

Research Article

**Cyanide removal by ozone oxidation in tapioca starch industrial wastewater for recycling purposes: effects of pH and starch concentration**

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**Abstract**

This research investigated the effects of pH and tapioca starch concentration on cyanide removal by ozone oxidation in recycling water of the tapioca starch production process. Wastewater reuse and recycle is an appealing alternative for the reduction of industrial operating costs. An acrylic column reactor equipped with a gas sparger located at the bottom of the column was used as a reactor. Experiments were performed at the total cyanide concentration of 30 ppm and ozone ( $O_3$ ) generation rates of 10.0, 15.0, 20.0 g  $O_3$  h<sup>-1</sup>. The liquid phase volumetric mass transfer coefficient ( $k_La$ ) and equilibrium ozone concentration ( $O_3^*$ ) were determined. The values of  $k_La$  were in the ranges of 3.17±0.03 min<sup>-1</sup>, 2.97±0.06 min<sup>-1</sup>, 2.48±0.13 min<sup>-1</sup>, and 2.05±0.03 min<sup>-1</sup> at pH 4, 5, 6 and 7, respectively.  $O_3^*$  increased when  $O_3$  generation rates increased at acidic pH values. At pH 5, 6, and 7, cyanide was rapidly oxidized by  $O_3$  and became plateau.

**Keywords:** environment, food waste, cassava, *Manihot esculenta*, bubble column reactor, Thailand.

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**Introduction**

Cyanide occurs during the production of native tapioca starch due to the naturally occurring cyanogenic glycosides, i.e. linamarin (97%) and lotaustralin (3%) from cassava roots. Once the plant cells are broken during the production process, the linamarase enzyme catalyzes the breakdown of these glycosides to hydrogen cyanide (HCN) in a pathway called cyanogenesis [1].

The total amount of cyanide entering the native starch production process varies from 28 to 43 kg HCN equivalent/day, depending on the root quality (based on the production capacity of 100 tons starch per day) [2]. Normally, the total cyanide compounds including bound cyanide, cyanohydrin, and free cyanide concentrations are measured as the product's quality control step [3]. The World Health Organization (WHO) specification states that the cyanide content of starch product must be less than 10 ppm, while the acceptable limit of the Japanese standard is as low as 0.1 ppm [4, 5]. Currently, the recycle and reuse of wastewater is a widespread measure to reduce the environmental and production cost in starch industry. However, a problem of cyanide accumulation arises from the reuse of wastewater within the production units. Cyanide content of the reused water ranges from 4 to 34 kg HCN equivalent per day.

Several methods, including oxidation, are applied to destroy and reduce cyanide in several industries [6]. Chemical oxidation is generally used to treat cyanide in most industrial processes [7]. Ozone ( $O_3$ ) is known as an extremely powerful chemical agent with redox potential of 2.07 V (in aqueous solution for pH= 0, at 25°C) [6]. The cyanide oxidation reaction by  $O_3$  is very rapid with complete decomposition and retains no harmful residue.

Several research efforts have investigated the cyanide removal efficiency in starch wastewater [3, 6, 8]. Cyanide removal efficiency depended upon various parameters, such as treatment dose and pH of starch slurry. Piyachomkwan *et al.* [3], found that when aeration ( $3 \text{ L kg}^{-1}$  dry starch) was applied to starch slurry at pH 5.5 and 7.0, only 14 and 18% of total cyanide in starch was removed, respectively. The cyanide removal efficiency increased to 33% once  $O_3$  ( $3.67 \text{ L kg}^{-1}$  dry starch) was applied. Somboonchai [8], studied kinetics of cyanide oxidation by  $O_3$  in tapioca starch production process. The level of initial cyanide concentrations of 10, 20, 30, 40 ppm and  $O_3$  generation rates of 7.4, 15.0, 22.6, 30.0  $\text{g O}_3 \text{ h}^{-1}$  were used. The results showed that the concentration of cyanide was sharply decreased during the first 30 seconds of reaction. The kinetics of cyanide oxidation was first order with respect to cyanide and zero order with respect to  $O_3$ . The rate constant obtained from first order and zero order equation of cyanide oxidation with  $O_3$  was 2.76 and  $2.74 \times 10^{-9} \text{ min}^{-1}$ , respectively.

