

EFFECT OF AMMONIA AND COD TO SULFATE RATIO ON TREATMENT PERFORMANCE OF CONCENTRATED LATEX PROCESSING WASTEWATER

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ABSTRACT

The objective of this study is to investigate the effects of ammonia nitrogen ($\text{NH}_3\text{-N}$) and COD to sulfate ratio on the treatment performance of concentrated latex processing wastewater (CLPW). In a batch experiment, the CLPW influent COD, % granular sludge and pH were controlled at about 1,200 mg/l, 10% and 7, respectively. The influent $\text{NH}_3\text{-N}$ concentrations varied at 100, 500, 1000 and 3000 mg/l, whereas COD/sulfate ratio varied at 0.3, 0.6, 1 and 1.3. Treatment performance was determined by methane generation rate and COD removal efficiency at different $\text{NH}_3\text{-N}$ and COD to sulfate ratio. The $\text{NH}_3\text{-N}$ concentration at 100 and 500 mg/l (which were presented in the real CLPW), did not result in inhibition on methane producing bacteria (MPB), since methane generation was detected. Inhibition on MPB was observed at 1,000 mg/l $\text{NH}_3\text{-N}$. Strong inhibition occurred at 3,000 mg/l $\text{NH}_3\text{-N}$, since % COD removal was relatively reduced to 7.98 % and no methane generation. The COD/sulfate ratio at lower than 0.6 resulted in inhibition of MPB, but in the real CLPW the ratio is normally higher than 0.6. Ammonia concentration higher than 1000 mg/l and COD to Sulfate at lower than 0.6 resulted in inhibition of methane producing bacteria in the treatment of CLPW.

KEY WORDS : Concentrate latex, Ammonia, COD to Sulfate ratio

INTRODUCTION

The Rubber industry in Thailand is of economic and social importance, especially in the southern region, due to its production volume, revenues from export and source of employment. Concentrated latex is one important product with an annual production capacity of about 0.96 million tons, and has a prosperous incremental trend (Rubber Institute, 2010). However, its production process has resulted in several environmental problems including air pollution, water pollution and solid waste production. Among these problems, water pollution has been paid much attention in Thailand (Tekasakul and Tekasakul, 2006).

Concentrated latex is derived from field latex and has a dry rubber content of approximately 60-70%.

After fresh latex is delivered to the concentrated latex mill, dry rubber content (DRC) and ammonia are determined. Ammonia, TMTD (Tetra methyl thiurum disulphide) /ZnO, and DAP (Diammonium phosphate) are then added to preserve latex quality, and the latex is then delivered to centrifugation process in order to separate rubber content and water. Products are classified depending on the level of ammonia requirement as well as process requests. The water layer (skim latex) still contains up to 8% (by wet weight) dry rubber. This water phase is discharged into a skimming tank for skim rubber production. Sulfuric acid is added to skim latex for coagulation (Mohammadi *et al.*, 2010; Jawjit *et al.*, 2010; Jawjit *et al.*, 2015). Concentrated latex serves as the raw material in the production of numerous products,

including gloves, condoms, baby pacifiers, elastic and adhesives.

Concentrated latex processing wastewater (CLPW) mainly contains BOD, COD, $\text{NH}_3\text{-N}$, Organic-N and phosphate. The COD from natural latex mills in Thailand was $9,710 \pm 2,600$ mg/l, whereas concentration of nitrogen in terms of TKN, NH_3 , NH_2 and NO_3 were about 550-1,300 mg/l, 200 mg/l, 40 mg/l and 10 mg/l, respectively (Jawjit *et al.*, 2010; Jawjit *et al.*, 2015; Tanikawa *et al.*, 2016). Several studies reported that ammonia use for latex preservation was human toxicity problem (Jawjit *et al.*, 2015; Tanikawa *et al.*, 2016; Thongnuekhang and Puetpaiboon, 2004). A schematic diagram of concentrated latex production indicating sources of wastewater is illustrated in Figure 1. Washing and centrifugation are the main sources of wastewater containing large amount of ammonia and phosphorus (from DAP- Diammonium Phosphate), whereas wastewater from skim latex production is highly acidic due to the use of sulfuric acid for coagulation. Table 1 presents the characteristics of concentrated latex processing wastewater (Jawjit *et al.*, 2015; Nugyen *et al.*, 2012).

