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Combination of sequencing batch reactor and vertical flow treatment wetlands: A full-scale experience for rum distillery wastewater treatment in a tropical climate

Stéphanie Prost-Boucle^{a,*}, Lucas Pelus^b, Emmanuel Becheau^c, Laurent Cervoise^d, Stéphane Troesch^e, Pascal Molle^a

^a INRAE - French National Research Institute for Agriculture, Food and Environment, Research Unit REVERSAAL, 5 rue de la Doua, CS 20244, 69625 Villeurbanne Cedex, France

^b ODE - Office de l'Eau Martinique, 7 avenue Condorcet, BP 32, 97201 Fort-de-France Cedex, Martinique (FWI)

^c Héritiers Crassous de Médeuil, Distillerie de Fonds Préville, 97218 Macouba, Martinique (FWI)

^d AGRO KEYS, Ravine Plate, 97280 Le Vauclin, Martinique (FWI)

^e ECOBIRD, ZAC des Balarucs, 12 Rue Toussaint Flechaire, 84510 Caumont sur Durance, France

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ABSTRACT

Distillery wastewater is a difficult type of wastewater to treat because of its low pH (pH = 3.3), high chloride and sulfate content, high organic matter concentrations (3.2 gTSS/L, 6.8 gBOD₅/L and 14.6 gCOD/L). Moreover, the rum production is intermittent over the season (5 months per year). The acidity of this type of wastewater, combined with the relatively underbalanced nitrogen concentrations, requires the addition of specific chemicals such as soda solution and urea, when a biological treatment technology is envisioned. In this full scale experiment, located in a tropical zone (Martinique island), the objective was to demonstrate the ability of treatment chain composed of a sequencing batch reactor (SBR) followed by vertical flow treatment wetlands (VFTWs). The objective was to use VFTWs for treatment polishing as well as sludge/water separation and sludge treatment over years. The VFTWs are planted with *Heliconia psittacorum* and *Cyperus papyrus*. The monitoring occurred during three rum production seasons. The treatment chain allows an overall average removal of TSS of 92% and 96% for organic matter (COD and BOD₅). The average annual load applied to the filter in operation was 32 kgTSS/m²/y, which is equivalent to 78 kgTSS/m²/y over the distillation period. The applied load was too high for the plants tested and lead to plant death and surface clogging of the filters. Nevertheless, this experiment allowed specifying the way to operate the whole treatment plant as well as acceptable daily loads that should be applied on the VFTWs. Even if the operation of the filters and the selection of plants in a tropical climate still need to be optimized, this combination of intensive and extensive processes is an effective and sustainable nature-based solution for rum distillery wastewater treatment.

1. Introduction

In rum production, sugar cane is harvested, and crushed or pressed to extract the juice, which is subsequently fermented in tanks. After fermentation, the distillate is collected and the alcohol is separated at the top of a column still. The liquid recovered at the bottom of the column, called the stillage, has to be treated before its release into the environment. This industrial effluent is very acidic (pH = 3 to 5) and contains about 100,000 mg/L of COD [1,2]. Rum distillery wastewater also has high concentrations of chlorides and sulfates. That said, it is a

wastewater that is free of pathogens and heavy metals. The rum production is temporary (4 to 5 months per year) which constitutes a challenge for biological treatment (startup of the process). It is common to count a wastewater production of around 11 L of effluent per liter of rum produced [1].

Even if the carbonaceous compounds in rum distillery wastewater are readily biodegradable, the high concentrations constitute a major challenge, particularly in the fragile natural environments of the Caribbean islands. The distilleries have equipped themselves with facilities to meet treatment requirements [1], which often include

* Corresponding author.

E-mail address: stephanie.prost-boucle@inrae.fr (S. Prost-Boucle).

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approaches such as direct land-application of stillage, use of aerated lagoons, methanization processes, membrane distillation [3] or irrigation of treated stillage or dispersion by sea outfall. However, these solutions are not always satisfactory because of the high infrastructure costs, complicated treatment process, and the fact that the management of the sludge is generally poorly taken into account. More recently, many researches focus on promoting circular economy while treating stillage [4,5].

Treatment wetlands have been successfully used for treatment of high-strength wastewaters [6], livestock wastewater [7], swine farm slurry [8,9], and high-COD and high-N wastewaters in tropical climates [10]. Different combination of treatment wetlands alone or coupled with conventional systems have been widely tested for winery effluent [11, 12] but less experiences for stillage which are more concentrated. Considering the high organic loads to be treated a full treatment of raw stillage with a treatment wetland would require too large area in the Caribbean regions where low slope lands are rare. Therefore, a system coupling intensive (e.g., sequencing batch reactor - SBR) and extensive (e.g., vertical downflow treatment wetland - VFTW) technologies appears preferable. It has been already implemented for winery wastewater [12]. The SBR treats the major part of the dissolved biodegradable compounds (total BOD₅ and COD). The VFTW aims at providing: i) the separation of the sludge produced and the treated water, ii) a completion of the effluent treatment, and iii) drying and mineralization of the sludge over a long period. They observed good removal performances while minimizing clogging risk on the treatment wetlands system in temperate climate. Designing 4 vertical flow systems in parallel with an annual load of 25 kgSS/m²/y on a two months operation per year (equivalent to 150 kgSS/m²/y over the production period), it raises the question of the acceptable load for a 5 months production period a year in tropical climate. When comparing to sludge treatment reed bed, design loads varying from 50 kgTSS/m²/y (temperate climate) to 130 kgSS/m²/y in tropical climate [13] with a full time production period and 6 to more than 10 beds in parallel. Beside sludge treatment reed beds only received the sludge production, while in the winery effluent experience all the water passes through the treatment wetland as a polishing step is targeted. Consequently, we still face a lack of experience to adapt the design (solid loading rates, number of beds, hydraulic loads) according to specific characteristics coming from rum distillery effluent production (influent characteristics, period of production, climate). If the advantages of managing and treating the sludge over a long period on a vertical flow treatment wetland seems relevant, several design and operational questions need to be addressed including issues regarding pH and nitrogen deficient influent that can cause problems specifically during annual start up.

