

Degradation of polyvinyl alcohol in aqueous solutions using UV-365 nm/S₂O₈²⁻ process

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Received: 29 October 2011/Revised: 13 June 2012/Accepted: 16 March 2013/Published online: 27 April 2013
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Abstract This investigation evaluates the effectiveness of UV-365 nm/S₂O₈²⁻ process in degrading polyvinyl alcohol in aqueous solutions. The effects of pH, Na₂S₂O₈ dosage, and temperature on the degradation efficiency of polyvinyl alcohol were studied. Under acidic conditions, the degradation efficiency of polyvinyl alcohol exceeded that under alkaline conditions. Additionally, a higher Na₂S₂O₈ dosage and a higher temperature were associated with a higher degradation efficiency of polyvinyl alcohol. The degradation rates of polyvinyl alcohol followed a pseudo-first-order kinetic model. Moreover, the observed degradation rate coefficient increased from 0.0078 to 0.4081 min⁻¹ when the temperature was increased from 10 to 55 °C. Also, the activation energy estimated using the observed degradation rate coefficients and the Arrhenius equation was 64 kJ/mol. At UV-365 nm, pH 3, an Na₂S₂O₈ dosage of 0.06 g/L, a temperature of 55 °C, and an initial polyvinyl alcohol concentration of 20 mg/L, around 100 % of polyvinyl alcohol was degraded, indicating that UV-365 nm/S₂O₈²⁻ process has great potential in degrading polyvinyl alcohol in aqueous solutions.

Keywords Degradation · Polyvinyl alcohol · UV irradiation · Persulfate

Introduction

Polyvinyl alcohol (PVA), a water-soluble polymer synthesized by the hydrolysis of polyvinyl acetate, is widely utilized in industrial applications, such as an adhesive and a sizing agent in textiles (Tokiwa et al. 2001). It is also used as ophthalmic lubricant in the pharmaceutical industry (Chou et al. 2010). PVA is produced in greater quantities than any other water-soluble synthetic polymers. The global production of PVA is around 650,000 tons per year (Tokiwa et al. 2001). The large amount of discarded PVA in wastewater has become a significant problem. Therefore, a considerable amount of scientific studies on the degradation of PVA has been carried out. Most of these studies have focused on electrocoagulation (Chou et al. 2010), biodegradation (Solaro et al. 2000), adsorption (Behera et al. 2008), the Fenton process (Kang et al. 2002), photo-Fenton oxidation (Giroto et al. 2006), chemical oxidation (Won et al. 2001), and photocatalytic degradation (Hsu et al. 2011). These methods have certain drawbacks, including long operating time and high capital costs. For example, Solaro et al. (2000) proposed that around 100 % of PVA was degraded after 70 days of incubation. Therefore, it is imperative to develop more effective methods for the degradation of PVA in wastewater.

Persulfate ion (S₂O₈²⁻) has attracted growing interest as an alternative oxidant in the chemical oxidation of contaminants (Huang et al. 2002; Liang et al. 2007; Li et al. 2009). The application of S₂O₈²⁻ offers several advantages. For example, S₂O₈²⁻ is a strong oxidant ($E^\circ = 2.01$ V) that can be activated to generate an even stronger oxidant termed as a sulfate radical (SO₄^{-•}) ($E^\circ = 2.4$ V) (Huie et al. 1991). Moreover, S₂O₈²⁻ is non-selectively reactive and relatively stable at room temperature. S₂O₈²⁻ can be

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activated to produce two $\text{SO}_4^{\cdot-}$ under UV irradiation in accordance with Eq. (1), leading to a great potential for degrading organic compounds in wastewater.



Therefore, UV/ $\text{S}_2\text{O}_8^{2-}$ process has been confirmed to be effective in degrading organic contaminants (Hori et al. 2005, 2007; Lau et al. 2007; Salari et al. 2009; Criquet and Leitner 2009; Lin et al. 2011). For example, Lin et al. (2011) found that phenol (0.5 mM) can be completely degraded within 20 min using UV/ $\text{S}_2\text{O}_8^{2-}$ process with a UV wavelength of 254 nm and an $\text{S}_2\text{O}_8^{2-}$ concentration of 84 mM.