In Thailand current wastewater treatment systems applied for concentrated latex mills are a combination of anaerobic pond and aerobic pond, such as aerated lagoon and oxidation pond (Phoolphundh *et al.*, 2013). However, it is regularly found that the treated effluent from these processes do not yet meet Thailand's industrial effluent standards (Chaiprapat and Sdoodee, 2007; Hatamoto *et al.*, 2012; Rubber Institute, 2010). Therefore, Anaerobic treatment, has been given more attention to its apply for concentrated latex mill.

However, some pollutants (e.g. $\text{NH}_3\text{-N}$, sulfate) presented in CLPW adversely affect treatment performance of anaerobic system. From Table 1, it can be observed that $\text{NH}_3\text{-N}$ concentration is relatively high (300-600 mg/l). Koster (1989) reported that $\text{NH}_3\text{-N}$ at 1,500 – 3,000 mg/l resulted in toxicity to methane producing bacteria (MPB). In the case of sulfate generated from the application of sulfuric acid in skim latex production, it also inhibits MPB (Oliver *et al.*, 1996), since sulfate is reduced to toxic sulfide by sulfur reducing bacteria (SRB)

The objective of this study is therefore to investigate effect of $\text{NH}_3\text{-N}$ and sulfate on treatment performance of the two-stage UASB applied to concentrated latex mill. To achieve this purpose, batch and continuous experiments were performed

Table 1. Characteristic of concentrated latex processing wastewater in Thailand.

Characteristic	Range	Unit
pH	3.9-4.4	mg/l
Suspended solid	265-856	mg/l
Volatile solid	154-292	mg/l
Soluble solid	7,780-8,650	mg/l
COD	3,890-4,860	mg/l
BOD	2,400-2,860	mg/l
Sulfate	1,247-4,140	mg/l
TKN	836-1397	mg/l
Ammonia-N	316-578	mg/l
Total-P	86.46-126	mg/l
Calcium	10.69-16.30	mg/l
Cobalt	nd ^a	mg/l
Nickel	nd	mg/l
Zinc	nd-0.274	mg/l
Copper	nd-0.022	mg/l
Iron	0.843-4.184	mg/l
Magnesium	0.168-0.173	mg/l
Chloride	7.4-17.1	mg/l

^a nd = not detected

with various $\text{NH}_3\text{-N}$ concentrations and COD/sulfate ratios. The two-stage UASB reactor was used as a model reactor system. Treatment performance (% COD removal, % $\text{NH}_3\text{-N}$ removal, % Sulfate removal) and biogas production were measured.

MATERIALS AND METHODS

Batch experiment

Serum bottle with a working volume of 1,000 ml was used as a reactor in a batch experiment (Figure 2). The bottle was sealed with aluminum screw caps and butyl rubber septum. The CLPW and 10% granular sludge plus additional nutrient solution and trace elements (Table 2) were fed to the bottle (Brummeler and Koster, 1990). The influent COD concentration and pH were controlled at about 1,200 mg/l, and 7, respectively. To investigate the effects of ammonia and sulfate, the influent $\text{NH}_3\text{-N}$ concentrations varied at 100, 500, 1000 and 3000 mg/l, whereas COD/sulfate ratio varied at 0.3, 0.6, 1 and 1.3. The temperature of the reactor was controlled at 35 °C. Methane production was measured by substitution of 5% NaOH.