Another important issue regarding treatment efficiency and sustainability of the operation is linked to the choice of plant in tropical climate. Plants need to have a certain degree of stress tolerance (water stress, anoxic stress, phytotoxic stress) while ensuring a fast and homogenous growth to play the mechanical role we are researching in sludge treatment wetlands. Indeed, the mechanical movement of the stems is indispensable to ensure water infiltration and air diffusion within the surface organic deposit for mineralizing the sludge [14,15]. The dense root system ensures homogeneous infiltration of the effluent, which discourages hydraulic short-circuiting. Common reed (*Phragmites australis*) is the most commonly used plant in treatment wetlands in Europe [14], but it is not endemic everywhere and is sometimes even considered an invasive plant. Thus, in tropical climates, the varieties *Heliconia pittacorum* and *Cyperus papyrus* have been investigated for their use in French VF wetlands for wastewater treatment [14,16,17] or sludge treatment [18]. Nevertheless, their use for stillage treatment is not validated.

The objective of this study was, while taking advantage of distillery treatment unit rehabilitation, to precise design and operation mode of a hybrid treatment chain that could ease the management of sludge and allows reusing treated water for banana trees irrigation as rum

production happen in dry season. The process combining SBR and VFTW has proven itself in temperate climates [12] for winery effluent, its adaptation to rum factories and tropical climate is a new field of application. The main objectives of this study are:

- to characterize the stillage and its impact on the treatment process;
- define operational conditions (nitrogen addition, pH control) that ensure good annual restarting of the treatment chain;
- to identify the optimal range of hydraulic, organic, and solids loading for treatment of rum distillery wastewater with VF treatment wetlands;
- to determine the suitability of alternative plants in French VFTW operating in tropical climates.

2. Material and methods

The experimentations took place at Rhum JM distillery, located on the island of Martinique (latitude 14.641528; longitude -61.024174; temperature 22–29 °C all the year; rainfall 1950 mm/y). Considering the high organic loads to be treated (200 m³/d and 3000 kgCOD/d), a system was designed that contains a buffer tank, a SBR and a VFTW as a final step.

2.1. Pretreatment and SBR operation

After pre-treatment by coarse screening to retain the residues of cane fibers, the stillage is discharged into a buffer tank of 850 m³ (storage capacity for 4 days of production). It is fed from Monday to Friday (6am to 8pm) and Saturday morning. It is not a treatment facility: it smoothes out the inflows to the SBR to attenuate peaks in pollutant load and avoid weakening its operation. It is not equipped with a mixing system.

The aeration basin (SBR: 4000 m³ and depth 1.5 to 1.8 m) is the first step in the stillage treatment. In such well-known microorganism-based process, the microbial cultures are not inoculated and develop spontaneously in the form of flocs (sludge). The separation of the sludge from the treated water takes place in the same reactor, by gravity settling. The biological treatment is carried out in four 6h-cycles; each cycle consists of three phases:

- 1 Feeding of stillage and stirring: 4h45 of mixing and aeration by three slow surface turbines (two slow turbines of 37 kW each and one slow turbine of 45 kW);
- 2 Stop aeration and mixing: floc decantation for 30 min;
- 3 Emptying for 15 to 45 min. This is the extraction of the treated effluent (supernatant) to the next stage: the treatment wetlands (VFTWs).

2.2. VFTW operation

The second stage of treatment consists of three vertical flow TWs in parallel (1330 m² each). They are composed of a top layer of 0.40 m of gravel (grain size 2/4 mm), followed by a 0.15 m thick coarse gravel layer (grain size 15/25 mm) and a drainage layer of 0.15 m of pea gravel (grain size 30/60 mm). Filters are in operation at a 3.5/7 days feeding/rest ratio. The selected filter receives four daily batches (SBR cycles) of 50 m³, for a nominal hydraulic loading rate of 0.15 m/d. The water is spread by 6 feeding points per bed. The filtration of total suspended solids (TSS) on the filter leads to the formation of a surface organic deposit. This sludge layer accumulates during the feeding phases and then mineralizes and dries out during the rest phases. The final objective is to use the sludge as fertilizer to be spread on the local crop plots. Design load, depending of the SBR sludge production rate, was expected to 75 kgTSS/m²/y (over the 5 months production period) corresponding to a real annual load of 31 kgTSS/m²/y. To allow drainage and passive aeration from the bottom, a drainage pipe network is implemented at the bottom of the filters. Filters are freely drained to favor passive

aeration. The three filters are planted as follows: 100% *Heliconia psittacorum* on VFTW1, 50%/50% *Heliconia psittacorum*/*Cyperus papyrus* on VFTW2, 100% *Cyperus papyrus* on VFTW3. The loads applied to each of the filters were different, so the combination of two plant species in VFTW2 allows a comparison of the behavior of each plant under the same received load conditions.

2.3. Treatment plant discharge

Local regulations require that the whole treated effluent be reused for irrigation (banana trees). Thus, the totality of the treated wastewater is sent to a storage basin for the irrigation. Since no legal discharge limit is defined, the objective of the research project was to evaluate how far we can go in discharge limits to be guaranteed.

2.4. Follow-ups carried out

The treatment plant and its experimental monitoring started during the 2019 distillation period and include 3 rum productions (2019–2021).

Daily flows are recorded at all points of the treatment plant: SBR inlet and outlet, and VFTW outlet. Online measurements (15 min time step) were done to follow the SBR functioning: water levels in buffer tank and SBR, and temperature, pH, redox, TSS inside the SBR.

Twenty-four hour proportional flow samplings were carried out every two weeks, using refrigerated samplers. Samples are taken at the different points of the treatment: 1) effluents extracted from the buffer tank and sent to the SBR, 2) effluents extracted from the SBR and sent to the VFTWs, 3) treated effluents at the output of the fed filter. They are immediately sent to INRAE laboratory, in coolers maintained at 4 °C, for an arrival within 48 h. The analyses concern the classical parameters of wastewater treatment (COD, dissolved COD, BOD₅, TSS, VSS, N–NH₄, N–NO₂, N–NO₃, TKN, TN, filtered TN, HCO₃⁻, P-PO₄, and TP. According to standard methods [19]). Dissolved (dCOD) is done by measuring COD after filtration at 0.45 μm. Additional parameters related to the quality of the effluent and the experimental nature of this monitoring are also measured: sulfides, chlorides, proteins, sugars.

Some analyses are also performed at the laboratory of Rhum JM distillery, several times a month:

- TSS by filtration and thermo balance (Gibertini Crystaltherm), for the calculation of the mass load;
- Ammoniacal nitrogen (N–NH₄⁺) by colorimetric kit for spectrophotometers (LDS 197845) at different stages of the treatment, in order to verify the availability and consumption of nitrogen by bacteria;
- COD by colorimetric kit for spectrophotometers (HACH LCK 114), to ensure the proper functioning of the treatment plant.

