# Autocatalytic Oxidation of β-Alanine by Peroxomonosulfate in the Presence of Copper(II) Ion

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Received 21 February 2007; revised 5 August 2007; accepted 11 August 2007

DOI 10.1002/kin.20288 Published online in Wiley InterScience (www.interscience.wiley.com).

ABSTRACT: Kinetics and mechanism of oxidation of  $\beta$ -alanine by peroxomonosulfate (PMS) in the presence of Cu(II) ion at pH 4.2 (acetic acid/sodium acetate) has been studied. Autocatalysis was observed only in the presence of copper(II) ion, and this was explained due to the formation of hydroperoxide intermediate. The rate constant for the catalyzed ( $k_2^{obs}$ ) and uncatalyzed ( $k_1^{obs}$ ) reaction has been calculated. The kinetic data obtained reveal that both the reactions are first order with respect to [PMS].  $k_1^{obs}$  values initially increase with the increase in [ $\beta$ -alanine] and reach a limiting value, but  $k_2^{obs}$  values decrease with the increase in [ $\beta$ -alanine].  $k_1^{obs}$  values increase linearly with the increase in [Cu(II)], whereas  $k_2^{obs}$  values increase with [Cu(II)]<sup>2</sup>. Furthermore,  $k_1^{obs}$  values are independent of [acetate], but  $k_2^{obs}$  values decrease with the increase in acetate. A suitable mechanism has been proposed to explain the experimental observation. The reaction has been studied at different temperatures, and the activation parameters are calculated. © 2007 Wiley Periodicals, Inc. Int J Chem Kinet 40: 44–49, 2008

## INTRODUCTION

The kinetics of oxidation of  $\alpha$ -amino acids by peroxomonosulfate has been studied extensively in buffered medium [1–4] (acetic acid/sodium acetate) and also in highly alkaline medium [5] (NaOH > 0.1 M). In buffered medium, the observation was the exhibition of autocatalysis of the product aldehyde. The mechanism proposed involves the interaction between aldehyde and peroxomonosulfate (PMS) to produce hydroperoxide, which in turn reacts with amino acids to give the product. The kinetics of oxidation of  $\alpha$ -amino acids in buffered medium and also in the presence of metal ions, such as Cu(II) and Ni(II), has also been studied recently [6]. Here, the presence of metal ion inhibits the autocatalysis because the PMS was not free to form hydroperoxide, which is responsible for the autocatalysis. PMS reacts with metal ions, such as Cu(II) [7] and Ni(II) [8], to form an intermediate that reacts with a faster step with amino acids to give the product.



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Although the kinetics of  $\alpha$ -amino acids with PMS has been studied extensively, but little information is available in the literature about the oxidation of  $\beta$ -amino acids, such as  $\beta$ -alanine, with PMS. Moreover, an  $\alpha$ -amino acid undergoes oxidative decarboxylation and deamination, whereas a  $\beta$ -amino acid undergoes oxidation by deamination only, because the removal of direct conjugation between NH<sub>3</sub><sup>+</sup> and COO<sup>-</sup> groups by the intervening CH<sub>2</sub> groups is responsible for the difficulty in decarboxylative deamination. Hence, it is proposed to study the kinetics of the oxidation of  $\beta$ -alanine in buffered medium and the results obtained are discussed in this paper.

## EXPERIMENTAL

Potassium peroxomonosulfate was obtained from Dupont (Lancaster, PA, USA) under the trade name "Oxone." The purity of the triple salt  $2KHSO_4 \cdot KHSO_4 \cdot K_2SO_4$  was estimated by iodometry and was found to be 98%. However, the presence of  $H_2O_2$  in an oxone sample was tested and it showed negative results, thus eliminating the chances of the hydrolysis of oxone. A fresh solution of peroxomonosulfate was prepared before starting the experiments.  $\beta$ -alanine was purchased from Loba-Chemie Indo Austranal Co. (Mumbai, India) and was used as such. All other chemicals used in this study were of the Analar grade and were used as such without any further treatment.

