# The Mechanism and Technology Parameters Optimization of Alkali-$H_2O_2$ One-Bath Cooking and Bleaching of Hemp

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**ABSTRACT:** Hemp is cooked and bleached in alkali-$H_2O_2$ one-bath. The analysis of physical and chemical properties related to acid, alkali, and hydrogen peroxide about various types of components in hemp such as hemicelluloses and pectin, and lignin components makes it ascertained. In this one-bath process, alkali and $H_2O_2$ have cooperative effects to remove most proportions of the noncellulosic components efficiently. The additive $MgSO_4$ and a stabilizer of $H_2O_2$ as auxiliary agents are introduced to protect cellulose from degradation and, meanwhile, they improve the efficiency of $H_2O_2$. The universal rotatable composite experiment design is employed to get the highly precise equation, in which the variables of NaOH and $H_2O_2$ concentrations, and the processing time are considered to be the main technological parameters of this alkali-$H_2O_2$ one-bath process. The residual gum content and residual lignin content of hemp fiber are the evaluation indexes. Based on regression equations, the figures are plotted to analyze the influences of parameters on the evaluation indexes; the optimization multicriteria mathematical model is simultaneously established to obtain the optimum parameters. Finally, an experiment is carried out to further test the acceptance of the calculated optimum values. It is shown that, for the hemp taken from Anhui Province of China, this alkali-$H_2O_2$ one-bath process can substantially remove gum and lignin from raw hemp. NaOH 10.5 g/L, $H_2O_2$ 9.8 g/L, and the processing time of 127 min with 0.1% $MgSO_4$ + 2.7 g/L stabilizer of $H_2O_2$ at 100°C are recommended to be the optimum parameters; then, a compromise is achieved among the lowest lignin content, the residual gum content, and the best strength and whiteness of hemp fiber. © 2005 Wiley Periodicals, Inc. J Appl Polym Sci 97: 2279–2285, 2005

**Key words:** additives; degradation; fibers; processing

## INTRODUCTION

Bast fibers are important fibers because they have biodegradable and ecofriendly characteristics. Efforts to exploit the wide use of bast fibers like hemp, coir, banana, sisal, and jute have been an area of interest.[1–6] Among these, hemp fiber has been undergoing a revival worldwide as a natural source of fibers for its easy availability at low cost—fast growing and not very demanding as to climate, soil quality, and nutrients.[7] As a textile fiber, hemp provides all the warmth and softness of other natural textiles but with a superior durability seldom found in other materials. Hemp blended with other fibers easily incorporates the desirable qualities of mixture of textiles.[8] However, hemp fibers consist of both cellulosic and noncellulosic substances, such as hemicellulose, pectin, lignin, and wax, and these noncellulosic substances affect the spinning, weaving, dyeing, and finishing of hemp fibers, yarns, and fabrics. So, to make good use of hemp fiber, removal of the most noncellulosic components is a prerequisite problem.

Usually, hemp fibers are processed with bacterial, chemical, and enzymatic methods. Bacterial processing depends on the weather, water quality, and other factors and often results in inconsistent fiber quality.[9] Enzymatic processing method was developed for flax in the 1980s,[10–12] but may not be a feasible method for processing hemp fibers. Chemical processing of these fibers is effective for removing noncellulosic substances, and considerable research has focused on this method to improve the quality of fiber.[13–16]

It is known that bleaching or similar treatments improve the appearance of fibers and hydrogen peroxide is often used to bleach the textile fibers.[17–21]

Earlier reports on the vast application of alkali-$H_2O_2$ one-bath process were employed in dyeing and finishing.[22–27] In this article, the principle of alkali-$H_2O_2$ one-bath process of cooking and bleaching of hemp is investigated. The corresponding experiments are carried out to prove that this treatment can remove the noncellulosic substances, especially lignin from the hemp fibers, more effectively than other processes. Additionally, the optimum technological parameters are obtained by the universal rotatable composite experiment design.