In order to evaluate the quality of the SBR treatment and the suitability of the sludge flocs for settling, the settling test is performed (Mohlman's V30 index [20]). It consists in taking 1 L of effluent from the SBR during the stirring phase and observe the supernatant after 30 min. Poor sludge settling (V30 > 300 mL [21]) will result in a risk of sludge discharge to the VFTWs and clogging of the filters. At the same time, TSS size analysed by laser granulometry (Cilas 1190 instrument) are performed by the INRAE LAMA laboratory on each sample. This measurement determines the size distribution of the sludge flocs formed in the SBR.

2.5. Additives

To control pH in the SBR, due to influent acidity and possible power cuts that stop aeration of the basin, soda solution had been made punctually in 2020, at a rate of 100 to 300 L/d to maintain pH above 6.5. After so no soda addition has been necessary.

Due to nitrogen deficiency for biological activity (see Section 3.1),

nitrogen addition is necessary and usually done in the form of nitrate to avoid overconsumption of oxygen during nitrification. As no such additives are available locally, urea additions were made, in the form of soluble granules (CO(NH₂)₂, 46% N composition, SCIC Martinique mineral nitrogen fertilizer), due to its ease of use (neutral, nontoxic and inexpensive product). 75 kg per day were added since mid-March 2021 (5 days per week), which meets two-thirds of the nitrogen needs for COD degradation.

The additives are poured directly into the SBR at one time per day and during a stirring phase.

2.6. Treatment plant performances

The water balance shows that the inlet and outlet volumes of each structure are similar (see Section 3.1), while considering the measurement uncertainties. The removal yields are therefore calculated on the basis of the concentrations.

In the SBR, the sludge production, also called conversion rate, is an important parameter of the treatment to better design the surface of the VFTW that are limited in surface TSS loads. Sludge production is represented by the biomass growth yield coefficient (*Y*). Due to the particularity of the influent and the high TSS production observed, the actual conversion rate has been determined. The quantity of produced TSS and the quantity of treated BOD₅ are calculated over periods without electrical failure, according to the following equations:

$$TSS_{produced} (kg / d) = TSS_{extracted} + TSS_{accumulated}$$

With:

$$TSS_{extracted} (kg / d) = V_{out} \times C_{out (TSS)}$$

$$TSS_{accumulated} (kg / d) = Volume_{SBR} \times \Delta C_{(TSS)}$$

Where: *V*_{out} is the volume of effluent extracted from SBR and sent to VFTW in m³/d; *C*_{out (TSS)} is the average TSS concentration of the effluent extracted from SBR and sent to VFTW in g/L; *Volume*_{SBR} is the volume of effluent into the SBR in m³; $\Delta C_{(TSS)}$ is the average variation of TSS concentration in SBR over the time period considered.

And:

$$BOD_{5treated} (kg / d) = V_{in} \times (C_{in (BOD5)} - C_{out (BOD5)})$$

Where: *V*_{in} is the volume of influent (stillage) sent to SBR in m³/d; *C*_{in (BOD5)} is the BOD₅ concentration of the raw effluent sent to the SBR in g/L; *C*_{out (BOD5)} is the BOD₅ concentration of the effluent extracted from SBR and sent to VFTW in g/L.

Then the coefficient *Y* is calculated by linear regression plotting cumulative TSS produced as a function of cumulative BOD₅ treated.

3. Results and discussion

3.1. Stillage characterization

In 2021, the SBR received a total of 26,000 m³ of stillage, for an average daily flow of 175 m³/d (0 to 640 m³/d). This daily flow represents 80% of the reference flow of 200 m³/d. This daily volume is equivalent to 4% of the volume of the aerated basin, i.e. a theoretical retention time of 22 days. The volume of treated water amounts to 26,500 m³. The small difference with the input is explained by the contributions of rainfall in the tropics in this particular case, which compensate for evaporation losses on the SBR and evapotranspiration on the VF treatment wetlands (data not shown).

The stillage sent to the SBR is highly loaded with organic matter (Table 1): the average concentrations are 14.6 gCOD/L, 3.3 gTSS/L and 6.8 gBOD₅/L in 2021. They are less than half of those for which the treatment plant was designed (15 gBOD₅/L in theory) but they should increase in future years due to the optimization of water use in the

Table 1

Stillage characteristics (concentrations) in 2021 ($n = 9$ samples). SD represents the standard deviation. No data is available for 2020 and 2022.

(mg/L)	TSS	BOD ₅	COD	dCOD ^a	TN	dTN ^a	TKN	NH ₄ -N	NO ₃ -N	TP	PO ₄ -P
Mean	3260	6843	14,641	10,954	262	110	261	20	1	145	57
SD	1010	1125	2973	1958	67	37	67	13	0	91	16
(mg/L)	TOC ^a	DOC ^a	HCO ₃ ⁻	Cl ⁻	SO ₄ ²⁻	Na ⁺	K ⁺	Mg ²⁺	Ca ²⁺	Sugars	
Mean	5895	4544	110	668	939	26	1378	115	191	926	
SD	1041	665	191	92	176	11	210	23	196	302	

^a d COD: dissolved Chemical Oxygen Demand; d TN: dissolved Total Nitrogen; TOC: Total Organic Carbon; DOC: Dissolved Organic Carbon.

distillation process. The average incoming BOD₅ load is of 1140 kg/d (versus 3000 kg/d at nominal load) over the production period.

The variability of concentrations is high (Table 1, Fig. 1a). These variations can be due to the type of cane used, the duration of fermentation, the volume of cleaning water used (about 15% of the total volume) or the storage time in the buffer tank.

Stillage appears unbalanced for biological activity: it is deficient in nitrogen and very slightly in phosphorus. A classical carbon/nitrogen/phosphorus (C/N/P) ratio of 100/5/1 is necessary for a biological treatment of the effluents (bioavailability ratio, where C is BOD₅; [22]). Depending on the form of nitrogen considered, this deficiency is more or less high. Stillage is low loaded in nitrogen that is easily assimilated by the treating biomass: 20 mg NH₄-N/L and 1 mg NO₃-N/L on average, i. e. a ratio of 100/0.3/0.8. However, it is the overall dissolved nitrogen concentration (dissolved TN = 110 mg/L) that should be considered: the ratio would therefore be of 100/1.6/0.8. Nitrogen additions were introduced in March 2021 on this basis. Initially overestimated by three times the requirement during one month, the urea dosage was then reduced by half for six weeks.

