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Case Report

Combined treatment of domestic and pulp and paper industry wastewater in a rice straw embedded activated sludge bioreactor to achieve sustainable development goals

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ABSTRACT

The pulp and paper industry has been recognized as one of the largest users of water worldwide. Water is used in nearly every step of the manufacturing process. It generates significant amounts of wastewater and leftover sludge, creating several problems for wastewater treatment, discharge, and sludge disposal. Adopting the most effective and economical treatment techniques before discharging wastewater is therefore crucial. Thus, this study aims to evaluate the performance of the activated sludge bioreactor system (ASBS) for the treatment of pulp and paper industry wastewater (PPIW). The PPIW was characterized. During the experiment, the domestic and PPIW wastewater were run at a fixed HRT of 1 day. Subsequently, the ASBS was evaluated by varying the HRT and OLR. The HRT was varied in the range of 3, 2, and 1 day. At a fixed HRT of 2 days, the maximum and minimum COD removal were 88.4 and 63.2%. Throughout the study, the ASBS demonstrated higher treatment efficiency in terms of COD removal. First order, Grau second order, and modified Stover Kincannon biokinetic models were applied for the study. The biokinetic investigation shows that the modified stover kinetic model was more appropriate for the description of the experimental data in terms of microbial growth parameters. Thus, the kinetic coefficients obtained in this study could be used for the bioreactor scale-up. The study has also proven that the biosorbent made from biomass waste can potentially help preserve non-renewable resources and promote zero-waste attainment and principles of a circular bioeconomy.

1. Introduction

Wastewater is generated in various activities such as pharmaceuticals, manufacturing, agriculture, oil and gas exploration, industrial applications like paper manufacturing plants, and domestic usage. However, the concentration of organic matter, nutrients, and other water contaminants vary from one wastewater source to another [1]. Industrial wastewater is a major source of water pollution due to its high organic content and it is generated from different industrial applications [2]. Recent estimations have indicated that industrial applications utilize about 16% of total freshwater consumption in industrial nations. One of the industrial applications with high water consumption is the pulp and paper industry. It is an important industrial sector and includes a wide range of products and manufacturing processes. These generate a high quantity of wastewater [3–5]. The wastewaters from this industry can be put to beneficial use when properly treated. In addition, effective treatment of these wastewater will reduce water body pollution [6]. This will counterbalance the disparity between water shortage, demand, and supply, especially in arid regions. Moreso, food production will be guaranteed due to the availability of water for irrigation purposes [7].

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In several nations, wastewater treatment has been used as a solution to supplement scarce freshwater supplies. Primary, secondary, and tertiary/advanced treatment procedures are the three main techniques used to treat wastewater [8]. Water is separated from suspended particles, floating, and settleable materials using primary methods; biodegradable trash is removed using secondary methods; non-biodegradable waste is removed using tertiary methods [9–11]. However, most of these traditional techniques are expensive and demand some expertise. Furthermore, most of these techniques include drawbacks that reduce their effectiveness [12]. Several conventional treatment processes have shortcomings such as high energy demand, and excess sludge leading to the generation of secondary pollution [13–15]. To be specific, (i) Coagulation/chemical precipitation requires a large number of chemicals and a high cost and dose of coagulants/precipitants with low biological oxygen demand (BOD) and COD removal efficiency [16]. (ii) Anaerobic filters and Algae treatment have high water demand with the added cost of support media [17]. (iii) Constructed wetland is faced with high investment costs, large space requirements and are difficult to control. (iv) Membrane bioreactor requires skilled operator and membranes susceptibility to fouling and foaming. It also has the disadvantage of high membrane costs, washing, potential replacement, and maintenance due to clogging [18–20]. (v) Electro-Fenton processes possess high infrastructure costs associated with the use of electricity and UV light. (vi) Advanced oxidation processes are economically not acceptable for large-scale effluents. They are faced with the challenge of high oxidant doses and investment costs [21].

The biosorption process using RSAC has great economic value. In Malaysia, about 2.5 million tonnes of rice straw are burnt by local farmers every year as agriculture waste, which leads to significant CO_2 emissions, destructive fires, and an increase in global warming impacts [22]. Utilizing rice straw can convert the agricultural by-product into a valuable and inexpensive organic source. Rice straw as a raw material in the paper packaging material industry and wastewater treatment application is a sustainable agro-waste and renewable resource. The reuse of rice straw is sustainable and creates a new life for paddy farmers by turning their paddy waste into wealth and allowing them to earn up to 20% supplement to their income per year (an average additional income of US$300 per year) boost farmers' incomes, help in reducing CO_2 emissions/leaves a lower carbon footprint in the entire supply chain, from cradle to cradle, End-of-Life (EOL) compliances and meet Sustainable Development Goals (SDG) [23]. Consequently, efforts to enhance their performance have intensified. This leads to the utilization of biosorbents such as rice straw activated carbon (RSAC) in the system. Rice straw as agricultural waste is generated from acres of rice fields. As an agricultural residue, rural farmers and ranchers do burn tonnes of it [24]. This leads to the air pollution index always exceeding unsafe levels and subsequently resulting in respiratory effects [25].

Studies investigating the combined use of the activated sludge and biosorption processes for organic matter removal from PPIW using RSAC are lacking. Hence, this study would treat PPIW in the combined system using waste material as a biosorbent. Hence, the objectives of this study are to investigate the sustainable approach to recycling rice straw in an eco-friendly way. The study will prepare the RSAC for COD biosorption. RSAC would be synthesized and characterized alongside PPIW. The study would also determine if the application of RSAC in ASBS would yield high removal efficiencies. The biosorption performance and biokinetics for COD removal from PPIW would be studied.

2. Materials and methods

2.1. Materials

The investigation only employed the purest substances available, and the study flow chart is depicted in Fig. 1 below. A local business located in the nearby Seri Iskandar, Tronoh, Perak, Malaysia provided the rice straw biomass. Wastewater from the pulping industry was obtained from a company in Kedah, Malaysia. Sludge that had been activated was acquired from the UTP sewage treatment facility [26].

