Application of Urea/H₂O₂ Activation-Oxidation System in Degradation of PVA and Desizing of Polyester/Cotton Fabric

Juchuan Shan,^{1,2} Yu Guan,³ Qingkang Zheng,² Jinshi Han,² Qingshu Liu,² Zhongyao Pu⁴

¹College of Polytechnic, Hebei University of Science and Technology, Shijiazhuang, Hebei 050018, China ²College of Light Industry, Textile and Food Engineering, Sichuan University Chengdu, Sichuan 610065, China ³Bureau of Quartermaster Military Representation of the General Logistics Department, Chengdu 610015, China ⁴Sichuan Textile Industry Research Institute, Chengdu 213164, China

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ABSTRACT: The viscosity and the UV–vis spectrum of PVA degradation aqueous solutions, the FTIR, and DSC spectrums of degradation products were measured to investigate and compare the effect of Fenton's reagent and urea/ H_2O_2 activate oxidation systems on the degradation of PVA. The results showed that the viscosity of PVA aqueous solution decreased and the degradation rate of PVA increased with the concentration of hydrogen peroxide in both activate oxidation systems. The FTIR and DSC spectrum of degradation products showed that the degradation products showed that the degradation products showed that the degradation products contained aldehyde, ketone groups. The aldehyde compounds and car-

INTRODUCTION

Polyvinyl alcohol (PVA) as a water-soluble polymer plays a significant role in many industrial applications, such as use as a sizing agent, an emulsifier, a colloid stabilizer, a coating agent, and an adhesive in the textile industry and in the building industry due to its high tensile strength, water solubility, and good film-forming property.^{1–3} PVA is very difficult to degrade biologically because its BOD/COD is only 0.064. The PVA in wastewater produced from the desizing processes in the textile industry can cause many ecological problems. For example, it still needs to be solved as to how to mobilize heavy metals from sediments in water streams and lakes. Furthermore, the foam produced during the treatment of PVA-rich wastewater in biological reactors makes the treatment very difficult, or even in some cases impossible, and results in the operation of PVA biological processes unsteadily as well as obtaining an unsatisfactory result.⁴ Size takes up to 70% of the compounds in the liquid effluents of textile industry.⁵ PVA can be biologically degraded by some specially adapted bacteria strains,⁶⁻¹⁰ such as Pseudomonas spp.¹¹ However, the degradation activity of

bon dioxide produced in the degradation systems were validated by Fehling reagents and clarifying limewater. When the urea/ H_2O_2 system was applied in the one-bath and one-step desizing and scouring process of polyester/cotton 65/35 fabric, the results of 94.7% degradation rate of PVA, the 99.0% desizing rate of PVA, the whiteness, and wicking height of the fabric were obtained. © 2009 Wiley Periodicals, Inc. J Appl Polym Sci 113: 860–867, 2009

Key words: degradation; polyester/cotton fabrics; activation-oxidation system; PVA

microorganisms in industrial waste water treatment facilities is usually limited because the other compounds in the effluent can negatively affect the process.¹² The traditional desizing methods, such as alkalinity desizing, acid desizing and hot water desizing, can partly remove the PVA on fabric. However, PVA cannot be degraded during these desizing processes; the high viscosity of the PVA waste water will make PVA difficult to be removed from fabric, and large quantities of hot water are needed to rinse the desized fabric. Some other desizing methods, such as ultrasonic desizing,¹³ plasma desizing,^{14–16} and supercritical carbon dioxide deszing,^{17–19} were already investigated, but these methods have disadvantages such as high costs, need for special hermetical equipment, and so on.

Activation-oxidation processes are the processes that hydroxyl radicals generated by activation hydrogen peroxide to oxidize majority complex chemicals in wastewater. Hydroxyl radical is a powerful oxidizing reagent with an oxidation potential of 2.33 V and exhibits a faster oxidation reaction rate compared with other conventional oxidants such as hydrogen peroxide.

Fenton's reagent is a mixture of H_2O_2 and ferrous iron. The ferrous iron can initiate and catalyses the decomposition of H_2O_2 to generate hydroxyl radical. The ability for oxidation of hydroxyl radicals is extremely strong; its electrode potential is 2.80 V

Correspondence to: Q. Zheng (shanjuchuan@126.com).

