The full scale
- 138 WWTP is a continuously fed AS system. The sludge retention time (SRT) of this
- 139 WWTP is kept at 30 days for optimal carbon removal. The food to mass ratio (F/M) of
- 140 the industrial installation is approximately 0.25 kgCOD.kgMLVSS<sup>-1</sup>.d<sup>-1</sup>.

141 During this study, thirteen different influent batches were used. The batches were taken

142 from a buffer tank before AS treatment. Table 2 summarizes the most important

143 parameters of the influent wastewater used in the current study. All influents were

144 stored at 4°C to minimize biological reaction, but were given time to equilibrate to 145 laboratory temperature before feeding to the reactors.

145 lab 146

> Fotal nitrogen (mg N.L<sup>-1</sup>) Conductivity (mS.cm<sup>-1</sup>) sCOD (mg O<sub>2</sub>.L<sup>-1</sup>) PO4-P (mg P .L<sup>-1</sup>) Chloride (mg.L<sup>-1</sup>) COD (mg O<sub>2</sub>.L<sup>-1</sup>) Batch Stage day F 1 1 - 39 1900 1850 10.4 0.42 3100 13.1 8.00 2 40 - 87 1680 1640 10.6 0.29 3350 11.1 7.50 I 88 - 144 1728 5.0 3250 6.95 3 1680 0.35 11.3 4a 144 - 154 1842 3500 7.08 1820 3.0 0.50 12.0 4b 155 – 181 182 – 212 1500 1480 0.9 0.70 2000 12.1 7.10 5 6 213 - 244 1890 1820 0.50 3000 6.80 0.5 13.1 7 7.30 Ш 245 – 275 1940 1796 0.3 1.00 3500 12.8 4000 8 276 - 313 1680 1625 0.6 1.10 13.7 7.02 9 314 - 343 1580 0.90 3500 10.1 1600 0.6 7.15 10 344 – 374 1252 1180 0.4 1.20 2400 9.5 7.47 375 - 402 11 1504 1464 0.6 1.35 3500 12.0 7.53 Ш 12 403 - 433 1290 1196 0.7 1.30 3550 12.2 7.82 950 7.41 13 434 - 450 996 0.6 1.30 3550 12.4 Average and standard 1600 1544 2.6 0.8 3246 7.3 11.9 deviation ± 285 ± 283 ± 3.7 ± 0.4 ± 530 ± 1.2 ± 0.3

147 Table 2. Composition of the influent batches used during stage I, II and III

148 Nutrients were added to satisfy the nutritional requirements of biomass in a ratio of

149 100:5:0.8 as COD/N/P (Eckenfelder et al. 2008). To avoid nitrification and associated

150 oxygen consumption, nitrogen was added as  $KNO_3$  instead of the commonly used  $NH_4^+$ .

151 Phosphorus was added as K<sub>2</sub>HPO<sub>4</sub>.

#### 152 Analytical methods

- 153 The chemical oxygen demand (COD) was measured with Hanna Instruments (HI)
- 154 (Belgium, Temse) COD Tests HI 93754A-25 low range and HI 93754B-25 medium
- range tubes. Total nitrogen was measured with the HI 94767A-50 test and chloride
- 156 concentration with the HI 3815 test kit, both from Hannah Instruments. pH and
- 157 conductivity were measured with the HI 9023 microcomputer pH meter, respectively HI
- 158 9033 multi range conductivity meter. Phosphorus was measured using PhosVer3
- 159 powder pillows from Hach Lange (Belgium, Mechelen).
- 160

161 Sludge volume (SV), sludge volume index (SVI), mixed liquor suspended solids

- 162 (MLSS) and mixed liquor volatile suspended solids (MLVSS) were measured in
- 163 accordance with the procedures described in Standard Methods for the Examination of
- 164 Water and Wastewater (1998). Settling rate, SV and SVI were determined with a 1L
- 165 graduated cylinder with a height of 34.5cm and an internal diameter of 6.1cm. Sludge
- samples were always taken at the end of the aeration step and before the settling phase.
- 167 So sludge was always in an endogenous state.
- 168

169 Dissolved Organic Carbon (DOC) was analyzed during the experiments with a Sievers

- 170 InnovOx Laboratory Total Organic Carbon Analyzer. All samples were filtered before
- 171 they were analyzed using glass microfiber filters of  $1.2\mu m$ .

#### 172 Image analysis

173 Image characterization of the AS was performed with a Motic BA 310 microscope.

- 174 Endogenous sludge, taken before the settling phase, was used. Images were taken with
- an EF-N Plan 10x0.25 ocular.