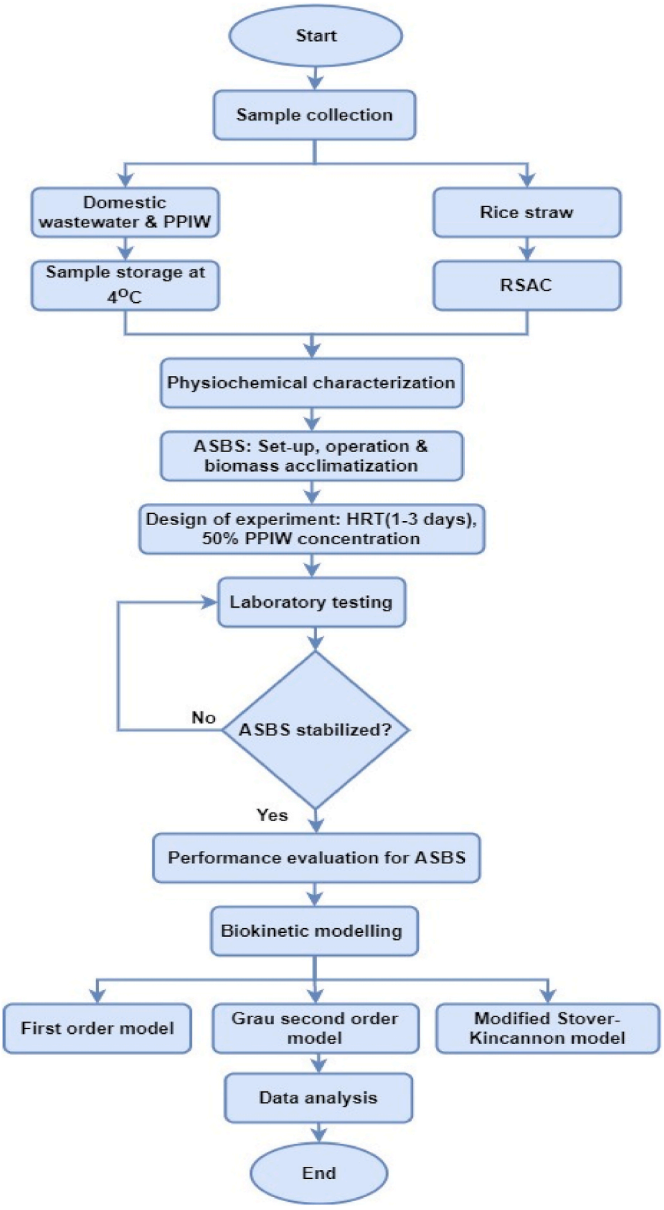

Fig. 1. Flowchart of the experimental study.

2.2. Activated sludge bioreactor system (ASBS) experimental set-up

These tests are carried out to learn more about the contents of wastewater. An aerobic activated sludge bioreactor system is used to treat the wastewater sample. Because biological reactors are inherently environmentally sensitive, it requires frequent and consistent monitoring, maintenance, and testing [27]. COD, MLSS, and MLVSS are among the factors that are being examined for the study. Table 1 depicts the variables of the ASBS that are implanted in the study.

Inside the lab, the ASBS was built up for the investigation of continuous mode. The system consists of an aerated influent tank connected by a 10 mm tube operated by an up-flow pump to an aeration tank, and the overflow is emptied into an effluent tank. To guarantee a minimal D.O. of 2 mg/L, several tube diffusers were inserted in the intended reactor, which was made of 5 mm thick acrylic glass. Silicone peristaltic pump tubing was used to link the reactors to the 20-litre

Table 1
ASBS operating variables.

Properties	Variables
Flow rate	5 L/d
Temp.	25 °C
Dissolved oxygen (DO)	2.0 mg/L
Sludge retention time (SRT)	40 days
F/M ratio	0.085
bCOD/BOD ratio	1.6
Organic Loading Rate (OLR)	0.2 kg BOD/m^3.d

influent tank. The reactor was constructed in a lab using biological biomass from the residential STP as the beginning biomass during the operation stage. The activated sludge characteristics are highlighted in Table 2.

The treatment plant's return sludge pipeline is where the biomass was obtained [28]. Utilizing Peristaltic Pump Silicone Tubing by Longer Pump, influent PPIW was constantly fed into the reactors at a rate of 5 L/d. Pulp and paper industry effluent generation and its treatment in a bench-scale ASBS is depicted in Fig. 2. Long-term aeration was used in the bioreactor. The biomass was stabilized for 15 days during the acclimatization period, and the sludge age was managed daily through sludge recycling and waste.

2.3. Preparation of RSAC

The rice straw used as depicted in Fig. S1 came from a small business in Perak, Malaysia. This became essential to heat-treat the rice husk at 800 °C and a heating rate of 10 °C/min for 2 hours in a microwave oven to raise the SiO_2 content and reduce the sintering effect, which is often reflected by a severe fall in the specific surface area. HCl and NaOH solutions were used in a chemical treatment approach to pre-clean the sample. It was then thoroughly cleaned with deionized water and dried in an oven for 2 h at 105 °C [29]. Before beginning the experiment, samples were stored in a tight container after cooling at room temperature [30]. The created activated carbon was then used in this investigation. Reagents used were NaOH, HCl, HCOH, H_2SO_4, H_2O_4, and HNO_3 purchased from Gouden Sdn Bhd, Ipoh, Perak, Malaysia. Further purification was not required as all reagents received were of technical grade. To prepare the required solutions, distilled water was used. Series 6000 heating and drying oven, UV-2700 digital spectrophotometer, water bath shaker (Spectralab), and portable pH meter were used [31].

2.4. Characterization of RSAC

The synthesized RSAC was characterized. The BET surface area and pore size of the RSAC were calculated using the N_2 adsorption-desorption using Micrometric ASAP 2020. To create a specimen molecular impression, the functional groups present in the raw and treated rice straw were determined by Fourier Transform Infra-red (FTIR) analysis (NICOLET iS10, Thermo Scientific, USA). The FTIR spectra were used to observe the particles' absorption and transmission, which was carried out using PerkinElmer Frontier as shown in Fig. S2a. The FTIR was conducted to investigate the effect of chemical pre-treatment on functional groups, especially hemicellulose, cellulose, and lignin.

Table 2
STP's activated sludge characteristics.

Parameter	Mean value
pH	8.03 ± 0.0485
Temperature (°C)	25.18 ± 0.2816
Total solids (mg/L)	9065 ± 28.8357
Suspended solids (mg/L)	6914 ± 11.2081
MLVSS/MLSS	0.88 ± 0.0449
Settleability	309.43 ± 22.1793
SVI	98.91 ± 1.3563

Samples were washed with acetone in a cantered glass funnel. The samples were transferred to a preheated oven at 80 °C for 1 hour [12]. One milligram of the sample was mixed with 100 mg of spectroscopy grade KBr (See Fig. S2b). Infrared images were acquired using PerkinElmer over a wavenumber range of 4000 to 4000 cm^{-1}, at 400 cm^{-1} resolution, and 100 iterations were performed to record the spectral range.