Almost all the studies using UV/ $\text{S}_2\text{O}_8^{2-}$ process are performed with 254 nm UV. But 254 nm light is not environmentally relevant because light of wavelengths below 290 nm reaching the earth is minimal (Sahoo et al. 2011). As pointed out by Goslich et al. (1997), the sun produces 0.2–0.3 mol photons at 1 m²/h in the range of 300–400 nm near the earth's surface with a typical UV flux of 20–30 W/m². Therefore, sunlight can be used as an economically and ecologically sensible light source in the wastewater treatment process. Accordingly, it would be ideal to adopt UV with a wavelength of 365 nm and verify the possibility of using sunlight in the UV/ $\text{S}_2\text{O}_8^{2-}$ process. The main objective of this investigation was to examine the feasibility of using UV-365 nm/ $\text{S}_2\text{O}_8^{2-}$ process in the degradation of PVA in aqueous solutions. Also, the degradation efficiency of PVA in relation to the main operating variables, which are pH, $\text{Na}_2\text{S}_2\text{O}_8$ dosage, and temperature, was examined. The findings of this investigation could be helpful for the treatment of wastewater contaminated with PVA. This investigation was conducted in Department of Chemical and Materials Engineering, Chang Gung University, Taoyuan, Taiwan, during 2010–2011.

Materials and methods

PVA with a molecular weight of 22,000 was obtained from Showa. Sodium persulfate ($\text{Na}_2\text{S}_2\text{O}_8$, 99 %) was obtained from Sigma. HNO_3 (65 %) was purchased from Fluka, and NaOH (99 %) was obtained from Mallinckrodt. Boric acid (99 %) was supplied by Showa, and potassium iodide (99.5 %) and iodine (99.9 %) were obtained from J.T. Baker. The aqueous PVA were prepared using deionized water.

Degradation of PVA using UV-365 nm/ $\text{S}_2\text{O}_8^{2-}$ process was conducted in a Pyrex batch cylindrical photoreactor with a 3-L capacity, containing aqueous PVA, as shown in Fig. 1. Two quartz tubes were placed vertically in the middle of the photoreactor; each tube contained a 8 W UV

lamp that was used as the UV irradiation source. The wavelength of the maximum emission induced from each UV lamp was 365 nm. The photoreactor, under which the photoreaction took place, had inlet and outlet ports for the bubbled nitrogen. Nitrogen was fed continuously into the photoreactor from the bottom at a flow rate of 400 mL/min, and a stirrer was used in the center of the photoreactor.

During all experiments, the temperature of the aqueous PAV in the photoreactor was maintained to a desired value using a temperature-controlled water bath. The pH of the aqueous PVA was adjusted by adding concentrated aqueous HNO_3 and NaOH. When the pH and temperature of the aqueous PVA had reached constant value, a known amount of $\text{Na}_2\text{S}_2\text{O}_8$ was added. Then, two UV lamps were turned on and degradation was sustained for 30 min. Analytic samples of 20 mL were withdrawn at regular time intervals.

After the sample was extracted, aqueous boric acid and iodine were added to the sample according to the method developed by Finley (1961). The amount of PVA in the sample was measured using a UV-Vis spectrophotometer (Jasco, V-630) at a maximum absorption wavelength of 690 nm with reference to a previously prepared calibration curve. This maximum absorption wavelength was determined by the product of the reaction of PVA with iodine in the presence of boric acid (Finley 1961) and did not alter during the degradation. The calibration curves at various pH values were plotted using the aqueous PVA with a

