Application of Deaerated Water in Swelling of Cellulose and Amylose and Cotton Desizing with Enzyme

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ABSTRACT: The effect of deaerated water on the swelling of cellulose and amylose and on the application in cotton desizing with enzyme that contains necessarily these swelling processes were studied. The total volume changes of the swelling of cellulose and amylose were measured by dilatometry. The total volume changes of cellulose and amylose were more negative in deaerated water. In deaerated water, the total volume changes are 142 ± 6% for cellulose and 229 ± 2% for amylose to those in air-present water. The rates of cotton desizing with two kinds of enzymes were studied by measuring the concentration of generated sugars by using HPLC. Higher efficiency of 140–150% was obtained in deaerated water than in water containing dissolved gases of air, oxygen, or nitrogen. © 1999 John Wiley & Sons, Inc. J Appl Polym Sci 74: 1693–1700, 1999

Key words: deaerated water; swelling of cellulose; swelling of amylose; cotton desizing

INTRODUCTION

The interaction between hydrophilic polymers and water was studied in our series of articles.[1–5] The degree of interaction between the polymer and water measured by dilatometry was different in air-present water from in deaerated water that was obtained by cooling in a sealed container after boiling.[3,4] Air-present water in equilibrium with air contains about 16 mg L^{-1} of nitrogen, 7–8 mg L^{-1} of oxygen, and some inert gases at room temperature. These days, the deaerated water, which is the water after these dissolved gases are removed, can easily be obtained with membrane technology.[6–9] Some other researchers have studied the application of deaerated water in food processing.[10–12]

In our previous article,[4] the effect of dissolved air in water on the swelling of hydrophilic polymers was investigated. Both the hydrobondings between water and polymers and the dissociation of ionic dissociable groups of polymers were increased in water with a low-dissolved oxygen concentration.

The effects of the deaerated water on the swelling of Konbu (*Laminaria Japonica*) by was examined by using the gravimetric and buffer capacity methods at different temperatures.[13] The absorbed water and amounts of soluble components from Konbu increased with time and showed simple saturation curves. At low temperatures, the values of the rate constants in deaerated water were slightly smaller than those in air-present water. The maximum values of the absorbed water and amounts of soluble components were greater than those in air-present water, this being more apparent at lower temperature. The efficiency of deaerated water was calculated to be 13 ± 4% per 10 mg L^{-1} of oxygen concentration.

In this article, to examine the application efficiency of deaerated water to textile finishing, we determined the effect of deaerated water on the kinetics of the swelling of cellulose and amylose

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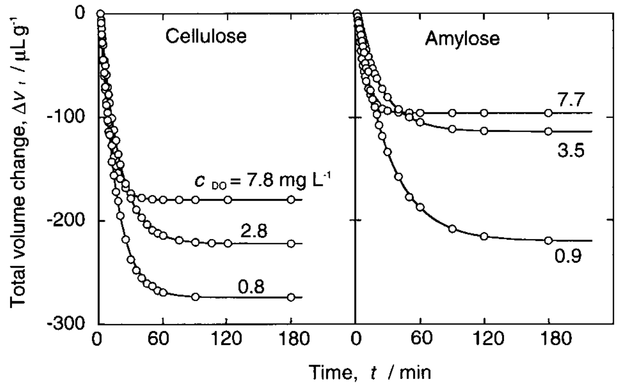

Figure 1 Time course of total volume change of the swelling of cellulose and amylose obtained by dilatometry at 25°C.

as an application model. The rates of cotton desizing with two kinds of enzymes were also studied by measuring the concentration of generated sugars.

EXPERIMENTAL

Water

The deaerated water was made by boiling water in a Myer flask and storing it sealed with a silicone-rubber stopper. As a criterion for the concentration of dissolved gases in water, the concentration of the dissolved oxygen, c_{DO}, was measured with a dissolved-oxygen meter (DO-8F, Horiba Co., Kyoto, Japan). c_{DO} of the deaerated water was 0.8 ± 0.1 mg L^{-1}. Deionized water in equilibrium with air was used for air-present water. c_{DO} of air-present water was 7.0 ± 1.0 mg L^{-1}. Water having different concentrations of c_{DO} was obtained by mixing deaerated water and air-present water in a glass syringe.