The pH of the stillage is 3.3 ± 0.2 and is explained by the distillation process (acidification). In addition, the conductivity of the stillage is high (5750 μ S/cm) due to the addition of ammonium sulfate to improve yeast growth and productivity during the fermentation stage before distillation. Sulfate and chloride levels averaged 940 mgSO₄²⁻/L and 670 mg Cl⁻/L respectively (Table 1). Sulfates are slightly lower than the reference of 1 to 2 g/L beyond which they are possibly damaging to

biological treatment [22,23]. For chlorides, the French reference CCTG [24] indicates that the connection of industrial effluents to a municipal wastewater treatment plant is possible only if they do not induce a concentration higher than 500 mg/L at the inlet; this value is exceeded here.

3.2. Overall performance of the treatment plant

The stillage treatment is efficient since a strong decrease in carbonaceous matter (Fig. 1a) is observed. Outlet concentrations are on average of 170 (SD: 127), 277 (SD: 323), 583 (SD: 489) and 346 (SD: 321) for respectively TSS, BOD₅, COD and dCOD, and compatible with banana plantation irrigation. Removal yields are high: 92% for TSS, and 96% for both COD and BOD₅ (Fig. 1b). These performances are stable whatever the applied load. The variation in nitrogen performances (dissolved TN, Fig. 1b) is explained by the addition of urea. Phosphorus analyses show consumption by the treating microorganisms for their metabolic needs.

The stillage is very poor in carbonates (HCO₃⁻, Table 1). This element is essential to appreciate the mineral carbon content to meet the needs of the autotrophic bacteria responsible for the treatment of nitrogen by nitrification [22]. Carbonates are produced during the non-aeration phases by denitrifying bacteria (drops in oxygen concentration and redox potential). Along the treatment, an increase in average HCO₃⁻ concentrations is observed (raw stillage = 110 mg/L; SBR outlet = 402 mg/L; VFTW outlet = 642 mg/L), highlighting the presence of

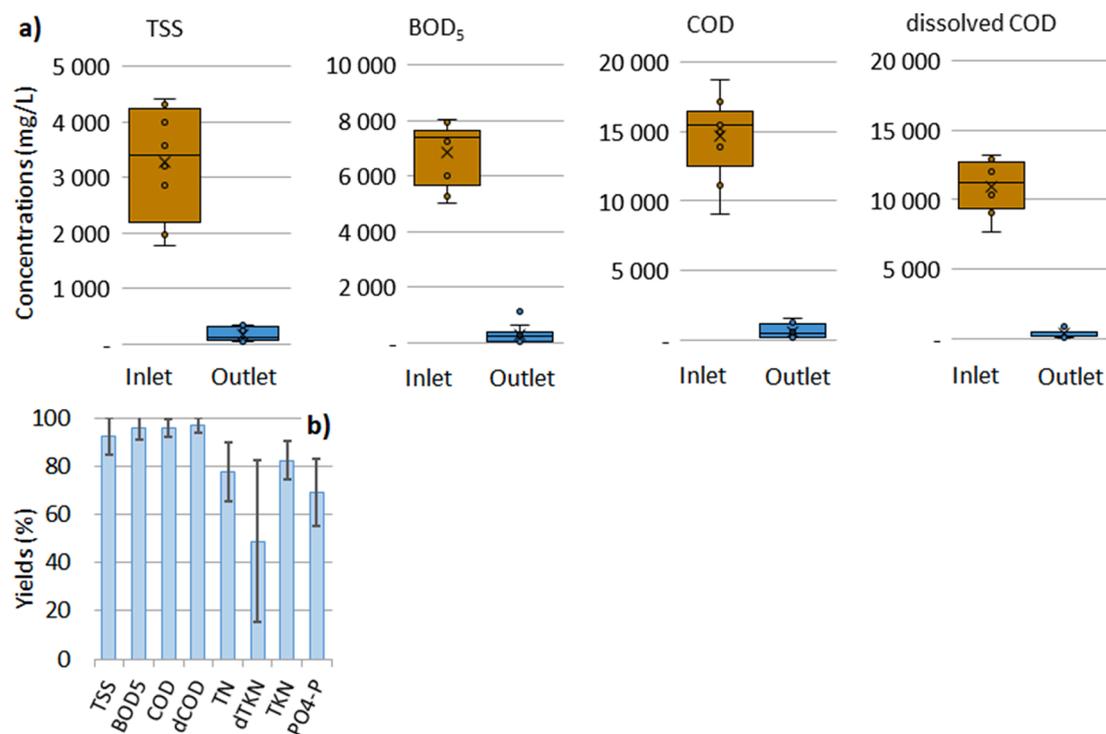


Fig. 1. Performance of the SBR in 2021: a) inlet and outlet concentrations and b) removal yields (error bars represent the standard deviation).

denitrification reactions mainly in the aeration basin but also in the French VF treatment wetlands at the end of the distillation period (high sludge deposit).

The performance objectives are met, but the treatment plant still needs to optimize some of its settings on both SBR and VFTWs in order to guarantee long-term treatment.

3.3. SBR performance

SBR was functioning with an average mass load of 0.07 kgDBO₅/kgVSS/d (0.01 to 0.12) and an average volume load of 0.28 kgBOD₅/m³ (0.06 to 0.58) corresponding to a low load [25]. Operation at high load would have resulted in high sludge production. A low load was preferred to avoid this inconvenience.

3.3.1. pH management: soda addition

In 2020, numerous electrical failures occurred, bringing the treatment plant to a complete halt for several hours a day. As a result, the effluent arrival and the stirrers were stopped. The lack of oxygen supply in the aeration basin led to significant pH drops, associated with these electrical failures. Additions of soda solution were able to remedy this problem from time to time. In 2021, 25 breakdowns were recorded during the distillation period (3 to 92 h of shutdown each) but the pH of the SBR remained rather stable (6.5 to 7.5). However, no soda was used but urea was added: the hypothesis is that urea would have allowed stabilizing the treating biomass (stress limitation) and thus the pH. Indeed, in May, without urea addition, the pH dropped rapidly during an electrical cut (Fig. 2a).