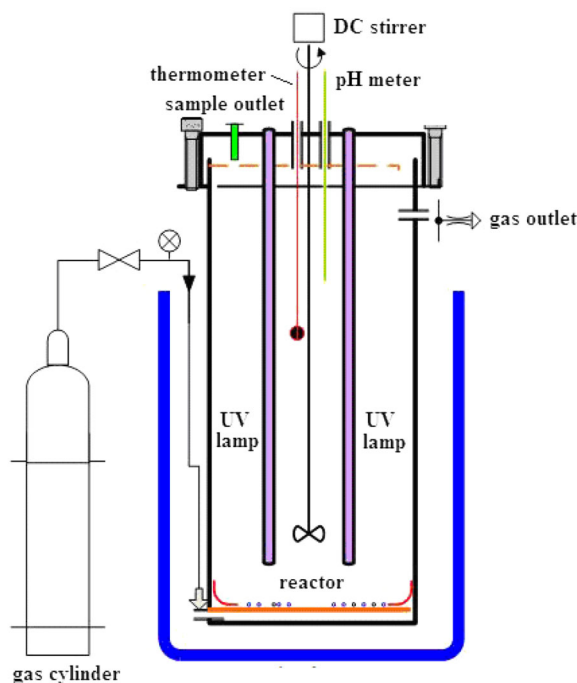


Fig. 1 Photoreactor for degradation of PVA using UV/ $\text{S}_2\text{O}_8^{2-}$ process

concentration of 0–30 mg/L. A linear dependence between the concentration of PVA and the absorbance at 690 nm was observed. Therefore, during the experiment, the degradation of PVA was monitored spectrophotometrically at this wavelength.

For the degradation of PVA using UV-365 nm/S₂O₈²⁻ process, the degradation efficiency of PVA (E) is defined as

$$E(\%) = \frac{C_0 - C}{C_0} \times 100 \quad (2)$$

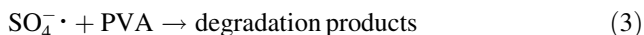
where C_0 is the initial PVA concentration and C is the concentration of PVA at time t . A higher E value represents a higher degradation efficiency of PVA. To evaluate the performance of UV-365 nm/S₂O₈²⁻ process in degrading PVA in aqueous solutions, the degradation efficiency of PVA is presented as functions of the main operating variables, which are pH, Na₂S₂O₈ dosage, and temperature.

Results and discussion

Performance of UV-365 nm/S₂O₈²⁻ process

To examine the performance of UV-365 nm/S₂O₈²⁻ process, control experiments for the degradation of PVA were carried out under different processes: (1) UV-365 nm, (2) S₂O₈²⁻, and (3) UV-365 nm/S₂O₈²⁻. These experiments were conducted at pH 3, with an initial PVA concentration of 20 mg/L and a temperature of 25 °C. The Na₂S₂O₈ dosage was 0.06 g/L under S₂O₈²⁻ and UV-365 nm/S₂O₈²⁻ processes. Figure 2 indicates that the self-photolytic degradation of PVA was not significant under UV-365 nm irradiation. After 30 min, the degradation efficiency of PVA was 1.9 %. When S₂O₈²⁻ was added, the significant degradation of PVA was observed. With UV-365 nm/S₂O₈²⁻ process, the degradation efficiency of PVA increased dramatically with the time and was observed to be 93.6 % after 30 min. However, with S₂O₈²⁻ process, the degradation efficiency of PVA (8.3 %) was much lower than that with UV-365 nm/S₂O₈²⁻ process, as measured after 30 min.

These observations implied that UV-365 nm/S₂O₈²⁻ process was associated with a higher degradation efficiency of PVA owing to the following mechanism (Hori et al. 2005, 2007; Lau et al. 2007; Salari et al. 2009; Criquet and Leitner 2009; Lin et al. 2011).



where $\text{SO}_4^- \cdot$ represents the sulfate radical. $\text{SO}_4^- \cdot$ was formed from photolytic decomposition of S₂O₈²⁻ (Eq. 1),