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and is only lower than that of fluoride in the oxidants. The high electronegativity and electron affinity energy (569.3 kJ) of \cdot OH make it easy to attack the materials with a high-density electronic cloud and has a high selectivity.^{20–23}

Fenton's reagent is widely applied in the treatment of wastewater containing PVA in recent years. However, iron ions may cause yellowness of fabric and affect the followed process of fabric when Fenton's reagent is used to desize the fabric. For these disadvantages, Fenton's reagent is difficult to apply in fabric desizing. Urea has the advantages of being inexpensive, without color, easy to obtained and degrade, so it was employed to reactivate H_2O_2 in the current experiment to investigate the effect of the urea/ H_2O_2 reactive oxidation system on the degradation of PVA.

In the acidic condition, hydrogen peroxide can decompose into hydroxyl free radicals in the presence of urea, and the hydroxyl free radicals will further promote hydrogen peroxide to decompose and yield peroxide hydroxyl-free radical, water, and oxygen.

$$\begin{array}{c} O & O \\ H_2N - \overrightarrow{C} - NH_2 + H_2O_2 \longrightarrow H_2N - \overrightarrow{C} - NH_2 + 2 \cdot OH \\ H_2O_2 + \cdot OH \longrightarrow H_2O + HO_2 \cdot \\ HO_2 \cdot + H_2O_2 \longrightarrow \cdot OH + H_2O + O_2 \\ RH + \cdot OH \longrightarrow R \cdot + H_2O \end{array}$$

RH is the organic matter.

When the pH value is higher than 10, hydrogen peroxide mainly generates HOO⁻. HOO⁻ and the combination of urea with hydrogen peroxide can accelerate the decomposition of hydrogen peroxide. The reaction formula is as follows:

$$\begin{array}{c} H_2O_2 + OH^- \longrightarrow HOO^- + H^+ \\ O \\ H_2N - C - NH_2 + HOO^- \longrightarrow H_2N - C - NH_2 \cdots HOO \\ H_2O_2 + OH^- \longrightarrow HOO^- + H_2O \end{array}$$

It would reduce environment pollution if PVA could be degraded into some innocuous and biodegradable materials during fabric desizing by Fenton's reagent and urea/ H_2O_2 systems. This paper was aimed at investigating the effect of Fenton's reagent and urea/ H_2O_2 oxidation reactive systems on PVA degradation. The urea/ H_2O_2 system was used in a one-bath, one-step desizing and scouring process of polyester/cotton fabric. For this purpose, the viscosity and UV–vis spectrum of the PVA degradation aqueous solution and FTIR and DSC spectrums of the degradation product were measured. The degradation rate of PVA in desizing waste water, the desizing rate of PVA on the fabric, the strength, wicking height, and whiteness of the desized fabric were also measured, and the $urea/H_2O_2$ system described was discussed.

EXPERIMENTAL

Materials

Boric acid, iodine, potassium iodide, hydrogen peroxide (30%), PVA1799, ferrous sulfate, sodium hydroxide, calcium hydroxide, sulfuric acid, potassium sodium tartrate, polyacrylamide (M_n , 3 × 10⁶). and urea were AR grade regents. Cornstarch was commercial product. Potassium permanganate and cupric sulfate pentahydrate were CP grade.

Sample preparation of PVA degradation solution

Fenton's reagent degradation system: The PVA aqueous solution contained PVA 1 g/L (2 g/L when the viscosity was measured), H_2O_2 0.8 mL/L, and the FeSO₄ was added according to the molar ratio 10 : 1 of H_2O_2 /FeSO₄ into the PVA aqueous solution, then the aqueous solution reacted 25 min under the condition of 70°C and pH value 3.5.

Urea/ H_2O_2 degradation system: The PVA aqueous solution contained PVA 1 g/L (2 g/L when the viscosity was measured), H_2O_2 20 mL/L, and the urea was added according to the ratio 1 : 1.875 of H_2O_2 (ml/L)/urea (g/L) into the PVA aqueous solution, then the aqueous solution reacted 30 min under the condition of 90°C and pH value 5.