At each restart in January, after 7 months with no effluent input and stirring reduced to 4 h/d, the SBR sludge level is less than 1 gTSS/L and pH is 5. More intense stirring and addition of soda are required during the week prior to effluent arrival to create conditions conducive to the development of sludge in the aeration basin for treatment. This

assumption is validated in 2022 (data not shown). Then the arrival of effluent, coupled with urea additions, allows the flocs to develop in 2 weeks.

3.3.2. TSS management: sludge production

The sludge rate in the aeration basin refers to the TSS concentration, linked to the biomass growth. Probe values are supplemented by laboratory measurements. The TSS concentration is less than 1 g/L after each production shutdown period (July to December) due to the absence of incoming pollutant load. Consequently, the treatment facility is restarted each year in January by a continuous stirring during the week preceding the distillation resumption. Theoretically, the TSS concentration must be between 3 and 5 g/L to ensure the treatment. The level of 4 g/L is reached two weeks after the re-feeding of the SBR (Fig. 2b). Besides, for major power cuts lasting more than 48 h, the TSS concentration dropped sharply. Then it returned to normal after only two days of restarting the brewers, confirming the resilience of the process for treating stillage.

A very high sludge concentration is observed in March-April 2021 (Fig. 2b): up to 12 gTSS/L in the aeration phase and 10 g/L in the supernatant during settling. At such a concentration, the TSS are so numerous that decantation cannot take place (flocs are interfered with each other). Moreover, they require more oxygen to ensure the treatment, which the brewers are no longer able to provide in sufficient quantity (drop in O₂ content, data not shown). For this reason, sludge extraction was introduced until the TSS returned to correct values (5 g/L). The action consists in programming the SBR emptying during the stirring period, without settling phase, in order to send the excess TSS to the VFTWs.

Consistent with a lower dosage of urea (in quantities reduced to the metabolic needs of the biomass), the sludge rate remained relatively stable until the end of the distillation period. Finally, the drop in TSS content in June is linked to the end of rum production and the absence of

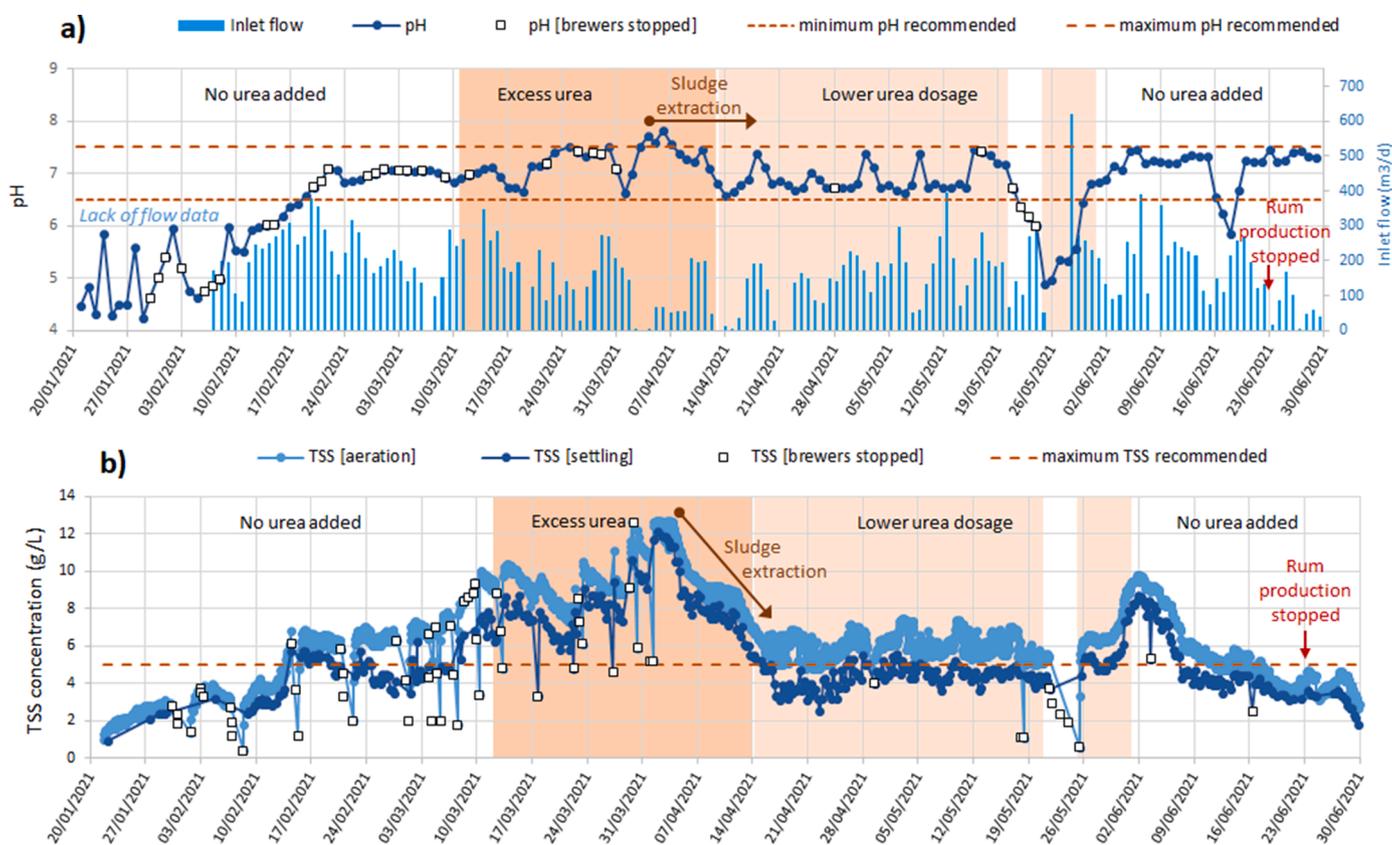


Fig. 2. Evolution of the parameters a) pH and b) TSS, in the SBR in 2021.

incoming stillage: microorganisms dye quickly (Fig. 2b).