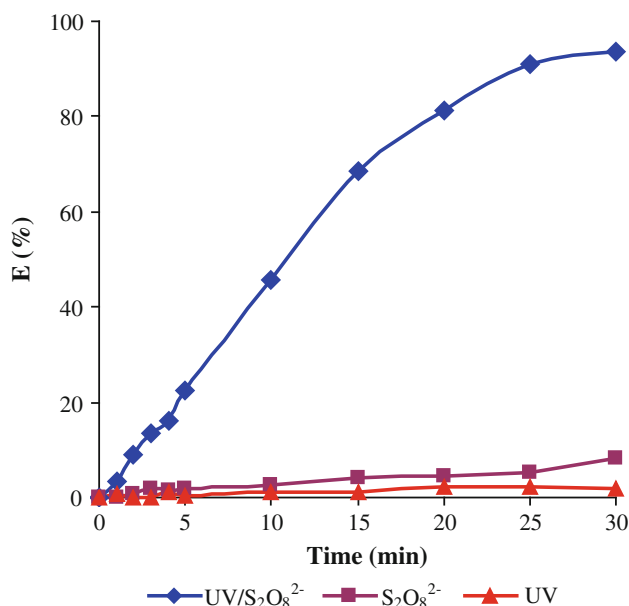


Fig. 2 Efficiency of UV/S₂O₈²⁻ process on degradation of PVA

and it can directly degrade PVA in aqueous solutions (Eq. 3). Therefore, in the presence of both S₂O₈²⁻ and UV-365 nm irradiation, more degradation of PVA occurred, as displayed in Fig. 2.

Degradation of PVA

To investigate the effect of pH on the degradation of PVA in aqueous solutions under UV-365 nm/S₂O₈²⁻ process, pH 3, 5, 7, 9, 11 were chosen to conduct degradation experiments with an Na₂S₂O₈ dosage of 0.06 g/L, a temperature of 25 °C, and an initial PVA concentration of 20 mg/L. Figure 3 reveals that the E values after 30 min for pH 3, 5, 7, 9, and 11 were 93.6, 85.9, 53.2, 37.2, and 22.9 %, respectively; clearly, the degradation efficiency of PVA after 30 min increased as pH declined. The results obtained in this investigation were also in agreement with the previous findings that the degradation efficiency of methyl *tert*-butyl ether (MTBE) by heat-assisted persulfate process (Huang et al. 2002) and the degradation efficiency of diphenylamine by persulfate (Li et al. 2009) increased as pH decreased. This behavior is attributable to the fact that sulfate radicals decayed rapidly owing to the reaction with hydroxyl ions (Hayon and McGarvey 1967). Additionally, carbon oxide formed from the degradation of PVA could lead to the formation of bicarbonate and carbonate ions under alkaline conditions, which may inhibit the degradation of PVA (Xu et al. 1989).