Continuous experiment

A two-stage UASB consists of one acid tank and one UASB reactor. The acid tank, which is made of PVC, is 26.5 cm. in diameter, 60 cm. in height and a

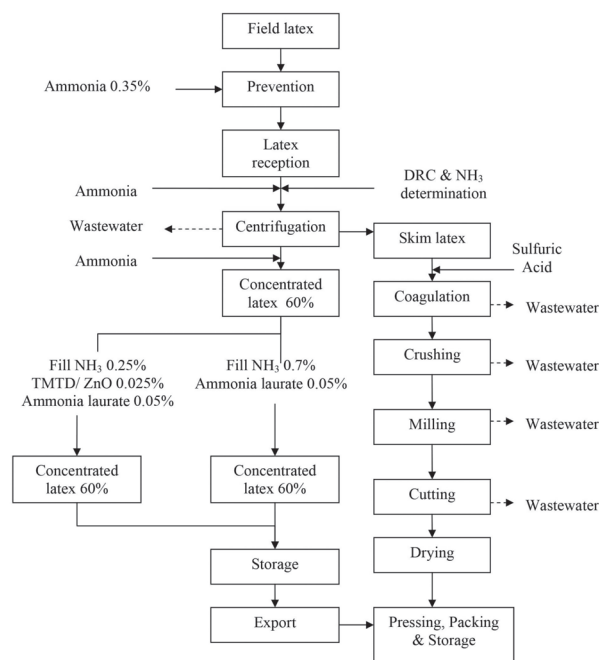


Fig. 1. Schematic diagram of concentrated latex production.

working volume of 24.8 liter. The UASB reactor consists of outer and inner columns. Both columns are made of Acrylic. The inner column is 6 cm. in diameter, 120 cm. in height and a working volume of 3.523 liter (excluding volume of a gas-solid separator installed at the upper zone of the column). Water sampling ports are installed at three different heights of the column. The outer column, which is 12 cm. in diameter, works as a water jacket by installation of a heater to control temperature of the UASB reactor. A schematic of the experimental set up of the two-stage UASB reactor is illustrated in Figure 3.

The reactor operated at 35 °C was continuously fed with real wastewater (collected from concentrated latex mill in Nakhon si thammarat, South Thailand). A nutrient and trace elements solution was added to the CLPW by preparing stock solution and use of 6 ml nutrient solution and 0.1 ml trace-solution for 1 l. The composition of these stock solutions, according to Hulshoff Pol (1989), was presented in Table 2.

Analytical procedures

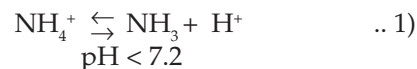
Analytical methods for each parameter are presented in Table 3.

RESULTS

Effect of NH₃-N on treatment performance inhibition

The influent NH₃-N concentrations were varied at 100, 500, 1000 and 3000 mg/l to investigate the inhibition on anaerobic activities. From Table 4, it was clear that COD removal efficiency decreased, as NH₃-N concentration was increased. It can also be observed that NH₃-N concentration at 1,000 -3,000 mg/l resulted in toxicity on MPB, since methane production was not produced in such concentrations. However, NH₃-N concentration in CLPW was in the range of 300-500 mg/l (Table 1). Therefore, it can be concluded that NH₃-N concentration in CLPW was not significantly toxic to MPB in anaerobic treatment.

The pH also plays an important role in the effect of NH₃-N. If pH is higher than 7.2, ammonium ion (NH₄⁺) would convert to toxic ammonia (NH₃) (equation 1). However, In case of CPLW pH is lower than 7.2 (NH₃-N concentration at 100 and 500 mg/l), the less toxic (non-toxic) NH₄⁺ form would dominate.



Accumulation of toxic NH₃-N damaged granular sludge (Yamaguchi *et al.*, 1997). This can be observed from the reduction of MLSS from its initial concentration of 3,896 mg/l to 3,518 mg/l, as NH₃-N concentration was increased to 1,000 mg/l. Damaged granular sludge resulted in increase of organic N, which consequently increased TKN concentration. Fig. 3 compares normal granular sludge and damaged granular sludge at high NH₃-N concentrations (3,000 mg/l).