Doubly distilled water was employed throughout the study; the second distillation was from alkaline permanganate solution in an all glass still.

The reaction mixtures containing all reactants except peroxomonosulfate in glass-stoppered Erlenmeyer flasks were immersed in the water bath thermostated at  $35 \pm 0.1^{\circ}$ C. Peroxomonosulfate was taken in another flask to attain the same temperature of  $35 \pm$ 0.1°C. The required volume of the oxidant solution was pipetted out into the reaction mixture. Experiments were carried out in acetic acid/sodium acetate buffered medium at various pHs ranging from 3.6 to 5.2. The rate of oxidation of  $\beta$ -alanine was followed by monitoring the concentration of unreacted oxone by iodometry. Autocatalysis was observed from the plot of  $\log_{10}$  (volume of thio) at various time intervals ( $V_t$ ) versus time (Fig. 1). The values of catalyzed and uncatalyzed rate constants were calculated. The relative standard errors of the above-mentioned rate constants for a single run and the relative standard errors of the mean were about 2%.

The stoichiometry of the reaction was determined under the kinetic conditions by taking a known excess concentration of the oxidant over  $\beta$ -alanine, that



Figure 1 Plot of log [PMS]<sub>t</sub> versus time [ $\beta$ -alanine] = 0.05 mol dm<sup>-3</sup>, [NaOAc] = 0.08 mol dm<sup>-3</sup> [Cu(II)] = 2.5 × 10<sup>-3</sup> mol dm<sup>-3</sup>, pH 4.2 ± 0.1.

is, [peroxomonosulfate]/[ $\beta$ -alanine] = 2.5 with [Cu<sup>2+</sup>] 2.5 × 10<sup>-3</sup> mol and pH 4.2 allowed to stand for 48 h. The experiment was performed with different initial concentrations of the oxidant and  $\beta$ -alanine by maintaining the ratio at 2.5. Then, the unreacted oxidant in each case was estimated by iodometry. After making corrections for self-decomposition of the oxidant, which was carried out under the same kinetic conditions, stoichiometry was calculated as  $\beta$ -alanine: PMS = 1:1. Thus, the stoichiometric ratio evident from the data corresponds to the reaction and can be written as

$$H_2N-CH_2-CH_2-COOH + PMS$$

$$\xrightarrow{Cu^{2+}}_{pH4.0}OHC-CH_2-COOH + Cu^{2+}$$

$$+HSO_4^- + NH_4^+ \qquad (1)$$

The product analysis was done under kinetic conditions as described below. The reaction mixture containing  $\beta$ -alanine at pH 4.2 and PMS in the presence of Cu(II) was allowed to stand for 48 h for the completion of the reaction. After completion of the reaction, the excess oxidant was destroyed by adding NaHSO<sub>3</sub> and then extracted with dichloromethane. The organic layer obtained was analyzed to identify the product. FT-IR and H<sup>1</sup> NMR data confirm the product as malonic semialdehyde, and the retention time at 9.423 min and 99.1% purity was confirmed by gas chromatography/mass spectrometry. The chromatographic conditions used for the oxidation kinetics to identify the product were EC5, 30 m  $\times$  0.2 mm, 0.25-µm column with nitrogen as a carrier gas with a flow of 1.0 mL/min. Injection and detection temperatures for the study were 280°C and 290°C, respectively. The oven temperature was initially started from 50°C and held for 2 min, and

the temperature was raised to  $280^{\circ}$ C with increments of  $15^{\circ}$ C/min and held at  $280^{\circ}$ C for 30 min.

#### **RESULTS AND DISCUSSION**

The rate of oxidation of  $\beta$ -alanine by PMS is very slow. However, the added Cu(II) ions even at a concentration of 2.5 mM enhance the rate of reaction appreciably; hence, the reaction has been studied only in the presence of Cu(II) ions at 35°C. pH of the reaction was maintained with acetic acid/sodium acetate at 3.6–5.2. The kinetics was investigated under pseudofirst-order conditions, that is, [ $\beta$ -alanine]  $\geq$  [PMS]. The rate of the reaction was measured by monitoring the concentration of unreacted PMS at various time points iodometrically.