Oxygen- (O_2) saturated water was obtained by bubbling oxygen gas into deaerated water by monitoring c_{DO}. c_{DO} of O_2-saturated water was > 20.0 mg L^{-1}.

Nitrogen- (N_2) saturated water was obtained by bubbling nitrogen gas into air-present water until c_{DO} was equilibrated near 0. c_{DO} of N_2-saturated water was 0.25 ± 0.05 mg L^{-1}.

Dilatometric Measurement of Swelling of Cellulose and Amylose

Cellulose powder (Cellulose Microkrisrallin Avicel 2330 from Merk) for thin-layer chromatography was used. Before the measurement, cellulose powder was dried for 3 days at 105 ± 5°C.

Amylose powder was kindly offered from the Laboratory of Physical Biochemistry of Osaka Prefecture University, Sakai, Osaka, Japan, with a mean degree of polymerization of 760. Amylose powder was preconditioned in a silica-gel desiccator for 1 month.

The total volume changes of cellulose or amylose and water system were measured by dilatometry. Details are in our previous study.[3] About 60 cm^3 of the sample water was added to 200 mg of cellulose or amylose. The time course of the total volume change of the system was measured. The total volume change is represented as Δv. Δv values correspond to the quantities of interaction between water and the polymer.[4] The temperature was maintained at 25.000 ± 0.001°C during the measurement.

Cotton and Desizing Enzyme

The gray cotton, with ends, 27; picks, 27; thickness, 0.42 mm; and weight, 148.9 gm^{-2}, was used in cotton desizing with enzyme in deaerated water and air-present water. It was rinsed with the sample water before desizing.

Cotton desizing enzymes, Aquazyme (30 L) and Termamyl (120 L), supplied from Novo Nordisk Bio-Industries, Inc., Chiba, Japan, were used. These are bacterial amylase that decompose starch into dextrin and sugars.[14] The specific activities of these enzymes are 30 KNU (Kilo Novo Unit) for Aquazime and 120 KNU for Termamyl.[14] The best pH ranges were 5–7 for Aquazime, 6–8 for Termamyl.[15] The desizing performed at pH 6.3 with Aquazime and 7.0 with Termamyl.

Ten grams of plain-woven cotton fabric was desized with Aquazime (0.1 g L^{-1}) and Termamyl

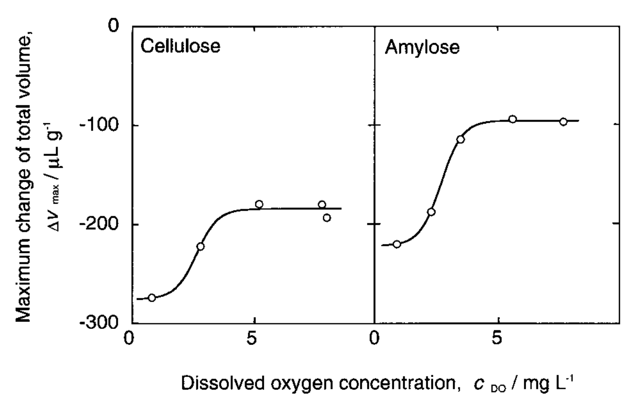

Figure 2 Effect of dissolved oxygen concentration on the maximum change of total volume of the swelling of cellulose and amylose obtained by dilatometry at 25°C.

Table I The First-Order Kinetic Parameters of the Total Volume Change of Cellulose Obtained by Dilatometry at 25°C

DO Conc. c_{DO} (mg L^{-1})	Maximum Change of Total Volume		Rate Constant	
	Δv_{max} (μL g^{-1})	$\Delta v_{max}/\Delta v_{max\,A}$[a]	k (min^{-1})	k/k_A[b]
8.0	−193.3 ± 15.6	1	0.120 ± 0.005	1
7.8	−179.8 ± 11.2	0.930 ± 0.110	0.117 ± 0.004	0.975 ± 0.055
5.1	−179.4 ± 13.3	0.928 ± 0.118	0.089 ± 0.004	0.742 ± 0.083
2.8	−222.2 ± 10.2	1.150 ± 0.081	0.057 ± 0.003	0.475 ± 0.141
0.8	−274.0 ± 10.0	1.417 ± 0.062	0.067 ± 0.003	0.558 ± 0.110

[a] $\Delta v_{max\,A}$ is Δv_{max} at $c_{DO} = 8.0$.
[b] k_A is k at $c_{DO} = 8.0$.