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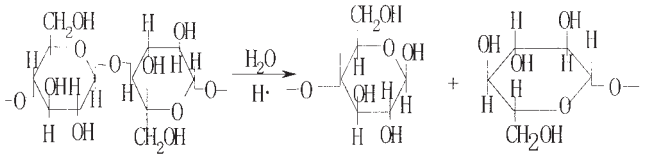
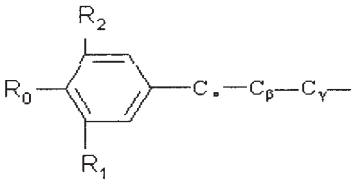


**Figure 1** Acid hydrolysis and degradation of cellulose.



**Scheme 1**

## COMPONENTS OF HEMP

### Cellulose

Cellulose is a polysaccharide and its molecular formula is $(C_6H_{10}O_5)_n$. In the cellulose, $\beta$-*d*-glucose residues join together by 1,4-glucosidic linkage to form the macromolecule.

### Reaction with inorganic acid

When acid solution is applied to cellulose, acid hydrolysis will occur. $\beta$-1,4-Glucosidic bond is an acetal, which is sensitive to the acid. With the suitable concentration of hydrion, time, and temperature, the glucosidic bond can break down, resulting in the decrease of polymerization and strength, and leading to the increase of reducing power. The resultant of part-hydrolysis is named as hydrocellulose and glucose is the end product after complete hydrolysis. The course of reaction is expressed in Figure 1.

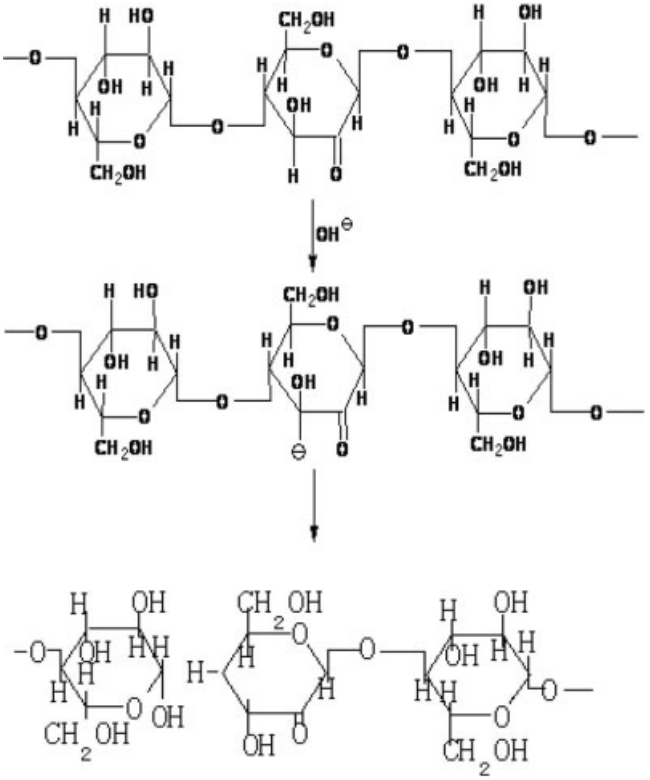


**Figure 2** Oxidative degradation of cellulose caused by $H_2O_2$.

### Physical and chemical reaction with alkali

In alkali solution, luster and elasticity of hemp fiber improve, resulting from the swelling of cellulose. The abilities of swelling related to several alkalis are listed as LiOH > NaOH > KOH > PbOH > CsOH. It can be seen that NaOH has a strong swelling ability of cellulose. The degree of swelling increases with the increase of alkali concentration in the same kind of alkali and at the same temperature. If the concentration keeps increasing, the metal ion concentration will increase. However, when the concentration of ion becomes extremely high, the radius of hydrated ion will decrease and lead to a drop in the degree of swelling.

At room temperature, cellulose is stable to alkali. However, in the boiling process, alkali degradation of cellulose will occur because of the removal of gums at higher temperature.

### Reaction with $H_2O_2$

Cellulose can be oxidized by $H_2O_2$ with the existence of alkali accompanied by a decrease of polymerization by 15%. The reaction is demonstrated in Figure 2.

### Lignin

Lignin is a complex amorphous polymer consisting of propane benzene crosslinked by ether bonds and C—C bonds. The constitutional formula is shown in Scheme 1.

These constitutional units combine to form the lignin macromolecule by various types of bindings. In general, the bindings have two forms. One is ether linkage amounting to two-thirds up to three-fourths.