Proximate and ultimate analyses of the RSAC were conducted to determine pH, moisture, fixed carbon, volatile matter, and ash [32]. The proximate value tests, greater heating value, ultimate analysis, and moisture content test was carried out in accordance with ASTM E1755-01, D4809-00, ASTM D3176-09, and ASTM E871-82 respectively. They were conducted using the LABsys Evo TGA analyzer, Ac-350 bomb calorimeter, and Leco CHNS-932 type analyzer. Carbon, Hydrogen, Nitrogen, Sulphur, and Oxygen. Chemical composition and analyses of RSAC were conducted to determine Hemicellulose, Cellulose Lignin using Chesson Method and functional groups using Fourier Transform Infrared spectroscopy (FTIR). RSAC characterization was carried out to investigate the surface morphology by using Scanning Electron Microscope (SEM). Supra 55 VP (Carl-Zeiss AG, Germany) as shown in Fig. S2b was used to analyze the surface morphology of RSAC using a Variable Pressure Scanning Electron Microscope with an accelerating voltage of 15 kV.

2.5. Basic concepts in the use of biological activated carbon

The biological activated carbon process, which is based on activated carbon technology, uses the synergistic effects of adsorption on activated carbon and biodegradation to purify raw water. Due to its high ability to absorb dissolved oxygen and organics in raw water and its substantial specific surface area and completely developed pore structure, activated carbon stands out. The right temperature and nutrition can be achieved by gathering or artificially immobilizing microorganisms. The microbes will then reproduce on the surface of the activated carbon and eventually form BAC, which may simultaneously perform the adsorption and biodegradable roles [33]. The biological activated carbon technique entails the interaction of activated carbon particles, DO, contaminants, and microorganisms in a water solution. A simplified model of the interactions between the four components is shown in Fig. 3 [17]. Simply put, activated carbon's adsorption produces the interaction with pollutants, and the outcome is determined by the properties of both the activated carbon and the pollutants. In the meanwhile, activated carbon can absorb DO, and contaminants will be removed by microorganisms that have attached to its surface and are eating DO. In essence, by utilizing biological activated carbon, the goal of eliminating contaminants from raw water can be accomplished through the interaction of these 4 elements [6].

2.6. Analytical methods

The analytical techniques and laboratory equipment employed in this investigation, as well as the Standard Methods of Water and Wastewater Examination (APHA, 2005). To assess the COD values in the influent and effluent samples, the pulp and paper wastewater was digested in the DRB200 Reactor. The COD absorbance was measured using a spectrophotometer by HACH, model number DR3900. The pH was measured using a HACH SensIONTM pH metre set. The ASBS performance efficiencies were recorded, and each measurement was made in three copies.

2.6.1. MLSS and MLVSS

Sludge and water that have been extracted from the clarifier throughout the wastewater treatment process and reintroduced back into an earlier stage of treatment are known as mixed liquor. Microorganisms in the mixed liquor break down wastes in raw water [34]. The total suspended particles in a sample of mixed liquor are measured using

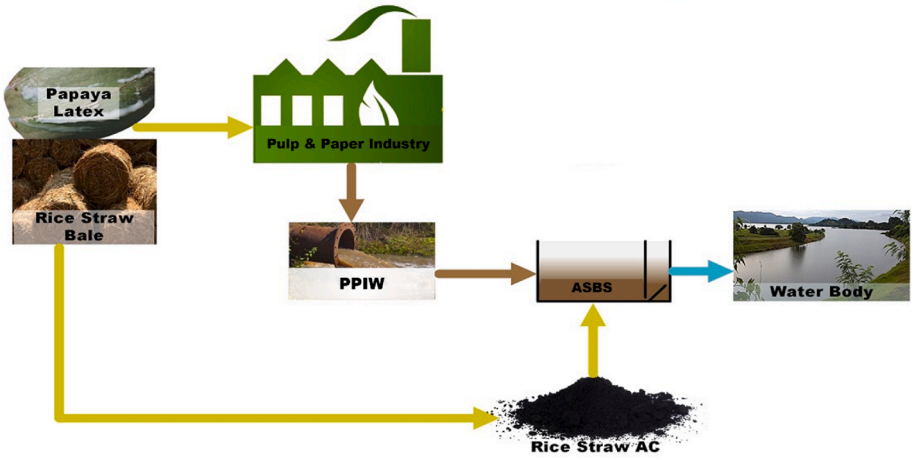

Fig. 2. PPIW generation and its treatment in a bench-scale ASBS [13].

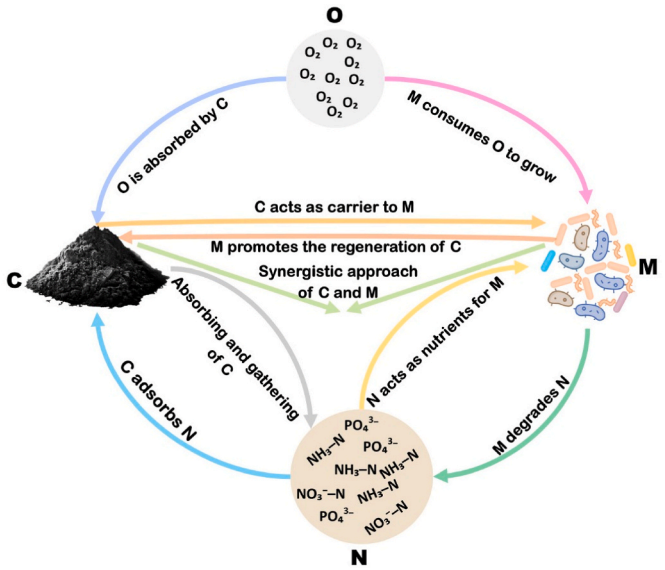

Fig. 3. Carbonated rice straw ASBS biosorption process interactions between activated carbon, microorganisms, contaminants, and dissolved oxygen [17].

the MLSS method using the equipment shown in Fig. S3a. Except for using mixed liquor as the water sample, this test is substantially identical to the total suspended solid (TSS) test. Although MLSS concentrations are frequently higher than 1000 mg/L, they shouldn't go above 4000 mg/L.