The rate of oxidation of  $\beta$ -alanine follows firstorder kinetics with respect to [PMS] only at lower concentrations of  $\beta$ -alanine < 0.05 M, as evidenced by log [PMS] versus time plots with  $r \ge 0.99$ . However, as the concentration of  $\beta$ -alanine is increased, the firstorder plot shows curvature toward the time axis, which may be due to the product that catalyzes the reaction. The oxidation of  $\beta$ -alanine can be represented by the autocatalysis reaction as

$$\beta$$
-Alanine + Cu(II) + PMS  $\rightarrow$  I<sub>1</sub> + Product (2)

$$I_1 + PMS \rightarrow I_2 + Product$$
 (3)

$$I_2 + \beta$$
-alanine · · · Cu(II)  $\rightarrow 2I_1 + Product$  (4)

Under the experimental condition, where [AA] > [PMS], the rate equation can be written with the assumption that  $[I_1] = [PMS]_0 - [PMS]_t$  as

$$-d[PMS]/dt = k_1^{obs}[PMS]_t + k_2^{obs}[PMS]_t + k_2^{obs}[PMS]_t([PMS]_0 - [PMS]_t)$$
(5)

The best values of  $k_1^{\text{obs}}$  and  $k_2^{\text{obs}}$  can be obtained from the nonlinear region. The kinetic constants are calculated at different PMS concentrations. Perusal of the results shows that the kinetic constants remain constant even after a threefold increase in [PMS]. This shows that the reaction is first order with respect to [PMS].

The effect of  $\beta$ -alanine on the rate constant values is studied at constant pH and by keeping other parameters at a constant value.  $k_1^{\text{obs}}$  value initially increases with the increase in [ $\beta$ -alanine] and then reaches a limiting value. Moreover, the plot at  $1/k_1^{\text{obs}}$  versus  $1/(\beta$ -alanine) is a good straight line (r = 0.99) with a positive intercept (Fig. 2a). But  $k_2^{\text{obs}}$  values decrease with the increase in [ $\beta$ -alanine] and the plot of  $k_2^{\text{obs}}$  versus [ $\beta$ alanine]<sup>-1</sup> is a straight line (Fig. 2b).



**Figure 2** (a) Plot of  $1/k_1^{obs}$  versus [β-alanine]<sup>-1</sup> at 294 K, [NaOAc] = 0.08 mol dm<sup>-3</sup>, [Cu(II)] =  $2.5 \times 10^{-3}$  mol dm<sup>-3</sup>, pH 4.2 ± 0.1,  $r^2 = 0.9948$ . (b) Plot of  $k_2^{obs}$  versus [β-alanine]<sup>-1</sup> at 294 K, [NaOAc] = 0.08 mol dm<sup>-3</sup>, [Cu(II)] =  $2.5 \times 10^{-3}$  mol dm<sup>-3</sup>, pH 4.2 ± 0.1,  $r^2 = 0.9953$ .

The effect of Cu(II) on the rate constant values is studied by keeping other parameters constant.  $k_1^{obs}$  and  $k_2^{obs}$  values are found to increase linearly with the increase in the Cu(II) ion concentration, and the plots of  $k_1^{obs}$  versus [Cu(II)] and  $k_2^{obs}$  versus [Cu(II)]<sup>2</sup> give straight lines (Fig. 3).

The effect of pH on the rate is studied by keeping other parameters constant. The rate of disappearance of [PMS] at pH 3.6 becomes simple first-order kinetics as shown by a good correlation between log [PMS] and time (r = 0.999). At all other pH values, the intermediate catalyzed oxidation also contributes to the rate as evidenced by the nonlinear regression with higher correlation coefficient (r = 0.99). Perusal of the results in  $\beta$ -alanine shows that the  $k_1^{\text{obs}}$  value first decreases and then increases with the increase in the pH values.

