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Performance evaluation of ICX reactor in treatment of paper mill wastewater: a case study in South Vietnam

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ABSTRACT

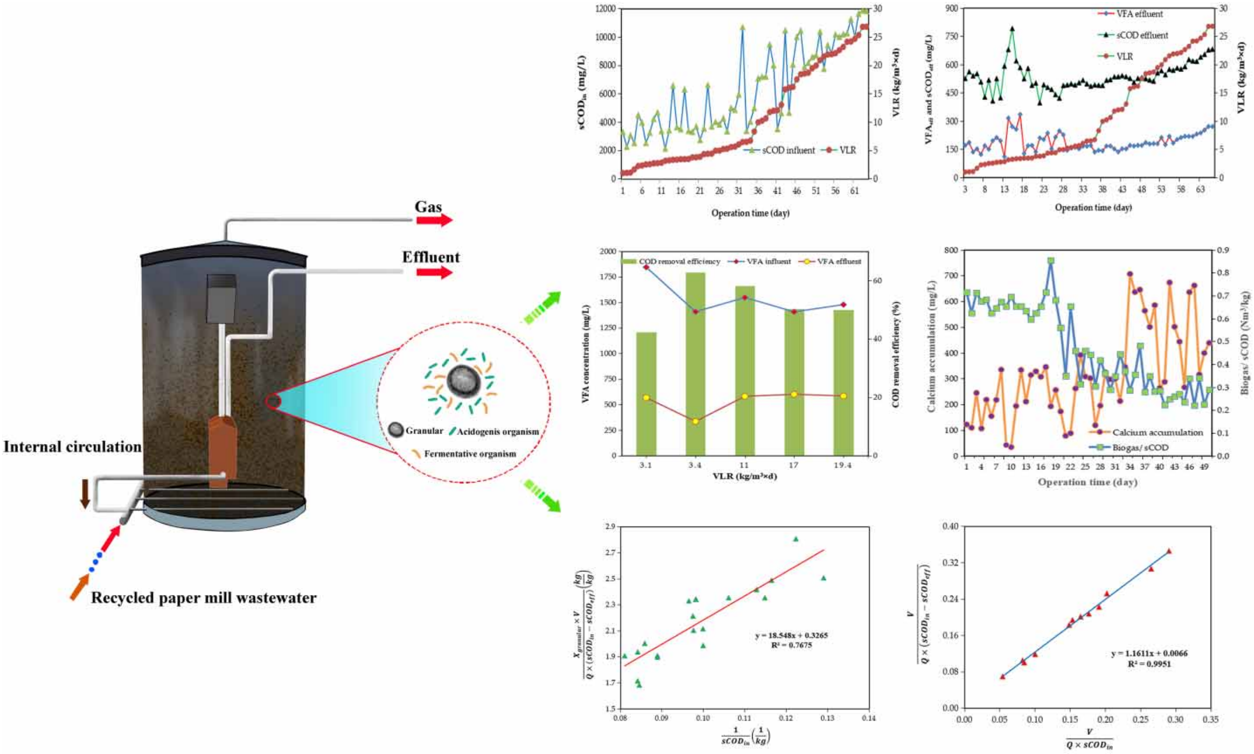
This study evaluates the performance of the Internal Circulation eXperience (ICX) reactor in treating high-strength paper mill wastewater in the south of Vietnam. The ICX reactor effectively managed organic concentrations (sCOD) of up to 11,800 mg/L. Results indicate a volumetric loading rate (VLR) of 26.8 $kg/m^3 \times day$, achieving processing efficiency exceeding 81% while consistently maintaining volatile fatty acids (VFA) below 300 mg/L. The study employed Monod and Stover–Kincannon kinetic modeling, revealing dynamic parameters including $K_s = 56.81$ kg/m^3, $Y = 0.121$ kgVSS/kgsCOD, $K_d = 0.0242$ 1/day, $\mu_{max} = 0.372$ 1/day, $U_{max} = 151$ kg/m$^3 \times$ day, and $K_B = 175.92$ kg/m$^3 \times$ day, underscoring the ICX reactor's superior efficiency compared to alternative technologies. Notably, the reactor's heightened sensitivity to VFA levels necessitates influent concentrations below 1,400 mg/L for effective sludge treatment. Furthermore, the influence of calcium on treatment efficiency requires post-treatment alkalinity maintenance below 19 meq/L to stabilize MLVSS/MLSS concentration. Biogas production ranged from 0.6 to 0.7 Nm^3 biogas/kg sCOD; however, calcium impact diminished this ratio, reducing overall treatment efficiency and biogas production. The study contributes valuable insights into anaerobic treatment processes for complex industrial wastewaters, emphasizing the significance of controlling VFA, calcium, and alkalinity for optimal system performance.

Key words: anaerobic wastewater treatment, ICX reactor, inhibition anaerobic, kinetic modeling, paper mill wastewater

HIGHLIGHTS

- ICX excels in treating high-strength paper mill wastewater (sCOD $>$11,800 mg/L).
- ICX maintains stability at a remarkable VLR of 26.8 kg/m$^3 \times$ day.
- Monod and Stover–Kincannon models reveal ICX's superior efficiency.
- ICX's heightened sensitivity to VFA levels ensures optimal sludge treatment.
- Calcium concentration significantly reduces biogas production in recycled paper mill wastewater.

GRAPHICAL ABSTRACT



1. INTRODUCTION

Pulp and paper production, being one of the world's largest industries and the fifth most energy-consuming sector globally (Osaki 2019), confronts a critical juncture necessitating sustainable practices aligned with global initiatives such as the 28th UN Climate Change Conference of the Parties (COP28) in 2023. As the industry endeavors to align with sustainability goals, recycling paper using recovered materials emerges as an imperative option. Approximately 17% of the 285 million tons of global waste paper produced in 2019 were exported for recycling in 2019 (Provost-Savard *et al.* 2023). In the United States, over 70% of carton boxes produced in 2022 were successfully recovered for recycling (Ketkale & Simske 2023).

The packaging paper industry, a major consumer of recovered cardboard, faces the dual challenge of meeting growing demand while minimizing environmental impact. With the global packaging paper market projected to grow nearly 3% annually, reaching a value surpassing 1.2 trillion USD by 2028 (Keskin *et al.* 2020), it becomes imperative to explore environmentally conscious approaches. In Vietnam, packaging paper production was estimated to reach 4.3 million tons per year by the end of 2022, with exports totaling about 1.2 million tons per year. This figure is expected to rise to 1.3 million tons per year by the end of 2023 (Vietnam Pulp and Paper Association). However, this growth raises concerns about freshwater ecotoxicity and the carbon footprint associated with wastewater treatment processes, underscoring the need for sustainable practices in the industry (Bui *et al.* 2022).

The potential freshwater ecotoxicity linked with packaging paper ranges from 3.75 to 11.58 kg1,4-DCB-eq/ton of paper. As production increases, so does the pollution load on the environment due to heightened wastewater discharge demands. Wastewater from the packaging paper industry exhibits substantial organic pollution content, with total chemical oxygen demand (COD) over 20,000 mg/L (Harif *et al.* 2021), alongside significant calcium content ranging from 1,000 to 2,000 mg/L (Wang *et al.* 2023). These environmental challenges underscore the importance of adopting sustainable practices and technologies to reduce the carbon footprint of wastewater treatment processes.