#### 176 Offline batch respirometric experiments

177 To investigate the variation of the AS kinetics in function of time during all stages,

178 offline batch respirometrie experiments were conducted. During the test, wastewater

- 179 was fed to endogenous excess sludge of the SBRs in a VER of 30% to compare the
- 180 substrate removal rate. During the test, filtered samples were taken in function of time
- 181 to analyse the removal of DOC. As mentioned in Kovarova-Kovar and Egli (1998), it
- 182 can be assumed that the concentrations of individual substrates in a complex mixture
- decrease linearly until the substrate is exhausted. Thus, the overall DOC removal rate
- 184 will be the sum of the individual removal rates that are still present in the mixture.
- 185 Those multicomponent DOC removal kinetics were described by the Eckenfelder186 equation (Eckenfelder et al. 2008):

$$\frac{S}{S_0} = e^{\frac{-K.X.t}{S_0}} \tag{1}$$

187 where X is the biomass concentration (mgMLVSS.L<sup>-1</sup>), t is time (d), K is a first order 188 rate coefficient (d<sup>-1</sup>).  $S_0$  = start DOC substrate concentration at  $t_0$  (mg.L<sup>-1</sup>) and S = DOC 190 substrate concentration at time t (mg.L<sup>-1</sup>)

- 189 substrate concentration at time t (mg. $L^{-1}$ ).
- 190 This equation describes in a simplified way the removal of substrate (expressed as 191 DOC) in a multicomponent mixture. The above equation is experimentally proven by
  - 6

192 Grau et al. (1975). K values were estimated via non-linear regression (equation 1) using193 GraphPad software.

### 194Results and discussion

#### 195 **Reactor performance**

196

197The evolution of the MLVSS concentration in all reactors are shown in Figure 1. From198the start of stage I to the end of batch 1 (day 1 - 39) the MLVSS was  $2.76 \pm 0.18$ 199gMLVSS.L<sup>-1</sup>. After switching to a new influent batch a decrease of MLVSS in all200reactors was observed, which can be explained by the lower influent COD (Table 2).201For the 3<sup>the</sup> and 4<sup>the</sup> batch, a stabilization in the MLVSS concentration was observed.202For stage I, F/M ratio was  $0.25 \pm 0.05$  kgCOD.kgMLVSS<sup>-1</sup>.d<sup>-1</sup> (Figure 2).

203

204 During stage II, different OLRs were imposed in the reactors (Figure 2 B). 7 different

influent batches were fed to the reactors (Table 2). Depending on the influent batch
 used, significant changes were observed in MLVSS concentrations. Higher sludge

207 concentrations were observed in SBR 2 and 3 compared to SBR 1. This can be

208 explained by the higher OLR in SBR 2 and 3. Changing the OLR influenced the F/M

ratio significantly. At the beginning of stage II, F/M increased 2 and 3 times for SBR 2

and SBR 3 respectively, as a result of the increased OLR. Due to sludge growth in SBR

211 2 and 3, F/M decreased slowly. The average F/M during stage II was  $0.32 \pm 0.06$ 

212 kgCOD.kgMLVSS<sup>-1</sup>.d<sup>-1</sup>, 0.38  $\pm$  0.03 kgCOD.kgMLVSS<sup>-1</sup>.d<sup>-1</sup> and 0.57  $\pm$  0.04

213 kgCOD.kgMLVSS<sup>-1</sup>.d<sup>-1</sup> for SBR 1, 2 and 3 respectively.

214 In period III, different VER were applied to SBR 2 and 3. During adaptation to the new

215 VER, outspoken changes occurred in the MLVSS concentration. At the end of the

feeding batch 11 (day 402), SBR 2 had a MLVSS concentration twice as high as SBR 3.

217 During batch 2 and 3, the same trend was observed. The decrease of MLVSS in both

reactors could be explained by the lower COD of the influent. As a result of this,
different F/M ratios were observed. SBR 2 evolved at the end of stage III to a F/M ratio

different F/M ratios were observed. SBR 2 evolved at the end of stage III to a of  $0.29 \text{ kgCOD.kgMLVSS}^{-1}$ .d<sup>-1</sup> and SBR 3 to  $0.66 \text{ kgCOD.kgMLVSS}^{-1}$ .d<sup>-1</sup>.