The effect of the acetate ion is studied by keeping other parameters constant at pH 4.2 (Fig. 4). Perusal of the results shows that  $k_1^{obs}$  values are independent of [acetate], but  $k_2^{obs}$  value decreases with the increase in acetate. The reaction has been studied at different temperatures, and the thermodynamic parameters are calculated and given in Table I



**Figure 3** (a) Plot of  $k_1^{obs}$  versus [Cu(II)] at 294 K, [β-alanine] = 0.05 mol dm<sup>-3</sup>, [NaOAc] = 0.08 mol dm<sup>-3</sup> pH 4.2 ± 0.1,  $r^2$  = 0.9941. (b) Plot of  $k_2^{obs}$  versus [Cu(II)]<sup>2</sup> at 294 K, [β-alanine] = 0.05 mol dm<sup>-3</sup>, [NaOAc] = 0.08 mol dm<sup>-3</sup>, pH 4.2 ± 0.1,  $r^2$  = 0.9995.



Figure 4 Plot of  $1/k_2^{\text{obs}}$  versus [acetate] at 294 K, [ $\beta$ -alanine] = 0.05 mol dm<sup>-3</sup>, [Cu(II)] =  $2.5 \times 10^{-3}$  mol dm<sup>-3</sup>, pH 4.2  $\pm$  0.1,  $r^2$  = 0.9929.

**Table I**Temperature Variation and ThermodynamicParameters for the  $Cu^{2+}$ -Catalyzed Oxidation of $\beta$ -Alanine by Peroxomonosulfate

Parameters	β-Alanine	
	$10^5 \times k_1^{\rm obs}$	$10^2 \times k_2^{\rm obs}$
Temperature (K)		
294	2.16	12.43
301	5.12	39.96
308	15.51	110.20
$E_{\rm a}$ (kJ mol <sup>-1</sup> )	45.8	46.4
$\Delta H^{\neq}$ (kJ mol <sup>-1</sup> )	44.0	49.0
$\Delta S^{\neq}$ (J mol <sup>-1</sup> deg <sup>-1</sup> )	-362.056	-289.27

Peroxomonosulfate ion  $HSO_5^-$  is a weak acid with pK<sub>a</sub> value of 9.4 [9,10] at moderate acidic pH, and PMS exists predominantly as  $HSO_5^-$ 

$$\mathrm{HSO}_{5}^{-} \xrightarrow{k_{\mathrm{d}}} \mathrm{SO}_{5}^{2-} + \mathrm{H}^{+} \tag{6}$$

 $\beta$ -Amino acids similar to  $\alpha$ -amino acids exist in the following equilibrium:



The pK<sub>a</sub> values [11] of  $\beta$ -alanine suggest that at the experimental conditions, that is, at pH 4.2, a zwitter ionic form is the active form of  $\beta$ -alanine. In aqueous solution, the zwitter ions lose a proton from the <sup>+</sup>NH<sub>3</sub> group and chelate with a metal ion of the transition metal ions; Cu(II) is one of the most kinetically labile ions [12]. The equilibrium of the copper– $\beta$ -alanine complex in aqueous medium can be represented as

$$Cu^{2+}H_2N-CH_2-CH_2-COO^{-} \xleftarrow{K_1} Cu(A)^{+} (8)$$
$$Cu(A)^{2+} + H_2N-CH_2-CH_2-COO^{-} \xleftarrow{K_2} Cu(A)_2 (9)$$

 $Cu(A)^+$  represents copper–mono- $\beta$ -alanine complex, and  $Cu(A)_2$  is the bis complex. A review of literature on  $Cu-\beta$ -alanine complexes shows that the formation of the bis complex is insubstantial and hence may be neglected; furthermore, the percentage of the mono complex increases with the increase in pH [13].