Presently, upflow anaerobic sludge blanket (UASB) technologies constitute approximately 67% of global wastewater paper and pulp mill treatment methods, with other high-load treatment technologies such as anaerobic baffled reactor, expanded granular sludge bed (EGSB), fluidized bed reactor, accounting for around 33% (Zieliński *et al.* 2023). Despite their widespread use, UASB methods encounter challenges in granular sludge retention, leading to operational inefficiencies, elevated carbon emissions and reduced effectiveness under low organic load rates. In response to these challenges, the internal circulation (IC) reactor emerges as a noteworthy alternative within the UASB technology domain. IC anaerobic technology overcomes UASB limitations with a unique design featuring two vertically stacked cells and two three-phase separators. A proficient three-phase separator ensures substantial retention of sludge mass and extends the average solids retention time. This leads to efficient biogas generation at high hydraulic velocities, retaining 77.4–81.1% of biomass and enhancing the overall efficiency and effective volume of the anaerobic system by 16–25% (Pan *et al.* 2017; Guo *et al.* 2018; Hao & Shen 2021). Moreover, IC characterized by a negative carbon emissions index compared to traditional UASB technology (Bui *et al.* 2022), is specifically tailored for paper wastewater treatment, demonstrating robust and consistent treatment efficiency within a compact footprint. Comprehensive studies validate IC technology's capability to manage COD levels up to 10,000 mg/L and volumetric loading rate (VLR) reaching 20–25 kg/m^3 day (Chen *et al.* 2019). However, the limited application of IC systems in practical settings is due to substantial space and material requirements, resulting in increased construction time and costs. The new generation of IC, Internal Circulation eXperience (ICX), could address this issue, particularly for small paper and pulp factories. Motivated by the promising capabilities of the IC reactor, ICX is designed to enhance the efficiency of paper wastewater treatment while reducing the system height. ICX has a single reaction compartment and two-phase separators to ensure stable granular sludge retention. Notably, ICX employs the down-flow technique, pumping wastewater from the bottom to create high-speed agitation of sludge particles. The treated water is then directed to the top-phase separator to reduce turbulent flow and separate biogas. Subsequently, sludge and treated wastewater move to the lower-phase separator, where the sludge layer acts as a filter, preventing sludge escape. The treated water exits the system through the riser, while the separated sludge is circulated back into the reaction sludge layer. This innovative design not only makes ICX accessible for small plants but also simplifies construction, shortening the startup time for wastewater treatment (Noordink *et al.* 2018; Hendrickx *et al.* 2019).

Despite these advances, there is a scarcity of studies investigating the application of ICX in wastewater, particularly in paper and pulp wastewater. This study presents the operation of one ICX in a paper and pulp mill in Vietnam, examining treatment capacities, and the effect of various factors such as VLR, volatile fatty acids (VFA), alkalinity, calcium ion, and biogas production yield. This nuanced examination aligns with the overarching objectives of COP28 and broader sustainable development initiatives, advancing the discourse on environmentally responsible practices in industrial processes.

2. EXPERIMENTAL

2.1. ICX reactor

The study was carried out at a paper mill wastewater in the South of Vietnam (Figure 1).

The experiment spanned 90 days, comprising 7 days for load and equipment testing, 1 day for O_2 removal in the ICX reactor (using N_2), and 12 days for sludge loading. The ICX reactor was inoculated with sludge to handle the theoretical pollutant load, estimated at approximately 120 tons of sludge with with a ratio of mixed liquor volatile suspended solids to mixed liquor suspended solids (MLVSS/MLSS) content of 0.87 (Tian *et al.* 2023). The sludge for system inoculation was sourced from an IC reactor at a different paper mill during wastewater treatment. After sludge inoculation, the VSS concentration was verified and the IC system was activated. As the ICX began biogas production, the ICX reactor was initiated. Since the sludge had already adapted from the operating IC reactor, it took only about 2 h for transportation and 3 h for sludge loading, making the ICX system operational after 8 h of IC. The design parameters of the ICX are presented in Table 1.

During operation, nutrient concentrations were maintained at a COD:N:P ratio of 650:5:1. Due to the high alkalinity in the wastewater, no NaOH supplementation was required; however, HCl acid (32%, Dong A Company, Vietnam) was added to the treatment process to maintain stable pH. The characteristics of paper production wastewater are described in Table 2.

2.2. Kinetic modeling

The study employs a comprehensive approach by integrating the Stover–Kincannon and Monod models in the investigation of anaerobic systems. The Stover–Kincannon model focuses on substrate removal rates, providing a crucial perspective on reactor performance. In contrast, the Monod model emphasizes the relationship between microbial growth and substrate concentration.

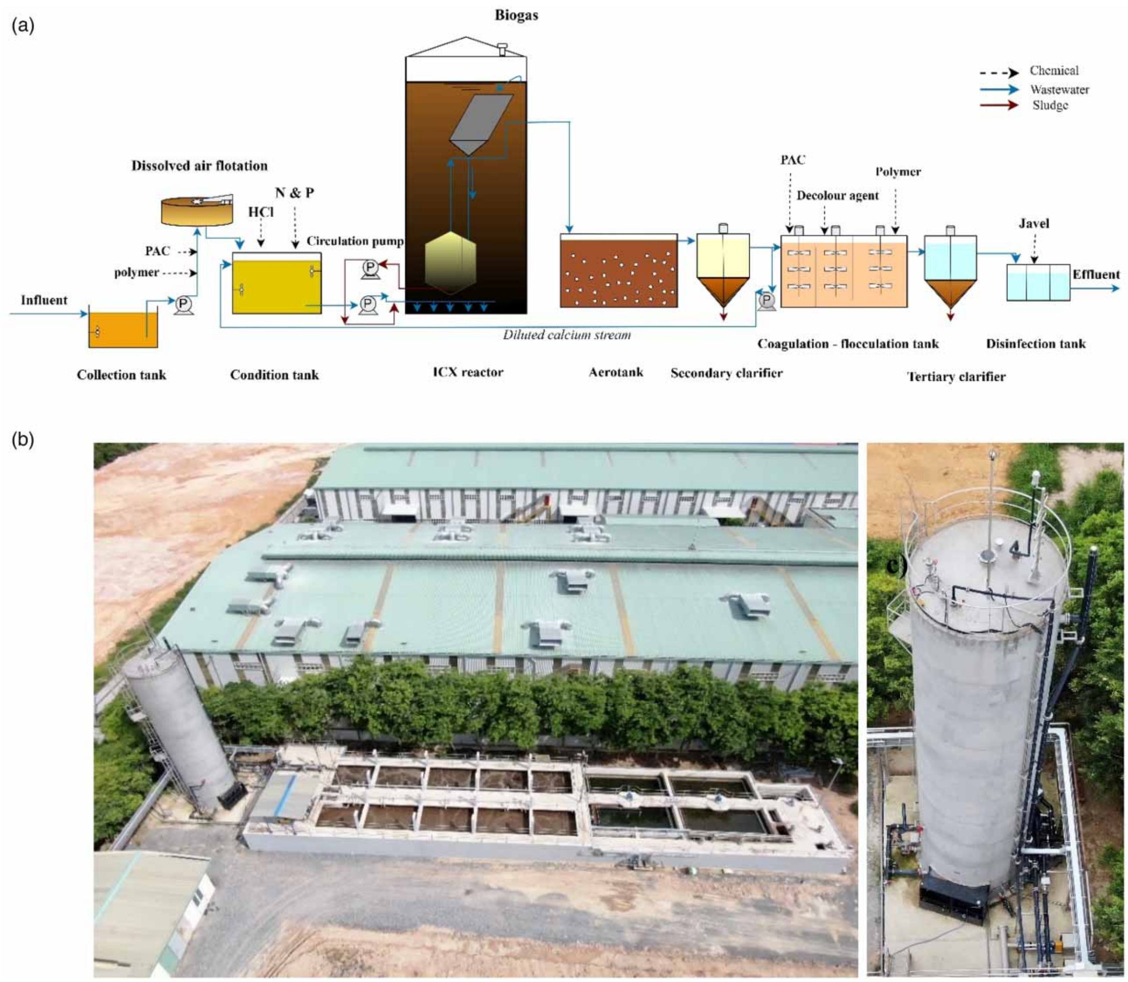


Figure 1 | The flowchart of (a) the actual process, and (b) the ICX reactor of the paper and pulp wastewater treatment plants in this study.