A test called MLVSS is used to calculate the level of volatile suspended solids in a sample of mixed liquor. Solids that burn up when the sample is heated to 550 °C in a furnace depicted in Fig. S3b are said to be volatile solids. Most of volatile solids in a sample of mixed liquor will be made up of organic material and bacteria. As a result, it is possible to tell whether there are enough bacteria available to digest the sludge by comparing the volatile solids concentration of mixed liquor to the total number of microorganisms in the water [22]. The first step of performing the MLSS and MLVSS experiment was to prepare wastewater samples with a dilution factor of 1:100. Then, three samples of 50mL for MLSS and three samples of 50mL for MLVSS were prepared. Sampled wastewater was shaken from an anoxic tank, aeration tank, and clarifier vigorously. Quickly, 10mL of wastewater sample was extracted using a pipette from a 1000mL volumetric flask and made up to 1000 mL and it was mixed well. Normal filter paper would have burnt off in a furnace at 550 °C thus microfiber filter (Whatman Filter Paper 934-AH) was used. For the MLVSS experiment, MLSS test samples were continued for burning at 550 °C for 1 h. It was then placed in a desiccator for 15–20 minutes to cool down. Then, pan together with filter paper and solids, WC were weighted.

The concentration of MLSS and MLVSS in mg/L was calculated using Equations (1) and (2), respectively.

$$MLSS = \frac{(B - A)}{V_{ML}} \times D_f \quad (1)$$

$$MLVSS = \frac{(B - C)}{V_{ML}} \times D_f \quad (2)$$

where,

A = Initial weight of glass microfiber filter
B = Weight of filter after oven drying at 105 °C (mg)
C = Weight of filter after ignition of volatile suspended solids at 550 °C (mg)
V_{ML} = Volume of diluted mixed liquor samples (L)
D_f = Dilution factor.

2.6.2. Chemical oxygen demand (COD)

COD is typically used to describe the organic content of wastewater and the toxicity of natural waterways. It is the quantity of oxygen needed to oxidize an organic compound (biodegradable or not) into CO_2 and H_2O when a strong oxidant ($K_2Cr_2O_7$) is present in an acidic environment and silver nitrate is acting as a catalyst [35]. It is possible to establish the relationship between BOD and COD to quickly estimate the value of BOD. For domestic wastewater, the common relationship between these two values is $BOD_5/COD = 0.5$ [14]. Homogenize 500 mL of the water sample to start the experiment and utilize distilled water as a control. A COD digestion cap Reagent vial was taken out from the equipment shown in Fig. S4a and kept at a 45-degree angle. 2mL of wastewater sample was extracted using a pipette into a vial and the vial cap was then tightened. Cap-held vial was placed over a sink. To combine the contents, it was gently flipped many times. Heat production is a sign of exothermic activity. The vial spent 2 h at 150 °C in a COD reactor that had been warmed. Finally, the vial was measured in triplicates with UV-VIS HACH Spectrophotometer depicted in Fig. S4b.

2.7. Growth biokinetic analysis in ASBS system

Kinetic models are essential in determining the relationship between variables, experimental designs, and results. This is a prominent part of the biological treatment system. Besides, kinetics is implemented to predict and monitor the performance of biological treatment systems

hence enhancing the quality of the operation [9]. The biological treatment has been profoundly known in the industry due to the information of many biochemical and metabolic processes obtained throughout the system activity. The relationship between substrate consumption and microbial growth has been defined with several biokinetics models such as the Monod model, Contois model, and Stover Kincannon model [18]. Three sequential procedures for microbial activity include ingestion, respiration, and growth rate. During ingestion, substrates are consumed by microorganisms. The kinetic model that is used in the study is First order, Grau second order, and modified stover.

3. Results and discussion

3.1. Influent wastewater characteristics

The influent wastewater (domestic, pulp, and paper) was subjected to a physicochemical examination. Their properties are depicted in Table 3. While Table 4 depicts the standard discharge limits for PPIW.

3.2. Characterizations of RSAC

The structural morphology of the materials has been described by the cubic particles on the surface of the RSAC as shown by the SEM images at different magnifications (see Fig. 4). The material showed an apparent physical change in the inner and outer surfaces.

The surface porosity of the RSAC was determined using nitrogen adsorption-desorption isotherms. The porous nature of the RSAC was confirmed by the higher BET surface area and pore volumes of the RSAC as highlighted in Table 5. The BET surface area of the material is 124.93 m^2/g. Interestingly, in comparison to what was previously reported by Ref. [23], the BET surface area achieved for RSAC is significantly greater.

By using FTIR spectroscopy, the functional groups in the RSAC were examined. The RSAC FTIR variation bonds spectrum is shown in Fig. 5. It was possible to see the typical peaks caused by both symmetric and asymmetric vibrations. The stretching of the OH bond from the water molecules in the RSAC is what causes the peak at 3403 cm^{-1} to appear. The OH bond is connected to the polyphenol and alcohol found in cellulose and lignin. The methylene contained in cellulose and hemicellulose is presented by the C–H stretching vibrations, which are responsible for the peaks at 2784 cm^{-1} and 2881 cm^{-1}. The absorption band associated with the C–O stretching of the free 1,4-benzenedicarboxylate contained inside the pore of the RSAC is shown in the peak at 1808 cm^{-1}. The C–H skeletal variation present in lignin is represented by 21 cm^{-1}. The asymmetric and symmetric stretching and bending vibrations of the carboxylate group, which may act as adsorption sites for the pollutants, are responsible for the intense peaks seen at 1590, and 1467 cm^{-1}. The FTIR spectrum for the RSAC is in good agreement with those previously reported [36]. Findings from the proximate and ultimate analysis are reported in Table 6.

Table 3
Domestic/pulp and paper wastewater characteristics and effluent standards.

Parameter	Mean	Mean
	PPIW	Domestic wastewater
pH	7.41 ± 0.48	7.19 ± 0.39
Temperature (°C)	25.3 ± 0.03	26.8 ± 0.08
TSS (mg/L)	612 ± 0.29	385 ± 0.41
COD (mg/L)	4103 ± 5.74	1017 ± 4.98
BOD_5 (mg/L)	1928 ± 6.18	426 ± 5.72
Ammoniacal Nitrogen (mg/L)	47.52 ± 1.87	28.13 ± 2.02
Phosphorus (mg/L)	105 ± 3.98	14 ± 4.15

*ND= Not detectable.

Table 4
Contaminants and their permissible limits for PPIW.

