THE INFLUENCE OF TEMPERATURE AND ORGANIC MATTER ON THE DECOMPOSITION KINETICS OF PERACETIC ACID IN AQUEOUS SOLUTIONS

L. KUNIGK, S.M. SILVA and C. H. JURKIEWICZ

Instituto Mauá de Tecnologia, Praça Mauá, 01, 09580-900, São Caetano do Sul, SP, Brasil.
kunigk@maua.br

***Abstract*– Peracetic acid (PAA) is a powerful sanitizer used by food industries all over the world. The main disadvantage of PAA is its decomposition rate. The main purpose of this paper is to study the decomposition of PAA between 20 and 40 °C with 0.0, 2.5 or 5.0 g·L^{-1} of beer, milk or tomato juice in its solutions. The observed decomposition of PAA is a first-order reaction for solutions free of organic matter with observed rate constants from 1.24·10^{-3} to 5.59·10^{-3}·h^{-1} and an activation energy (E_a) of 62.11 kJ·mol^{-1}. However a pseudo first-order reaction was observed in the presence of organic matter and the rate constants between 1.13·10^{-3} and 2.18·10^{-2} h^{-1}. The E_a calculated by the observed decomposition rate constants are 42.3 and 61.2 kJ·mol^{-1} when there was 2.5 and 5.0 g/L beer in solution, respectively. PAA solutions contaminated with 2.5 and 5.0 g·L^{-1} milk have showed E_a values between 9.7 and 55.3 kJ·mol^{-1}, respectively. For tomato juice solutions, the Arrhenius equation slope was not constant. The different E_a obtained indicate that different mechanisms are taking place at the various tested operating conditions. This work also proposes a mathematical equation to estimate the PAA concentration for solutions free of organic matter.**

***Keywords*– peracetic acid, decomposition kinetics, beer, milk, tomato juice.**

I. INTRODUCTION

PAA is a powerful sanitizer with a reduction potential of 1.06V that has been introduced in the Brazilian food industry in the 90´s. Commercial formulations of this sanitizer contain mixtures of acetic acid, hydrogen peroxide (PH), water and PAA as shown by the following chemical equation (Zhao *et al.*, 2008b, Musante *et al.*, 2000, Kunigk *et al.*, 2001; and Yuan and Heiningen, 1997a):

$$CH_3COOH + H_2O_2 \overset{H^+}{\rightleftharpoons} CH_3COOOH + H_2O$$

PAA has many advantages when compared to sodium hypochlorite, the most commonly used sanitizer in Brazil (Kunigk *et al.*, 2001). One of the many advantages of PAA is that its decomposition produces nothing else than acetic acid and oxygen which are non-toxic compounds and therefore affect neither the final product nor the industry biological waste treatment process. It was reported that PAA in solution may be consumed by hydrolysis, spontaneous decomposition or decomposition catalyzed by transition metal ions (Yuan and Heiningen, 1997b, Kitis, 2003, Veschetti, *et al.*, 2003 and Zhao, *et al.*, 2008a).

Spontaneous decomposition of PAA occurred at a pH range of 5.9-10.2 (Koubek *et al.*, 1963 *apud* Zhao *et al.*, 2008a). Zhao *et al.* (2008a) reported that in an acid condition and at a temperature range of 55 to 95°C the spontaneous decomposition of PAA is a second-order reaction with respect to PAA concentration. However at a temperature below 55 °C the spontaneous decomposition of PAA is so insignificant that it could be negligible and hydrolysis became the predominant consumption factor for PAA. Zhao *et al.* (2007) reported that the hydrolysis of PAA in an acidic environment is first order with respect to PAA concentration, water and H^+ concentration.

Rucker and Cates (1988) reported that the decomposition of peracetic acid is affected by the solution´s pH and the observed decomposition rate of peracetic acid at 30 °C increases with pH ranging from 0.921·10^{-3} h^{-1} at 5.34 to 15.5·10^{-3} h^{-1} at 8.90.

Temperature also affects the decomposition of PAA. Greenspan and MacKellar (1948) observed that at room temperature, diluted solutions of PAA (10 g/L), lost half of their sanitation power within 6 days. Kunigk *et al.* (2001) have shown that increasing the temperature, a more rapid decomposition occurred. At 45 °C the concentration was halved in 72 hours, but at 25 °C the loss in 240 hours was only 33%. They concluded that temperature has an important role in the shelf life of PAA solutions.