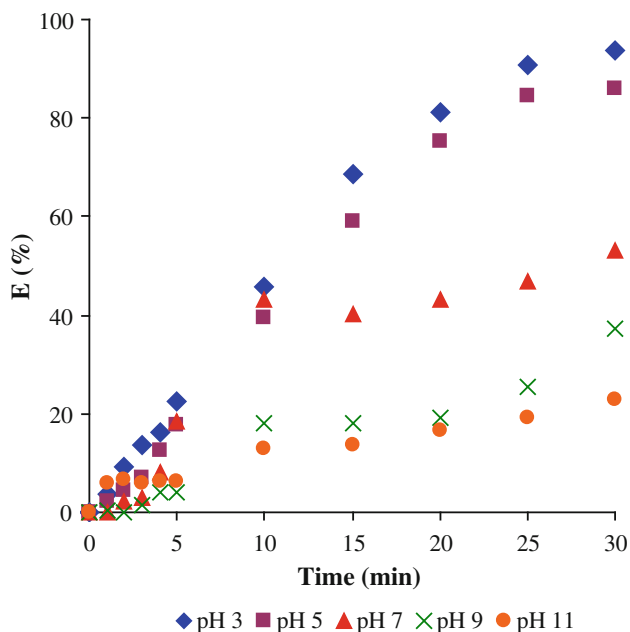


Fig. 3 Effect of pH on degradation of PVA

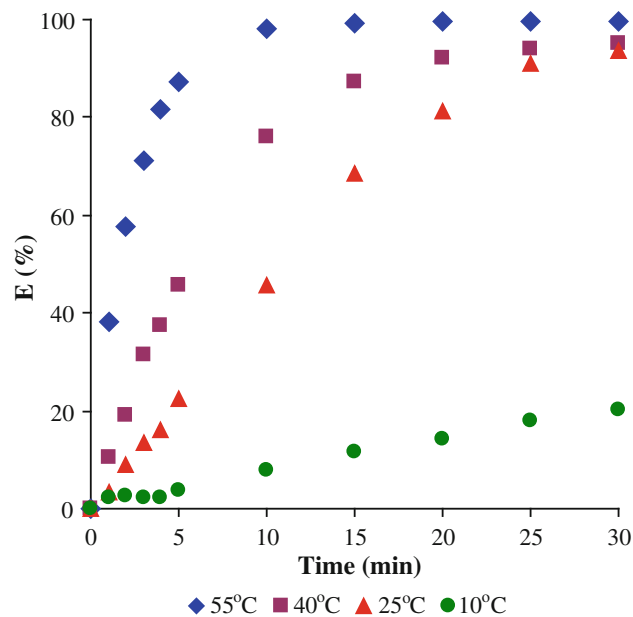


Fig. 5 Effect of temperature on degradation of PVA

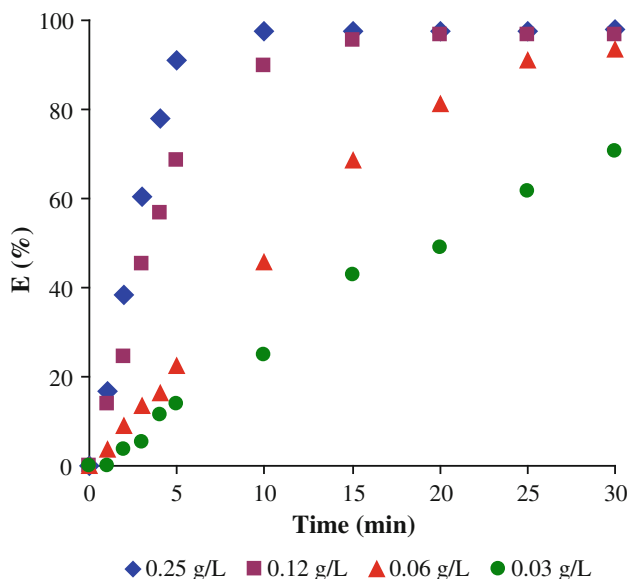
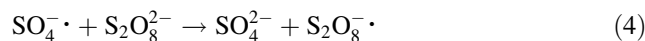


Fig. 4 Effect of $\text{Na}_2\text{S}_2\text{O}_8$ dosage on degradation of PVA

Figure 4 presents the effect of $\text{Na}_2\text{S}_2\text{O}_8$ dosage on the degradation efficiency of PVA at pH 3, with a temperature of 25 °C and an initial PVA concentration of 20 mg/L. As shown in Fig. 4, after 15 min, the E values with $\text{Na}_2\text{S}_2\text{O}_8$ dosages of 0.03, 0.06, 0.12, and 0.25 g/L were 42.9, 68.5, 95.5, and 97.5 %, respectively. After 30 min, the E values with $\text{Na}_2\text{S}_2\text{O}_8$ dosages of 0.03, 0.06, 0.12, and 0.25 g/L