Methods of measurement

Measurement of viscosity

The conventional dilution-type Ubbelohde viscometer (Chengdu, China) with capillary length of 110.0 mm and diameter of 0.50 mm was used. A stopwatch (Chengdu, China) with an accuracy of 0.01 s was used manually to measure the flow time of the solution. All measurements were performed in a water bath (25°C \pm 0.01°C). The flow time of pure water (t_0) or aqueous solution of degraded PVA (t_i) was measured by using a thoroughly cleaned viscometer with a solvent and dried. For the purpose of accuracy, at least three parallel determinations for each concentration were done. Relative viscosity η_r was calculated by $\eta_r = \eta_i/\eta_0 = t_i/t_0.^{24}$

Measurement of PVA degradation rate

Quantitative determination of PVA concentration in the solution was according to the method procedure described by Finley.²⁵ The degradation rate of PVA in aqueous solution or desizing waste water was calculated by:

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Rate degradation of PVA (%) =
$$\frac{C - D}{C} \times 100$$

where C is the concentration of PVA in aqueous solution before degradation and D is the concentration of PVA in aqueous solution after degradation.

The desizing rate of PVA on the fabric was calculated by:

Rate desizing of PVA (%) =
$$\frac{A - B}{A} \times 100$$

where A is the concentration of PVA on the fabric before desizing and B is the concentration of PVA on the desized fabric.

Measurement of whiteness, wicking height, and strength

The whiteness of the fabric was measured according to AATCC 110-2000; wicking height of the fabric was measured according to FZ/T01071-1999; and strength of the fabric was measured according to ISO13934.1/2-1999.

UV spectral analysis

The UV spectrum of the PVA before and after degradation was obtained by the spectrophotometer (UV760ultraviolet–visible light spectrophotometer, Shanghai Precision Scientific Instrument, Shanghai, China). The scanning range was from 500 nm to 200 nm.

FTIR spectrum analysis

Sample preparation: The PVA aqueous solution contained PVA 50 g/L, H_2O_2 40 mL/L, and the FeSO₄ was added according to the molar ratio 10 : 1 of $H_2O_2/$ FeSO₄ into the PVA aqueous solution, then the aqueous solution reacted 25 min under the condition of 70°C, pH value of 3. After the end of the reaction, the pH value of PVA aqueous degradation solution was adjusted to 10 and was centrifuged to remove the deposited ferric hydroxide. The upper aqueous solution was dried under vacuum conditions to remove water, and a solid powder sample was obtained.

The PVA aqueous solution contained PVA 4 g/L, H_2O_2 40 mL/L, and the urea was added according to the ratio 1 : 1.875 of H_2O_2 (mL/L)/urea (g/L) into the PVA aqueous solution, then the aqueous solution reacted 30 min under the condition of 90°C, pH value 5. After the end of the reaction, the PVA aqueous degradation solution was dried under vacuum conditions, and a solid powder sample was obtained. The sample was then purified by methanol.

The samples were ground with KBr. The powdered mixture was pressed at 20 MPa to form a uniform disk. The IR spectra were received by the Tensor 27 infrared spectrometer (Bruker, German).

Differential scanning calorimeter spectrum analysis

Sample preparation was the same as sample preparation in FTIR spectrum analysis.

The sample was measured in the form of powder. The DSC curve was obtained by the thermal analyzer (model: CDR-4P Shanghai Precision Instrument, Shanghai, China) with a heating rate 10°C/min, the compensation differential thermal units range was 40 mW, and scanning temperature ranged from room temperature to 600°C, under the normal atmosphere.

One-step, one-bath desizing/scouring/ bleaching process of the fabric

The composition of the padding bath was hydrogen peroxide 60 mL/L; the ratio of H_2O_2 (ml/L)/urea (g/L) was 1 : 0.6; oil release agent 3 g/L, scouring agent 12 g/L.

Process condition of desizing, scouring, and bleaching for the polyester/cotton fabric: padding liquid at room temperature (two padding and two rolling), the pH value of paddling bath was 5. The mangle expression was 100%) \rightarrow steaming (100°C × 60 min) \rightarrow hot water washing three times (80–85°C) \rightarrow cold water washing twice \rightarrow dry.

RESULTS AND DISCUSSION

Effect of hydrogen peroxide concentration on the viscosity of PVA aqueous solution and the degradation rate of PVA

When the same solvent and measurement condition was employed, the viscosity of polymer aqueous solution mainly reflected an internal friction of macromolecules in the solution. The greater the viscosity of polymer solution is, the higher is the molecular weight of polymer.