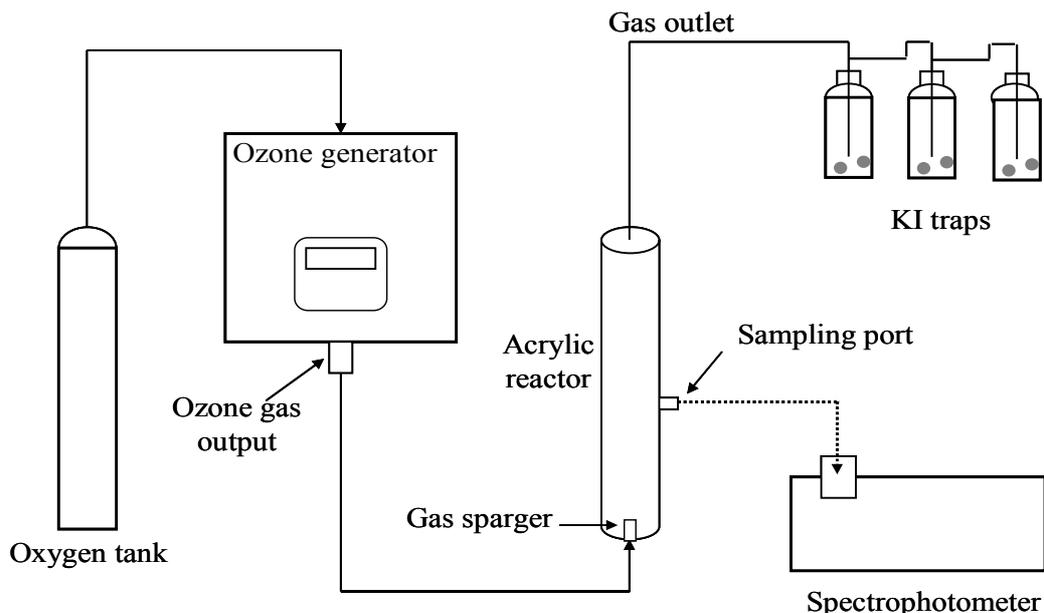
In the actual cassava starch process, the wastewater stream is reused to reduce the amount of freshwater required, as well as to reduce starch loss, resulting in a fluctuation in pH values and starch content in the process. This research, therefore, aimed to study the effect of pH, starch concentration and  $O_3$  concentration in aqueous phase on the cyanide removal by  $O_3$  oxidation in a designed bubble column reactor.

## Materials and Methods

### *Experimental setup*

Experiments were conducted in a 2-L acrylic bubble column (i.e., 6.4 cm internal diameter and 1 m height) equipped with an  $O_3$  gas sparger at the bottom of the column (Fig. 1). The  $O_3$  generator (Model # Prominent OZVa 5) with a maximum  $O_3$  generation rate of  $30 \text{ g O}_3 \text{ h}^{-1}$  was used to generate the required  $O_3$  dose. Residual  $O_3$  in gas phase was removed by passing outlet gas through KI solution.

Experiments were conducted under a steady-state countercurrent flow. Cyanide solution with an initial concentration of  $0.030 \text{ g L}^{-1}$  was fed at a flow rate of  $12 \text{ L h}^{-1}$  at 0.8 m height from the bottom of reactor. Three levels of  $O_3$  generation rates of 10.0, 15.0, and  $20.0 \text{ g O}_3 \text{ h}^{-1}$  were fed upward with flow rate of  $150 \text{ L h}^{-1}$ . Dye solution and air were fed countercurrent to cyanide solution and  $O_3$ . Cyanide content was evaluated as total cyanide concentration using enzymatic methods [9].



**Figure 1. Schematic Diagram of Experimental Setup.**

#### ***Mass transfer and equilibrium concentration of ozone***

Three sets of experiments were conducted to determine the mass transfer of  $O_3$  from gas to liquid phase at the  $O_3$  generation rate of 10.0, 15.0, and 20.0  $g O_3 h^{-1}$ . The gaseous mixture of oxygen and  $O_3$  was bubbled in 2 L of water for 20 minutes. Samples were collected at a sampling port located at 0.5 m height from the bottom and analyzed for dissolved  $O_3$  concentration by photometer (Model # Dulcotest DT1) [10]. The experiments were carried out with 2 replications.