221

222 During all stages a good COD effluent quality was observed. An average COD of  $62 \pm$ 

14 mgO<sub>2</sub>.L<sup>-1</sup>, 29  $\pm$  10 mgO<sub>2</sub>.L<sup>-1</sup> and 31  $\pm$  11 mgO<sub>2</sub>.L<sup>-1</sup> for stage I, II and III respectively.

Although wide variation of OLR and VER were imposed to the reactors, a stable

effluent quality was achieved which was well below Flemish discharge limits i.e. 125

226 mgO<sub>2</sub>.L<sup>-1</sup>.





Figure 1: Evolution of the MLVSS in the three SBR systems and the influent COD, a

229 dotted line represents a new feeding batch.



230 231

Figure 2: Evolution of (A) the average F/M and (B) the OLR for every influent batch in each SBR. A dotted line represents a new feeding batch. 232

- Sludge activity 233
- 234

235 A respirogram showing the evolution of the specific OUR (sOUR) was constructed

- 236 during each cycle to monitor the evolution of the sludge activity. A typical (
- 237

238 Figure 3A) consisted of 4 distinctive sOUR levels, representing four different

- 239 carbon removal rates. After the fourth level, the sludge reached the endogenous
- 240 state. The average sOUR values for level 1, 2, 3 and 4 in
- Figure 3A are respectively 43.4, 13.1, 5.6 and 2.6 mg  $O_2$ .(gMLVSS.h)<sup>-1</sup>. The 241
- endogenous respiration rate was 1.6 mg O<sub>2</sub>.(gMLVSS.h)<sup>-1</sup>. The corresponding DOC 242
- removal is expressed as  $S/S_0$  in this figure. 243



244 245

Figure 3: (A) Typical specific oxygen uptake rate profile for the chemical
wastewater during a SBR cycle. 4 sOUR levels are identified. (B) Evolution of the
average sOUR for the different rate levels during the first 16 batch feeding cycles
for 1 SBR.

250

For every cycle an average sOUR value was calculated for each level defined in

Figure 3A.

Figure 3B gives an overview of the evolution of these average sOUR values during the first 16 cycles. During adaptation of the continuous sludge to batch feeding, significant

first 16 cycles. During adaptation of the continuous sludge to batch feeding, significant
 changes in the sOUR profile were observed. Most changes were observed during the

256 first fifteen cycles, after which stabilization occurred (results not shown).

257 During the first 15 cycles, the sOUR for the most rapidly treatable components (level 1)

increased 5 times (36 to 190 mg  $O_2.(gMLVSS.h)^{-1}$ ). For the second (level 2), third

(level 3) and fourth (level 4) sOUR level, sOUR increased 2 times. In combination with
the higher activity, the time to reach the endogenous rate decreased by 43.9% from 13.1
h to 7.36 h. A similar trend was observed in all 3 SBRs.

262

### 263 Carbon removal kinetics

264

265 DOC removal kinetics were determined during the three different stages using equation 266 1. The seed sludge had a DOC removal rate coefficient K of  $0.058 \text{ d}^{-1} \pm 0.003 \text{ d}^{-1}$ 

1. The seed sludge had a DOC removal rate coefficient K of  $0.058 \text{ d}^{-1} \pm 0.003 \text{ d}^{-1}$ (Figure 4). After 39 days, this value increased 17.5 times to 1.01  $\text{d}^{-1} \pm 0.11 \text{ d}^{-1}$ . The

increased DOC removal rate corresponds to the significant increase observed in the

source levels (Figure 3B). As shown in this research and also mentioned before in

270 literature by Grau et al. (1975) this equation can be used to describe the kinetics of a

271 multicomponent substrate removal by a mixed culture. As shown in Figure 4, DOC

kinetics doesn't change anymore during stage II and III. As a conclusion it can be

273 noticed that changing the OLR and VER, which leads to different F/M ratio's, did not

274 influence the removal kinetics for this wastewater.



🗔 seed sludge 🎆 SBR 1 🛄 SBR 2 🕅 SBR 3

Figure 4: DOC removal kinetics for different influent batches in the 3 SBRs. Error bars
represent the standard error.