No information was found on the decomposition kinetics of PAA solutions contaminated with organic matter usually present in the food industry. The main purpose of this paper is to present the results obtained in experiments carried out to study how organic matter affects the reduction of PAA concentration during its storage when their aqueous solutions are contaminated with 0.00, 2.5 or 5.0 g.L^{-1} beer, milk or tomato juice at 20, 30, 35 and 40 °C, rather than to make a rigorous study of its decomposition kinetic. This study is aimed at showing that organic matter reduces the PAA shelf-live and this must be taken in consideration for equipment sanitation operations. It is also important to note that the concentrations of organic matter that have been chosen for the simulation of contamination of PAA solutions are several decimals above the values which may be expected in the food industry but can illustrate what could happen.

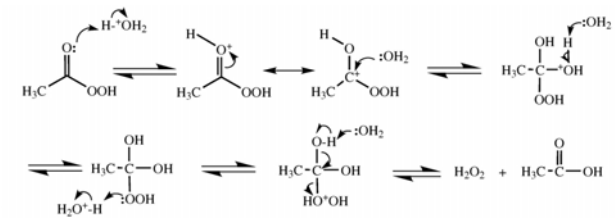

Figure 1. Reaction mechanisms of PAA hydrolysis (Zhao *et al.*, 2008a).

II. METHODS

A commercial formulation of PAA sanitizer containing around 300.0 g·L^{-1} acetic acid, 200.0 g·L^{-1} PAA and 100.0 g·L^{-1} hydrogen peroxide was used in the experiments. The above product was diluted with distilled water in order do obtain concentrations of PAA equal to about 300 mg·L^{-1}. The solutions were then maintained in 500 mL Erlenmeyers at constant temperatures (20.0, 30.0, 35.0 and 40.0 °C) in a water bath in static condition. The Erlenmeyers had been previously rinsed with a diluted HCl solution and then rinsed with de-ionized water before use. The concentrations of PAA were measured at regular intervals using the iodometric methodology as proposed by Greenspan and MacKellar (1948) and used by Yuan and Heiningen (1997a,b), Musante *et al.* (2000), Kunigk *et al.* (2001), Veschetti *et al.* (2003), Zhao *et al.* (2008a) and Zhao *et al.* (2008b) in their works. All chemicals used in the experiments (potassium permanganate, potassium iodide, sodium thiosulfate, sulfuric acid and so on) were analytically pure. The effect of organic matter in PAA decomposition rate was studied adding 0.00, 2.5 or 5.0 g·L^{-1} of beer, milk or tomato juice in PAA solution and then the methodology described above was used to determine the PH and PAA concentration. No type of chelator was used in any of the PAA solution. Control samples with the organic matter but without PAA were prepared in the same conditions as described above. The sodium thiosulfate volumes obtained from these titrations were subtracted from those volumes obtained from the titration of the PAA solutions contaminated with the same organic matter. Each datum was the average of three to five measurements.

The kinetic constants were fitted according to the experimental data using Origin 6.0 software.

III. RESULTS AND DISCUSSION

Kunigk *et al.* (2001), Zhao *et al.* (2007) and Zhao *et al.* (2008a) reported that PAA decomposition occurs by hydrolysis and decomposition kinetics can be expressed by a first-order reaction in an acid environment and below 55 °C with respect to PAA concentration and the mechanism is shown in Fig. 1.

The PAA concentration reduction could also occur by spontaneous reaction or by transition metal ions catalyzed decomposition as reported by Yuan and Heiningen (1997a), Brasileiro *et al.* (2001) and Unis (2010). The mechanism of spontaneous decomposition of PAA is shown in Fig. 2. The peracetic acid decomposition catalyzed by transition metal ions (Zhao *et al.*, 2008a) is shown in Eq. (1).

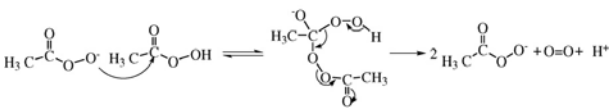

Figure 2. Mechanism of spontaneous decomposition of PAA. (Zhao *et al.*, 2008a).

$$CH_3COOH \xrightarrow{M^{n+}} CH_3COOH + O_2 + other\ products \quad (1)$$

Therefore the decomposition mechanism of peracetic acid can be very complex. In this work, the hydrolysis mechanism couldn´t be used to explain the decomposition of PAA because hydrogen peroxide concentration didn´t increase as can be seen in Fig. 3 to 6. However, PAA decomposition can be expressed by a first-order reaction for PAA solutions without organic matter and by a pseudo first-order reaction for the other condition studied as can be seen in Fig. 7 to 13 and mathematical Eq. (2) was used to represent its decomposition kinetic.

$$Ln[PAA] = Ln[PAA]_o - k \cdot t \quad (2)$$

where: $[PAA]$ is the PAA concentration; $[PAA]_o$ is the initial PAA concentration (mg·L^{-1}); t is the storage time (h) and k is the reaction rate constant (h^{-1}) for Eq. (2). Tables 1 to 4 show the equations parameters for each studied condition.