#### ***Cyanide oxidation with ozone***

Experiments were conducted in a bubble column operated under a steady-state countercurrent flow with  $O_3$  generator rates of 10.0, 15.0 and 20.0  $g O_3 h^{-1}$  with a constant flow rate of 150  $L h^{-1}$  and an initial cyanide concentration of 30 ppm with a flow rate 12  $L h^{-1}$ . Samples were collected from the sampling port located at 0.8 m from the bottom and analyzed for residual  $O_3$  concentration and cyanide content. The experiments were carried out with 2 replications.

### **Results and Discussion**

#### ***Mass transfer and equilibrium concentration of ozone***

Mass transfer of  $O_3$  using volumetric mass transfer coefficient ( $k_L a$ ) and equilibrium ozone concentration ( $O_3^*$ ) were estimated. Three levels of  $O_3$  generation rates of 10, 15 and 20  $g O_3 h^{-1}$  were bubbled through 2 L of DI water at different levels of pH. Dissolved  $O_3$  concentrations were analyzed by a photometer.

The  $k_L a$  and  $O_3^*$  were calculated at  $O_3$  consumption equal to zero using the following equation [11]:

$$\frac{dO_3}{dt} = k_L a (O_3^* - O_3) - r_L \quad (1)$$

Where  $k_L a$  = volumetric mass transfer coefficient,  $min^{-1}$

$O_3^*$  = equilibrium ozone concentration, mg L<sup>-1</sup>

$O_3$  = ozone Concentration at any time, mg L<sup>-1</sup>

$r_L$  = ozone consumption in the aqueous phase, mg L<sup>-1</sup> min<sup>-1</sup>

Table 1 presents the  $k_L a$  and  $O_3^*$  obtained from the model. At a constant pH, the  $k_L a$  value is constant. The  $O_3^*$  and  $k_L a$  values decreased with increasing pH. These results can be explained by the reactions in aqueous phase as follows [11]:



The OH<sup>-</sup> has an influence on self-decomposition rate of O<sub>3</sub>. At a high pH value, O<sub>3</sub> is not stable and decomposes rapidly. Thus, the  $O_3^*$  at high pH values were lower than those at low pH values. On the other hand,  $O_3^*$  values did not depend on the O<sub>3</sub> generation rate. They depend on process parameters, e.g. flow rate, energy input, reactor volume and physical parameters, e.g. bubble size of O<sub>3</sub>, density, surface tension, kinetic viscosity [11]. However, these parameters were controlled during the experiment; therefore, the  $k_L a$  values were constant at each pH value.

**Table 1. Liquid Phase Volumetric Mass Transfer Coefficient ( $k_L a$ ) and Equilibrium Concentration of Ozone ( $O_3^*$ ).**

pH	Ozone generation rate (g O <sub>3</sub> h <sup>-1</sup> )	$k_L a$ (min <sup>-1</sup> )	$O_3^*$ (mg L <sup>-1</sup> )
4.00	10.0	3.14±0.07 <sup>a</sup>	3.97±0.05 <sup>a</sup>
4.00	15.0	3.19±0.02 <sup>a</sup>	4.00±0.01 <sup>a</sup>
4.00	20.0	3.18±0.01 <sup>a</sup>	4.00±0.01 <sup>a</sup>
5.00	10.0	2.99±0.15 <sup>a</sup>	2.76±0.03 <sup>b</sup>
5.00	15.0	2.91±0.09 <sup>a</sup>	3.65±0.07 <sup>c</sup>
5.00	20.0	3.02±0.24 <sup>a</sup>	4.00±0.01 <sup>a</sup>
6.00	10.0	2.37±0.25 <sup>b</sup>	1.61±0.01 <sup>d</sup>
6.00	15.0	2.62±0.46 <sup>b</sup>	2.01±0.01 <sup>c</sup>
6.00	20.0	2.46±0.21 <sup>b</sup>	3.93±0.06 <sup>a</sup>
7.00	10.0	2.08±0.28 <sup>c</sup>	1.05±0.08 <sup>f</sup>
7.00	15.0	2.04±0.07 <sup>c</sup>	1.88±0.05 <sup>c</sup>
7.00	20.0	2.02±0.22 <sup>c</sup>	2.24±0.09 <sup>e</sup>

Values followed by the same letter are not significantly different (P>0.05).