Table 1 | Initial design values and operation parameters for the ICX reactor

Factor	Unit	Design of ICX reactor	Remarks
Granular sludge retention time	Day	60–90	Optimize 35 °C
X	g/L	50 (43.5)	MLSS (MLVSS)
C	mg/L	9,000	Total COD
SST	–	0.041	COD_{ss}/COD_t influent
R		0.92	Fraction of COD_{ss} removed
Total volume/water volume	m^3	192/188	The overall volume of the tank comprises both water and biogas
Surface area	m^2	12.6	The tank is cylindrical with a diameter of 4 m
Height reactor/water	m	16/15	The tank's height is designed to accommodate a range of water levels, including the maximum allowable level
Velocity	m/h	≥ 6	This calculation considers input wastewater flow and internal circulation
Volume biomass	m^3	163	The total volume is determined by measuring from the bottom to the top water levels, taking into account the first three phases
HRT	h	~ 2 h	It includes both the internal circulation flow and the input water

Table 2 | Composition of the raw wastewater and ICX influent

Type wastewater	Q (m^3/h)	tCOD (mg/L)	sCOD (mg/L)	pH	TSS (mg/L)	Ca^{2+} (mg/L)	SO_4^{2-} (mg/L)	TN (mg/L)	TP (mg/L)	VFA (mg/L)
Raw	<500	4,500–14,000	3,000–11,800	6.8–7.1	2,000–3,000	400–2,200	50–150	30–55	10–17	1,450–4,960
Dissolved air flotation effluent	<500	3,100–12,200	2,800–11,800	6.5–6.8	200–230	400–22,00	50–148	28–30	9–12	1,650–6,500
[a]Vietnam Standard	–	<100	–	6–9	<50	–	–	–	–	–

[a]National technical regulation on the effluent of pulp and paper mills.

The Stover–Kincannon model depicts the substance removal rate at a steady state as a function of the organic loading rate, formulated in the following equation (Tian *et al.* 2023).

$$\frac{V}{Q \times (\mathrm{sCOD_{in}} - \mathrm{sCOD_{eff}})} = \frac{K_B}{U_{\max}} \times \frac{V}{Q \times \mathrm{sCOD_{in}}} + \frac{1}{U_{\max}} \tag{1}$$

where U_{max} is the maximum rate constant of substrate consumption (g/L × day); K_B is the saturation constant (g/L × day); V is the empty reactor bed volume (L); Q is the influent flow rate (L/day).

The Monod describes the relationship between microbial growth rate and substrate concentration. It is based on the premise that microbial growth is limited by the availability of a single limiting substrate as follows (Jijai *et al.* 2016).

$$\frac{Q \times (\mathrm{sCOD_{in}} - \mathrm{sCOD_{eff}})}{V \times X} = \frac{1}{Y \times \theta_c} + \frac{K_d}{Y} \tag{2}$$

where Y is the biomass yield (kg VSS/kg sCOD); K_d is the endogenous decay coefficient (1/day); X is the microorganism concentration (kg/m^3); θ_c is the cell-residence time (day) or using Equation (3) which is modified from Equation (2) to predict effluent substrate concentration (Equation (4)) in the ICX reactor.

$$\frac{V \times X}{Q \times (\mathrm{sCOD_{in}} - \mathrm{sCOD_{eff}})} = \frac{K_s}{q_{\max} \times \mathrm{sCOD_{in}}} + \frac{1}{q_{\max}} \tag{3}$$

$$\mu_{\max} = q_{\max} \times Y \tag{4}$$

where q_{max} is the maximum substrate consumption rate (kg sCOD/kgVSS $^{\times}$day); K_s is the half velocity constant (kgCOD/m^3); μ_{max} is the maximum specific growth rate (1/day).

2.3. Analytical method

The water analysis was collected at the effluent of each tank, which covered diverse water quality parameters, encompassing COD or total COD (tCOD) total nitrogen (TN), total phosphorus (TP), total solids (TS), volatile solids (VS), total suspended solids (TSS), volatile suspended solids (VSS), alkalinity, VFA, calcium, sulfate ion, and pH. The VFA analysis was conducted based on insights from our previous study (Nhat-Ha & Manh-Ha 2019), while other parameter analyses strictly followed the standard methods of water and wastewater by the American Public Health Association (Rice *et al.* 2012). While anaerobic granulars were taken at the valves at 1, 3, 5, 7, 9, 11, and 13 m according to the height of the ICX reactor.

Gas production was systematically measured daily using a thermal flow meter (Combimass ECO Binder Group, Germany) and the outcomes were verified through validation using a gas chromatograph (GC-8A Shimadzu) with a flame ionization detector.

The removal efficiency of soluble chemical oxygen demand (sCOD), denoted as H, was calculated using the following equation.

$$H = \frac{Q_{\mathrm{in}} \times \mathrm{sCOD_{in}} - Q_{\mathrm{out}} \times \mathrm{sCOD_{out}}}{Q_{\mathrm{in}} \times \mathrm{sCOD_{in}}} \tag{5}$$

To maintain stable operation and control of ICX under organic load conditions, the organic load entering the ICX tank is regulated by the VLR, as depicted by the following equation:

$$\mathrm{VLR} = \frac{Q_{\mathrm{in}} \times \mathrm{sCOD}_{\mathrm{in}}}{V_{\mathrm{ICX}}} \quad (6)$$

3. RESULT AND DISCUSSION

3.1. Performance of the wastewater treatment system

In Figure 2(a), ICX impressively removes over 70% of sCOD, dealing with levels from 2,500 to 11,800 mg/L within 12 days of starting the treatment for paper wastewater. The first 12 days show efficiency below 70% as the granular sludge in ICX gets used to the wastewater. The highest removal rate hits 85%, even at sCOD levels of 10,000–11,000 mg/L, reaching 89% at COD levels of 8,000 mg/L.

This lines up with EGSB technology, data presented in Table 3, which typically achieves less than 77% removal of sCOD in paper mill wastewater (Liang *et al.* 2021). Yet, ICX is 10 times faster than EGSB, operating in just 2 h vs. 20. The VLR, evaluating the system's capability with flow rate and sCOD, gives a clearer picture of the overall sCOD removal efficiency in 24 h. Practical observations show VLR for packaging paper ranging from 2 to 26.8 kg/m^3 × day (see Figure 2(b)). Results indicate removal efficiency below 70% for VLR under 5 kg/m^3 × day, but it jumps over 80% for VLR between 5 and 26.8 kg/m^3 × day.