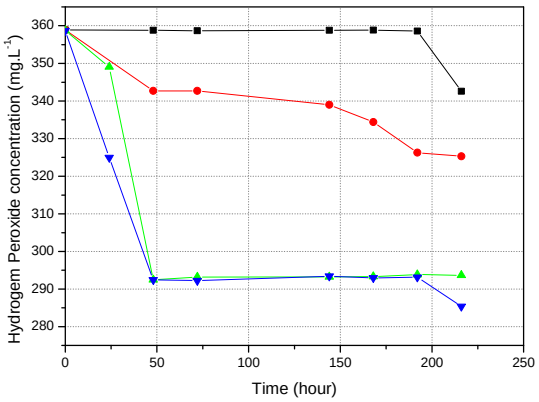

Figure 3. Hydrogen peroxide concentration in solutions free of organic matter with initial pH: 3.48 (-■- 20°C; -●- 30°C; ▲ 35°C; -▼- 40°C).

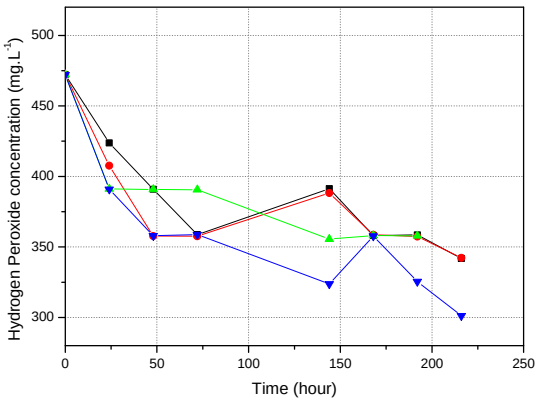

Figure 4. Hydrogen peroxide concentration in solutions with 5.0 g/L of beer with initial pH: 3.51 (-■- 20°C; -●- 30°C; ▲ 35°C; -▼- 40°C).

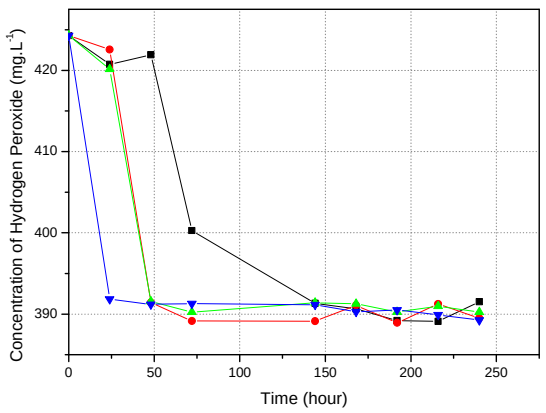

Figure 5. Hydrogen peroxide concentration in solutions with 5.0 g/L of milk with initial pH: 4.48 (-■- 20°C; -●- 30°C; ▲ 35°C; -▼- 40°C).

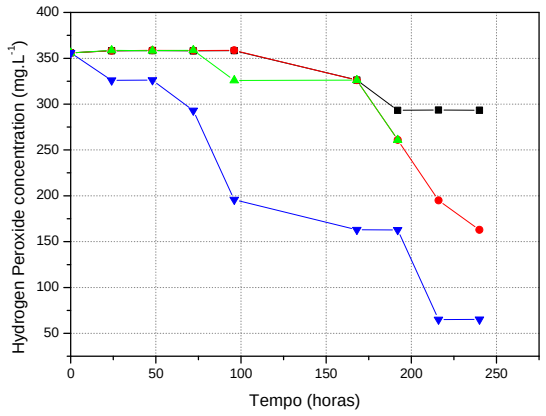

Figure 6. Hydrogen peroxide concentration in solutions with 5.0 g/L of tomato juice with initial pH: 4.42 (-■- 20°C; -●- 30°C; ▲ 35°C; -▼- 40°C).

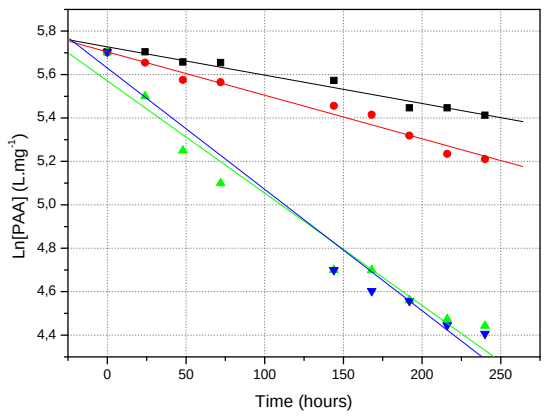

Figure 7. PAA concentration in solutions free of organic matter with initial pH: 3.48 (-■- 20°C; -●- 30°C; ▲ 35°C; -▼- 40°C).