(0.02 g L^{-1}) by using a fiber : liquor ratio of 1 : 10 in a glass syringe. The glass syringe was incubated at 100 rpm at 25°C.

The efficiency of cotton desizing was determined by measuring the concentrations of generated sugars in the desizing bath after each treatment time. The concentrations of each of the generated sugars, glucose (G1), maltose (G2), maltotriose (G3), maltopentaose (G5), and maltohexsaose (G6), were analyzed by using high-performance liquid chromatography (HPLC). HPLC was carried out by using L-6200 and RI Detector L-3300 from Hitachi, Tokyo, Japan. The separations of 10 μL of sample were performed by G615-S column (divinylstyrene polymer resin, 5 mm$\phi \times$ 150-mm length) with a flow rate of 0.5 mL min^{-1} of water at 60°C.

RESULTS AND DISCUSSION

Dilatomeric Measurement of Swelling of Cellulose and Amylose

Figure 1 shows the results of the swelling of cellulose and amylose in different c_{DO} water obtained by dilatometry. The total volume change, Δv_t, is plotted against time, t. The results of cellulose are on the left side and those of amylose are on the right side. Δv_t decreases with time. The values of Δv_t at $t = \infty$ are more negative in lower c_{DO} water for both cellulose and amylose.

Δv_t value at $t = \infty$ is represented as a maximum change of total volume, Δv_{max}. The plots of Δv_{max} against c_{DO} are shown in Figure 2. Δv_{max} is more negative at lower c_{DO} and shows a sigmoid curve.

Treating the change of the total volume as an apparent first-order reaction (A $\xrightarrow{k}$ B), the rate of total volume change can be represented by $d\Delta v_t/dt = k(\Delta v_{max} - \Delta v_t)$, in which k is the rate constant and Δv_{max} is the maximum value of total volume change. These apparent first-order kinetic parameters obtained are listed in Table I for cellulose and Table II for amylose.

Δv_{max} of cellulose is more negative at lower c_{DO}. This is similar to the result for amylose. The total volume change measured by dilatometry shows the degree of the hydration of water and polymer.[3,4] In deaerated water, the hydration is

Table II The First-Order Kinetic Parameters of the Total Volume Change of Amylose Obtained by Dilatometry at 25°C

DO Conc. c_{DO} (mg L^{-1})	Maximum Change of Total Volume		Rate Constant	
	Δv_{max} (μL g^{-1})	$\Delta v_{max}/\Delta v_{max\,A}$[a]	k (min^{-1})	k/k_A[b]
7.7	−96.3 ± 3.6	1	0.135 ± 0.004	1
5.6	−93.7 ± 2.4	0.973 ± 0.047	0.088 ± 0.003	0.733 ± 0.069
3.5	−114.2 ± 3.8	1.186 ± 0.042	0.044 ± 0.002	0.367 ± 0.167
2.3	−187.5 ± 4.2	1.947 ± 0.022	0.033 ± 0.002	0.275 ± 0.276
0.9	−220.0 ± 3.4	2.285 ± 0.018	0.034 ± 0.002	0.283 ± 0.262

[a] $\Delta v_{max\,A}$ is Δv_{max} at $c_{DO} = 7.7$.
[b] k_A is k at $c_{DO} = 7.7$.

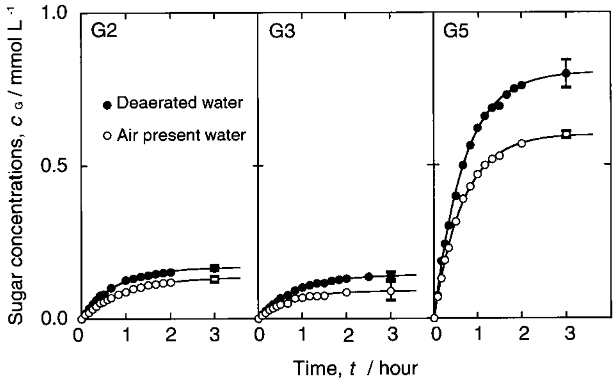

Figure 3 Time course of sugar concentration change in treatment bath of cotton desizing with Aquazime in deaerated water and air-present water at 25°C.