PPIW Parameters	Permissible limit (EPA, 2002)
pH	5–9
BOD	40
TSS	35
Total Phenols	0.50
COD	120
Electrical conductivity (EC)	1000
Sulphate	250
Chlorophenol	3.0
Cadmium	0.01
Total nitrogen	143
Nickel	0.10
Phosphorus	200
Na^+	200
Cl^-	1500
Copper	0.50
Iron	2.00
Manganese	0.20
Zinc	2.00

ND = All units are in mg/L except for pH and EC ($\mu mhoscm^{-1}$).

3.3. Impact of ASBS on organic matter removal

3.3.1. COD removal

The substrate utilization of bacteria was investigated by monitoring the COD concentrations from the ASBS. The bioreactor includes domestic wastewater with PPIW. The influent and effluent result alongside removal efficiency is presented in Fig. 6. The HRT was varied in the range of 3, 2, and 1 day at fixed PPIW concentration. The SRT was fixed at 40 days with decreasing HRT. From the figure during acclimatization, it would be observed that there were fluctuations during the startup period. The highest influent COD was observed at 3991 mg/L from sampling day 1 and the lowest was 3954 mg/L from sampling day 15. Evidently, the reactor took a long time to reach a steady state, thus the extreme differences in concentrations of wastewater samples. The overall steady state results show that COD removal increased in the range of 66–91% with decreasing HRT in the range of 3-1 days. The increased COD removal in the aerobic unit may be a result of organic matter being oxidized to CO_2, H_2O, and biomass when oxygen is present as an electron acceptor. The energy needed for cell upkeep and synthesis is released through the oxidation of organic materials [10]. Endogenous respiration, which occurs because of the aerobic unit's substrate running out, enables microorganisms to feed on their own cytoplasm for survival. Thus, endogenous respiration and organic matter oxidation—which provided energy for cell maintenance and synthesis—were the primary processes causing significant COD elimination [37]. These observations are in consonance with that reported by Ref. [16] who reported a COD removal of 87% in the bioreactor treating an industrial effluent. Additionally, the application of the influent feed into the aerobic unit provides comparatively higher portion of readily biodegradable organic matter for oxidation.

It is well known that when HRT is decreased, the hydraulic loading rate increases. This implies that more wastewater flows into the bioreactor which could reduce the interaction and biodegradation time between the substrate and the microorganisms and cause incomplete biodegradation [38]. A similar observation was made by Ref. [4] who noted that COD removal decreased with decreasing HRT in the range of 3, 2 and 1 day in a subsurface flow constructed wetland. A study also observed that COD removal decreased from 97.2 to 91.1% with decreasing HRT from 3 to I hour in a submerged MBR [29]. A similar observation was made by Ref. [31] who noted that the COD substrate removal rate increased with decreasing HRT in a membrane anaerobic system (MAS) treating palm oil mill effluent (POME). On the contrary, a study reported that the residual effluent COD concentration increased with decreasing HRT in the range of 9-3 days. This could be attributed to incomplete biodegradation of organic matter at short HRT because of

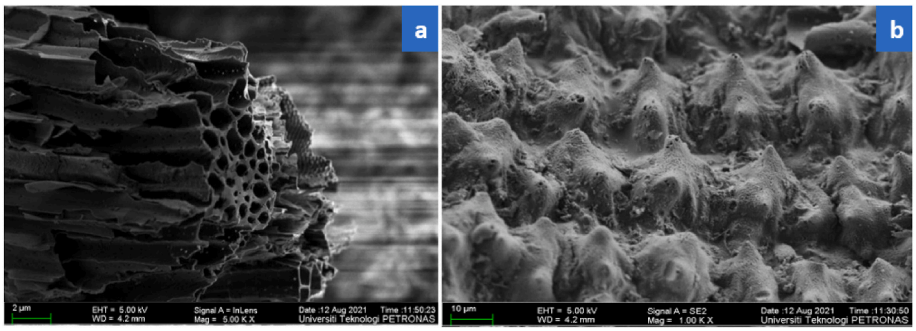

Fig. 4. SEM images of RSAC @ (a) 2μm (b) 10μm.

Table 5
RSAC surface and structural properties.

Parameter	Values
pH	7.48
Total pore volume (cm^3/g)	0.610
Mean pore size (nm)	2.306
BET surface area (m^2/g)	124.93
Mean pore radius (Å)	79.21

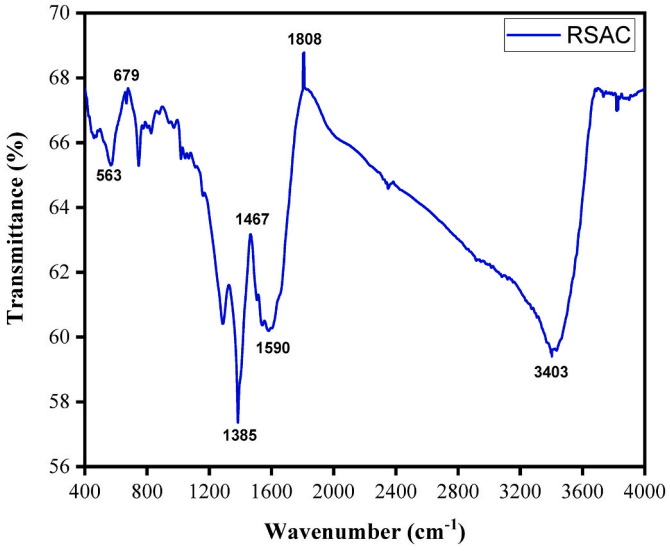

Fig. 5. FTIR spectra of RSAC.

Table 6
Proximate and ultimate analysis summary of RSAC.

	Proximate analysis				
RSAC	pH	Vm (wt %)	FC (wt %)	Ash (wt %)	Moisture (wt %)
	6.73	80.07	9.2	4.0	0
	Ultimate analysis				
	C	H	N	S	O
	46.81	10.56	0.52	0.83	41.28

higher hydraulic loading [5]. The increase of effluent substrate concentration whenever the HRT was changed was caused by shock loading and the disruption of the acclimation state of the biomass. The subsequent decrease in the effluent substrate concentration thereafter was due to the restabilization of the biomass and exogenous substrate consumption [22].