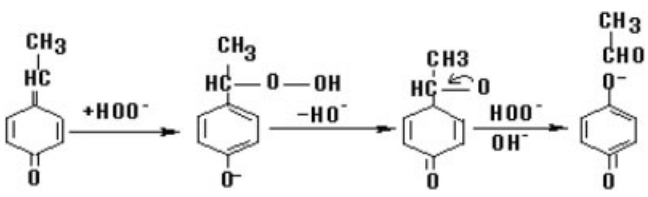


**Figure 3** Reaction of side-chains in lignin structure.

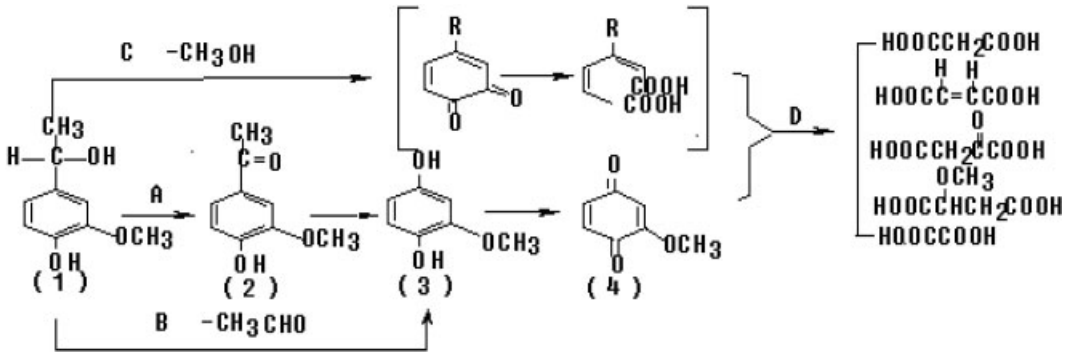



**Figure 4** Reaction of benzene ring and side-chains cracking simultaneously.

Among these ether linkages, $\alpha$-aryl ether, $\alpha$-alkyl ether, $\beta$-aryl ether, and methyl-aryl ether present in a larger proportion and are easy to crack and to be connected with chemical reactions. The other form is C—C binding, which is stable and amounts to one-fourth up to one-third of the bonds.

### Reaction with alkali

Because of the complex structure of lignin, it is stable in strong lye. Practically, cold strong lye in any concentrations from 0.25% up to 2.5% has no effect on the lignin. However, the boiling alkali solution from 0.1% up to 2% can dissolve 20% of lignin.

When lignin reacts with alkali at high temperature, ether linkages such as $\alpha$-aryl ether, $\alpha$-alkyl ether, and $\beta$-aryl ether will break down under the action of nucleophilic reagent $OH^-$, thereby degrading the lignin.

### Reaction with $H_2O_2$

Lignin reacts with $H_2O_2$ in the following three forms:

#### Reaction of benzene ring

The constitutional unit of lignin-benzene ring is colorless, which can be changed into a colored substance during the boiling process. In alkali-$H_2O_2$ one-bath process, $H_2O_2$ will destroy these quinine structures, changing the colored structure to colorless, then disintegrating them into low-molecular aliphatic compound.

#### Reaction of side-chains

When the conjugated double bond presents in the side-chains, it is a colored substance by nature. By $H_2O_2$ treatment, $H_2O_2$ can destroy side-chains, altering the colored conjugated double bonds to colorless, and further disintegrating the side-chains. Nonconjugated double bonds can also be spilt by $H_2O_2$. All these can separate the lignin out from cellulose, as shown in Figure 3.

#### Benzene ring and side-chains cracking simultaneously

Figure 4 shows that the resultant of Reaction A, methoxyl paradioxybenzene, will then be Oxygenized (4). Reaction B can form (3) owing to the stripping of acetaldehyde after oxidation. O-quinone can be produced after Reaction C—the benzene ring is oxygenized and methyl methoxyl appears as methyl alcohol.