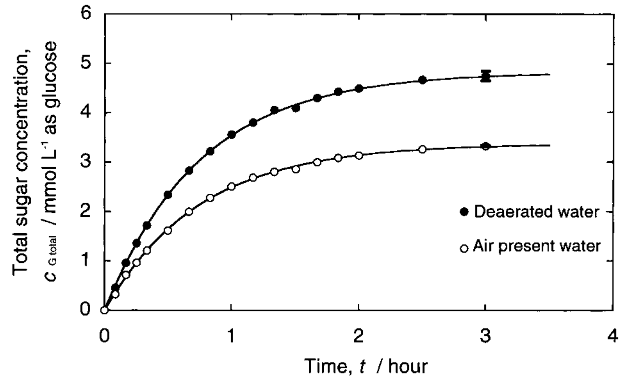

Figure 4 Time course of total sugar concentration in cotton desizing in deaerated water and air-present water with Aquazime at 25°C.

thought to be 142 ± 6% for cellulose and 229 ± 2% for amylose to the hydration in air-present water.

The rate constants, k, at low c_{DO} is smaller than those in high c_{DO} water for both cellulose and amylose. This tendency is similar to the results in our previous article.[13]

The Determination of Sugar Concentration Produced in Cotton Desizing with Enzyme

As described previously, the hydration of cellulose and amylose are more accelerated in low c_{DO} water than in air-present water. Because cotton desizing with enzyme involves these swelling processes, we compared the results of the cotton desizing with enzyme in deaerated water to the results in air-present water.

The kinetics of cotton desizing with Aquazime was investigated. Three kinds of sugars, G2, G3, and G5, were detected by HPLC analysis in desizing bath. Figure 3 shows the plots of sugar concentrations, c_G, against the treatment time, t. c_G increased with t. c_G in deaerated water is higher than that in air-present water. All these results apparently show simple saturation curves.

The concentration of G2, G3, and G5 are represented as c_{G2}, c_{G3}, and c_{G5}. These values are reduced to G1 concentration, c_{G1}. Total sugar concentration, $c_{G\,total}$, was calculated as follows, $c_{G\,total} = 2 \times c_{G2} + 3 \times c_{G3} + 5 \times c_G$. The plots of $c_{G\,total}$ against t are shown in Figure 4. They apparently show simple saturation curves.

Treating the change of the sugar concentration with time as an apparent first-order reaction, first-order kinetic parameters, the maximum sugar concentrations at $t = \infty$ and the rate constants k are calculated and listed in Table III. Table III shows the results of each sugar detected by HPLC and total sugars calculated from each sugar concentration.

The maximum sugar concentrations in deaerated water and air-present water are represented as $c_{GD\,max}$ and $c_{GA\,max}$, respectively. The rate constants in deaerated water and in air-present water are represented as k_D and k_A. The ratios of

Table III Apparent First-Order Kinetic Parameters of Cotton Desizing with Aquazime at 25°C

	Maximum Glucose Concentration (mmol L^{-1})			Rate Constant, k (min^{-1})		
Sugar	Deaerated Water, $c_{GD\,max}$	Air-Present Water, $c_{GA\,max}$	$c_{GD\,max}/c_{GA\,max}$	Deaerated Water (k_D)	Air-Present Water (k_A)	k_D/k_A
G2	0.169 ± 0.008	0.136 ± 0.009	1.24 ± 0.01	0.022 ± 0.003	0.018 ± 0.002	1.25 ± 0.05
G3	0.143 ± 0.013	0.090 ± 0.003	1.59 ± 0.02	0.020 ± 0.004	0.024 ± 0.002	0.82 ± 0.03
G5	0.807 ± 0.045	0.602 ± 0.011	1.34 ± 0.01	0.024 ± 0.003	0.025 ± 0.005	0.96 ± 0.05
Total	4.824 ± 0.099	3.376 ± 0.017	1.43 ± 0.00	0.022 ± 0.002	0.022 ± 0.000	0.99 ± 0.00

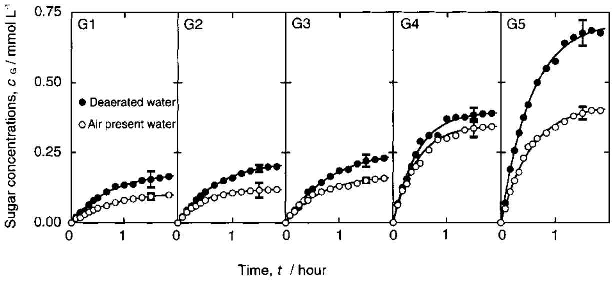

Figure 5 Time course of sugar concentration in treatment bath of cotton desizing with Termamyl in deaerated water and air-present water at 25°C.