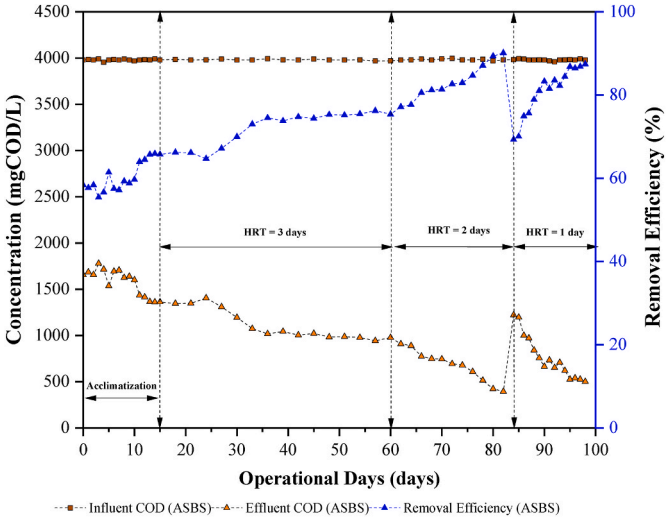

Fig. 6. The experimental time course of ASBS in terms of COD concentrations and removal efficiency.

3.3.2. *MLSS, MLVSS and MLSS/MLVSS ratio*

3.3.2.1. MLSS. Mixed liquor suspended solid is the amount of organic and inorganic materials present in polluted water overall. It is an important parameter in determining the total suspended solids in a sample of mixed liquor. The organic material and bacteria in a sample of mixed liquor would make up the volatile solids [27]. The MLSS and MLVSS were monitored in the aerobic and anoxic units and the obtained steady-state data was used for the determination of the biokinetic coefficients. During the startup period as depicted in Fig. 7, the average of mixed liquor solids in the reactor was approximately 3479.7 mg/L. It would also be observed that on the earlier sampling days, the MLSS values obtained for effluents were slower, compared to the later sampling days. It was also observed that the MLSS did not vary significantly until the 30% contamination phase began. As time passed, the pattern shifted, and later effluent had the lowest value for MLSS. It was observed that on sampling day 58, the mixed liquor solids concentration was 3846.3 mg/L. It decreased from 4130 mg/L on the previous sampling day. This was caused by the malfunction of the mixer, whereby it stopped working temporarily. This situation has caused the particles in the wastewater to settle and caused the influent wastewater pumped into the reactor at a lower concentration. These decrements could be considered natural, and they did not affect the treatment performance significantly. However, the reduction of mixed liquor suspended solids could be originated from the reduced amount of biomass in the reactors. The continuous reduction of MLSS concentration has depicted that the reactor could not manage to operate under this decreasing HRT. These contaminations have caused bacteria to gradually decrease, and the

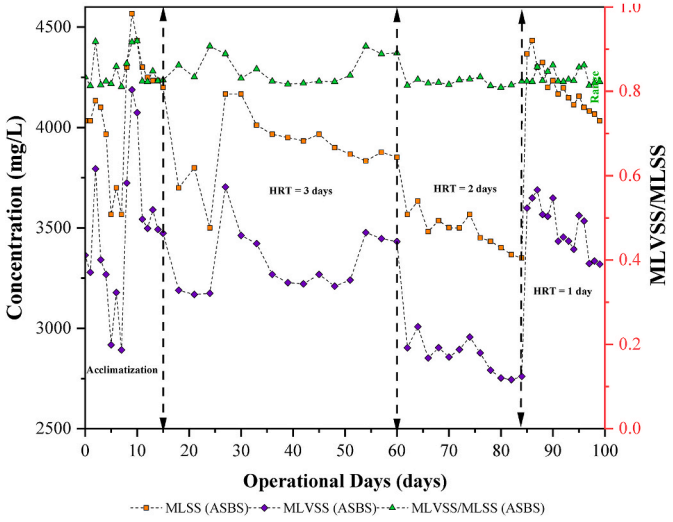

Fig. 7. Graph of mixed liquor volatile suspended solids.

biomass has failed to digest these nutrients sourced from the pulp and paper wastewater at overwhelming amounts [39].

As sample days increased, there were fluctuations in the MLSS values, which later stabilized within a specific PPIW concentration. This might be because more PPIW concentrations are present, which impact the concentration of biomass because the change always manifests right away after the addition [15]. The MLSS value fluctuates erratically before stabilizing within this range. This is consistent with earlier findings published in the literature [22]. The decline observed in most bioreactors is due to carbon contained in reactor feeds restricting biomass development. The decline is in line with the finding in the study by Ref. [8], which showed that MLSS increases when carbon source feed concentrations fall.

According to several research, larger MLSS values show clear benefits for boosting volumetric loading, solids retention time, or membrane bio-fouling rate, while particle size decreases as MLSS concentrations rise. All effluents had final MLSS values that were greater than the MLSS values recorded at the beginning sampling days under operational conditions for longer sampling times and increased PPIW concentration. According to earlier studies, the current study's increase in MLSS values produced effluents with lower viscosity, BOD, COD, appropriate filterability, and increased permeability [13]. However, higher MLSS concentrations aggravate settlement because the sludge layer forms quickly and deposits quickly at high MLSS concentrations, which affects the concentrations at which it is suited for filtering. When MLSS is raised, the viscosity rises. Numerous investigations have demonstrated that it is a mechanism capable of removing more suspended particles and turbidity at lower MLSS concentrations. At low MLSS concentrations compared to high concentrations, the process' mean flux value increased. System performance is adversely impacted by the sudden increase in MLSS value because normalized permeability decreases with rising MLSS concentrations.

3.3.2.2. MLVSS. In terms of MLVSS or volatile suspended solids, the concentration in the reactor has continuously shown that the PPIW contamination was excessive for the biological growth of bacteria, and it was damaging to the treatment system. The amount of MLVSS obtained would show the number of microorganisms or microbes retained in the mixed liquor sample. MLVSS value was high during the treatment time, as can be seen from the MLVSS rise for PPIW contaminated wastewater and sample periods displayed in Fig. 7. During the 2-day HRT, the lowest volatile suspended solids concentration which was 2820 mg/L recorded during this stage was also observed during previous HRT stages such as during sampling day 76. During this period, the reactor was operated according to standard, and no setback was encountered that could impact the condition of the reactor. This has shown that the MLVSS rate within the reactor was in a normal condition and has not drastically dropped [32]. During the 1-day HRT, a similar reduction trend was also observed for volatile suspended solids in the reactor. The concentration reduction has depicted that the bacteria in the reactor have reduced, and it was suspected to show further consequences in the next contamination stage.