Curves in Figures 1 and 2 show that when Fenton's system was used to degrade PVA, the relative viscosity of PVA aqueous solution was decreased, with the concentration of hydrogen peroxide increasing. The reason may be that with the increase of H_2O_2 , the more H_2O_2 could be activated by Fe^{2+} to produce hydroxyl radicals that could react with PVA and more PVA macromolecules would be broken into small molecules. The viscosity of degraded PVA aqueous solution dropped to a low point when the concentration of hydrogen peroxide was 0.8 mL/ L. After this point, the viscosity of degraded PVA aqueous solution with the concentration of hydrogen peroxide increasing tended to a constant value. As the same reason, when $urea/H_2O_2$ activation oxidation system was used to degrade PVA, the viscosity of degraded PVA aqueous solution was also decreased, with the concentration of hydrogen



Figure 1 Effect of H_2O_2 concentration in Fenton's reagent system on the viscosity of PVA aqueous solution.

peroxide increasing. Before the concentration of H_2O_2 2 mL/L, the viscosity of the degraded PVA aqueous solution dropped rapidly, which showed that the urea also could activate H_2O_2 to degrade PVA. After the concentration of H_2O_2 2 mL/L, the viscosity of the degraded PVA aqueous solution tended to decline slowly with the concentration of hydrogen peroxide increasing.

As shown in the curves in Figures 3 and 4, the degradation rate of PVA was increased with the concentration of hydrogen peroxide increasing in both Fenton's reagent and urea/ H_2O_2 systems. When the concentration of hydrogen peroxide was 0.4 mL/L in Fenton's reagent activation oxidation system, the degradation rate of PVA nearly arrived 100%. It suggested that Fenton's reagent was an excellent reagent to degrade PVA. When the concentration of hydrogen peroxide was 20 mL/L in the urea/ H_2O_2 activation



Figure 2 Effect of H_2O_2 concentration in the urea/ H_2O_2 system on the viscosity of PVA aqueous solution.



Figure 3 Effect of H_2O_2 concentration in Fenton's reagent system on the degradation rate of PVA in Fenton.

oxidation system, the 100% degradation rate of the PVA could also be obtained. The ability of the urea/ H_2O_2 activation oxidation system to degrade PVA seems lower than that of Fenton's reagent.

Possible chemical structure characterization of degraded PVA products

UV–vis spectra of PVA, urea, and the degraded PVA aqueous solution are shown in Figures 5 and 6. When Fenton's reagent system was used, the absorption peak of degraded PVA aqueous solution became strong, with hydrogen peroxide increasing at the greatest absorption wavelength. After degradation, a medium-intensity and symmetrical peak appeared at the absorption band λ_{max} 250–300 nm on the curve, and this absorption peak was coursed by the $n \rightarrow \pi^*$ transition in some compounds, which



Figure 4 Effect of H_2O_2 concentration in the Urea/ H_2O_2 system on the degradation rate of PVA.

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Figure 5 The UV–vis spectra of PVA aqueous degradation solution in Fenton's reagent system.

indicated that the degraded products may contain aldehyde-carbonyl group or conjugate carbonyl group. When the urea/H₂O₂ system was used, the absorption peak of the degraded PVA aqueous solution appeared at the absorption band $\lambda_{max} < 220$ nm, which resulted from $n \to \sigma^*$ and $n \to \pi^*$ transition in some compounds. The result showed that the degraded products might contain alcohol, amine, or conjugate groups. The absorption peak of the degraded PVA aqueous solution was much higher than that of pure urea aqueous solution near the λ_{max} 200 nm. Therefore, the PVA macromolecules could also be broken into low molecular weight materials, such as some small molecular alcohols, polyalcohols, or conjugate group materials, when the Fenton's reagent system was used.