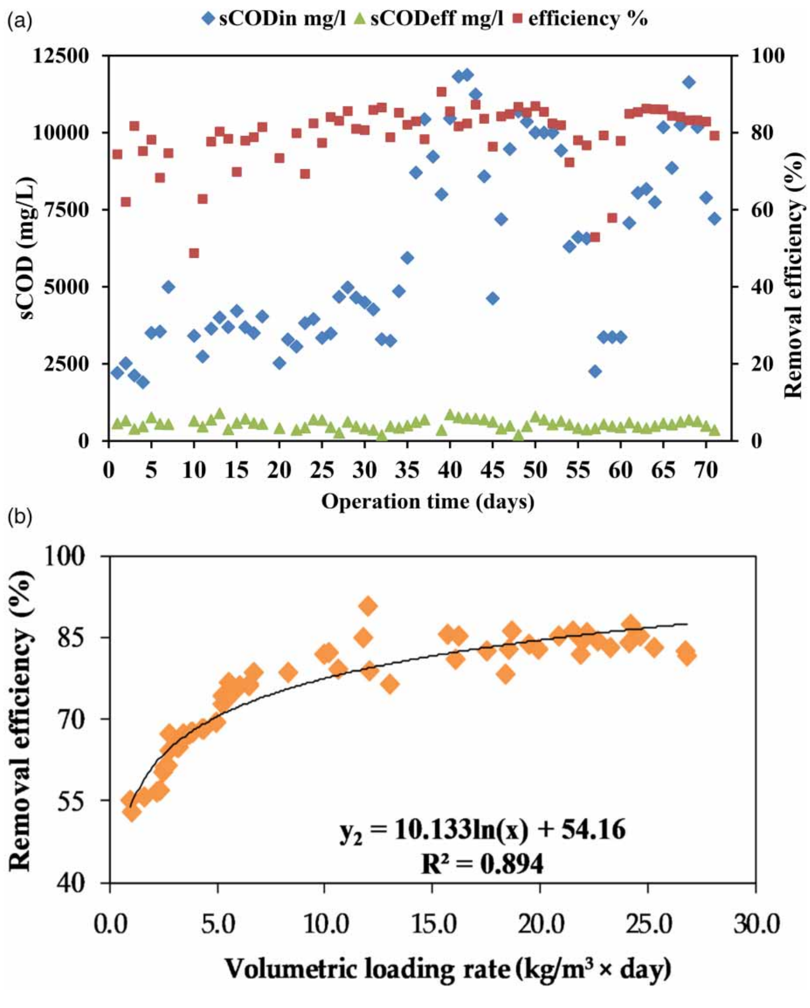


Figure 2 | Change in (a) sCOD and (b) VLR during the digestion of the ICX reactor.

Table 3 | VLR of some anaerobic paper mill wastewater treatment

Type anaerobic	Wastewater	VLR (kg/m^3/day)	Removal efficiency sCOD	Notes
ICX	Paper mill	5–26.8	70–89%	This study
IC	Pulp and paper (case study)	5–14	75–78%	Nhat-Ha & Manh-Ha (2019)
	Molasses	2.5–18.94	89–96%	Luo *et al.* (2016)
EGSB	Pulp and paper (case study)	6–21	65–80%	Liang *et al.* (2021)
UASB	Recycled pulp and paper	< 8.31	70–80.63%	Bakraoui *et al.* (2020)

The top efficiency is 89% at VLR of 24.2 kg/m^3 × day, slightly dropping to 81% at VLR of 26.8 kg/m^3 × day. This confirms ICX's suitability for treating organic loads when sCOD exceeds 5 kg/m^3 × day. Extended retention times in UASB reactors for paper wastewater start impacting COD treatment efficiency at over 96 h (Bakraoui *et al.* 2020). In contrast, ICX manages this in just 12 h. ICX has a higher VLR than other anaerobic technologies like EGSB. For paper mill wastewater, IC has a VLR range of 2.5–18.94 kg/m^3 × day, treating leachate from a molasses wastewater with up to 96% efficiency (Luo *et al.* 2016), with IC's retention time at 15.84 h. So, ICX might be around 41% more effective. During tests, ICX's VLR can reach up to 50 kg/m^3 × day with efficiency up to 72% (Hendrickx *et al.* 2019). Keeping VLR between 25 and 35 kg/m^3 × day could hit up to 80% efficiency for the paper mill wastewater in Allard Emballages, France (Noordink *et al.* 2018). However, for paper mill wastewater in Vietnam, the suitable VLR should stay under 26.8 kg/m^3 × day due to falling treatment efficiency, linked to the high organic sCOD concentration in the paper industry (sCOD up to 11,800 mg/L) and the high calcium concentration (Nhat-Ha & Manh-Ha 2019).

The role of VLR within the ICX system is evaluated through the fluctuations in VFA, specifically propionic and butyric acids. VFAs play a pivotal role in anaerobic digestion, significantly impacting the performance of acetogenic and methanogenic bacteria. The accumulation of VFAs results from the acidogenic phase during the conversion of organic compounds. Consequently, monitoring VFA dynamics in the ICX reactor becomes a crucial tool for assessing system stability.

Figure 3(a) reveals that the increase in sCOD in the influent stream minimally affects the stable processing efficiency of the system and VFA concentrations. In addition, VFA stability hinges on VLR fluctuations. With VLR ranging from 2 to 26.8 kg/m^3 × day, VFA concentrations fluctuated between 120 and 300 mg/L. These findings indicate that the anaerobic digestion system maintains stability when VFA concentrations remain below 300 mg/L. The dynamics of VFAs in the ICX reactor during the treatment of paper and packaging wastewater align with prior studies on IC systems (Nhat-Ha & Manh-Ha 2019). ICX demonstrates stability even with a VLR of 26.8 kg/m^3 × day, achieving an 81% treatment efficiency with consistent VFA concentrations at 250 mg/L.

As VFA concentrations reach 350 mg/L, sludge disintegration occurs, resulting in a decrease in COD treatment efficiency to approximately 50–60% (Figure 3(b)). This finding is consistent with previous studies by Batstone & Steyer (2007) using batch UASB, where VFA concentrations were maintained within the 300–600 mg/L range for wine production wastewater

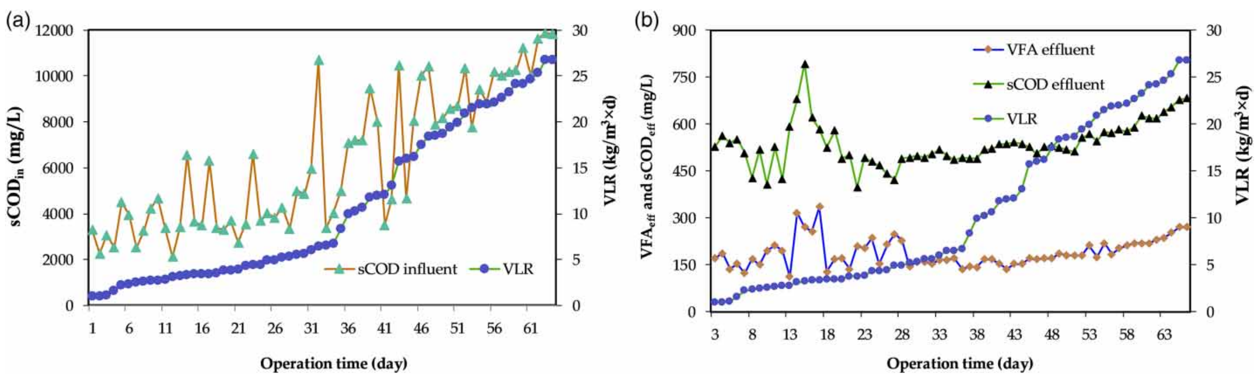


Figure 3 | The variation of VFA, sCOD (a) influent and (b) effluent in an ICX reactor.

and below 500 mg/L. Another study by Heydari *et al.* (2019) on a continuous UASB system indicates that maintaining VFA concentrations below 180 mg/L during the startup phase contributes to sustaining stable treatment capabilities. However, when VFA concentrations exceed 450 mg/L with an organic loading rate of 2.69 kg COD/m^3 × day, treatment efficiency decreases to approximately 60%.

In the case of EGSB treating simulated dairy wastewater, when VFA < 200 mg/L with VLR < 14 kg/m^3 × day, the system remains stable. However, when the VLR increases to 28 kg/m^3 × day, the system experiences shock loading, with VFA levels ranging from 500 to 600 mg/L (Mills *et al.* 2023). Another study utilizing EGSB for *Cassava* alcohol wastewater treatment showed that during the startup phase with VLR ranging from 1.55 to 5.37 kgCOD/m^3 × day, stable VFA concentrations were maintained below 300 mg/L. However, when the VLR increased to 16.14 kgCOD/m^3 × day, methane production efficiency decreased by 60%, with VFA levels ranging from 450 to 500 mg/L (Xu *et al.* 2023). These findings indicate that high-rate anaerobic technologies such as EGSB and ICX need to maintain VFA concentrations below 300 mg/L for stable operation. ICX demonstrates superior capability in handling higher loads and maintaining stability compared to UASB and EGSB, as it can sustain stable VFA levels even at higher loads. Table 4 summarizes the stable VFA concentrations observed in various anaerobic treatment systems.