On the basis of the experimental results, the following reaction scheme for the oxidation of  $\beta$ -alanine is proposed:

$$Cu(II) + PMS \xrightarrow{k_1}$$
 Intermediate (10)

Intermediate  $\xrightarrow{\text{fast}}$  Cu(II) + SO<sub>4</sub><sup>2-</sup> + O<sub>2</sub>  $\uparrow$  (11)

Intermediate +  $\beta$ -alanine  $\xrightarrow{\text{fast}}$  Product (12)

$$\operatorname{Cu(II)} + \beta$$
-alanine  $\underbrace{\frac{K_{a}}{k_{a}}}_{\mu_{a}} \operatorname{Cu(A)}$  (13)

$$Cu(A) + Intermediate \xrightarrow{\kappa_2} Product$$
 (14)

$$PMS \xrightarrow{\kappa_3} Intermediate 1$$
(15)

Amino Acid	$10^5 \times k_1 \text{ mol}^{-1} \text{ dm}^3 \text{ s}^1$	$10^5 \times k_2 \text{ mol}^{-1} \text{ dm}^3 \text{ s}^1$	$10^5 \times k_3 \text{ mol}^{-1} \text{ dm}^3 \text{ s}^1$
β-Alanine	320.0	3650.0	1.6

Table IIKinetic Constants for the  $Cu^{2+}$  Catalyzed Oxidation of  $\beta$ -Alanine by Peroxomonosulfate

Intermediate 
$$1 + \beta$$
-alanine  $\xrightarrow{\text{rast}}$  Product (16)

 $\beta$ -alanine + PMS  $\xrightarrow{\kappa_4}$  Product

The rate equation for the above scheme at constant pH can be written as

$$-d/dt[PMS]/[PMS] = k_1^{obs} = k_1[Cu(II)]$$
$$+k_2[Cu]^2[\beta-alanine] + k_3 + k_4[\beta-alanine] \quad (17)$$

The values of  $k_1$ ,  $k_2$ , and  $k_3$  are calculated from the different plots and are tabulated in Table II.

The experiment carried out in the absence at  $\beta$ alanine shows that PMS is quite stable in the presence of Cu(II) at pH 4.2, and only in the presence of  $\beta$ alanine the oxidation is observed. This can be explained by the fact that the reaction between Cu(II) and PMS may be the rate-limiting step in which the reactive intermediate is formed, which in turn reacts with  $\beta$ alanine rapidly. This means the rate-limiting step is the formation of reactive copper or PMS intermediate.

Available information shows that in acidic medium the mode of reaction of PMS with a metal ion is through the production of radicals, such as  $HSO_5^{-\bullet}$  [14–16]  $SO_4^{-\bullet}$  [17], or OH<sup>•</sup> [18].

$$\begin{split} \mathbf{M}^{n+} + \mathbf{HSO}_5^- &\to \mathbf{M}^{(n-1)+} + \mathbf{H}^+ + \mathbf{SO}_5^{-\bullet} \\ \mathbf{M}^{n+} + \mathbf{HSO}_5^- &\to \mathbf{M}^{(n-1)+} + \mathbf{OH}^- + \mathbf{SO}_4^{-\bullet} \\ \mathbf{Cu}(\mathbf{I}) + \mathbf{HSO}_5^- &\to \mathbf{Cu}(\mathbf{II}) + \mathbf{OH}^{\bullet} + \mathbf{SO}_4^{2-} \end{split}$$

Extension of such metal ion initiated radical mechanism to this system can be ruled out, because the Cu(II)-catalyzed oxidation is observed only in the presence of  $\beta$ -alanine. Furthermore, the presence of radical mechanism was ruled out completely on the basis of the experimental observations that the rate of oxidation is not affected by (i) the introduction of O<sub>2</sub> into the reaction mixture, (ii) the modification of the surface area of the reaction vessel, and (iii) nonpolymerization of acrylonitrile added to the reaction mixture.