Effect of COD/sulfate ratio on treatment performance inhibition

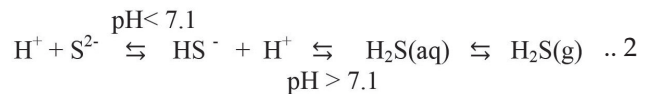
From Table 1, sulfate concentration in CLPW was in the range of 1,250 – 4,000 mg/l. Sulfate presented in CLPW resulted from the application of sulfuric acid (H₂SO₄) in skim latex production (Figure 1). In anaerobic treatment, sulfate is reduced to toxic sulfide by sulfate reducing bacteria. Different COD/sulfate ratios were therefore set at 0.3, 0.6, 1.0 and 1.3 by adding sodium sulfate (Na₂SO₄). Results presented in Table 5 indicated that every COD:sulfate ratio did not inhibit treatment performance, since concentrations of remaining hydrogen sulfide were low. This can be explained because the

Table 2. Composition of nutrient solution and trace elements supplied to the wastewater

Substrate	mg/l
Nutrient solution	
- NH ₄ Cl	1044
- K ₂ HPO ₄	169.8
- (NH ₄) ₂ SO ₄	169.8
- MgCl ₂ 6 H ₂ O	150
- KCl	270
- Yeast	19.8
Trace Element	
- FeCl ₂ 4H ₂ O	0.2
- H ₃ BO ₃	0.005
- ZnCl ₂	0.005
- CuCl ₂ 2H ₂ O	0.0038
- MnCl ₂ 4H ₂ O	0.05
- (NH ₄) ₆ Mo ₇ O ₂₄ 4H ₂ O	0.005
- AlCl ₃ 6H ₂ O	0.009
- CoCl ₂ 6H ₂ O	0.2
- NiCl ₂ 6H ₂ O	0.0092
- Na ₂ SeO ₃ 5H ₂ O	0.0164
- EDTA C ₁₀ H ₁₆ N ₂ O ₈	0.1
- Resazurine	0.02
- 36% HCl	

generated sulfide reacts with Zn²⁺ (from application of ZnO in concentrated latex production), and becomes Zinc sulfide (Anotai *et al.*, 2007). According to McCarty (1964), sulfide concentration at 50-100 mg/l did not inhibit activity of anaerobic bacteria. Sulfide concentration which resulted in toxicity to the bacteria was reported at 200 mg/l upwards. It should be noted that based on morphology granular sludge has a membrane that is covered. This makes granular sludge more tolerant to toxic substances than normal anaerobic bacteria.

It can also be observed that the degradation of organic substances reflected by % COD removal was in the same range (87-90%) in every COD/sulfate ratio. Never the less, it was found that a low COD/sulfate ratio resulted in more sulfate reduction than that of a high COD/sulfate ratio. An absence of methane was found at COD/sulfate ratio of 0.3 and 0.6, because in this condition methane producing bacteria (MPB) was dominated by sulfate reducing bacteria (SBR) (Yamaguchi *et al.*, 1997; Rattanapan *et al.*, 2009). At such COD/sulfate ratios, pH was lower than 7.1, Biogas mainly produced H₂S (equation (2)), which was trapped by 5% NaOH.



In the case of pH, it is obvious that the pH was raised as COD/sulfate ratio was increased. This is because the reaction of SRB generates HS⁻ and HCO₃⁻, which results in higher alkalinity (equation (3)), the SBR in anaerobic reactor produces H₂S, which is a strong malodorous gas. Such malodorous H₂S is generated in most conditions, because the SBR is tolerant to changes of acidity, alkalinity and temperature (Saritpongteeraka and Chaiprapat, 2008). Formation of H₂S largely depends on pH as mentioned above in equation (2).