3.2. Effect of parameters on ICX performance

The concentration of VFA, calcium, and alkaline of the input to the ICX reactor were varied according to the dilution between the influent (after the conditioning tank) and the effluent (after the secondary clarifier), please see Figure 1.

3.2.1. VFA influence

As illustrated in Figure 4, the fluctuations in influent VFA concentration have a significant impact on both the effluent VFA concentration and COD removal efficiency. An increase in removal efficiency and a decrease in effluent VFA concentration were observed, reaching optimal values at an influent VFA concentration of 1,400 mg/L and a VLR of 3.4 kg/m^3 × day. However, at higher influent VFA concentrations, the COD removal efficiency dropped below 60% and the effluent VFA concentration remained around 600 mg/L, leading to the destabilization of granular sludge, which could potentially transition into activated sludge over extended periods. This instability in granular sludge aligns with findings in the study by Castilla-Archilla *et al.* (2021) over a 9-day period with an influent VFA concentration of approximately 1,500 mg/L in the EGSB reactor. Researchers such as Wong *et al.* (2008) and Al-Sulaimi *et al.* (2022) have indicated that influent VFA concentrations can significantly affect the stability of methanogenic bacteria, crucial for biogas production. Methanogenic bacteria require over 200 h to release CH_4 when the VFA concentration exceeds 20 g/L. Based on these results, it is recommended to maintain influent VFA concentrations below 1,400 mg/L in the ICX reactor to ensure optimal system stability and performance.

3.2.2. Calcium concentration influence

In the context of paper mill wastewater treatment, calcium ion emerges as a crucial factor due to their long-term impact on the biomass stability within the anaerobic digestion system. This is attributed to the precipitation of $CaCO_3$ in the sludge matrix. Chen *et al.* (2023) proved that calcification phenomena led to a significant reduction in COD removal efficiency, dropping from 98 to 41.2% over a 200-day period, subsequently impacting the biogas production efficiency. Figure 5 illustrates the fluctuation in calcium concentrations in this wastewater, showing a tendency to increase with sCOD. This trend may be attributed not only to the initial calcium content in the influent wastewater but also to the accumulation process within

Table 4 | VFA thresholds for stable anaerobic digestion processes

Technology	Type wastewaters	VLR (kg/m^3 × day)	Stable VFA (mg/L)	References
ICX	Recycled paper	5–26.8	<300	This study
UASB	Oil	<2.69	<180	Heydari *et al.* (2019)
EGSB	Dairy	<14	<200	Mills *et al.* (2023)
EGSB	*Cassava* alcohol	<16.5	300	Xu *et al.* (2023)

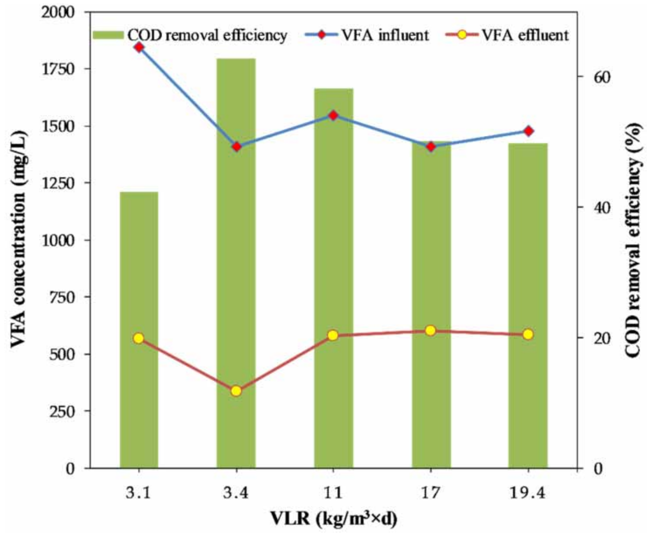


Figure 4 | Effect of VFA on the removal performance of the ICX reactor.

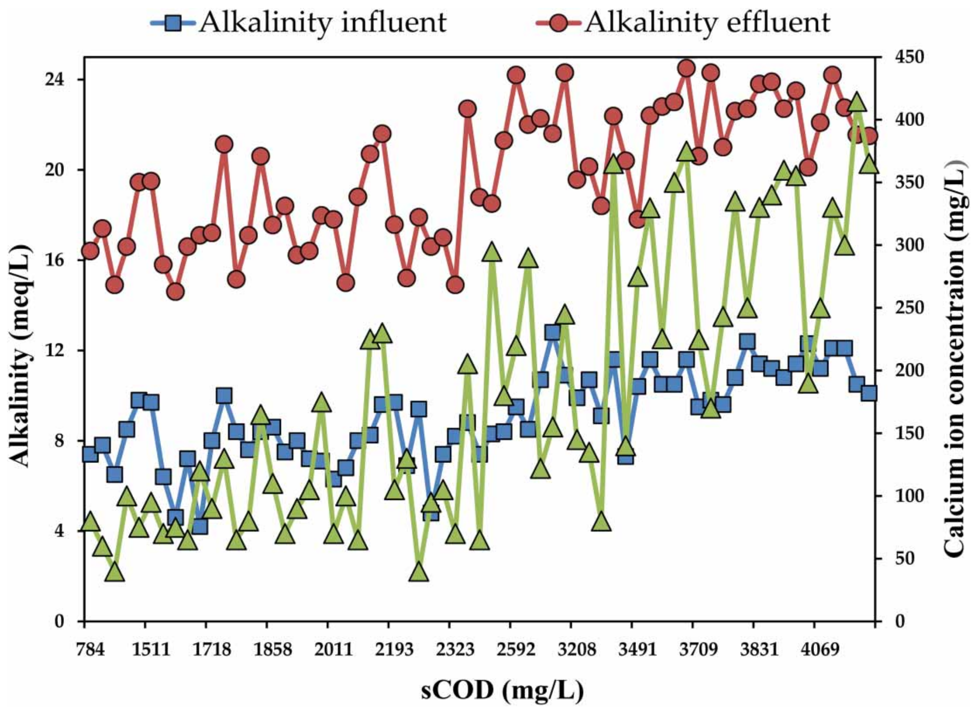


Figure 5 | Variation of alkalinity and calcium ion in the ICX reactor.

the sludge particle composition. In this ICX reactor, the influent calcium concentration and pH are maintained below 800 mg/L and 6.8, respectively (Wang *et al.* 2023).

3.2.3. Alkalinity concentration influence

Typically, the alkalinity increases with the rise in sCOD due to the formation of CO_2, which can exist in the solution as ions ion CO_3^{2-}, HCO_3^- (Chen *et al.* 2023). However, in the ICX reactor, the role of alkalinity in relation to the concentration of calcium ions and the stability of sludge is more apparent. As shown in Figure 5, when the effluent alkalinity in the ICX system is maintained below 19 meq/L, the accumulation of calcium in the biomass remains in the range of 70–160 mg/L.

At this concentration, it aligns with the development of granular sludge, proven to enhance the production of protein extracellular polymeric substances and maintains a MLVSS/MLSS ratio in the sludge of 0.85 with influent sCOD less than 5,000 mg/L (Wang *et al.* 2023). Nevertheless, when the effluent alkalinity in the ICX exceeds 19 meq/L, the accumulation of calcium in the biomass fluctuates between 200 and 400 mg/L. At this point, the MLVSS/MLSS ratio decreases to about 0.72 (reaching 0.69 at the bottom of the 1 m depth after 1 month of operation). Despite this, the sCOD removal efficiency is still maintained above 70%, a result consistent with the findings of Zhou *et al.* (2019). However, the prolonged variations in VSS over time led to an increase in sludge density, necessitating additional energy for sludge recirculation, consequently diminishing the effective treatment volume of the reactor. This issue has also been addressed in the study by Diamantis & Aivasidis (2018) involving an EGSB reactor. In their case, maintaining a velocity of > 5 m/h through an increased VLR > 15 $kg/m^3 \times day$ successfully induced calcium precipitation in the range of 15–33%. However, this maintenance strategy did not yield the expected results for the ICX system in this study. With a water flow maintained at 6.5–8.5 m/h, calcium precipitation reached 40–60%, leading to the excessive accumulation of calcium in the sludge. This not only increased the energy requirement for recirculation but also reduced the sludge's lifespan. Practical operation of the ICX reactor has demonstrated the need for additional acid supplementation to maintain alkalinity below 19 meq/L for sludge stability. However, this approach incurs significant costs and environmental and health risks for operators. In addition, the primary influencing factor is the direct relationship between influent sCOD and calcium. The use of recirculated water after treatment in the production process dilutes both sCOD and calcium in the wastewater.