Besides, the sludge production is calculated for each characteristic period of the monitoring: restart of the treatment facility, urea addition, sludge extraction, end of distillation (Fig. 3). Located between 0.6 and 2 kg of produced TSS per kg of treated BOD₅ (average of 1), the biomass growth Y coefficient varies during the 2021 follow-up (Fig. 3a). It tends to decrease over time and according to the environmental conditions. On the other hand, it seems to be higher than some references for domestic wastewater (0.4 to 0.5 kg TSS produced/kg BOD₅ treated in FNDAAE-34 [26]) but it is quite consistent with other references (0.8 to 1 kg TSS produced/kg BOD₅ treated according to Duchène [27]). The quality of the effluent, different from that of a domestic wastewater, and the very high stillage concentrations, can explain this difference. This sludge production rate varies during the monitoring, as it is directly related to the incoming mass load (Fig. 3b): the higher the pollutant load to treat, the higher the TSS production.

The biomass yield coefficient, on average 1 kg TSS produced/kg BOD₅ treated (Fig. 3), is quite high. This is due to the nature of the effluent (good biodegradability, presence of sugars), the climate (constant heat ~25 °C) and the low mass loading (extended aeration). Only regular sludge extractions would limit the sludge production and keep the TSS level below 5 g/L, and therefore the TSS overload of the VFTW.

3.3.3. Sludge quality and settle ability: urea addition

The V30 settling test allows a better characterization of the SBR behavior. It is systematically very high (about 940 mL/L), which indicates very poor settling. It improved only slightly with the addition of urea. This result may seem bad (V30 > 300 mL [21]), but the abnormally high levels of TSS and the absence of a reference for this type of influent make it difficult to interpret the V30. The water-sludge separation of the V30 tests is flat and marked. However, as during the previous year, the supernatant is cloudy, with the presence of turbidity and fine particles. This indicates a non-optimal sludge flocculation and the presence of colloidal particles.

The granulometric analysis of the flocs that produced in the SBR showed an average diameter of 15 to 35 μm. These are small flocs compared to the 10–100 μm range generally encountered in activated sludge processes [22]. Considering that particles larger than 40 μm are retained on the surface of French VFTWs [28], there is a risk of migration of these particles within the filter media and thus of internal clogging. Although floc size did not increase significantly following urea additions (results not shown), the monitoring of this experimental treatment plant must continue in order to determine precisely what affects the quality of the sludge in the SBR and if urea additions are of interest to address this problem.

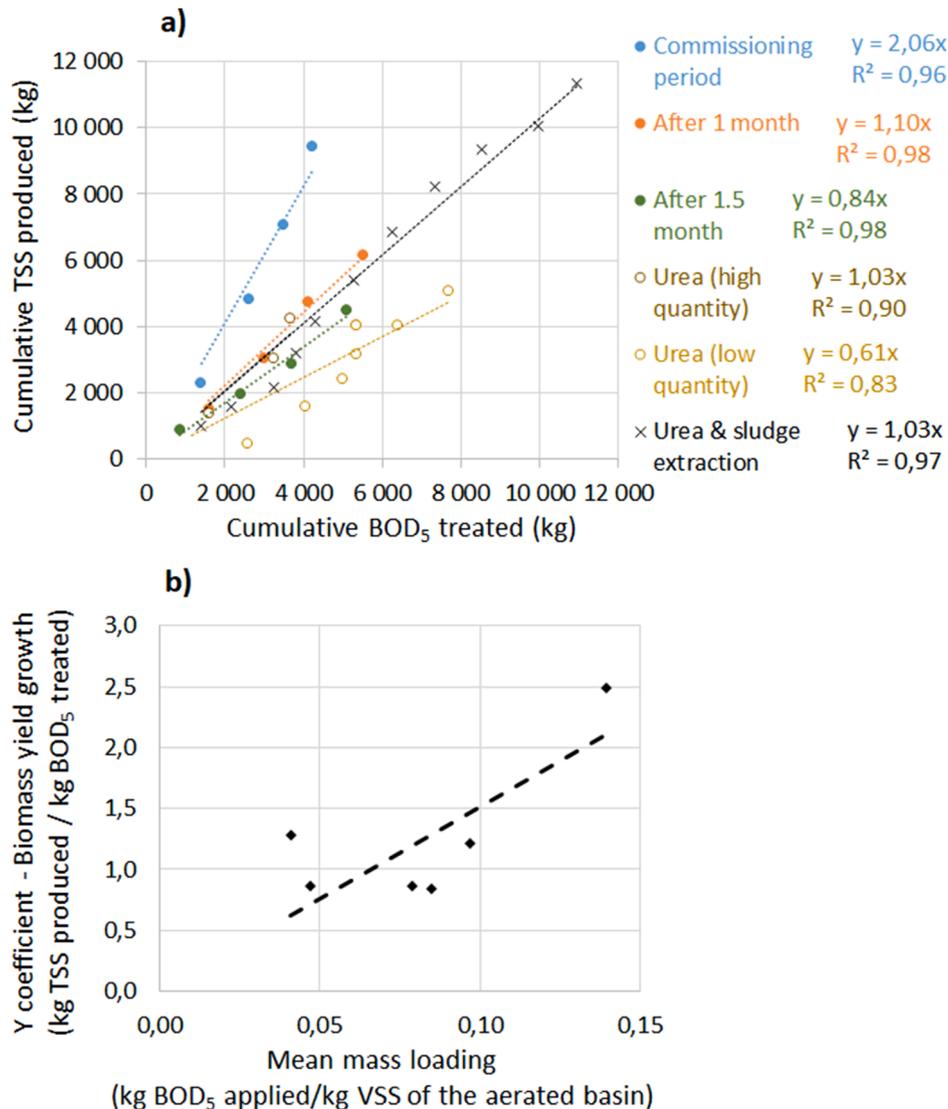


Fig. 3. Sludge production (TSS, biomass yield coefficient Y) at the aeration basin in 2021: a) as a function of treated BOD₅, and b) as a function of SBR mass loading.

3.3.4. Oxygenation and redox

Oxygen content varies according to the progress of the current cycle: 4–6 mgO₂/L in the stirring phase, <0.5 mg/L in the settling phase. With regard to the continuous recordings, the redox potential in the SBR is –200 to +200 mV EHN throughout the monitoring campaign. The conditions are thus generally rather reducing. This may explain the turbidity of the supernatant observed during V30 tests and the difficulties in filtering the effluent (possible fermentation reactions and formation of polysaccharides with strong clogging impact).

3.3.5. Effluent concentrations and SBR performances

The performance of the biological treatment of the aeration basin is stable and correct: 82 and 87% for the parameters total BOD₅ and dissolved COD respectively (Fig. 4a). COD removal yields are lower (44%) because this parameter integrates the particulate COD of the sludge produced in the SBR.