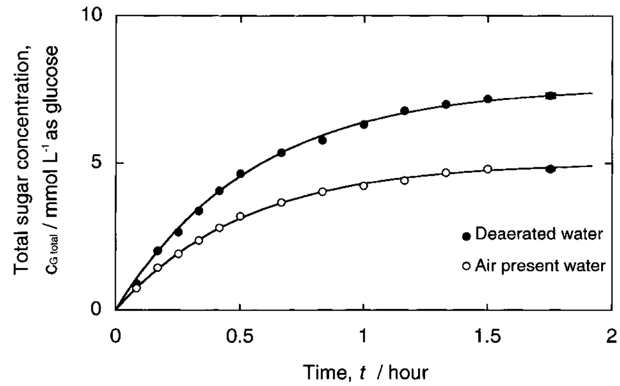

Figure 6 Time course of total sugar concentration in treatment bath of cotton desizing with Termamyl in deaerated water and in air-present water at 25°C.

these values in deaerated water to those in air-present water, namely, $c_{GD\,max}/c_{GA\,max}$ and k_D/k_A, are also listed in Table III.

For all sugars, $c_{GD\,max}$ is higher than $c_{GA\,max}$. $c_{GD\,max}/c_{GA\,max}$ of detected sugars are values of 124–159% and $c_{GD\,total}/c_{GA\,total}$ of total sugar is 143%. More starch is decomposed into sugars in deaerated water than in air-present water. The rate constants are different for each sugar. For total sugar concentration, k_D/k_A is nearly 1.

Figure 5, Figure 6, and Table IV show the results of cotton desizing with Termamyl in air-present water and deaerated water.

Sugars G1, G2, G3, G4, and G5 were detected by HPLC. Figure 5 shows the time course of each sugar concentration in desizing solution. All the sugar concentrations, c_G, are increased with treatment time, t. c_G in deaerated water is higher than that in air-present water. The sugar concentrations apparently show simple saturation curve.

Each sugar concentration was reduced to the concentration of glucose, G1, and the total sugar concentration, $c_{G\,total}$, was calculated as follows, $c_{G\,total} = c_{G1} + 2 \times c_{G2} + 3 \times c_{G3} + 4 \times c_{G4} + 5 \times c_{G5}$. Figure 6 shows the plots of $c_{G\,total}$ against treatment time, t. $c_{G\,total}$ in deaerated water are higher than that in air-present water. $c_{G\,total}$ apparently shows simple saturation curves.

The apparent first-order kinetic parameters are listed in Table IV. The ratios of $c_{GD\,max}$ to $c_{GA\,max}$ are 144–171%, and that of calculated total sugar ratio is 152%. More starch was decomposed into sugars in deaerated water than in air-present water. Although k for each sugar shows a different tendency between in deaerated water and air-present water, k of total sugar in deaerated water is similar to k in air-present water.

The Effect of Varieties of Dissolved Gas on Cotton Desizing with Enzyme

The effect of the kind of dissolved gases on cotton desizing with Aquazime was investigated by the

Table IV Apparent First-Order Kinetic Parameters of Cotton Desizing with Termamyl at 25°C

	Maximum Glucose Concentration, $c_{G\,max}$ (mmol L^{-1})			k (min^{-1})		
Sugar	Deaerated Water ($c_{GD\,max}$)	Air-Present Water ($c_{GA\,max}$)	$c_{GD\,max}/c_{GA\,max}$	Deaerated Water (k_D)	Air-Present Water (k_A)	k_D/k_A
G1	0.179 ± 0.028	0.109 ± 0.011	1.64 ± 0.09	0.023 ± 0.005	0.022 ± 0.006	1.06 ± 0.12
G2	0.221 ± 0.013	0.120 ± 0.026	1.84 ± 0.17	0.023 ± 0.003	0.039 ± 0.014	0.60 ± 0.13
G3	0.259 ± 0.022	0.168 ± 0.008	1.54 ± 0.02	0.020 ± 0.004	0.026 ± 0.003	0.79 ± 0.03
G5	0.396 ± 0.025	0.347 ± 0.031	1.14 ± 0.02	0.038 ± 0.007	0.038 ± 0.002	0.98 ± 0.03
G6	0.716 ± 0.046	0.418 ± 0.022	1.71 ± 0.02	0.030 ± 0.004	0.030 ± 0.005	0.98 ± 0.04
Total	7.602 ± 0.075	5.015 ± 0.036	1.52 ± 0.00	0.031 ± 0.001	0.032 ± 0.001	0.94 ± 0.00