Despite the signs of detrimental effects of PPIW in the reactor, the HRT was further reduced to obtain more observations on the effects of increment. The fall in biomass concentration brought on by the biosorbents' reactivity and potential rapid increase in toxicity was what caused the MLVSS readings to decline. This contrasts with the results of [40], which showed that biomass and MLVSS concentration were raised by using kenaf powder as a bio-sorbent. The concentration of all effluents increased from Day 15 to Day 60, even though MLVSS concentrations did not change much during this time. This demonstrates that the bacterial population and sorbents in the reactors have both undergone environmental adaptation. The amount of sludge after 30 minutes of settling, temperature, SVI, biomass content, pH, and sorbent characteristics all had an impact on the variations in MLVSS concentration that were seen. The numbers continued to rise during the sampling days until Day 60 when the greatest levels were reached. The conclusions of the current study are consistent with those drawn in a study by Ref. [29] that suggested that the accumulation of precipitated pollutants may be to blame for the rise in MLSS concentration inside reactors. Thus, lowering the MLVSS/MLSS ratio as a result. When MLVSS concentration is 2000 mg/L, the effects of hazardous contaminants are more pronounced. Additionally, it was said that the economics and system performance will be impacted by the buildup of these harmful pollutants in reactors. However, from Day 84 to the final day of sampling, the MLVSS levels started to decrease. This considerable decrease in MLVSS indicates that the reactor's biomass was unable to survive due to high MLSS. The decline might be caused by the lag phase that sludge from all reactors is going through. The MLVSS value reaching its peak near the conclusion of the sampling period is evidence of the RSAC's successful pollutant removal which is the ideal environment for bacterial growth. This is in line with a study that was previously mentioned by Ref. [11].

3.3.2.3. MLSS/MLVSS ratio. To analyze sludge behavior, determination of MLVSS to MLSS is required. Results obtained for the effluent on Day 30 were 0.84. This rate was consistent with those found when a mixture of domestic wastewater and PPIW was evaluated [7]. These ratios continued to rise until day 62 when they abruptly dropped to 0.69. This attests to the fact that more minerals have accumulated in the reactors during the wastewater treatment process using activated sludge [21]. The minimal ratio for the effluent was 0.48, 0.42, and 0.38 while the ratio of maximal ratio was 0.96 for the effluent. The maximal ratio for the effluent was 0.96. This proves the effectiveness of the biosorbent for PPIW treatment. Unexpectedly, the maximum ratio for the blended effluent matches what is frequently considered to apply to sewage treatment plants [41].

3.4. Biokinetics for pulp and paper wastewater treatment in the ASBS

3.4.1. First order substrate removal kinetic

The steady-state data were fitted into the plot in Fig. 8. The substrate removal rate (k_1) was found as 31.9 per day with a correlation coefficient (R^2) of 0.548. The differences between the two slopes presented were observed to be significant as the intercept for slope k_1 was 677.7 and the intercept for slope k_2 was 1335. The obtained k_1 value in this study is in close agreement with the report of a study that reported k_1 constants of 33.533, 16.990, 13.988, and 7.891 per day for BOD_5 concentrations of 0.5–2.5 g/L, respectively in a moving bed biofilm sequencing batch reactor (MBSBR) treating a simulated sugar industry

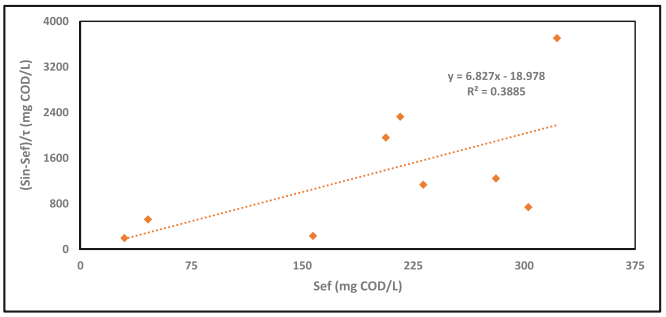

Fig. 8. First-order kinetics for COD in ASBS.

wastewater [42]. However, the low correlation coefficient of 0.548 makes the first-order model unsuitable for the description of the experimental data. In comparison with other studies, it was reported a k_1 of 59.59 per day for COD removal in an adapted SBR treating poultry wastewater whereas a k_1 of 0.615 per day for COD removal in a UASB reactor treating aquaculture wastewater [25]. Several differences in the kinetic coefficients reported in literature have been largely attributed to the bioreactor configuration, wastewater characteristics, type of microorganisms and environmental factors.

3.4.2. *Grau second-order model*

To determine the kinetic coefficients (m, n, and k_s), Fig. 9 was created. Calculating n and m requires using the straight line's intercept and slope, respectively. According to the regression line's equation, the m value corresponding to COD removal was 0.047 ± 0.016 while the n corresponding value was 1.21 ± 0.02. The value of k_s is influenced by the influent COD concentration as well as the biomass concentration in the bioreactor. It goes up as substrate removal effectiveness goes up. For this research, the k_s value of COD was 0.317 per day. Research by Ref. [43] revealed k_s as 3.582 per day. The coefficient of correlation (R^2) of the plot for COD was 0.9980. Similarly, with remarkable (R^2) 0.995 correlation coefficients, m and n values were observed to be 0.0391 and 1.0102, respectively. Previous research reveals that Grau's second-order model constants observed for COD while treating sugar industry wastewater were $k_s = 10^{-3}$. The characteristics of the model and the readings of the coefficients indicate a strong correlation coefficient ($R^2 = 0.97$). The findings attributed to this model align well with the COD. A study computed k_s values as 0.44 d^{-1} for suspended growth while the best correlation coefficient ($R^2 = 0.9999$) was attained at a concentration of 560 mg/L of the influent substrate. The m and n values nearly match those found in a study where m and n were found to be 1.317 and 0.063, respectively. The Grau second-order model, on the other hand, was inadequate for simulating the specified digestion process. The substrate removal constant reported by Ref. [44] was perfectly within the range of ks values noted in other studies.