3.4. VFTW performance

3.4.1. Hydraulic and organic loads

The average hydraulic load during the follow-up is of 0.13 m/d on the filter in operation, close to the nominal load of 0.15 m/d. However, the applied organic loads are very high and variable due to the difficulties of sludge rate stability in the SBR and the poor settling of the flocs. Fig. 4b illustrates the robustness of the French VF treatment wetlands, i.e. the excellent stability of the performances for the carbonaceous parameters whatever the applied load:

- The average COD load sent to the filters is 920 g/m²/d (up to 1900 g/m²/d).
- TSS loading averages 640 g/m²/d (up to 1200 g/m²/d). For VFTW fed exclusively with raw domestic wastewater, Molle et al. recommends 150 gTSS/m²/d in temperate climate [17] and have tested a maximum of 140 gTSS/m²/d in tropical climate [29]. In sludge treatment wetlands, Torrens et al. studied high loads up to 230 gTSS/m²/d [6]. Loads applied in this experimentation are therefore significantly higher than these feedbacks.

The cumulative load of 75 kgTSS/m²/y recommended by the design company is similar to the 78 kg/m²/y load that was applied during the production period to the filters in 2021 (corresponding to 32 kg/m²/y for the whole year). It is far superior to other feedbacks from sludge treatment wetlands: 28 kg of dry matter/m²/y tested by Torrens et al. [6] for pig slurry, and 25 to 30 kg of dry matter/m²/y ordered by Troesch [30], or a maximum of 60 kg of dry matter/m²/y recommended by Nielsen & Larsen for sludge extracted from aeration basins [31], but lower than experiments done in tropical climate for sludge treatment read beds [13].

This high organic load, in linked of the number of bed in parallel and the plant used, caused plants death and consequent increase in the level of liquid sludge on the filters (see Section 3.4.3). As a result, they are clogged on the surface, with a permanent ponding and too slow water draining.

3.4.2. Effluent concentrations and VFTW performance

The VF filters ensure the finishing of the stillage treatment despite strong variations of the received loads. TSS are retained at over 85% (Fig. 4b). COD is treated at 91%, with acceptable variations (73–98%). More globally, particulate pollution is very well treated (filtration), while dissolved compounds are less so due to permanent ponding and lack of oxygen into the filters.

TN and TKN concentrations decreased between the inlet and outlet of the VFTW (Table 2), as did organic nitrogen (influent 357 mg/L vs effluent 9 mg/L) with a removal efficiency of 96%. But this was not the case for NH₄-N: the filters produced ammonium in significant quantities (ammonification of organic matter). The low oxygenation conditions do not allow a full nitrification. Such an emission of ammonium would be harmful in case of direct discharge to the receiving water body.

Unsurprisingly, phosphorus is not treated by this type of treatment process (Table 2). The particulate fraction of TP is retained in the sludge, while the dissolved part of P (orthophosphates PO₄-P) is not treated.

3.4.3. Infiltration capacity and sludge accumulation

The death of the plants did not allow to take advantage of their mechanical role for the infiltration. Consequently, it led to an

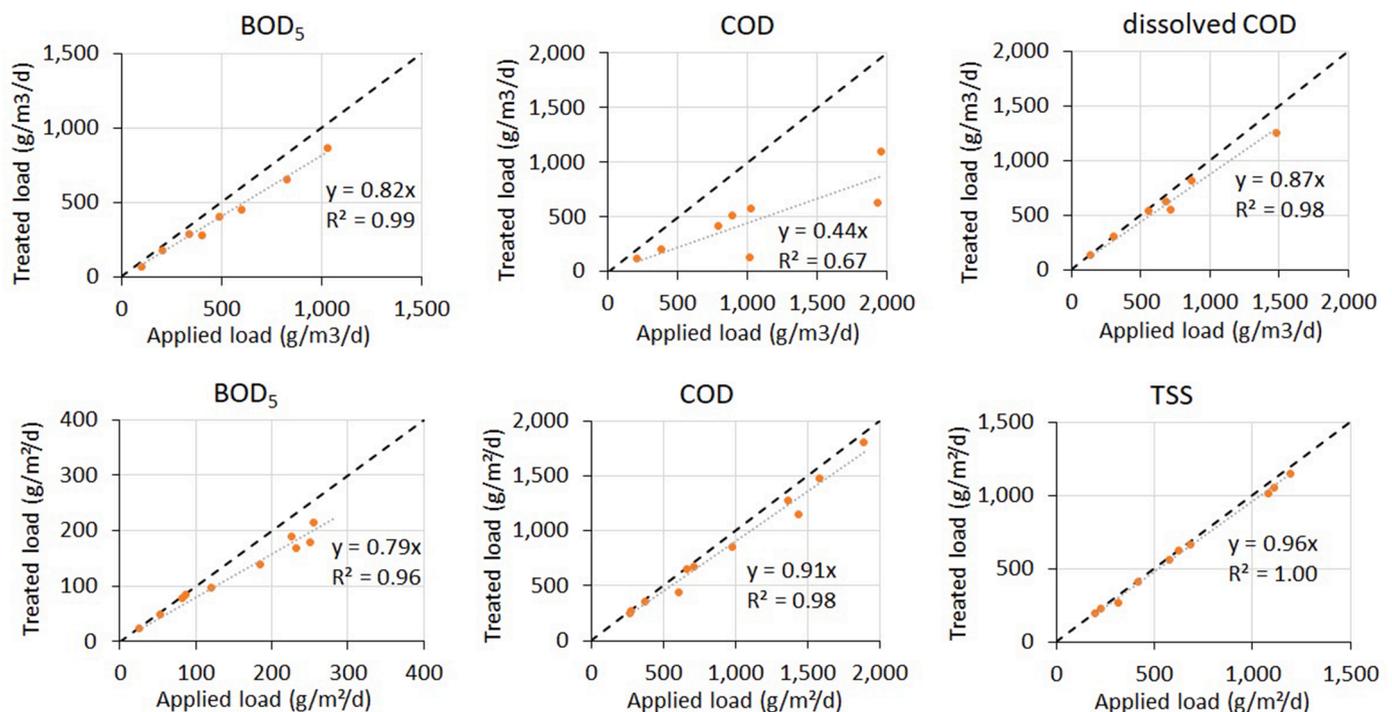


Fig. 4. Applied and treated loads in 2021: a) in BOD₅ and COD on the SBR, and b) in BOD₅, TSS and COD on the VFTW in operation.