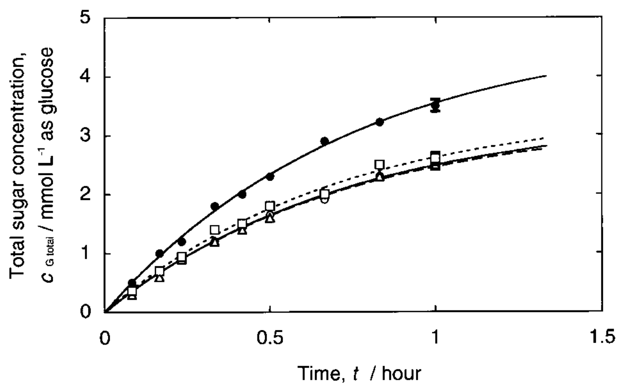

Figure 7 Effect of dissolved gas on cotton desizing with Aquazime at 25°C. (—●—) Deaerated water, (—○—) air-present water, (—△—) O_2-saturated water, (—□—) N_2-saturated water.

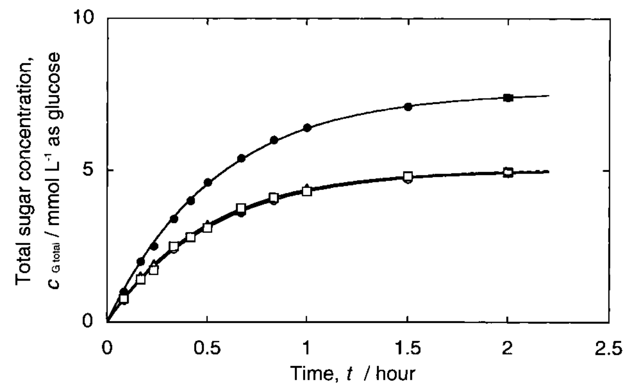

Figure 8 Effect of dissolved gases on cotton desizing with Termamyl at 25°C. (—●—) Deaerated water, (—○—) air-present water, (—△—) O_2-saturated water, (—□—) N_2-saturated water.

experiments by using deaerated water, air-present water, N_2-saturated water, and O_2-saturated water. The treatment bath solution was kept at a pH of 6.3.

The results are shown in Figure 7. All the sugar concentrations in treatment bath are reduced to G1 concentration; total sugar concentration, $c_{G\,total}$, was calculated as follows: $c_{G\,total} = c_{G1} + 2 \times c_{G2} + 3 \times c_{G3} + 4 \times c_{G4} + 5 \times c_{G5}$. Total sugar concentration, $c_{G\,total}$, was plotted against treatment time, t.

As $c_{G\,total}$ shows simple saturation curve, apparent first-order kinetic parameters are calculated and listed in Table V.

$c_{G\,total\,max}/c_{GA\,total\,max}$ of deaerated water is 143%. $c_{G\,total\,max}$ in N_2-gas-saturated water and in O_2-gas-saturated water are the same as $c_{GA\,total\,max}$. The values of k for each water are similar.

The results of cotton desizing with Termamyl are shown in Figure 8 and Table VI. $c_{G\,total\,max}$ in N_2-gas-saturated water and O_2-gas-saturated water are the same as $c_{GA\,total\,max}$ in air-present water, whereas $c_{G\,total\,max}/c_{GA\,total\,max}$ was 152 ± 2%.

From the above results, we can conclude that the efficiency of cotton desizing is affected by the presence or absence of dissolved gases in water, regardless of the varieties of dissolved gas.

With these experiments using Aquazime and Termamyl, rate constants k had similar values in deaerated water, air-present water, O_2-saturated water, and N_2-saturated water. This suggests that both Aquazime and Termamyl were not affected by dissolved gases such as inactivation by dissolved oxygen.