3.3. Biogas production

As depicted in Figure 6(a), the biogas production potential ranges from 0.22 to 0.83 Nm^3 biogas/kg sCOD, showing a gradual decrease as the sCOD removal efficiency increases, possibly due to the rise in influent sCOD. When the influent sCOD increases, it leads to an elevation in calcium content in the wastewater (Figure 5), resulting in the accumulation of Ca^{2+} in the sludge flocs. This accumulation enhances $CaCO_3$ precipitation, reducing CO_2 and making it challenging to release CH_4 from the sludge flocs. The biogas production rate during the stable phase ranges from 0.6 to 0.7 Nm^3 biogas/kg sCOD. If the sludge accumulates calcium at levels of 100–300 mg/L, the biogas production rate decreases to approximately 0.3–0.45 Nm^3 biogas/kg sCOD. For higher calcium accumulation (>400 mg/L), the biogas production rate further decreases to about 0.22–0.3 Nm^3 biogas/kg sCOD (Figure 6(b)). The biogas production rate of the ICX in this system is comparable to other studies on sludge, achieving around 70% as observed by Noordink *et al.* (2018) and 0.33–0.6 Nm^3 biogas/kg sCOD (Umiejewska 2019) and 0.38 Nm^3 biogas/kg (Musa *et al.* 2018).

Monitoring the graph depicted in Figure 6(b) reveals a notably higher biogas production efficiency/sCOD compared to previous anaerobic digestion studies, reaching 0.85 on day 16. Remarkably, the system demonstrates resilience in sustaining biogas production even when subjected to high calcium levels. This suggests that biogas generation is influenced by the nature of the sludge, with floc sludge yielding higher biogas output compared to granular sludge (Wang *et al.* 2018). Furthermore, biogas production results in the formation of large-sized sludge particles (3–3.5 mm), medium-sized particles (1.5–2 mm), and small-sized particles (0.5–1 mm) with correspondingly highest specific biogas production rates of 0.031, 0.016, and 0.006 m^3 /kgVSS day, respectively (Wu *et al.* 2016). In addition, due to the higher VLR in the ICX reactor compared to UASB technology, there is more effective transport of COD into the sludge bed, leading to improved biogas production rates (Afridi *et al.* 2019). Moreover, the predominant *Methanosarcina* and *Methanosaeta* bacterial strains play a crucial role in the conversion of organic matter into biogas. These strains demonstrate a higher density within the ICX reactor compared to other anaerobic digestion systems, even under conditions of high sludge retention times and turbulent flow (Andrén 2018). The empirical data obtained from this ICX system, featuring a sludge retention time exceeding 80 days and an average hydraulic velocity of 8.5 m/h, may provide conducive conditions for the maintenance and proliferation of both *Methanosarcina* and *Methanosaeta* strains, which were mentioned in the investigation of Owusu-Agyeman *et al.* (2019).

3.4. Kinetic coefficients of different kinetic models

3.4.1. Monod kinetic model

The Monod kinetic model serves to elucidate the interdependency between two sets of variables: those related to substrate concentration (sCOD) and the microbial growth rate, typically measured through MLVSS (Jin *et al.* 2022).

Mass in the ICX system consists mainly of granular sludge, with a small fraction of conventional activated sludge. Due to the challenges in measuring the mass of sludge discharged, the outflowing sludge is determined through the TSS parameter.

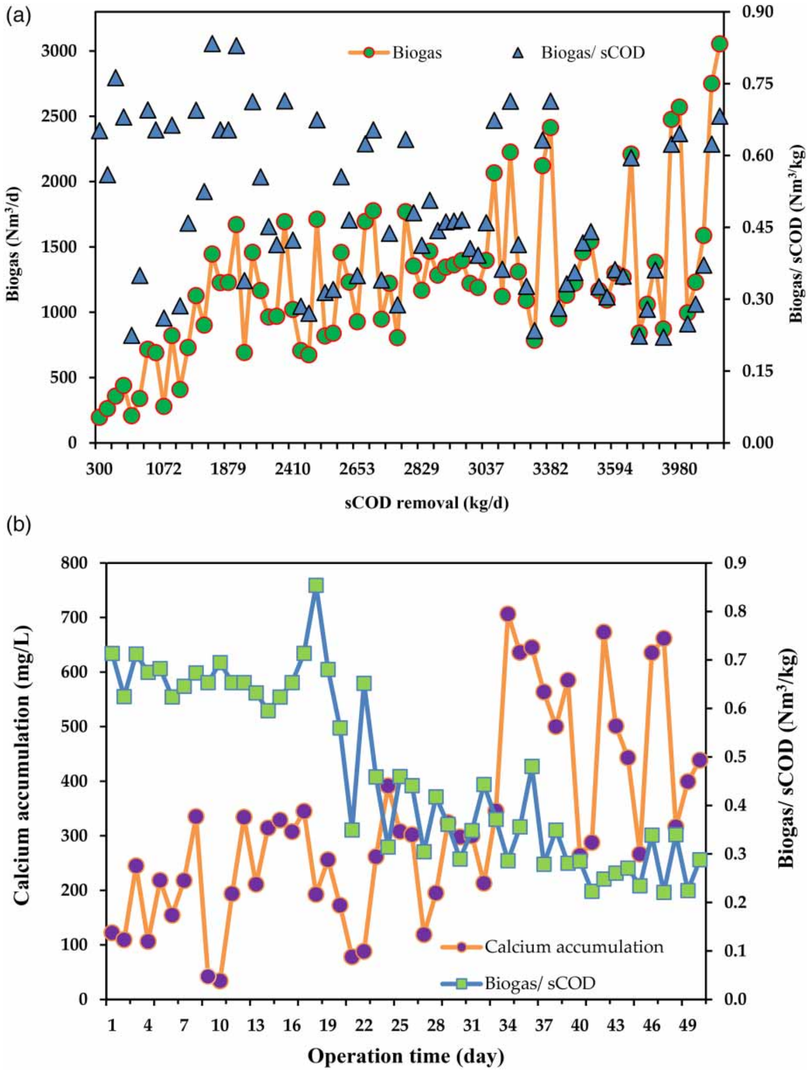


Figure 6 | (a) Relationship between sCOD vs. biogas production and (b) calcium concentration vs. biogas production.

The sludge is then measured by the suspended volume index in an Imhoff cone, followed by multiplying it by the specific gravity of the sludge to determine the sludge decay coefficient. Thus, the units of the two charts will differ. Yield (Y) and decay coefficients (K_d) are determined based on Equation (2) and Figure 7. The results indicate $Y = 0.121$ kgVSS/kgsCOD, the decay coefficient for floculant sludge $K_{dfloc} = 0.0021$ 1/day and for granular sludge $K_{dgra} = 0.021$ 1/day. Therefore, the total decay coefficient is $K_d = 0.0242$ 1/day. The model results align well with previous studies on anaerobic digestion (Table 4). Compared to the UASB system treating aquaculture wastewater, the K_d coefficient of the ICX system is higher while the Y coefficient is lower (Jijai *et al.* 2016). This is supported by the study of Hao & Shen (2021) on the ICX system, where the three-phase separator is improved, retaining the majority of floculant sludge (96–97%) and in this study, 98% of sludge is retained. Other studies on improved UASB, using filters containing polyvinyl alcohol and soft polyurethane, also enhance the biomass retention capability. However, the processing capacity does not improve VLR < 9 kg/m^3 $\times$ day (Hua *et al.* 2017; Patel & Rana 2022). Another study of Campos *et al.* (2014) shows that ICX has lower Y and K_d than UASB when removing phenolics from wastewater with a hydraulic retention time (HRT) > 6 days, having $Y =$ 0.37 kgVSS/kgsCOD and $K_d = 0.0075$ 1/day.