Table 2Influent and effluent concentrations for the French VF treatment wetland in 2021 ($n = 11$ samples). SD represents the standard deviation.

(mg/L)		TSS	BOD ₅	COD	dCOD ^a	TN	dTN ^a	TKN	NH ₄ -N	org N ^a	NO ₃ -N	TP	PO ₄ -P
Wetland influent (SBR effluent)	Mean	5692	1292	7285	866	360	12	358	1	357	1	94	26
	SD	2150	485	2111	794	147	7	146	1	146	1	30	13
Wetland effluent	Mean	170	277	583	346	57	52	46	37	9	10	33	27
	SD	127	323	489	321	41	38	30	28	8	16	23	28

^a d COD: dissolved Chemical Oxygen Demand; d TN: dissolved Total Nitrogen; org N: organic Nitrogen (= TKN - NH₄-N).

accumulation of liquid sludge (less than 5% of dryness) on the filter. The evolution of the surface deposit height is monitored for each of the three filters (Fig. 5). A camera pass in the drains at the end of the rum production confirms the absence of migration of sludge particles at the bottom of the filters.

3.4.4. Plant behavior

While well established before the first season of production (2020), the plants had degraded rapidly and *Heliconia psittacorum* and *Cyperus papyrus* were dead after 3 months of operation. After emptying the sludge and replanting before 2021 season, *Heliconia psittacorum* almost died and dried out after 2.5 months while *Cyperus papyrus* showed to be more resistant and died after 3 to 4 months of feeding (Fig. 6). This opposite observation between 2020 and 2021 can be explained by the age of the plants: in 2021 *Heliconia psittacorum* had just been replanted 6 months earlier while *Cyperus papyrus* is more than 18 months old because they survived by natural regrowth (without replanting).

At each annual restart of the treatment plant, the load increase in January for only one week was too rapid for young plants, whereas it should be gradual over several weeks. The sludge quantity accumulated on the top of the filters led to a permanent ponding: it is certainly conditions of very low oxygenation which led to the death of the plants (Fig. 6).

While the common reed (*Phragmites australis*) is not endemic to the Martinique island, specific criteria in other plants are sought in tropical areas, including resistance to water stress (especially outside the distillation period: June to December) and temporary anoxic stress (during large inputs of organic matter). Plant development must be rapid and homogeneous, and the stems fine and tall to facilitate mechanical infiltration of effluents, while allowing easy mowing [15,16,29]. Other plants and stress tests are currently being conducted at Rhum JM distillery to determine acceptable TSS loads on the VFTWs. These additional tests will be the subject of specific publications.

4. Conclusions and perspectives

Stillage are industrial effluents easily biodegradable but highly concentrated in BOD₅ and COD (and chlorides and sulfates), deficient in nitrogen, and very acid (pH of 3.3). Moreover, they are only produced over a short annual period (5 months). Therefore, each restart of the treatment process in January is the most sensitive phase for the treatment plant operation, and requires easy-to-use and inexpensive additives (soda solution, and even urea).

The effluents are well treated by the aeration basin (SBR): average yields of 82% for total BOD₅ and 91% for dissolved COD. The four daily aeration cycles seem to be adapted according to oxygen and redox measurements. The biomass yield coefficient is high (about 1 kg TSS produced/kg BOD₅ treated) a. The acidity of the stillage, the imbalance of the C/N ratio which characterizes them, and the delicate management of the sludge rate, imply an optimization of the operation of this treatment facility to stabilize TSS concentration around 5 g/L and favor settling.

As a result, high TSS loads are sent to the French VF constructed wetlands (640 gTSS/m²/d, i.e. the equivalent of 78 kgTSS/m²/y), which causes significant ponding and the death of *Heliconia psittacorum* and *Cyperus papyrus* plants. The management of the sludge in the SBR is therefore essential for the sustainability of the filters. In addition, experiments are underway with new loads applied to the VFTWs and tests of other local plant varieties resistant to the stress induced by the high treatment loads.

NBS impacts and implications

- **Environmental:** the pollution generated by the discharge of liquid effluents from distilleries (stillage) in the rivers and on the very sensitive coasts of Martinique is very important. A treatment plant using an ecological process (treatment wetlands) is preferred for its ease of operation, with no use of chemicals, and high performance.
- **Economic:** tourism and rum production are the main local economic activities. The important volumes of stillage, highly polluting, must

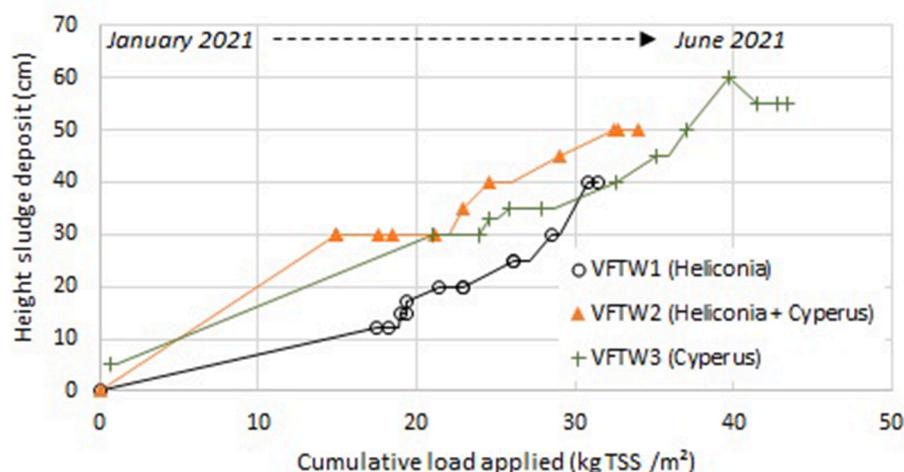


Fig. 5. Applied TSS loads and sludge height on the VFTWs in 2021.



Fig. 6. Status of *Heliconia* and *Cyperus* plants in the VFTWs in 2020 and 2021.

be treated to protect the sensitive environment and to perpetuate tourism. The studied distillery wants to renew its old stillage treatment unit, and promote circular economy while reusing treated water for banana trees irrigation as rum production happen in dry season.

- **Social:** the preservation of sustainable tourism depends largely on the quality of its bathing water. This regional demonstrator will be replicated for, at least, the Caribbean zone. The combination of an SBR and treatment wetlands reduces the footprint of the treatment plant to maintain agricultural land.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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