From Table 5, it was found that COD/sulfate ratios in batch experiments were decreasing. This reflects that toxic sulfide generated from sulfate reduction reaction adversely affect the growth of anaerobic bacteria. Like wise, the lowest % COD removal was also reported in this ratio. It was also found that sulfate removal efficiency was decreasing, as COD/sulfate ratio was raised. Higher sulfate removal efficiency reflects better sulfate reduction reaction.

Application of the two-stage UASB with real wastewater at latex mill

The two-stage UASB was applied at latex mill with

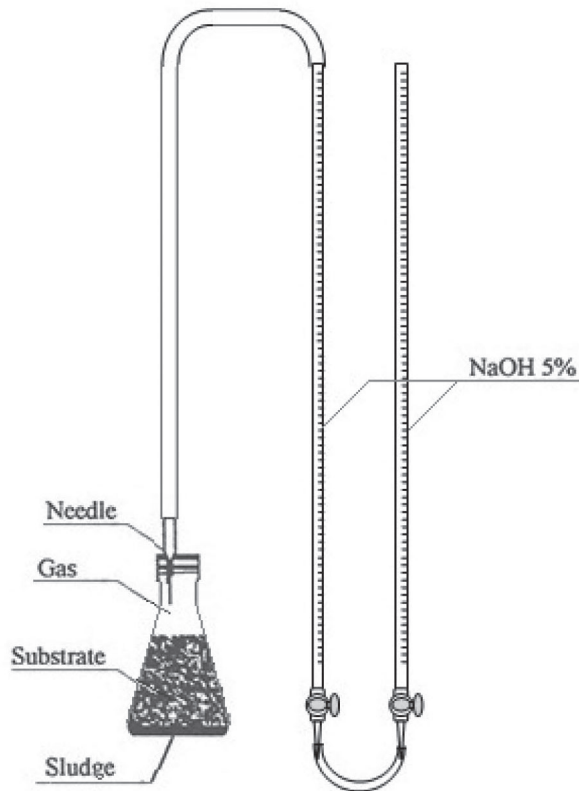


Fig. 2. Serum bottle used as a reactor in batch experiment

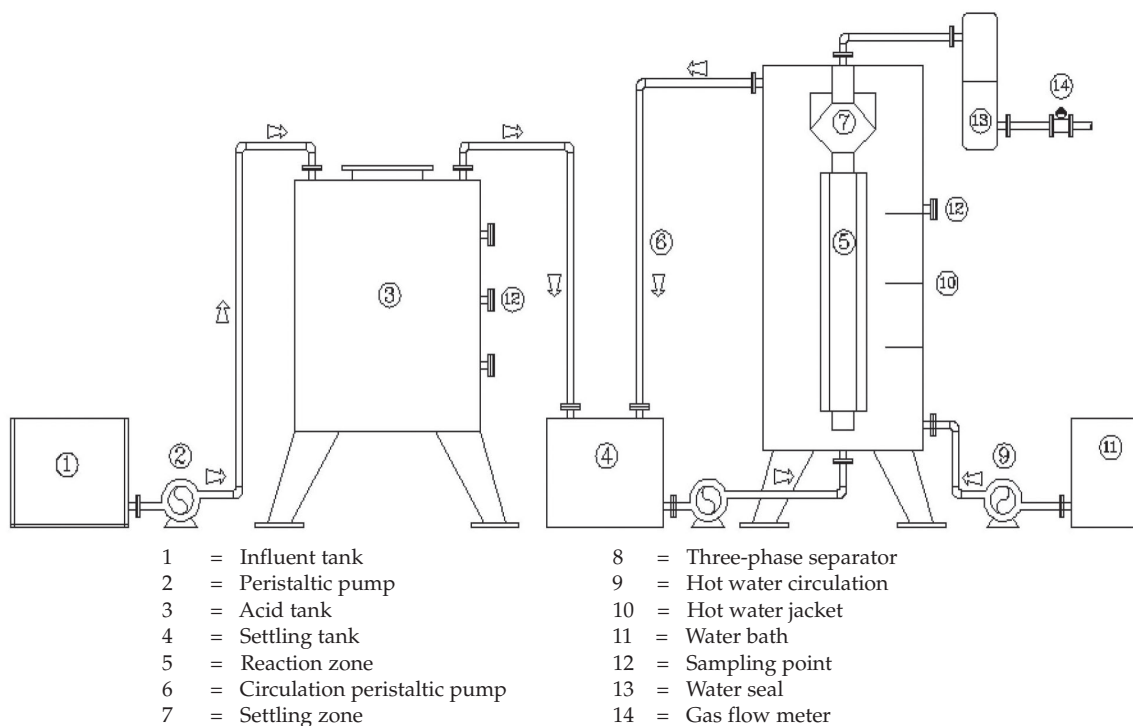


Fig. 3. Comparison of (3.1) normal granular sludge, and (3.2) damaged granular sludge at 3,000 mg/l $\text{NH}_3\text{-N}$ (captured by Olympus stereo microscope model DP 12 SZ-CTV; magnification $\times 46$)

Table 3. Analytical methods of wastewater parameters.

Parameter	Method	Code
pH	pH meter	–
COD	Closed reflux, Titrimetric method	-5220 C
Ammonia nitrogen	Titrimetric method	^a 4500-NH ₃ E ^a
Sulfate	Turbidimetric Method	4500-SO ₄ ²⁻ E ^a

^aStandard Method for the Examination of water and Wastewater (APHA, 2017)

Table 4. Treatment performance of concentrated latex mill at $\text{NH}_3\text{-N}$ 100, 500, 1000 and 3000 mg/l