In Figure 8, the saturation coefficient (K_s) is determined to be 56.81 kg/m^3 in the ICX reactor treating paper mill wastewater, indicating its robust capability in managing wastewater with elevated levels of organic pollutants, akin to those commonly found in confectionery or winemaking facilities (Ramos-Vaquerizo *et al.* 2018). Furthermore, q_{max} is calculated

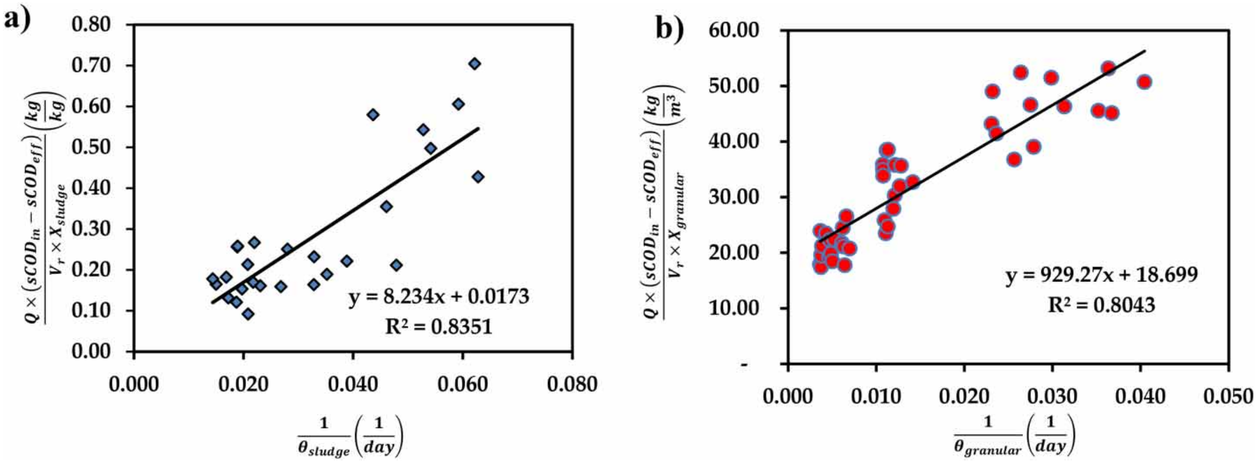


Figure 7 | Determination of yield coefficient (Y) and decay rate constant of the Monod model for (a) floculant sludge (K_{dfloc}) and (b) granular sludge (K_{dgra}).

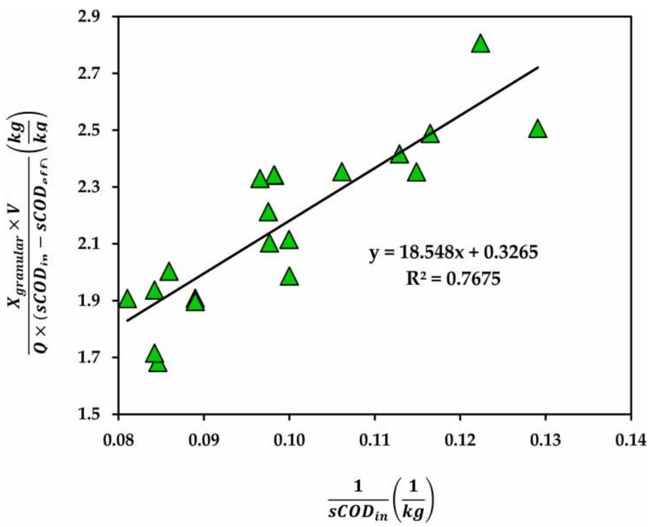


Figure 8 | Determination of maximum substrate consumption rate (q_{max}) and half velocity constant (K_s) of the Monod model.

as 3.063 kgCOD/kgVSS × day, resulting in the highest growth rate according to the Monod model, wherein $\mu_{max} = q_{max} \times Y = 0.372$ 1/day.

The evaluation of the relationship between substrate and biomass typically relies on K_s and μ_{max}. Calculations based on the Monod model suggest that the μ_{max} value is notably lower than K_s ($K_s/\mu_{max} = 152$) in this context. This implies that the growth rate of biomass is stable under high organic loads in the ICX, thereby offering an advantage for biological technologies by mitigating excess biomass treatment costs.

In contrast, for UASB technology treating seafood wastewater, with $K_s = 1.079$ kg/m^3 ($K_s/\mu_{max} = 37.2$), a low COD load and high biomass growth rate are apparent compared to the ICX (Jijai *et al.* 2016). Similarly, the Sonic Anaerobic-moving bed biofilm reactor (MBBR) technology exhibits a K_s/μ_{max} ratio of 1, indicating a mismatch between COD load and biomass growth rates for industrial applications (Som & Yahya 2021). The K_s coefficient of the ICX surpasses that of other anaerobic digestion technologies and is comparable to the hybrid Sonic Anaerobic-MBBR technology.

Monitoring the washout and decay of microbial cell populations in a system is typically represented by the coefficient K_d. Under favorable environmental conditions, negligible decay occurs and K_d can be considered to only represent the washout of cells. In such cases, the specific growth rate (μ) is calculated by the following equation.

$$\mu = \mu_{max} - K_d \quad (7)$$

In this study, the μ value in the ICX reactor is calculated as 0.3478. This value tends to be higher compared to other anaerobic digestion reactors such as UASB (Campos *et al.* (2014)), which reported μ values of 0.1925 for coffee production wastewater and 0.162 for urban wastewater (Singh & Vaishya 2017). Faekah *et al.* (2020) employed anaerobic fixed film reactor up-flow anaerobic filter (UAF) granular sludge systems to eliminate contaminants from synthetic rubber wastewater. The authors presented a higher endogenous decay coefficient K_d compared to conventional technologies due to longer growth and attachment times on the media. However, this technology exhibited a lower $\mu_{max} = 0.011$ 1/day and biomass yield $Y = 0.027$ kgVSS/kgCOD, resulting in extended adaptation periods.

In another study utilizing an EGSB reactor to remove organic contamination from simulated wastewater (Yoochatchaval *et al.* 2008), a Y coefficient of 0.121 kg VSS/kgsCOD was determined, indicating biomass yield efficiency comparable to ideal laboratory growth conditions. These results highlight the effectiveness of the ICX system not only in robust microbial growth but also in maintaining efficient granular sludge with a high specific growth rate (μ) compared to other anaerobic granular sludge technologies.