The greater value of the maximum concentration of the total sugar, $c_{G\,total\,max}$, in deaerated water than in air-present water is thought to be caused by the acceleration of the swelling of substrates in deaerated water.

The maximum concentration of total sugar should be the same value in deaerated water and

Table V Effects of Dissolved Gases on Cotton Desizing with Aquazime at 25°C

	Maximum Total Glucose Concentration		First-Order Rate Constant	
Water	$c_{G\,total\,max}$ (mmol L^{-1})	$c_{G\,total\,max}/c_{GA\,total\,max}$ [a]	k (min^{-1})	k/k_A [b]
Air present	3.376 ± 0.017	1	0.022 ± 0.000	1
O_2 saturated	3.301 ± 0.085	0.98 ± 0.03	0.023 ± 0.003	1.02 ± 0.13
N_2 saturated	3.465 ± 0.100	1.03 ± 0.03	0.024 ± 0.002	1.06 ± 0.11
Deaerated	4.824 ± 0.099	1.43 ± 0.03	0.022 ± 0.002	0.99 ± 0.07

[a] $c_{GA\,total\,max}$ is $c_{G\,total\,max}$ in air present water.
[b] k_A is k in air present water.

Table VI Effects of Dissolved Gases on Cotton Desizing with Termamyl at 25°C

	Maximum Total Glucose Concentration		First-Order Rate Constant	
Water	$c_{G\ total\ max}$ (mmol L^{-1})	$c_{G\ total\ max}/c_{GA\ total\ max}$ [a]	k (min^{-1})	k/k_A [b]
Air present	5.015 ± 0.036	1	0.032 ± 0.001	1
O_2 saturated	5.033 ± 0.064	1.00 ± 0.01	0.034 ± 0.000	1.04 ± 0.03
N_2 saturated	5.073 ± 0.113	1.01 ± 0.02	0.032 ± 0.001	0.99 ± 0.03
Deaerated	7.602 ± 0.075	1.52 ± 0.02	0.031 ± 0.001	0.94 ± 0.04

[a] $c_{GA\ total\ max}$ is $c_{G\ total\ max}$ in air present water.
[b] k_A is k in air present water.

in air-present water or with Aquazime and Termamyl, because the content of starch on the fabric was the same. These results, however, showed different values for Aquazime and Termamyl, or in deaerated water and other water. Assuming that the swelling process has a metastable state, the state in deaerated water is thought to be differ from that in air-present water. Namely, it is thought that starch is decomposed to clusterlike masses of starch of different sizes, and the clusters swell individually.

The ratio of the maximum change of total volume, Δv_{max}, of cellulose in deaerated water to that in air-present water is 1.42, as shown in Table I. The ratio of the maximum concentration of total sugar, $c_{G\ total\ max}$, in deaerated water to that in air-present water is 1.43 for Aquazime, as shown in Table III, and 1.52 for Termamyl, as shown in Table IV. These values are very similar. It is suggested that cotton fabric in cotton desizing is swollen in a similar state as cellulose. On the other hand, the ratio of Δv_{max} in deaerated water to air-present water of amylose was 2.29 in Table II. This value is different from the $c_{G\ total\text{-}max}$ ratio of 1.43 for Aquazime and 1.52 for Termamyl, as shown in Table III and IV. This suggests that the swelling state of amylose is different from the swelling of cotton fabric. From these results, we concluded that the higher effect of desizing in deaerated water than in air-present water is caused mainly by the accelerated swelling of cotton fabric in deaerated water, and not the swelling of the starch.

The different $c_{G\ total\ max}$ with Aquazime and Termamyl, however, are thought to be caused by the difference of decomposition from starch to sugars with these two enzymes. Those were not detectable by HPLC measurement used in this experiment.

CONCLUSIONS

The effect of deaerated water on the swelling of cellulose and amylose and on the application in cotton desizing with enzyme which necessarily contains these swelling processes were studied. The results are as follows.

- The hydration of cellulose and amylose is accelerated in deaerated water.
- In cotton desizing with enzyme, higher efficiency of 140–150% was obtained in deaerated water than in water containing dissolved gases of air, oxygen, or nitrogen.
- The efficiency of deaerated water was caused by the higher degree of the hydration of cellulose.

In the textile finishing processes, the swelling of fiber and substrate is necessarily required. For swelling that is more accelerated in deaerated water than in air-present water, deaerated water has a variety of uses in textile finishing.

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