Figure 2 further presents experimental results of  $O_3^*$  at three  $O_3$  generation rates. The  $O_3^*$  values increased as  $O_3$  generation rate increased. It can be described by Henry's Law [7], as shown in equation 4.

$$H_C = \frac{C_G}{O_3^*} \quad (4)$$

where  $H_C$  = Dimensionless Henry's Law constant

$C_G$  = Gas concentration in reactor,  $\text{mg L}^{-1}$

$O_3^*$  = Equilibrium ozone concentration in liquid phase,  $\text{mg L}^{-1}$

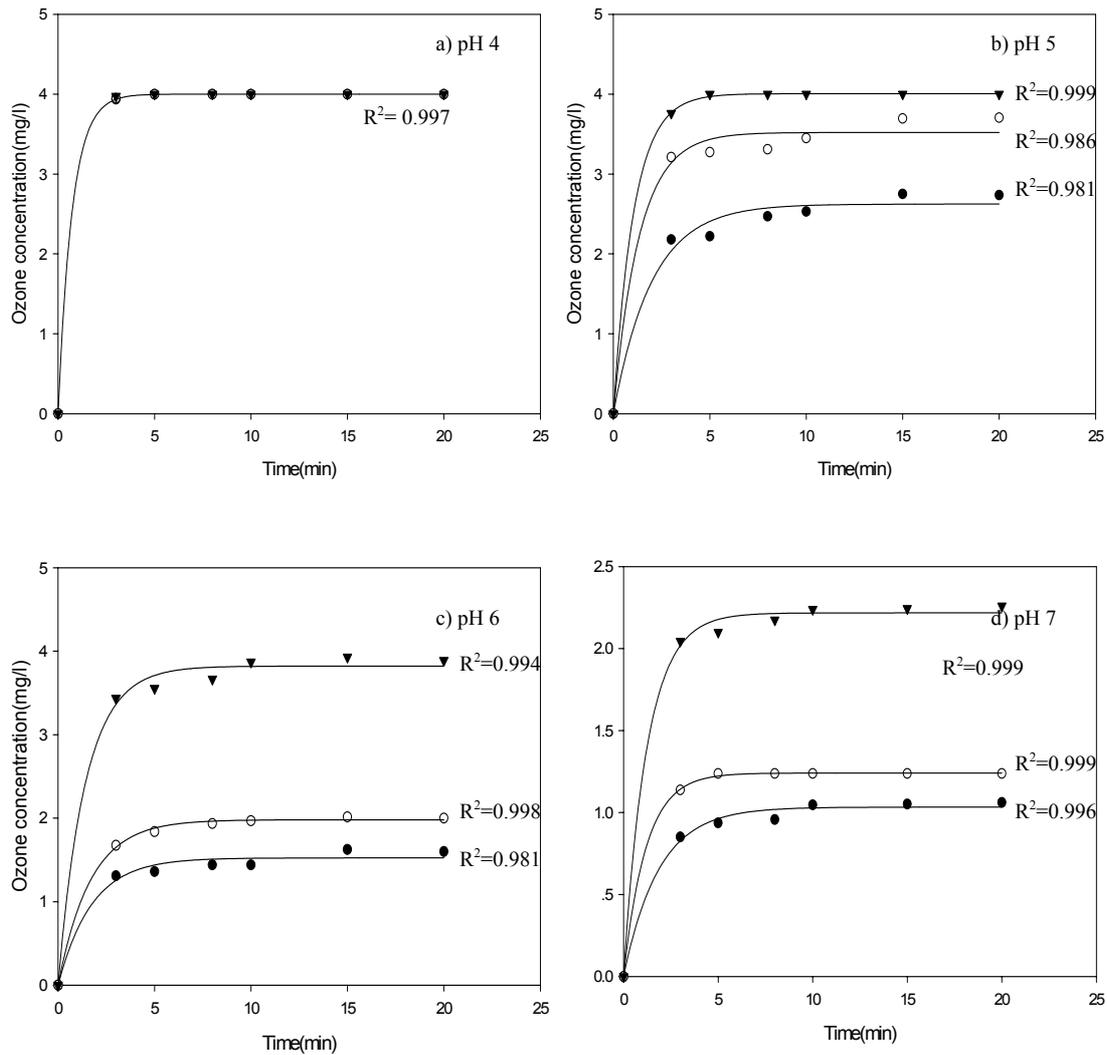
The dimensionless Henry's Law constant is a function of temperature, pH and ionic strength [11]. In this research,  $H_C$  was constant as temperature and ionic strength were similar at each pH value. Consequently, high  $O_3$  generation rate resulted in high gas concentration in the reactor; therefore,  $O_3^*$  was increased. However, at pH 4,  $O_3^*$  was  $4 \text{ mg L}^{-1}$  at every  $O_3$  generation rate, because  $O_3$  dissolved very well at acidic pH. In addition, it is not stable and decomposes rapidly at high pH values [12].