3.4.3. *Modified Stover-Kincannon kinetic models*

The maximum removal rate constant (μ_{max}) and the saturation value constant (K_B) were obtained from the intercept and slope of the plot of V/Q (S–S) vs V/QS, in Fig. 10. The μ_{max} and K_B for COD removal were found as 0.149 g/L/d and 454.63 g/L./d, respectively, with a correlation coefficient of 0.816. The μ_{max} obtained in this study was lower than many values obtained in literature while the K_B was higher. A study obtained μ_{max} and K_B values of 7.5 and 8.2 g/L/d in a UASB reactor treating agricultural effluent. It has been reported that the K_B value in the modified Stover Kincannon model, increases with an increase in substrate removal with respect to initial substrate concentration and microbial population in the reactor. A study also demonstrated that both μ_{max} and K_B values increased with increasing influent substrate concentration. The authors obtained a μ_{max} and KB values of 16.69 and 16.631 gBOD_5/L, 19.024 and 17.880g BOD_5/L, 23.195 and 21.356 gBOD_5/L, 25.583 and 21.579 gBOD_5/L for inf1uent BOD_5 concentrations of 0.5–2.5 g/L, respectively in a biofilm reactor treating simulated biorefinery wastewater. However, the obtained K_B in this study was close to the report by Ref. [30] who operated two reactors (Rl and R2) with anaerobic granular sludge and activated sludge, respectively in full-scale biogas lift reactors (BLRs) treating food industry wastewater. The authors obtained the kinetic constants μ_{max} and KB as 33.78 and 33.89 kg COD/m^3 d for Rl, 243.90, and 268.85 kg COD/m^3 d for R2. Additionally, another study by Ref. [45] demonstrated that the surface area of a submerged media can influence the substrate removal rate in upflow anaerobic fixed bed reactor treating winery wastewater and packed with three different media with a specific surface area of 800, 320 and 305 m^2/m^3, respectively.

The coefficients found here are compared to the substrate removal kinetic constants reported in the literature summary highlighted in Table 7. Many researchers have employed various substrates and bioreactors, and they have obtained varying values of kinetic constants. The Grau Second order and Modified Stover Kincannon models, rather than the first order model, provided a better explanation of the biokinetics of COD degradation when the regression and biokinetic coefficients were examined. Although the microbial population in the reactor grew, there was a significant decline in the values of ks as HRT dropped. The variance in the mean biokinetic constants between studies suggests that factors such as substrate concentration, substrate type, reactor configuration, operational conditions, and the existence of microbial consortia can all have a major impact on kinetic coefficient values.

4. Conclusion

This study used rice straw-activated carbon as a biosorbent to evaluate the performance of the ASBS in the treatment of PPIW. The study objectives were accomplished through a series of conceptualized experiments. The influence of HRT on ASBS was noticed for COD removal, especially at short HRT. However, the maximum loading rate was not attained at all HRT conditions investigated. This suggests that the

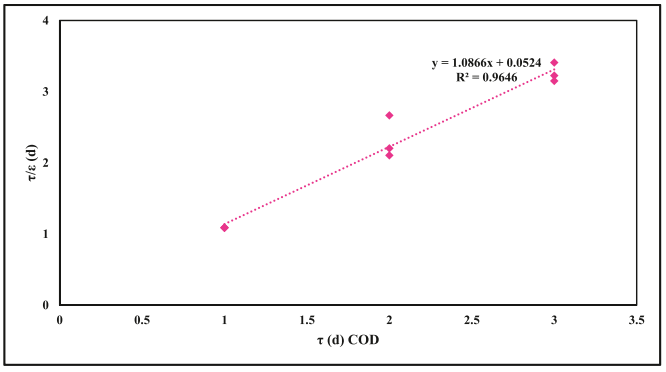

Fig. 9. Second-order kinetics for COD in ASBS.

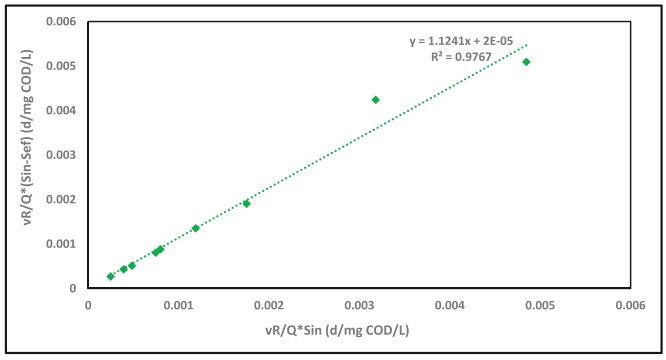

Fig. 10. Modified stover kinetics for COD in ASBS.

Table 7
Kinetic constants for COD removal in ASBS.

Wastewater	Kinetic model							Ref.
	First order model		Grau second-order model			Modified Stover–Kincannon model		
	k_1 (1/d)	R^2	c	d	k_S (1/d)	U_{msr} (g/L/d)	K_V (g/L/d)	
Pulping industry wastewater	0.0419	0.8624	49.022	3.418	0.0773	1.923	3.2	[28]
Paper and pulp wastewater	1.297	0.570	0.381	0.886	0.993	2.011	0.38	[1]
Pulp and paper industry wastewater	0.084	0.812	4.042	0.741	0.586	1.901	3.025	[3]
Pulp wastewater	1.2	0.78	1.5	2.3	2.19	0.06297	0.0263	[17]
Biorefinery wastewater	0.1581	0.8046	9.1742	0.518	21.0	0.0110	0.0104	[13]
Synthetic wastewater	0.297	0.882	0.884	0.7503	23.25	5.38	8.76	[44]
Palm oil mill effluent	0.8015	0.9218	1.853	0.793	0.0218	0.423	0.1905	[45]
PPIW	3.801	0.7204	1.109	0.0482	29	18	18.218	This study

bioreactor has the capacity to tolerate lower HRT conditions. The embedded carbonated rice straw provided a large surface area for biomass immobilization and accumulation. This was evident in the high concentration of MLVSS in the ASBS. The achievement of complete nitrification and denitrification indicates the good oxygen mass transfer capacity of the bioreactor and non-inhibitory microbial influence. The underlying principle for effective organic matter removal in this study was the operational sequence and the inclusion of rice straw in the aerobic units. The mechanisms responsible for high substrate removal in the aerobic unit of the bioreactor were organic matter oxidation, nitrification, cell synthesis, and endogenous respiration. Thus, the ASBS can be used to increase the footprint efficiency wastewater treatment. The kinetic coefficients were determined using the First order kinetic model, Grau second-order model, and Modified Stover Kincannon models. The substrate removal and microbial growth parameters were best described by the Modified Stover Kincannon model. This observation suggests that the substrate utilization rate in ASBS was a function of the organic loading rate whereas the maximum specific growth rate relied on the single limiting substrate in the wastewater. Thus, the model is more suitable for the prediction of effluent substrate concentration in ASBS.

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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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.cscee.2022.100261.

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