The foregoing discussion suggests that some reactive intermediates from Cu(II) and PMS are responsible for the reaction. The hydroperoxo Cu-(II) ion [16], CuOOH<sup>+</sup>, is formed by the action of  $H_2O_2$  on Cu(II) ions at neutral pH. Cu-(II) peroxide can be prepared by the action of  $H_2O_2$  on alkaline solution of Cu(II) salts [17,18]. Available information on the catalyzed self-decomposition of PMS suggests that the reaction proceeds through  $H_2O_2$  as an intermediate in strong acidic medium [19] and  $HO_2^-$  in alkaline medium [10]; moreover, in the recent literature formation of nickel peroxide in acetic acid/sodium acetate buffer [8] is reported. Therefore, we can propose the mechanism as

$$Cu(II) + HSO_5^{-} \xrightarrow{rds}_{H_2O} CuO_2 + SO_4^{2-} + H^+$$
(18)

$$CuO_2 + \beta$$
-alanine  $\xrightarrow{fast}$  product (19)

The experimental results suggest that the oxidation by PMS proceeds through deamination of  $\beta$ -alanine to the corresponding oxo acids. The other intermediate from PMS, such as CuO<sub>2</sub>, also follows the same mechanism of the intermediate-catalyzed oxidation of  $\beta$ -alanine in the presence of Cu(II) ion.

The oxidative decarboxylation of  $\alpha$ -amino acids by PMS at pH 4.2 shows that aldehyde catalyzes the reaction, and the catalytic effect is attributed to the formation of hydroperoxide, which in turn reacts with  $\alpha$ -amino acids to give the product. The observation of  $k_2^{obs}$  showing an inverse dependence on [ $\beta$ -alanine] can be explained by the fact that the formation of hydroperoxide from the oxo intermediate may be the ratedetermining step.

$$OHC-CH_2-COOH + Cu^{2+}$$

$$\xrightarrow{K} OHC-CH_2-COO^{-}\cdots Cu^{+} + H^{+} \quad (20)$$

$$OHC-CH_2-COO^-\cdots Cu^+ + HSO_5^-$$

$$\xrightarrow{k_2} \text{Hyderoxide(I)}$$
(21)

 $Hydroperoxide(I) + H_3^+N - CH_2 - CH_2 - COOH$ 

$$\xrightarrow{\text{fast}} \begin{array}{c} \text{OHC--CH}_2\text{--COO}^-\cdots\text{Cu}^+ \\ & + \\ \text{OHC--CH}_2\text{--COO}^- \end{array}$$
(22)

$$OHC-CH_2-COO^- + H^+$$
(23)

Therefore, the kinetic constant,  $k_2^{\text{obs}} = K k_2 [\text{Cu(II)}]_f$ , by substituting the concentration of  $[\text{Cu(II)}_f]$  and by approximation

$$k_2^{\text{obs}} = \frac{k_2 K [\text{Cu}(\text{II})]_{\text{T}}^2 [\text{H}^+] \{K_1 + [\text{H}^+]\}}{[\text{H}^+] \{K_1 + [\text{H}^+]\} K_a K_1 K_2 [\beta-\text{alanine}]_{\text{T}}}$$
(24)

The above equation explains all the experimental observations. The inhibitory effect of the acetate ion on  $k_2^{obs}$  can be explained by the following reaction:

$$\Theta HC - (CH_2) - CH_2 - COO \cdots Cu^+ + OAc^-$$
$$\Longrightarrow OHC - CH_2 - COO + Cu(OAc)^+$$
(25)

The effect of acetate on  $k_2^{obs}$  also supports the fact that the copper complex of the resultant oxo acid is involved in this reaction. The structure of aldehyde obtained from  $\beta$ -alanine involves a carboxyl group and a hydrogen ion. Furthermore, keto-enol tautomerism is well known in oxo carboxylic acid [20] as

$$OHC-CH_2-COOH \longrightarrow HO-CH=CH-COOH$$
 (26)

The presence of  $Cu^{2+}$  may inhibit the equilibrium. This may be the reason for observation of autocatalysis only in the presence of  $Cu^{2+}$  in the oxidation of  $\beta$ -alanine. The outcome from the oxidation of  $\beta$ -alanine is that the aldehyde group of oxo acid also reacts with PMS resulting in a more reactive intermediate.

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