# 278 Sludge settling

#### 278 279

280 Increasing the substrate gradient in the system by decreasing the length of the feeding 281 phase from continuous to pulse feeding had a strong positive impact on the sludge settleability. Figure 5 shows the evolution of the average SVI, after 30 minutes settling 282 283 for the 3 SBRs. The most significant changes in the SVI evolution were found between day 8 and 17. The SVI<sub>30</sub> gradually decreased in the three SBRs from 300 mL.gMLVSS<sup>-</sup> 284 <sup>1</sup> on day 1 to 85 mL.gMLVSS<sup>-1</sup> on day 20. After day 20, the SVI<sub>30</sub> improved gradually 285 to a stable value of 40 mL.gMLVSS<sup>-1</sup> at day 120. The SVI<sub>30</sub>/SVI<sub>5</sub> ratio evolved slowly 286 287 from day 20 to 100 to a value that remained stable between 0.85 and 0.95. The settling rate evolved from 0,02 m.h<sup>-1</sup> to a rate between 10 and 13 m.h<sup>-1</sup> in all SBRs 288 289 corresponding to a good settling sludge (Martins et al. 2004).

290

Throughout stage I, good settling sludge was formed. Changing the OLR at the beginning of stage II had no influence on the SVI<sub>30</sub>. During 220 operation days, SVI<sub>30</sub>

stayed almost equal in all three reactors with values between 25 and 45 mL.gMLVSS<sup>-1</sup>.

294 Changing the influent batch had no negative impact on the SVI<sub>30</sub>, although between day

108 and 204 a less favourable  $SVI_{30}/SVI_5$  ratio was obtained. Nevertheless, a settling

rate between 9.5 and 13.8 m.h<sup>-1</sup> was observed in all 3 reactors during the whole stage.

297

298 The  $SVI_{30}$  of the sludge in both reactors remained unchanged after changing the VER in

- stage III. SBR 2 and 3 had a low SVI with values between 45 and 25 mL.gMLVSS<sup>-1</sup>.
- After cycle 400, the  $SVI_{30}/SVI_5$  ratio became 1. A settling rate of 12 to 13.5 m.h<sup>-1</sup> was
- 301 measured in both SBRs.



302

Figure 5: Evolution of the average  $SVI_{30}$  and  $SVI_{30}/SVI_5$  in the three SBR systems, a dotted line represents a new feeding batch. Error bars represent the standard deviation between the three SBRs.

306

#### 307 Sludge morphology

Figure 6 illustrates the morphological changes of the sludge. The first picture in Figure6 shows the seed sludge from the industrial WWTP. The sludge had a loose and

310 irregular structure. After 10 days of SBR operation, more compact flocs appeared. After

311 20 days dark spots were formed. Between day 20 and 80, the formed flocs became

312 larger and darker. After 80 days robust and stable flocs were formed. This

313 morphological adaptation was observed in all three SBRs.

At the beginning of stage II, flocs with a diameter of 100µm were observed in the sludge. During the transition to a new OLR, flocs became bigger in all reactors. At the end of stage II, al reactors had a good settleable sludge (Figure 5). This corresponds to

the well-defined sludge flocs that were observed during the microscopic investigation.

318

During stage III, the microscopic structure of the sludge in SBR 2 and 3 underwent no

320 significant changes, which is in correlation to the sludge settling characteristics. Well-

321 shaped flocs were preserved. In accordance to granular sludge, granule size has to be at

322 least 0.2mm (de Kreuk et al. 2007). Although the floc size of the granules was not

- measured it can be observed that a substantial amount of the flocs had a size bigger than
- 324 0.2mm.
- 325



326 Day 240 Day 375 Day 450
 327 Figure 6: Evolution of the sludge morphology (40x magnified) during the whole
 328 experiment. The white scale bar represent a length of 500µm.

#### 329 Discussion

330

331 In this study the effect of a changing feeding pattern, from continuous to batch/pulse, on 332 the characteristics of AS was studied. Van den Broeck (2009) mentioned a significantly 333 different transition period for different sludge characteristics (SVI, sOUR, microscopic 334 structure) which was also confirmed in this study. Changing the feeding pattern resulted 335 in rapid and significant improvement of the microbial kinetics expressed as sOUR. The 336 adaptation time corresponded to half a sludge age. This is significantly faster than 337 reported in Van den Broeck et al. (2009) who showed an adaptation of approximately 338 20 days for the sOUR with a sludge age of 20 days after changing the feeding pattern 339 and influent composition of domestic AS. Earlier research from Chudoba et al. (1973, 340 1985), Martins et al. (2003), Van den Eynde et al. (1983) and Verachtert et al. (1980) 341 using synthetic wastewater and Van den Eynde et al (1982) using industrial wastewater 342 already stressed the importance of the feeding pattern for the settling characteristics of 343 the sludge which is also confirmed in this study with a real petrochemical industrial 344 chemical wastewater.