At  $O_3$  generation rate of 10, 15 and  $20 \text{ gO}_3 \text{ h}^{-1}$ ,  $O_3$  concentrations in gas phase were 1.39, 2.08 and  $2.78 \text{ mmol L}^{-1}$ , respectively.  $O_3$  dissolved in water can be estimated by  $O_3$  solubility coefficient in  $\text{mg L}^{-1}$  in water to  $\text{mg L}^{-1}$  in gas. Equation 5 was used to calculate of the solubility ratio(s) at different temperatures. In this research, the experiments were performed at  $30^\circ\text{C}$ .

$$\log_{10}s = -0.25 - 0.013T (^\circ\text{C}) = 3.302 - 0.013T (\text{K}) \quad (5)$$

The solubility ratio at  $30^\circ\text{C}$  was  $0.23 \text{ mg L}^{-1}$  water per  $\text{mg L}^{-1}$  gas. Therefore, the theoretical  $O_3$  concentration in liquid phase at  $O_3$  generation rates of 10, 15 and  $20 \text{ gO}_3 \text{ h}^{-1}$  should be 0.32, 0.48 and  $0.64 \text{ m mole L}^{-1}$ , respectively. However, the measured value of  $O_3$  concentration in liquid phase (shown in Table 2) was much less than the theoretical values.

$O_3^*$  (calculated from eq. 1) were less than the  $O_3$  concentrations in liquid phase in any pH values (Table 2). The real  $O_3$  concentration is influenced by a number of factors in the water. Temperature is one factor influencing the solubility. Further,  $O_3$  can be partially decomposed to  $\text{OH}^\cdot$  radicals. When the pH value increases, the formation of  $\text{OH}^\cdot$  radicals increased. These hydroxide ions act as an initiator for the decay of  $O_3$ .



**Figure 2. Concentration of O<sub>3</sub> Dissolved in Water at Three O<sub>3</sub> Generation Rates.**

● 10 g O<sub>3</sub> h<sup>-1</sup>, ○ 15 g O<sub>3</sub> h<sup>-1</sup> and ▼ 20 g O<sub>3</sub> h<sup>-1</sup>  
 a) pH 4, b) pH 5, c) pH 6, d) pH 7

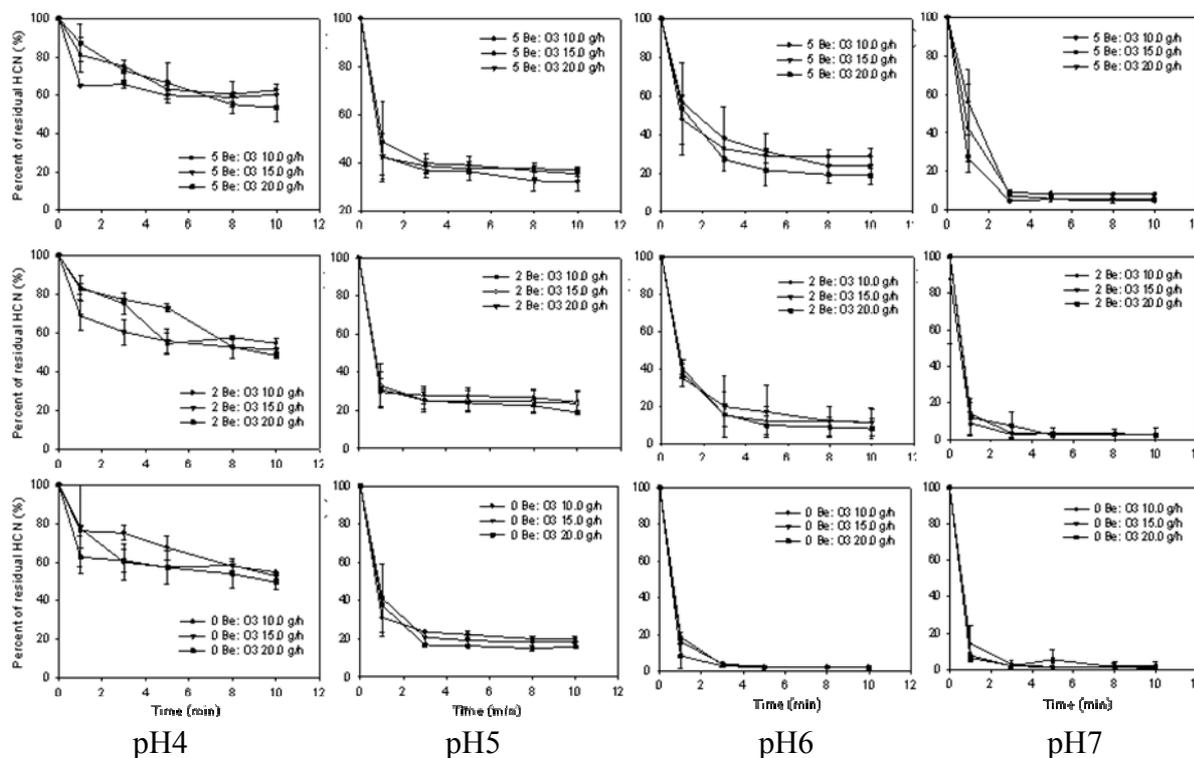
**Effect of pH and starch concentration on cyanide oxidation by O<sub>3</sub>**