$\text{NH}_3\text{-N}$ (mg/l)	COD removal (%)	$\text{NH}_3\text{-N}$ removal (%)	TKN (mg/l)	MLSS (mg/l)	pH	Vol. gas (l CH ₄ /g COD removed)
100	70.17	55.9	61.25	3,601.18	6.46	0.273
500	57.82	48.434	319.6	3,422.73	6.86	0.158
1000	37.32	40.386	667.39	3,518.36	7.15	nd
3000	7.98	71.35	1,667.26	3,392.55	7.63	nd

nd = not detected

Table 5. Treatment performance of concentrated latex mill at different COD/sulfate ratio

Ratio COD/ sulfate	COD removal (%)	Sulfate removal (%)	Sulfide (mg/l)	MLSS (mg/l)	pH	Vol. gas (l CH ₄ /g COD removed)
0.3	87.67	55.87	20.16	3,601.18	6.89	nd
0.6	89.32	55.62	18.88	3,422.73	7.01	nd
1.0	90.96	45.81	17.26	3,518.36	7.17	0.225
1.3	89.32	36.03	19.20	3,392.55	7.24	0.49

nd = not detected

Table 6. Influent and effluent after treating by the two-stage UASB.

Time	Influent (mg/l)(Mean \pm SD.)				Effluent (mg/l) (Mean \pm SD.)				Efficiency (%)			
	COD	SS	NH ₃ -N	SO ₄ ²⁻	COD	SS	NH ₃ -N	SO ₄ ²⁻	COD	SS	NH ₃ -N	SO ₄ ²⁻
Start up												
0-200 day	3,988.81 \pm 161.30	365.32 \pm 74.74	374.85 \pm 76.27	3812.35 \pm 578.12	1499.60 \pm 656.21	108.68 \pm 54.72	162.48 \pm 47.98	1879.23 \pm 523.26	62.35 \pm 16.6	69.04 \pm 16.94	64.12 \pm 16.02	50.56 \pm 14.23
Steady State (after 200 day)	3,566.17 \pm 933.61	378.24 \pm 101.31	364.95 \pm 78.54	3735.38 \pm 614.11	357.64 \pm 243.16	23.6 \pm 11.71	39.74 \pm 24.45	1,269.6 \pm 20.67	87.26 \pm 17.57	90.15 \pm 17.51	87.88 \pm 8.68	65.21 \pm 7.75