were 70.7, 93.6, 96.9, and 97.9 %, respectively. This finding implied that the degradation of PVA increased significantly as the $\text{Na}_2\text{S}_2\text{O}_8$ dosage was increased from 0.03 to 0.12 g/L. However, further increase in the $\text{Na}_2\text{S}_2\text{O}_8$ dosage resulted in saturation, that is, no obvious increase in the degradation of PVA. Similar result was obtained for the degradation of perfluorooctanoic acid (PFOA) using UV-254 nm/ $\text{K}_2\text{S}_2\text{O}_8$ process (Hori et al. 2005). More $\text{Na}_2\text{S}_2\text{O}_8$ could produce more $\text{SO}_4^{\cdot-}$ radicals (Eq. 1), leading to more degradation of PVA. However, at higher $\text{Na}_2\text{S}_2\text{O}_8$ dosages, the reaction of $\text{SO}_4^{\cdot-}$ with $\text{S}_2\text{O}_8^{2-}$ (Eq. 4) occurred (Yu et al. 2004), resulting in saturation of $\text{SO}_4^{\cdot-}$ concentration.



According to a previous study (Yu et al. 2004) and our results, the $\text{SO}_8^{\cdot-}$ should be less reactive than $\text{SO}_4^{\cdot-}$.

To investigate the enhancement of the degradation efficiency of PVA by raising temperature, this experiment was performed at pH 3, with an $\text{Na}_2\text{S}_2\text{O}_8$ dosage of 0.06 g/L and an initial PVA concentration of 20 mg/L. The results are displayed in Fig. 5. After 15 min, the E values with temperature of 10, 25, 40, and 55 °C were 11.8, 68.5, 87.2, and 99.1 %, respectively. After 30 min, the E values with temperature of 10, 25, 40, and 55 °C were 20.4, 93.6, 95.2, and 99.6 %, respectively. This result revealed that higher temperature significantly enhanced the degradation of PVA during an initial period (0–15 min). As pointed out by Li et al. (2009), the degradation of diphenylamine by persulfate was significantly influenced by temperature; that is, the

Table 1 Comparison of three methods for degradation of PVA

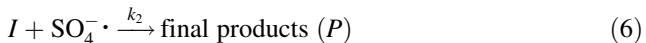
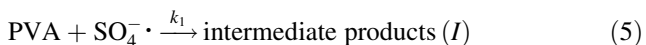
	Electrocoagulation (Chou et al. 2010)	Photocatalytic degradation (Hsu et al. 2011)	UV-365 nm/S ₂ O ₈ ²⁻
PVA concentration (mg/L)	100	20	20
Volume of PVA solution (L)	0.5	3	3
Temperature (°C)	25	25	25
pH	6.5	3	3
Operating time (min)	120	120	30
Degradation efficiency (%)	96	100	100

higher the temperature, the faster the degradation of diphenylamine. When temperature was increased, more SO₄^{-•} radicals were formed (Eq. 1) and the reaction of SO₄^{-•} with PVA was accelerated (Eq. 3). Accordingly, a higher temperature was associated with a higher degradation efficiency of PVA.

Table 1 summarizes the difference of three methods for the degradation of PVA. As described in “Introduction”, electrocoagulation and photocatalytic degradation would have a long operating time, thus leading to an increase in the operating cost. The UV-365 nm/S₂O₈²⁻ process could provide an advantage of a reduced operating time owing to that the UV-365 nm/S₂O₈²⁻ process offers a faster degradation rate of PVA. However, the UV-365 nm/S₂O₈²⁻ process has a disadvantage that more energy consumption was needed for UV irradiation. If the appropriate adjuvant for activating S₂O₈²⁻ was found for reducing the time of UV irradiation, the UV-365 nm/S₂O₈²⁻ process would offer a decrease in the operating cost when compared with other two methods.