Effects of cyanide oxidation by O<sub>3</sub> at 3 levels of O<sub>3</sub> generation rates and constant initial cyanide concentration were investigated. Three levels of O<sub>3</sub> generation rates of 10.0, 15.0 and 20.0 g O<sub>3</sub> h<sup>-1</sup> and initial cyanide concentration of 30 ppm were used. The experiments were performed in a steady state flow bubble column reactor operating under countercurrent flow. Starch concentrations were varied to be 0, 2, and 5 Be' (i.e., equivalent to 0, 36, and 92 g/l, respectively).

**Table 2.  $O_3^*$  Estimated from Equation 1 at Three Levels of  $O_3$  Generation Rates and Four Levels of pH Values.**

pH	Ozone generation rate (g $O_3$ h <sup>-1</sup> )	$O_3^*$ (m mole L <sup>-1</sup> )
4.00	10.0	0.083 <sup>a</sup>
4.00	15.0	0.083 <sup>a</sup>
4.00	20.0	0.083 <sup>a</sup>
5.00	10.0	0.058 <sup>b</sup>
5.00	15.0	0.076 <sup>c</sup>
5.00	20.0	0.083 <sup>a</sup>
6.00	10.0	0.034 <sup>d</sup>
6.00	15.0	0.042 <sup>c</sup>
6.00	20.0	0.082 <sup>a</sup>
7.00	10.0	0.022 <sup>f</sup>
7.00	15.0	0.039 <sup>e</sup>
7.00	20.0	0.047 <sup>c</sup>

Values followed by the same letter are not significantly different (P>0.05)



**Figure 3. Cyanide Oxidation at pH 4, 5, 6 and 7 Different  $O_3$  Generation Rates of 10.0, 15.0 and 20.0 g  $O_3$  h<sup>-1</sup> with Initial Cyanide Concentrations of 30 mg L<sup>-1</sup>. (Starch concentration 0 Be = 0 g/l, 2 Be =36 g/l and 5 Be =92 g/l).**

Cyanide concentration sharply decreased during the first minute of reaction. However, it can be observed that the final cyanide concentration treatments were not zero and percent of residual cyanide were higher at lower pH. It was the result of the oxidation capability of O<sub>3</sub> with cyanide. According to Henry (1996), only free cyanide can be oxidized by O<sub>3</sub> as shown in Eqs. (6) and (7).



Theoretically, 2 moles of cyanide are hydrolyzed by 5 moles of O<sub>3</sub>. With pK<sub>a</sub> of 9.2, at low pH values, [CN<sup>-</sup>] is very small, cyanide destruction practically stops.

The percentage of residual cyanide increased when the amount of starch increased. Partial O<sub>3</sub> oxidized starch in solution instead of oxidizing CN<sup>-</sup> [13]. However, oxidation efficiency increased when hydraulic retention time increased as the flow rate of cyanide solution decreased.