Compared with that of the PVA aqueous solution degraded by Fenton's reagent, no absorption peak appeared at 275 nm on the UV–vis spectra curves of PVA aqueous solutions degraded by the urea/ H_2O_2 system. It suggested that the degradation mechanism of PVA would be different when Fenton's reagent and the urea/ H_2O_2 system were used. The result of

Fe²⁺ + H₂O₂ → •OH

·OH + $PVA_{1799} \longrightarrow -CH_2COOH +$

·OH + $PVA_{1799} \longrightarrow -CH_2COOH +$

— CH₂CHO +

Fenton's system

·CH₂CHO +

Urea/H2O2 system



Fehling reagent and the clarifying limewater verified test showed that PVA was degraded to give out aldehyde compounds and carbon dioxide in both degradation systems. However, the amount of aldehyde compounds and carbon dioxide produced in Fenton's reagent system was more than that in the urea/H₂O₂ system. It may be that the amount of aldehyde compounds in the degraded products when the urea/H₂O₂ system was used to degrade PVA was less than that in the degraded products when Fenton's reagent system was used. It also may be that the aldehyde compounds in the degraded products would form hydrogen bonds with small molecular alcohols or polyalcohols in the urea/H₂O₂ system.

In Fenton's system, PVA was degraded to alcohol, aldehyde, ketone, and acid that was of small molecular weight. However, in the urea/ H_2O_2 system, in addition to these products, there was possibly incompletely degraded polyvinyl alcohol. The reaction mechanism showed that the ability of degradation of PVA by hydroxyl free radicals in Fenton's system was higher than that in the urea/ H_2O_2 system.

n << 1700

Г ́ ОН

+ - C-|| 0



Urea + H₂O₂ → ·OH







wave number/cm⁻¹

Figure 7 FTIR spectra of degraded products (a) FTIR spectra of PVA1799; (b) FTIR spectra of PVA degradation product obtained by the urea/ H_2O_2 system; (c) FTIR spectra of PVA degradation product obtained by the Fenton's reagent system.

The infrared spectrum of PVA1799 in Figure 7 shows a broad peak around 3400 cm⁻¹, indicating stretching of hydroxyl groups and peaks at 2930 cm⁻¹ corresponds to C–H stretching. The band for C-O stretching and O-H bending was observed at 1082 cm⁻¹. The corresponding bending and wagging of CH₂ vibrations and C-H wagging are around 1431 cm⁻¹. The infrared spectra curves of degraded products obtained by the urea/H2O2 system and Fenton's reagent system were similar. The characteristic peak near the 3400 cm⁻¹ of the degraded PVA product became wider and weaker than that of PVA, which indicated that the molecule chain of PVA was broken into different molecular weights and different alcohol structures. Because the PVA molecule chain was broken and the amount of -CH₂- reduced in the degraded products, the peaks of the degraded PVA products near the 2930 and 1431 cm⁻¹ on the infrared spectrum were weaker than that of PVA. The new characteristic peak of the PVA degradation products appeared at 1620–1750 cm⁻¹ on the infrared spectrum in Figure 7. This indicated that degradation products contained some aldehyde, ketone, carboxy groups, and the hydroxyl group absorbance peak of PVA at the 1081 cm⁻¹ was greatly weakened, which indicated that part of PVA was oxidized into aldehyde, ketone materials, and carboxylic acids by the both activation oxidation systems.

Thermal properties of degraded PVA products

The curve (c) in Figure 8 shows hat PVA had two endothermic peaks at 225.8 and 263.1°C, which were

the peaks of melting of PVA and conjugated double bonds formed by dehydration of PVA intermolecular separately. After 263.1°C, PVA was continually decomposed with the temperature increasing and gave out a high exothermic peak at 520°C on the curve (c).

Compared with the DSC curve (c) of PVA, the DSC curve (b) of PVA degradation product showed a newly wide endothermic peak at 111.9°C and no the dehydration peak of PVA intermolecular at 263.1°C, which suggests that PVA had degraded by Fenton's reagent into some small molecule materials such as alcohol, acid, aldehyde, and ketone materials, and the melting peak at 225.8°C as well as the dehydration peak of PVA intermolecular at 263.1°C would not appear on the DSC curve (b) of PVA degradation product. The exothermic peak on the curve (b) of the degraded PVA product appeared from nearly 320 to 550°C, which was earlier and wider than that on the curve (c) of PVA. It might be that the small molecules in the degraded PVA products could release more heat easily when they were oxidized than did PVA. From the above results, it could be inferred that PVA was not often contained in the degradation product.