345

346 A stable and robust AS system was formed during the first adaptation period. Changing 347 the VER, OLR and influent composition had no significant influence on the settleability of the sludge. During the whole period, SVI values between 25 and 45 mL.gMLVSS<sup>-1</sup> 348 349 were achieved. Carbon removal rate kinetics showed no significant difference between 350 the reactors, but there was a significant improvement during adaptation. In accordance 351 to the F/M ratio, a higher ratio was obtained with the highest VER of 60% and F/M 352 ratios 2 to 3 times higher than in the industrial installation. The bad settling sludge was 353 turned over into a good settling sludge in 20 days which was also achieved in Martins et 354 al. (2004) Martins showed an adaptation time of 7 days for the SVI after changing the 355 feeding pattern from continuous to pulse feeding with a SRT of 10 days. This is 0.7 356 times the sludge age which is almost the same as for this wastewater with a value of 357 0.67. Since the industrial company struggles with bad settling sludge (Figure 5), 358 changing the feeding pattern could mean a beneficial improvement for the settling. The

- obtained settling properties in the present study correlated to the characteristics of granular sludge. As defined in de Kreuk et al. (2007), granular sludge has a settling rate higher than  $10 \text{m.h}^{-1}$  and a SVI<sub>30</sub>/SVI<sub>5</sub> equal to 1 which is also obtained in this study. The observed effects can, to a large extent, be explained by the kinetic selection theory of Chudoba et al. (1973, 1985), who have shown that the activity of microorganisms depends on substrate composition and concentration. This implies that (among many other factors) the feeding pattern in the reactor exerts an important influence on
- 366 microbial dynamics in the AS.
- 367

Growing interest last years is observed for the SBR technology to treat wastewater since
some important benefits can be addressed to the SBR (Mace and Mata-Alvarez 2002):
(i) lower cost than conventional biological treatment methods (ii) lower footprint (iii)
flexible for wide swings in hydraulic and organic loadings (iv) easier to control

372 filaments and settling problems and (v) dynamic process control.

- 373 Nevertheless, the number of SBR reactors is still low in industry, especially in the 374 (petro)chemical industry. In the authors opinion, a lack of information is still present in 375 literature about SBR treatment of this type of wastewater. Mostly fear for sludge 376 inhibition is referred to as a reason to choose for other systems than SBR (USEPA 377 2000). Interestingly, different researchers stated the benefits of the SBRs, compared to 378 the CAS system, to remove difficult degradable (toxic) components. For example, 379 Papadimitrou (Papadimitriou et al., 2009) compared the adaptation of activated sludge 380 for wastewater that contained phenol and cyanide. They clearly showed the superior 381 removal capacity of the SBR system to remove both pollutants. Furthermore, the authors showed better performance to shock loading of these chemicals in the SBR and 382 383 inactivation and disintegration of the sludge with phenol concentrations above 800 384  $mg.L^{-1}$  was seen in the CAS system. In addition, the study clearly showed better settling properties in the SBR. Moreno and Buitrón (2012) (Moreno-Andrade and Buitrón, 385 386 2012) investigated the degradation of 4-methylaniline in a SBR. They demonstrated the 387 ability of AS to adapt to this difficult degradable chemical. In (Moreno-Andrade and 388 Buitrón, 2004) the capability of the SBR is presented to degrade 4-chlorophenol. In the 389 present study it is shown that inhibition of activated sludge was not relevant for the 390 petrochemical wastewater used. The activated sludge showed a good performance even 391 when subjected to triplication of the OLR and doubling of the VER.
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393 To conclude, the researchers have demonstrated that activated sludge from a CAS 394 system could quick and easily adapt to pulse fed activated sludge in a SBR reactor. 395 Furthermore, activated sludge characteristics evolved positively. In this research, lab 396 scale SBR results were compared with the full scale results. Future work has to be done 397 to compare the lab scale SBR results with a lab scale CAS system to exclude effects like 398 different degrees of mixing in the full scale plant, substrate gradients in the perfectly 399 mixed processes, differences in pH or nutrients addition and variable influent 400 composition of the wastewater or different organic load rate or oxygen concentration in 401 the reactor.

### 402 Conclusions

The results of the experiments allow the authors to conclude that batch feeding,
significantly influenced the characteristics of the microbial population of the AS in a
positive way. In SBR systems more advantageous treatment rates and improved settling
characteristics can be achieved in comparison to continuous flow systems with the

- chemical wastewater. As a result of this higher activity due to changing the feeding pattern, the industrial treatment plant could be built more compact and higher wastewater flows could be treated.

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