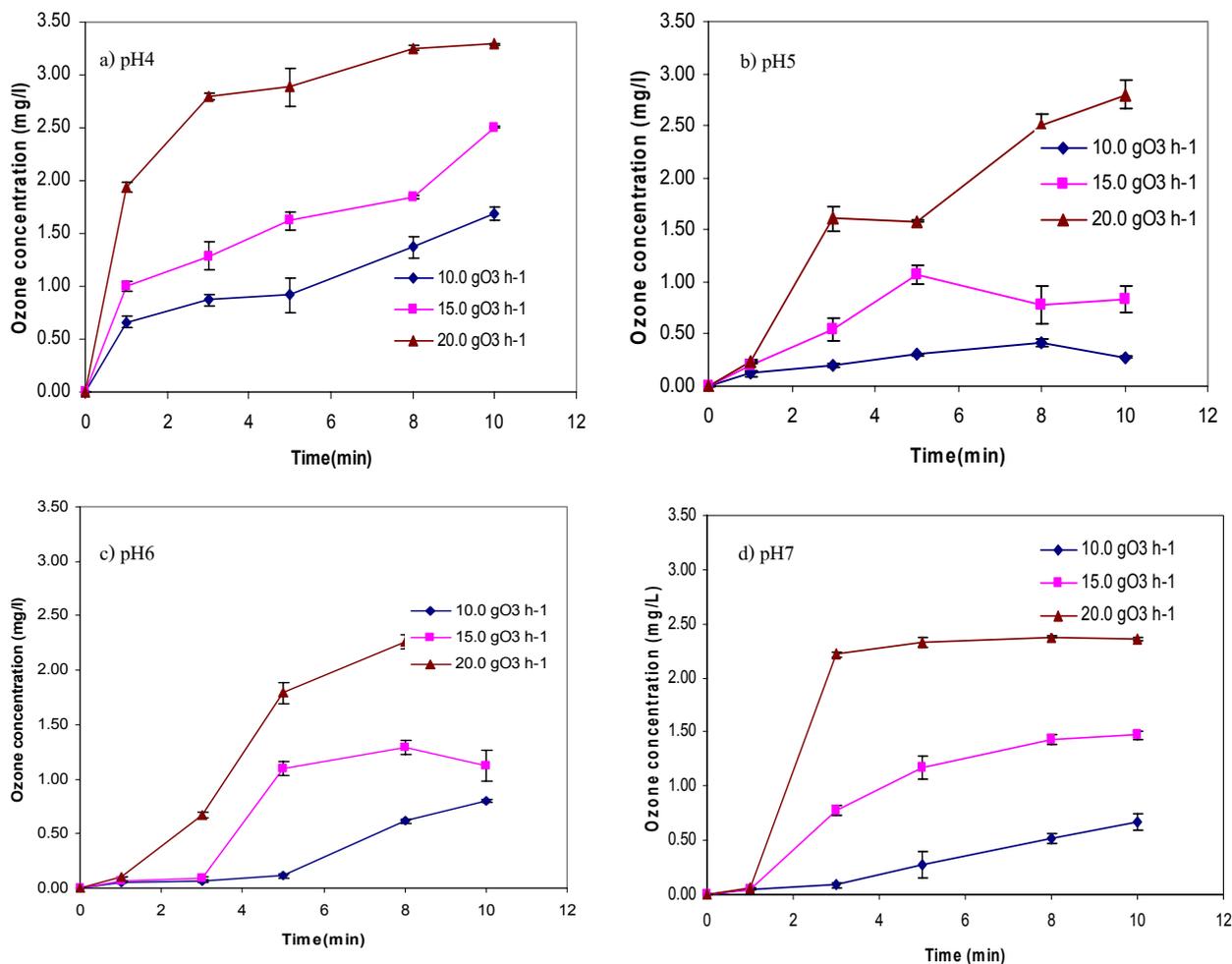
**Table 3. Oxidation Efficiency at Different O<sub>3</sub> Generation Rates of 10.0, 15.0 and 20.0 g O<sub>3</sub> h<sup>-1</sup> with Initial Cyanide Concentrations of 30 mg L<sup>-1</sup> at pH 4, 5, 6 and 7.**