From Table 1 it can be seen that increasing the temperature from 20 to 40 °C the observed rate constant of PAA decomposition has increased 4.5 times going from $1.24 \cdot 10^{-3}$ to $5.59 \cdot 10^{-3}$ h^{-1}, respectively. Therefore, temperature has an important role on observed rate constants. Kunigk *et al.* (2001) found a variation in rate constant of 5.6 by increasing temperature from 25 to 45 °C. The difference between the variations of the observed rate constants in these two studies can be explained by the fact that it was used a different commercial PAA formulation was used (unpublished data). Therefore the formulation composition of sanitizers can affect their decomposition. The smaller decomposition rate constant obtained in this work could mean that this product is less affected by temperature than the product used by Kunigk *et al.* (2001). Rucker and Cates (1988) found an observed first order constant for decomposition of peracetic acid solutions at pH 7.1 ranging from $4{,}572 \cdot 10^{-2}$ h^{-1} at 20 °C to 1,632 h^{-1} at 50 °C. This difference is due mainly to the higher pH in Rucker and Cates' work (7.1) to 3.48 in our study.

The organic matter used in this work could have copper, iron and manganese in beer (Zufall and Tyrell, 2008); catalase, peroxidase and metallic ions such as copper, iron, cobalt, manganese and zinc in milk (Neville *et al.*, 1995) and metallic ions such as copper, iron, cobalt, magnesium, manganese and zinc in tomato juice (Hernández *et al.*, 2005). All these transition metal

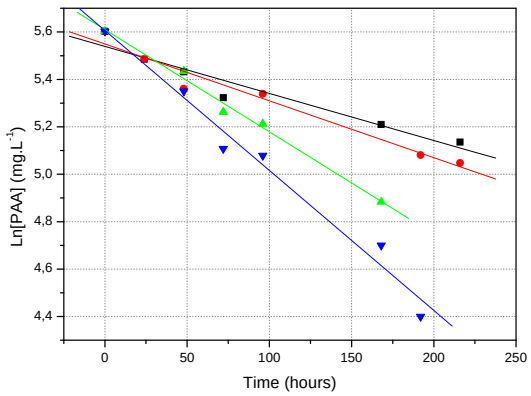

Figure 8. PAA concentration in solutions with 2.5 g/L of beer with initial pH: 3.50 (-■- 20°C; -●- 30°C; ▲ 35°C; -▼- 40°C).

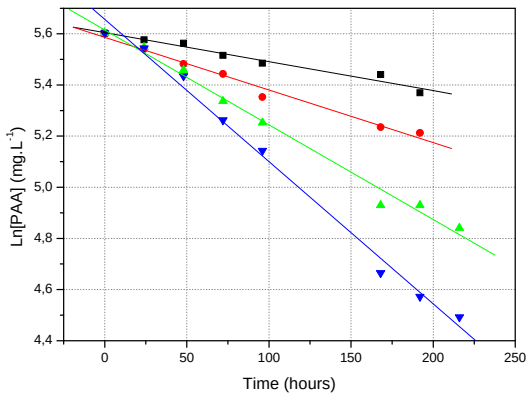

Figure 9. PAA concentration in solutions with 5.0 g/L of beer with initial pH: 3.51 (-■- 20°C; -●- 30°C; ▲ 35°C; -▼- 40°C).

ions can catalyze the decomposition of PAA. However the observed decomposition kinetics could be represented by a pseudo first-order reaction and the slopes of the equations, shown in Tables 2 to 4, are observed rate constants of PAA decomposition.

As it can be seen from Tables. 2 to 4, increasing the temperature from 20 to 40 °C, the observed rate constants of the PAA decomposition, were increased by a factor of 2.98 for the solutions with 2.5 $g \cdot L^{-1}$ of beer and by a factor of 4.92 for those with 5.0 $g \cdot L^{-1}$ of this same contaminant. However, the observed rate constants of the PAA solutions with 2.5 and 5.0 $g \cdot L^{-1}$ milk had increased 1.30 and 4.44 times respectively at the same temperature variation. In solutions containing 2.5 and 5.0 $g \cdot L^{-1}$ tomato juice, the observed rate constants were increased respectively 2.74 and 2.65 times, when the temperature rose from 20 to 40°C. These two last observed rate constants values can not be considered as different between them ($p<0.05$). Hence, tomato juice concentration did not affect the decomposition kinetics of PAA. As can be seen in these Tables, the presence of beer and milk in PAA solutions promoted the greatest influence on the observed rate constants. Lafontaine *et al.*, (2008) showed in their work that at 6 °C, PAA degraded much faster in ballast water (from St. Lawrence lower estuary) than in fresh water (St. Lawrence River water). They also reported that the rate of decay of PAA in water was accelerated in the presence of sediments. Without sediments, PAA were reduced by 30% in fresh water (0.0 Practical Salinity Units – PSU) after 7 days, but 92% in waters at salinity of 7 PSU and above. Figure 7 shows that PAA was reduced by 28% at 20 °C after 10 days. This Figure and Lafontaine *et al.*, (2008) work shows that PAA is more stable in distillated water than in river water.