When the urea/ H_2O_2 system was used to degrade PVA, the DSC curve (a) of the degraded PVA products also gave out no the melting peak at 225.8°C and the dehydration peak of PVA intermolecular at 263.1°C The new endothermic peak at 185°C on the curve (a) of the degraded PVA indicated that the degraded products were not the original PVA. The exothermic peak on the curve (a) of the degraded PVA products appeared from nearly 380°C to 580°C, which was also earlier and wider than that on the curve (c) of PVA. All of these showed that the urea/ H_2O_2 system also could degrade PVA the same as





| Results of Polyester/Cotton (65/35) Fabric Treatment by the Urea/H ₂ O ₂ System | | | | | |
|---|-----------------------------|---|-------------------|------------------------------------|-----------------------------------|
| | Desizing rate of PVA (%) | Degradation rate of PVA in desizing aqueous solution (%) | Whiteness (Wh) | Capillary effect (cm/30 min) | Strength of fabric warp (N) |
| Treated Untreated | 99.0 | 94.7 | 86.29 84.33 | 13.2 0 | 1,565 1,484 |

 TABLE I

 Results of Polyester/Cotton (65/35) Fabric Treatment by the Urea/H₂O₂ System

The strength data of warp was obtained under the condition of materials: liquid 1 : 30, the concentration of NaOH 1 g/L, and boiling 1 h.

the Fenton's reagent system. However, the endothermic peak and the exothermic peak on the curve (b) of degradation products of PVA degraded by Fenton's reagent system appeared earlier than the endothermic peak and the exothermic on the curve (a) of degradation products of PVA degraded by the urea/ H_2O_2 system, which may contribute to that the ability of Fenton's reagent to degrade PVA as higher than that of the urea/ H_2O_2 system. The molecule weight of degradation products was smaller by Fenton's reagent system was used to degrade PVA than by the urea/ H_2O_2 system.

Application of the urea/ H_2O_2 system in desizing, scouring, and bleaching process of polyester/cotton fabric

Fenton's reagent could be used in the desizing process of fabric and to degrade PVA effectively, but the iron ion deposited on fabric decreased the whiteness of fabric and led to serious loss of textile strength. The urea had a good solubility, without color, was easily washed out, and did not deposit on fabric to affect the processes of fabric when it was used as the reactivate of H_2O_2 .

The urea/ H_2O_2 system was applied in the onebath, one-step desizing, scouring process of polyester/cotton 65/35 fabric. After the desizing, scouring process of polyester/cotton, the desizing rate, the capillary effect, the strength and the whiteness of the fabric, and the degradation rate of PVA in the desizing aqueous solution were measured and the data were listed in Table I.

From the data in the Table I, it could be found that the desizing rate of the fabric and degradation rate of PVA in desizing waste water were 99% and 94.7% separately, the whiteness of fabric increased from 84.33 to 86.29, and the capillary effect of the fabric increased from 0 to 13.2 cm/30 min, which indicated that it was possible to degrade the PVA on fabric and in desizing liquid at the same times by using the urea/ H_2O_2 system in fabric desizing process.

CONCLUSIONS

The urea/ H_2O_2 activation oxidation system could degrade PVA effectively. The viscosity of PVA degradation solution and the rate of degradation of PVA in the degradation solution were affected by hydrogen peroxide concentration in the urea/ H_2O_2 activation oxidation system. The UV–vis, FTIR, and DSC spectrum of the PVA degradation products degraded by the urea/ H_2O_2 activation oxidation system showed that the degradation products were aldehyde, ketone materials, carbon dioxide, and some small polyalcohols.

After the one-bath, one-step desizing, scouring, and bleaching process in the urea/ H_2O_2 activation oxidation system, the PVA desizing rate of the polyester/cotton fabric and degradation rate of PVA in desizing waste water was 99% and 94.7% separately. The treated fabric showed good wicking height and whiteness, which showed that it was possible to use the urea/ H_2O_2 activation oxidation system in the one-bath, one-step desizing, scouring, and bleaching process of fabric.

Compared with Fenton's reagent activation oxidation system, the urea/ H_2O_2 activation oxidation system when used in the one-bath, one-step desizing, scouring process of fabric had the advantages of good solubility, without color, was easily washed out, was easily degraded, and did not deposit on fabric to affect the processes of fabric, besides degrading PVA on the fabric and in desizing waste water in the one-bath, one-step desizing, scouring, and bleaching process of fabric.

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