optimum HRT (24 hrs in acid forming tank, and 48 hrs in UASB tank) and pH influent control at 7 ± 0.5 which were determined in above-mentioned analyses. Results of COD, SS, NH₃-N and sulfate are steady state shown in Table 6

At the initial stage (start up) efficiencies of COD, SS, NH₃-N and SO₄²⁻ removal were not high, because it was the stage that bacteria assimilated to the wastewater. COD and SS removal efficiencies were then higher until the system reached the steady state. The methane production are 0.112 CH₄/g COD removed.

DISCUSSION

This study aimed to investigate the effect of NH₃-N and COD/sulfate ratio on treatment performance of concentrated latex mill. In the batch experiment, it was found that inhibition on anaerobic bacteria was initially observed at 1,000 mg/l NH₃-N. Strong inhibition occurred at 3,000 mg/l NH₃-N, since % COD removal was relatively reduced from 37.32 mg/l (at 1,000 mg/l NH₃-N) to 7.98 mg/l. NH₃-N concentration at 100 and 500 mg/l, which were in the range of NH₃ presented in the real CLPW, did not result in inhibition on MPB, since methane generation was still detected.

In the case of COD/sulfate ratio, it was found that every COD/sulfate ratio did not inhibit treatment performance, since generated sulfide concentration (17-20 mg/l) was not high enough to cause toxicity. This is because sulfide combined with Zn²⁺ (from ZnO) and Ca²⁺ (from CaO). The COD removal efficiencies more than 80% were reported at every COD/sulfate ratio. Sulfate removal efficiency at low COD/sulfate ratio was higher than that of high COD/sulfate ratio. Nevertheless, methane generation was not detected at COD/sulfate ratio of 0.3 and 0.6, because in this condition methane producing bacteria (MPB) was dominated by sulfate reducing bacteria (SBR). Produced biogas was therefore mainly H₂S. Nevertheless, methane was generated at COD/sulfate ratio of 1.0 and 1.3. Therefore, COD/sulfate should be kept higher than 0.6, if methane production is required.

The two-stage UASB was then applied with real wastewater at latex mill. It was found that methane production was about 0.112 \pm 0.03 l CH₄/g COD removed (16.257-22.76 m³CH₄/d), and average COD and SS removal efficiency were 81.08 % and 94.22 %, respectively. In case of SS removal efficiency, the result reveals that the two-stage UASB is capable to overcome the limitation of SS removal of the single-stage UASB treating concentrated latex effluent (Nguyen, 1999). This is because in the two-stage UASB the HRT is longer than that of in the single-stage UASB. Rubber particles contained in concentrated latex wastewater is a complex polymer which is slowly degrading substance, and cannot be completely hydrolyzed in the single-stage UASB. Different conditions controlled for different group of bacteria (acid and methane former) in separate reactors in the two-stage UASB also enhance the SS treatment efficiency.

These results indicated that application of the two-stage UASB to concentrated latex processing wastewater is feasible. Never the

Table 7. The Condition in two-stage UASB.

Condition	COD/SO ₄ ²⁻ inf	COD/SO ₄ ²⁻ eff	Alkalinity (mg/l as CaCO ₃)	pH	CH ₄ /g COD removed
Start up	1.05	0.800	1,808.53	7.06	0
Steady State	0.95	0.281	2,067.67	7.46	0.112

less, combination with other treatment systems (e.g. oxidation pond, aerated lagoon) is necessary in order to meet Thailand's industrial effluent standard (in case of COD).

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