The PAA half-life, $t_{½}$, was calculated using Eq. (3) for non-contaminated and contaminated solutions. It was admitted that $[PAA]_o$ is the initial PAA concentration and $[PAA]_o/2$ is the final PAA concentration. Therefore peracetic acid half-life, $t_{½}$, was calculated by:

$$t_{½} = \frac{0.6931}{k} \qquad (3)$$

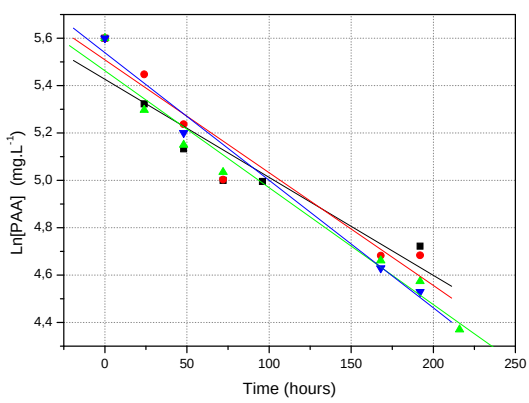

Figure 10. PAA concentration in solutions with 2.5 g/L of milk with initial pH: 3.73 (-■- 20°C; -●- 30°C; ▲ 35°C; -▼- 40°C).

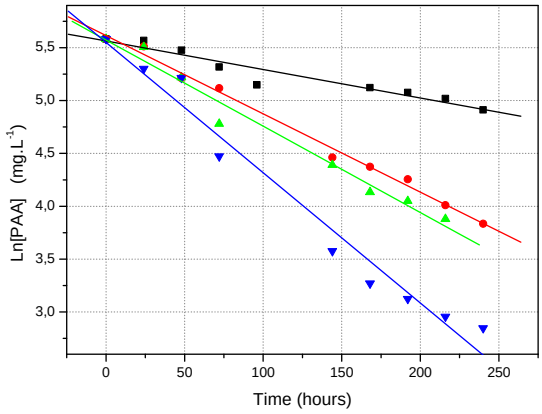

Figure 11. PAA concentration in solutions with 5.0 g/L of milk with initial pH: 4.48 (-■- 20°C; -●- 30°C; ▲ 35°C; -▼- 40°C).

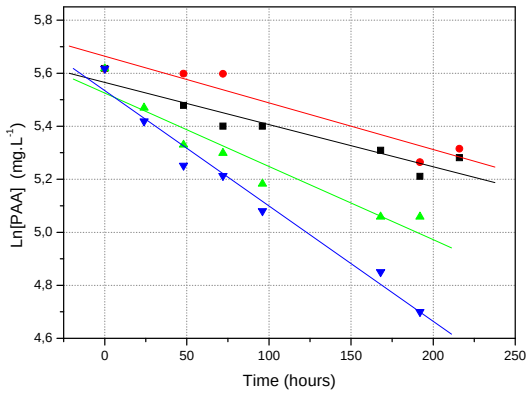

Figure 12. PAA concentration in solutions with 2.5 g/L of tomato juice with initial pH: 3.47 (-■- 20°C; -●- 30°C; ▲ 35°C; -▼- 40°C).

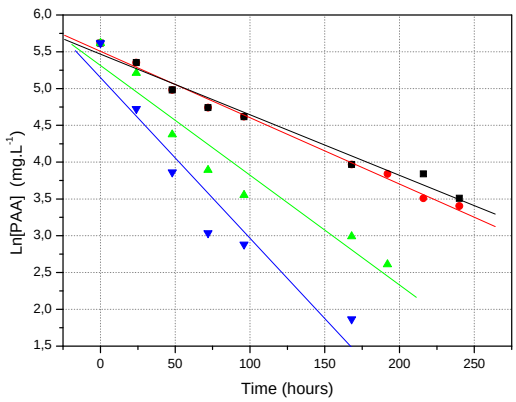

Figure 13. PAA concentration in solutions with 5.0 g/L of tomato juice with initial pH: 4.42 (-■- 20°C; -●- 30°C; ▲ 35°C; -▼- 40°C).