3.4.2. Kincannon–Stover model

The results obtained through the Kincannon model are presented in Equation (1) and Figure 9. The coefficients, $U_{max} = 151$ kg/m^3·day and $K_B = 175.92$ kg/m^3·day, were calculated with a correlation coefficient $R^2 = 0.9951$. These values are significantly better than those reported in many previous pulp and paper studies (as shown in Table 5). In practice, the ICX exhibits a VLR of 26.8 kg/m^3 × day and a small volume (retention time of approximately 2 h), compared to EGSB (with a retention time of 12 h or more) studied at VLR 1–8 kg/m^3 × day when treating poultry slaughterhouse wastewater, which had $U_{max} = 33.6$ and $K_B = 44.9$ kg/m^3 × day (Njoya *et al.* 2021). For an UASB system treating brewery wastewater with an average COD concentration of 2,005 mg/L, $U_{max} = 13.61$ and $K_B = 18.51$ kg/m^3 × day have been reported (Enitan *et al.* 2014). In addition, the anaerobic membrane bioreactor (AnMBR) technology, which features relatively high COD removal rates, demonstrated $U_{max} = 89.3$ and $K_B = 102.3$ kg/m^3 × day (Wang *et al.* 2009). Therefore, the ICX has proven to be superior in both treatment capacity and removal efficiency, surpassing EGSB and UASB granular sludge technologies and even outperforming the representative AnMBR. The summary of kinetic coefficients of anaerobic wastewater treatment technologies is presented in Table 5.

Through dynamic kinetic modeling and comparison with established models like the Monod and Kincannon–Stover, the ICX demonstrates several advantages. Notably, it effectively retains sludge and boasts a high sludge yield coefficient. This characteristic proves particularly beneficial in treating recycled paper mill wastewater, which often contains high levels of calcium, leading to potential inorganic accumulation within the system. The elevated sludge yield coefficient and specific

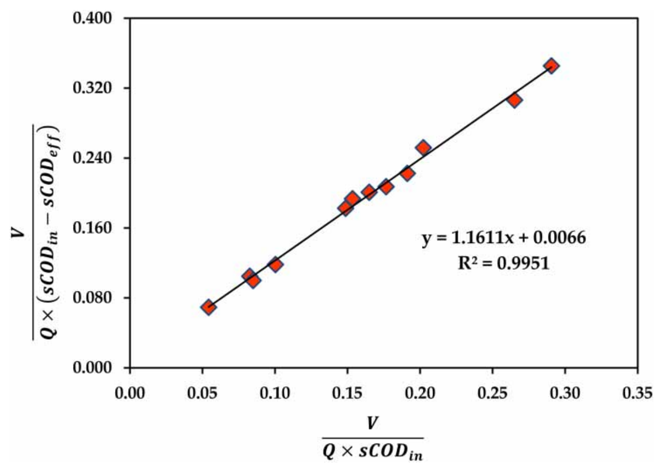


Figure 9 | Stover–Kincannon model plot U_{max} and K_B.

Table 5 | Kinetic coefficients of anaerobic wastewater treatment

Technology	Wastewater	Monod	Stover–Kincannon	Source
ICX	Paper mill	$K_s = 56.809$ kg/m^3 $Y = 0.121$ kgVSS/kgsCOD $K_d = 0.0242$ 1/day $\mu_{max} = 0.372$ 1/day	$U_{max} = 151$ kg/m^3 × day $K_B = 175.92$ kg/m^3 × day	This study (full-scale)
Hybrid-UASB	Pulp and paper	$K_d = 0.1615$ 1/day $Y = 0.082$ kgVSS/kgsCOD	$U_{max} = 34.5$ kg/m^3 × day $K_B = 104.3$ kg/m^3 × day	Hemalatha & Keerthinarayana (2017)
Anaerobic filter	Pulp and paper	–	$U_{max} = 86.21$ kg/m^3 × day $K_B = 104.15$ kg/m^3 × day	Yilmaz *et al.* (2008)
UASB	Seafood	$K_s = 1.079$ kg/m^3 $Y = 0.0463$ kgVSS/kgsCOD $K_d = 0.0056$ 1/day $\mu_{max} = 0.029$ 1/day	$U_{max} = 15.34$ kg/m^3 × day $K_B = 15.47$ kg/m^3 × day	Jijai *et al.* (2016)
	Coffee wastewater	$K_s = 1.504$ kg/m^3 $Y = 0.37$ kgVSS/kgsCOD $K_d = 0.0075$ 1/day $\mu_{max} = 0.2$ 1/day	–	Campos *et al.* (2014)
UAF	Rubber wastewater	$K_s = 84.1$ g/m^3 $Y = 0.027$ kgVSS/kgsCOD $K_d = 0.1705$ 1/day $\mu_{max} = 0.027$ 1/day	$U_{max} = 6.57$ kg/m^3 × day $K_B = 6.31$ kg/m^3 × day	Faekah *et al.* (2020)
Down-flow EGSB	Poultry slaughterhouse	–	$U_{max} = 33.6$ kg/m^3 × day $K_B = 44.9$ kg/m^3 × day	Njoya *et al.* (2021)
Anaerobic-MBBR	Milk permeate	–	$U_{max} = 89.3$ kg/m^3 × day $K_B = 102.3$ kg/m^3 × day	Wang *et al.* (2009)
Sonic anaerobic-MBBR	Palm oil mill	$K_s = 0.361$ kg/m^3 $\mu_{max} = 0.327$ 1/day	–	Som & Yahya (2021)

growth rate of the ICX facilitate the accumulation of organic matter, resulting in higher MLVSS/MLSS ratios compared to alternative technologies when addressing paper wastewater.

In addition, the ICX showcases resilience against shock loading and exhibits superior processing efficiency when faced with high organic loads. Its reaction rate surpasses that of many other anaerobic wastewater technologies, further enhancing its appeal as a viable treatment option.

3.5. Challenges and limitations

While the ICX proves effective in treating high pollutant loads, such as the VLR of 26.8 kg/m^3/day for pulp and paper mill wastewater, and demonstrates the capability to reduce area and volume requirements compared to traditional anaerobic technologies, it is not exempt from encountering challenges and limitations. The compact design of the ICX results in a lower dilution factor, making it highly susceptible to factors that impede microbial activity. Empirical observations highlight that exposure to VFAs exceeding approximately 2 h can impede microbial activity within the ICX. Therefore, it is crucial to maintain influencing factors within permissible thresholds.

Moreover, the ICX's compact design presents challenges concerning the accumulation of calcium and carbonate. The reduced accumulation time per unit volume exacerbates this issue and the compact separator unit is prone to clogging due to calcium buildup, posing a threat to the system's long-term stability.

In addition, the ICX's reliance on IC technology necessitates investment in circulation pumps, leading to increased energy consumption. Improper pump selection may result in the fragmentation of sludge particles, further complicating operational efficiency. Consequently, ensuring the stable operation of the ICX relies heavily on managing wastewater composition and the operational expertise of personnel overseeing the system.

4. CONCLUSION

The ICX reactor exhibits exceptional efficiency in treating high-strength paper mill wastewater, managing organic concentrations (sCOD) up to 11,800 mg/L. Operating stably at a VLR of 26.8 $kg/m^3 \times day$, the system achieves processing efficiency exceeding 81%, while consistently maintaining VFA below 300 mg/L. Employing Monod and Stover–Kincannon kinetic modeling, dynamic parameters, including $K_s = 56.809\ kg/m^3$, $Y = 0.121$ kgVSS/kgsCOD, $K_d = 0.0242$ 1/day, $\mu_{max} = 0.372$ 1/day, $U_{max} = 151\ kg/m^3 \times day$ and $K_B = 175.92\ kg/m^3 \times day$, underscore the ICX reactor's superior efficiency compared to alternative technologies. The ICX reactor's heightened sensitivity to VFA levels necessitates maintaining influent concentrations below 1,400 mg/L for effective sludge treatment. In addition, calcium significantly influences treatment efficiency, requiring post-treatment alkalinity maintenance below 19 meq/L to stabilize MLVSS/MLSS concentration. While the observed biogas production rate ranges from 0.6 to 0.7 Nm^3 biogas/kg sCOD, the impact of calcium in paper mill wastewater diminishes this ratio, reducing overall treatment efficiency and biogas production. This research significantly contributes to the body of knowledge in wastewater treatment, offering valuable insights for addressing challenges related to complex industrial wastewater.

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DATA AVAILABILITY STATEMENT

All relevant data are included in the paper or its Supplementary Information.

CONFLICT OF INTEREST

The authors declare there is no conflict.

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