pH	Starch concentration (Be)	Oxidation efficiency (%)		
		10.0 g/h	15.0 g/h	20.0g/h
4	0	45.2±0.3 <sup>a</sup>	47.2±1.6 <sup>a</sup>	50.5±3.8 <sup>a</sup>
4	2	45.4±2.8 <sup>a</sup>	48.6±0.9 <sup>a</sup>	51.7±1.7 <sup>a</sup>
4	5	37.3±2.8 <sup>a</sup>	39.71±0.2 <sup>a</sup>	46.6±7.2 <sup>a</sup>
5	0	80.3±1.8 <sup>b</sup>	81.69±3.1 <sup>b</sup>	84.2±0.2 <sup>b</sup>
5	2	75.7±5.8 <sup>c</sup>	76.36±6.1 <sup>c</sup>	81.0±0.8 <sup>c</sup>
5	5	62.5±1.1 <sup>d</sup>	64.50±1.3 <sup>d</sup>	67.6±4.0 <sup>d</sup>
6	0	97.6±0.7 <sup>e</sup>	97.55±0.2 <sup>e</sup>	97.9±0.0 <sup>e</sup>
6	2	88.6±7.6 <sup>f</sup>	88.8±7.0 <sup>f</sup>	92.1±5.3 <sup>f</sup>
6	5	71.2±4.0 <sup>g</sup>	76.4±0.9 <sup>g</sup>	78.5±8.2 <sup>g</sup>
7	0	98.1±2.6 <sup>e</sup>	98.9±1.6 <sup>e</sup>	99.3±2.1 <sup>e</sup>
7	2	95.8±1.2 <sup>e</sup>	97.6±0.4 <sup>e</sup>	98.1±1.4 <sup>e</sup>
7	5	91.7±0.4 <sup>h</sup>	94.3±0.1 <sup>h</sup>	95.1±0.7 <sup>h</sup>

Values followed by the same letter are not significantly different (P>0.05)

Table 3 shows that the oxidation efficiency increased at high pH values but decreased at high starch concentration. This result was similar rate constant of cyanide decomposition. This can be explained in terms of the hydrolysis of cyanide ions. Thus at low pH values when the concentration

of  $\text{CN}^-$  is small, cyanide destruction practically stops. Moreover, the study of mass transfer of  $\text{O}_3$  showed that the equilibrium of  $\text{O}_3$  concentration was higher at low levels of pH. Normally, high content of  $\text{O}_3$  should provide high cyanide reduction. But in this study, at high pH  $\text{O}_3$  can reduce cyanide better than at low level of pH. This can be postulated that the  $\text{O}_3$  in any level of pH was excess to react with cyanide as shown in Figure 4.



**Figure 4. Residual  $\text{O}_3$  Concentration at Different  $\text{O}_3$  Generation Rate of 10.0, 15.0 and 20.0  $\text{gO}_3 \text{ h}^{-1}$  with Initial Cyanide Concentrations of 30  $\text{mg L}^{-1}$  a) pH 4, b) pH 5, c) pH 6, d) pH 7.**

## Conclusions

Ozone was used for cyanide oxidation in the recycling water during tapioca starch processing.  $\text{O}_3$  is known to be a powerful oxidant, high oxidation and disinfection potential. The oxidation of cyanide by  $\text{O}_3$  in aqueous solutions was studied using an acrylic column reactor. The experiment was performed at cyanide concentrations of 30  $\text{mg L}^{-1}$  and  $\text{O}_3$  generation rates of 10.0, 15.0 and 20.0  $\text{gO}_3 \text{ h}^{-1}$ . The liquid phase volumetric mass transfer coefficient ( $k_L a$ ) and equilibrium  $\text{O}_3$  concentration were determined for calculating the rate of  $\text{O}_3$  transfer from gas phase to liquid phase comfortable to reduce cyanide in solution. It was found that the values of  $k_L a$  were increased when pH value decreased. However, the values of  $k_L a$  quite constant at difference  $\text{O}_3$  generation rate. Equilibrium  $\text{O}_3$  concentration was increased when  $\text{O}_3$  generation rate increased and pH decreased. In the cyanide oxidation part, cyanide content was sharply decreased at the first minute of reaction and after that the cyanide concentration slightly decreased. However, the percentage of residual cyanide increased

when the amount of starch and acidic pH values increased. In this research, O<sub>3</sub> generation rates were comfortable to reduce cyanide in solution.

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