Kinetics of degradation of PVA

The behavior described in the above section may be interpreted quantitatively by the following kinetic model:



In this mechanism, SO₄^{-•} attack may involve the formation of different intermediate products, and then in other steps, these intermediate products also were degraded and final products were produced. The corresponding kinetic equations for S₂O₈²⁻, PVA, and SO₄^{-•} are:

$$-\frac{d[S_2O_8^{2-}]}{dt} = \Phi I_a \tag{7}$$

$$-\frac{d[PVA]}{dt} = k_1[PVA][SO_4^{-\bullet}] \tag{8}$$

$$-\frac{d[SO_4^{-\bullet}]}{dt} = k_1[PVA][SO_4^{-\bullet}] + k_2[I][SO_4^{-\bullet}] - 2\Phi I_a \tag{9}$$

In Eq. (7), Φ is the quantum yield of the photochemical decomposition of S₂O₈²⁻, I_a is the radiation intensity absorbed by S₂O₈²⁻.

According to steady state approximation with Eq. (9), the concentration of SO₄^{-•} can be calculated:

$$[SO_4^{-\bullet}] = \frac{2\Phi I_a}{k_1[PVA] + k_2[I]} \tag{10}$$

In the early stage of the process, we can assume:

$$[PVA]_0 = [PVA] + [I] \tag{11}$$

The following relationship is obtained by substituting Eqs. (10) and (11) into Eq. (8):

$$-\frac{d[PVA]}{dt} = \frac{2k_1\Phi I_a}{(k_1 - k_2)[PVA] + k_2[PVA]_0} [PVA] \tag{12}$$

The difference between k₁ and k₂ is supposed to be negligible (k₁ = k₂), thus it can be written as:

$$k_2[PVA]_0 \gg (k_1 - k_2)[PVA] \tag{13}$$

Also, in the initial part of the process, Φ and I_a are assumed to be constants. Accordingly, Eq. (12) can be rewritten as follows:

$$-\frac{d[PVA]}{dt} = \frac{2k_1\Phi I_a}{k_2[PVA]_0} [PVA] \tag{14}$$

or more simply:

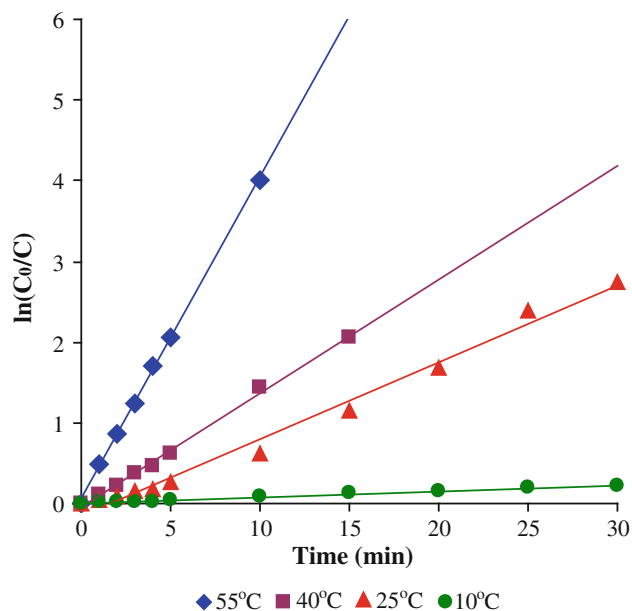


Fig. 6 Kinetics of degradation of PVA at various temperatures

Table 2 k values at various temperatures

Temperature (°C)	k [L(min) ⁻¹]	R^2
10	0.0078	0.9967
25	0.0876	0.9863
40	0.1362	0.9972
55	0.4081	0.9994

$$\frac{dC}{dt} = -kC \quad (15)$$

where C represents the concentration of PVA at time t and k is the observed degradation rate coefficient. The integration of Eq. (15) yields Eq. (16) as follows:

$$\ln\left(\frac{C_0}{C}\right) = kt \quad (16)$$

The slope of the plot of $\ln(C_0/C)$ versus time gives the k values. Here, C_0 is the initial PVA concentration. Figure 6 shows the kinetics of the degradation of PVA as a pseudo-first-order kinetic model fitting for various temperatures. Then, the k values for the degradation of PVA at various temperatures were determined graphically; they are listed in Table 2. The agreement between experimental data and the results obtained using the model (Eq. 16) was evaluated from coefficients of determination (R^2). The high values of R^2 at all