Equation (3) is a rate-constant function only. Therefore, for PAA non contaminated solutions, half-life went from 124 to 559 hours when the solutions were stored at 40 °C and 20 °C, respectively. So, the variation obtained is 77.8%. Kunigk *et al.* (2001) found a variation of 68.0% when storage temperature increased from

25 to 40 °C and a variation of 82.0% when storage temperature increased from 25 to 45 °C. Yuan *et al*. (1997b) showed that half-live at 25, 50 and 70 °C were 672, 144 and 48 hours, respectively at pH 7.0.

Table 5 shows the half-life variation of PAA solutions contaminated with organic matter which were stored between 20 and 40 °C. This table shows that the temperature and type of organic matter have affected the half-life variation of peracetic acid. For instance, if temperature of PAA solutions contaminated with 2.5 $g \cdot L^{-1}$ milk were raised from 20 to 40 °C, the variation of PAA half-life was 66.4%. However, with 2.5 $g \cdot L^{-1}$ milk, the same temperature variation, promoted a variation in the half-life of only 23.2% and 63.5% when there were 2.5 $g \cdot L^{-1}$ tomato juice.

Table 1. Mathematical equations that represent PAA decomposition kinetics in solutions free of organic matter.

Condition	Temp. (°C)	Linear coefficient $[PAA]_o$	Equation slope k (h^{-1})	r	Eq N°
free of organic matter	20	5.727	$1.24 \cdot 10^{-3}$	0.989	4
	30	5.699	$2.08 \cdot 10^{-3}$	0.996	5
	35	5.571	$5.17 \cdot 10^{-3}$	0.981	6
	40	5.630	$5.59 \cdot 10^{-3}$	0.982	7

Table 2. Mathematical equations that represent PAA decomposition kinetics in solutions contaminated with beer.

Condition	Temp. (°C)	Linear coefficient $[PAA]_o$	Equation slope k (h^{-1})	r	Eq. N°
2.5 g/L	20	5.54	$1.98 \cdot 10^{-3}$	0.966	8
	30	5.55	$2.40 \cdot 10^{-3}$	0.981	9
	35	5.61	$4.31 \cdot 10^{-3}$	0.995	10
	40	5.61	$5.90 \cdot 10^{-3}$	0.989	11
5.0 g/L	20	5.61	$1.13 \cdot 10^{-3}$	0.983	12
	30	5.59	$2.06 \cdot 10^{-3}$	0.992	13
	35	5.62	$3.70 \cdot 10^{-3}$	0.995	14
	40	5.66	$5.56 \cdot 10^{-3}$	0.996	15

Table 3. Mathematical equations that represent PAA decomposition kinetics in solutions contaminated with milk.

Condition	Temp. (°C)	Linear coefficient $[PAA]_o$	Equation slope k (h^{-1})	r	Eq. N°
2.5 g/L	20	5.43	$4.14 \cdot 10^{-3}$	0.927	16
	30	5.51	$4.76 \cdot 10^{-3}$	0.967	17
	35	5.46	$4.94 \cdot 10^{-3}$	0.982	18
	40	5.54	$5.39 \cdot 10^{-3}$	0.993	19
5.0 g/L	20	5.59	$2.77 \cdot 10^{-3}$	0.989	20
	30	5.62	$7.40 \cdot 10^{-3}$	0.996	21
	35	5.57	$8.16 \cdot 10^{-3}$	0.988	22
	40	5.55	$1.23 \cdot 10^{-2}$	0.986	23

Table 4. Mathematical equations that represent PAA decomposition kinetics in solutions contaminated with tomato juice.

Condition	Temp. (°C)	Linear coefficient $[PAA]_o$	Equation slope k (h^{-1})	r	Eq. N°
2.5 g/L	20	5.57	$1.59 \cdot 10^{-3}$	0.946	24
	30	5.66	$1.76 \cdot 10^{-3}$	0.953	25
	35	5.53	$2.76 \cdot 10^{-3}$	0.954	26
	40	5.53	$4.35 \cdot 10^{-3}$	0.987	27
5.0 g/L	20	5.47	$8.24 \cdot 10^{-3}$	0.988	28
	30	5.51	$9.03 \cdot 10^{-3}$	0.995	29
	35	5.31	$1.49 \cdot 10^{-2}$	0.966	30
	40	5.15	$2.18 \cdot 10^{-2}$	0.958	31

Table 5. Half-life of PAA solutions contaminated with organic matter.