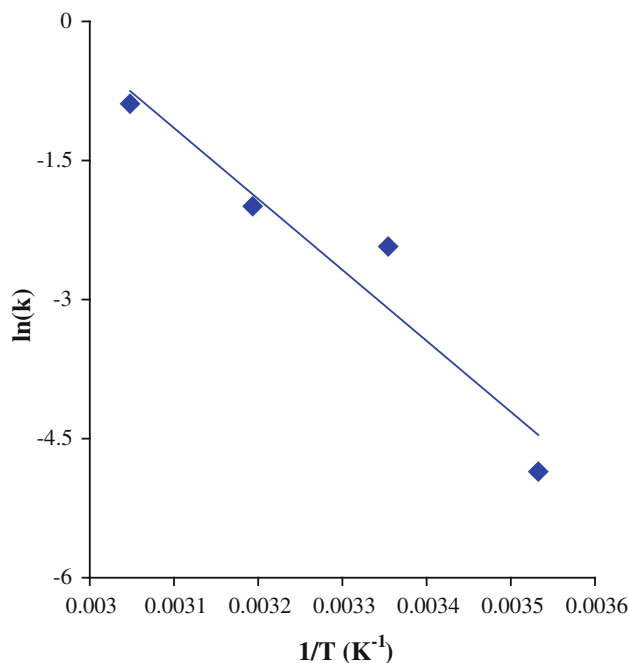


Fig. 7 Regression of Arrhenius equation for degradation of PVA

temperatures revealed that the degradation of PVA using UV-365 nm/S₂O₈²⁻ process obeyed the pseudo-first-order kinetic model.

To understand the relationship between the temperature and the rate of the degradation of PVA, we assume that the k values can be expressed by the Arrhenius equation (Huang et al. 2002; Liang et al. 2007) as follows:

$$k = A \exp\left(-\frac{E_a}{RT}\right) \quad (17)$$

where A denotes the proportionally constant of the degradation of PVA, E_a denotes the activation energy of the degradation of PVA, R denotes the gas constant ($=8.134$ J/mol K), and T denotes the temperature. Thus, the activation energy of the degradation of PVA can be determined using Eq. (17). By taking the natural logarithm of both sides of Eq. (17), we obtain

$$\ln(k) = \ln(A) - \frac{E_a}{R}T \quad (18)$$

According to Eq. (18), the plot of $\ln(k)$ versus $1/T$ should provide a straight line with a slope of $-E_a/R$, as illustrated in Fig. 7. An activation energy of 64 kJ/mol was determined using the slope of the fitted equation ($R^2 = 0.9244$).



Conclusion

This investigation elucidated the feasibility of degrading PVA in aqueous solutions using UV-365 nm/S₂O₈²⁻ process, as well as the effects of pH, Na₂S₂O₈ dosage, and temperature on this process. Experimental results demonstrated that the degradation efficiency of PVA in the acidic aqueous solution was higher than that in the alkaline aqueous solution. Moreover, the degradation efficiency of PVA increased with Na₂S₂O₈ dosage and temperature. A pseudo-first-order kinetic model can describe the degradation of PVA using UV-365 nm/S₂O₈²⁻ process. Additionally, the activation energy for the degradation of PVA using UV-365 nm/S₂O₈²⁻ process was 64 kJ/mol, determined using the observed degradation rate coefficient (*k*) and the Arrhenius equation. At pH 3, an Na₂S₂O₈ dosage of 0.06 g/L, a temperature of 55 °C, and an initial PVA concentration of 20 mg/L, the degradation efficiency of PVA was around 100 %. Therefore, the proposed approach based on UV-365 nm/S₂O₈²⁻ process is highly promising for the degradation of polymers in aqueous solutions.

Acknowledgments The authors would like to thank the National Science Council of the Republic of China, Taiwan, for financially supporting this research under Contract No. NSC 100-2815-C-182-019-E.

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