Organic matter	Organic matter concentration (g/L)	Temperature (°C)	Half-life (h)	Half-life variation (%)
Beer	2.5	20	350.1	66.4
		40	117.5	
	5.0	20	613.4	79.7
		40	124.7	
Milk	2.5	20	167.4	23.2
		40	128.6	
	5.0	20	250.2	77.5
		40	56.3	
Tomato juice	2.5	20	435.9	63.5*
		40	159.3	
	5.0	20	84.1	62.2*
		40	31.8	

* no significant difference were observed between values ($p \leq 0.05$).

Another important parameter to consider in kinetics studies is the activation energy, calculated by Arrhenius equation:

$$k = k_o \cdot \exp(-\frac{E_a}{R \cdot T}) \qquad (4)$$

Figures 14 to 17 show the Arrhenius plots of the experimental data of this work. Table 6 shows the frequency or pre-exponential factor, $\boldsymbol{k_o}$ (h^{-1}) and the activation energies, $\boldsymbol{E_a}$ ($J \cdot mol^{-1}$), from the PAA decomposition with and without organic matter. Figures 14 to 17 also show that PAA solution, without organic matter and those contaminated with beer and milk had a unique slope for all temperature ranges. Therefore, the activation energies in these cases were constant. Figure 17 shows that PAA solutions contaminated with tomato juice had two slopes for the temperature range studied in this work. Levenspiel (1999) observed that a change in the activation energy with temperature indicates a shift in the controlling mechanism of reaction.

Reactions with high activation energies are very temperature-sensitive; reactions with low activation energies are relatively temperature-insensitive (Levenspiel, 1999). So the higher the activation energy, the lower must be the temperature variation to double the rate constant of PAA decomposition. Therefore, Table 6 shows that PAA solutions contaminated with 2.5 or 5.0 $g \cdot L^{-1}$ beer or milk are less temperature-sensitive than PAA solutions without organic matter. On the other hand, PAA solutions contaminated with 2.5 or 5.0 $g \cdot L^{-1}$ tomato juice between 30 and 40 °C, are more temperature-sensitive than PAA solutions without organic matter. The greatest activation energies were observed in solutions contaminated with tomato juice.

Table 6 shows that the activation energy of peracetic acid decomposition, in solutions free of organic matter, 62.1 $kJ \cdot mol^{-1}$, is close to the values obtained by Kunigk *et al*. (2001) and Zhao *et al*. (2007), 66.2 and 60.4 $kJ \cdot mol^{-1}$, respectively. However, it is lower than the 99.65 $kJ \cdot mol^{-1}$ reported by Rucker and Cates (1988) who have studied the peracetic acid decomposition at a pH of 7.1.

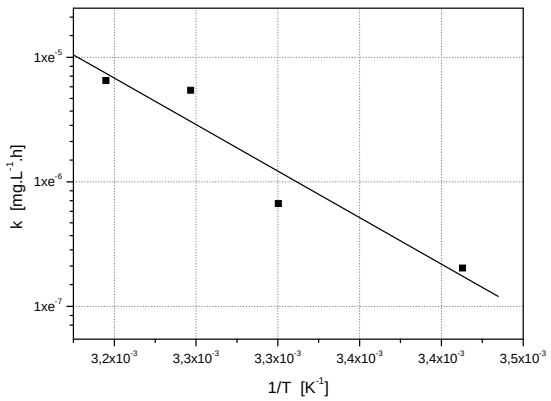

Figure 14. The influence of temperature (T) on the rate constant (k) for decomposition of PAA solutions without organic matter.

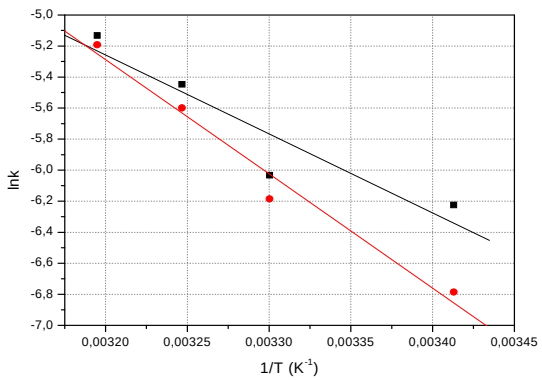

Figure 15. The influence of temperature (T) on the rate constant (k) for decomposition of PAA solutions contaminated with beer (beer concentration:-■- 2.5 g·L; -●- 5.0 g·L).

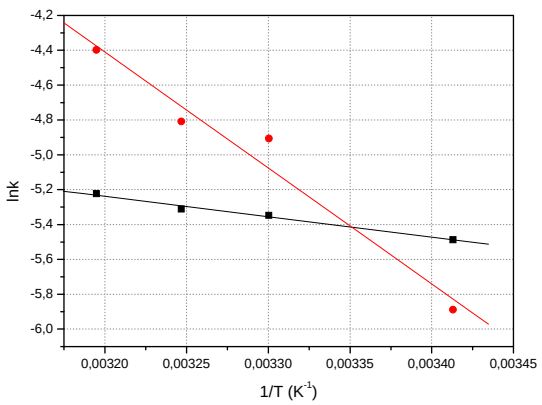

Figure 16. The influence of temperature (T) on the rate constant (k) for decomposition of PAA solutions contaminated with milk (milk concentration:-■- 2.5 g·L; -●- 5.0 g·L).

The PAA concentrations can be estimated by combining Eqs. (1) and (3), using 300 mg·L^{-1} as PAA initial concentration and data form Table 6. Therefore the equation used to estimate PAA concentration in solutions free of organic matter is:

$$Ln[PAA] = 5{,}70 \cdot [1{,}38 \cdot 10^{8} \cdot \exp(-7470{,}53 \cdot \frac{1}{T})] \cdot t \qquad (5)$$

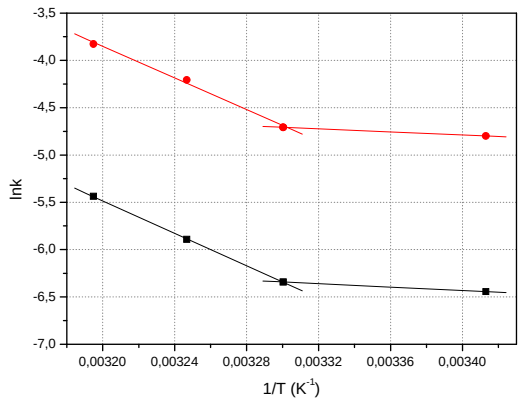

Figure 17. The influence of temperature (T) on the rate constant (k) for decomposition of PAA solutions contaminated with tomato juice (tomato juice concentration:-■- 2.5 g·L; -●- 5.0 g·L).

Table 6 – Pre-exponential factor and activation energies of equation (31) for PAA decomposition kinetics

Contamination	Organic matter concentration [g.h^{-1}]	Ln (k_o) [L.h^{-1}·mg^{-1}]	E_a [kJ·mol^{-1}]
Free of organic matter		18,74	62,11
Beer	2.5	11.01	42.27
	5.0	18.26	61.19
Milk	2.5	-1.50	9.72
	5.0	16.88	55.30
Tomato juice from 20 to 30 °C*	2.5	-3.37	7.50
	5.0	-2.03	6.76
from 30 to 40 °C	2.5	21.97	71.34
	5.0	22.92	69.54

* linear regression with two points only.

Table 7 – Mathematical equations that represent correlation between *Ln*[*PAA*] calculated from Eq. (4) and experimental values for PAA solutions free of organic contamination.

Temperature	Linear coefficient	Slope	r	Eq. N°
20	0.459	0.950	0.989	34
30	-1.619	1.285	0.996	35
35	0.750	0.913	0.987	36
40	-0.077	1.021	0.982	37

Table 7 shows the linear regression slopes obtained from the correlation between *Ln*[*PAA*] calculated from Eq. (4) and experimental values. These slope values can be considered as being 1.00 ($p<0.05$). Therefore, *Ln[PAA]* calculated from Eq. (4) and the experimental data for the same temperature cannot be considered as being different and the Eq. (4) can be used to estimate the PAA concentration for any temperature and storage time.

IV. CONCLUSIONS

We can see that with the increase of temperature and the organic matter concentration, the observed rate constant of PAA decomposition increases. The decomposition of PAA, can be represented by a first-order reaction when the solution is not contaminated with organic matter and a pseudo first-order reaction when there is organic mat-

ter present. It was also shown that the observed rate constant is affected by temperature according to the Arrhenius equation. The decomposition activation energy of PAA in aqueous solutions prepared from a commercial formulation is 62.1 $kJ \cdot mol^{-1}$, when there was no organic matter and 42.3 and 61.2 $kJ \cdot mol^{-1}$ when there was 2.5 and 5.0 $g \cdot L^{-1}$ beer in the solution. PAA solutions contaminated with 2.5 and 5.0 $g \cdot L^{-1}$ of milk, had activation energies of 9.7 and 55.3 $kJ \cdot mol^{-1}$, respectively. As in the solutions containing tomato juice, the slope obtained by the Arrhenius equation was not constant; this could indicate a shift in the controlling mechanism of the reaction. The different Ea values obtained can also indicate that different reactions are taking place at the various tested operating conditions. This work also proposes a mathematical equation to estimate the PAA concentration when its solutions do not have organic matter contamination.

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