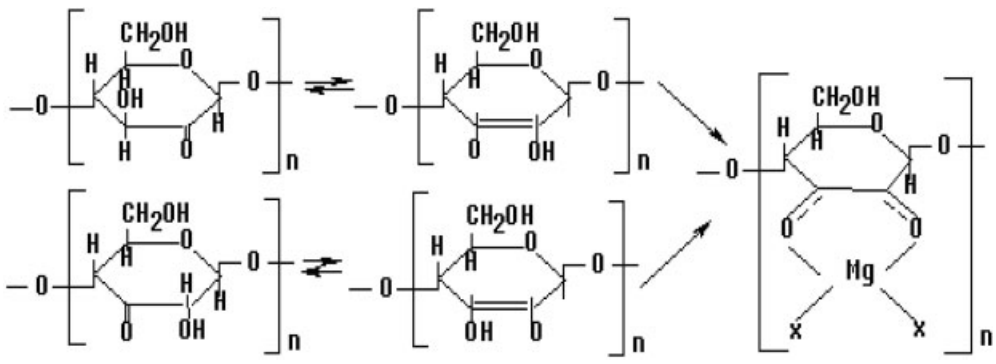



**Figure 5** The coordinate reaction between the cellulose and $Mg^{2+}$.

**TABLE I**
**Levels of the Variables for Alkali- $H_2O_2$ One-Bath Processing**

| Variable | Levels | | | | |
|---|---|---|---|---|---|
| | $-r(-1.682)$ | $-1$ | 0 | $+1$ | $+r(+1.682)$ |
| X1: NaOH Concentration (g/L) | 8 | 8.8 | 10 | 11.2 | 12 |
| X2: $H_2O_2$ Concentration (g/L) | 8 | 8.8 | 10 | 11.2 | 12 |
| X3: Treating Time (min) | 90 | 100 | 120 | 140 | 150 |

Reaction D shows that O-quinone and parabenzoquinone are oxygenized to split and then form a series of lower acid.

### Reaction with oxygen

In the alkaline medium, $H_2O_2$ will decompose to produce oxygen, as seen as $HOOH \rightarrow 2HO\bullet + O_2$. Oxygen is the oxidant due to its two unpaired electrons having strong reaction tendency to organics, occurring as a free-radical reaction.

The reaction between oxygen and lignin is virtually a free-radical reaction because, in the alkaline medium, oxygen can attack C-negatron produced by the auto-oxidation of lignin units of phenolic structure and olefin alcohol forms. During this reaction, $HOO^-$ formed in the auto-oxidation reaction of lignin as a nucleophilic reagent adds to carbonyl and conjugated carbonyl by additive reaction. The initial free-radical reaction between phenolic lignin structure and oxygen can induce $HOO^-$. However, it cannot change the lignin's structure greatly or minimize the macromolecular structure. During further reaction, greater changes will take place, leading to the oxidation of benzene ring or elimination of side-chains.

### Hemicellulose and pectins

Hemicellulose is an amorphous matter in the interfiber and intermicrofibril regions as a cementing and packing constituent, which has a lower degree of polymerization from 80 to 200 and a lower molecular weight. As a result, it is prone to be hydrolyzed by inorganic acid and to be dissolved in the dilute hot alkali solution, particularly to be easily oxygenized by oxidant.

Pectin is a complicated polysaccharide that varies in structure,[28] which has two forms of raw pectin and mature pectin. The latter can be dissolved in boiling water. Although the former is difficult to be dissolved, it is easily hydrolyzed by acid and alkali treatment.

**TABLE II**
**Experimental Results of the Universal Rotatable Composite Experiment Design for Three Variables of One-Bath Processing**

| Experimental combinations | $X_0$ | $X_1$ | $X_2$ | $X_3$ | $X_1X_2$ | $X_1X_3$ | $X_2X_3$ | $X_1^2$ | $X_2^2$ | $X_3^2$ | $Y$ (%) | $Y'$ (%) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 4.27 | 2.74 |
| 2 | 1 | 1 | 1 | −1 | 1 | −1 | −1 | 1 | 1 | 1 | 4.89 | 3.04 |
| 3 | 1 | 1 | −1 | 1 | −1 | 1 | −1 | 1 | 1 | 1 | 4.20 | 3.56 |
| 4 | 1 | 1 | −1 | −1 | −1 | −1 | 1 | 1 | 1 | 1 | 6.18 | 3.61 |
| 5 | 1 | −1 | 1 | 1 | −1 | −1 | 1 | 1 | 1 | 1 | 5.08 | 3.19 |
| 6 | 1 | −1 | 1 | −1 | −1 | 1 | −1 | 1 | 1 | 1 | 5.96 | 3.27 |
| 7 | 1 | −1 | −1 | 1 | 1 | −1 | −1 | 1 | 1 | 1 | 4.65 | 3.89 |
| 8 | 1 | −1 | −1 | −1 | 1 | 1 | 1 | 1 | 1 | 1 | 5.23 | 4.16 |
| 9 | 1 | $-r$ | 0 | 0 | 0 | 0 | 0 | $r^2$ | 0 | 0 | 3.68 | 3.39 |
| 10 | 1 | $r$ | 0 | 0 | 0 | 0 | 0 | $r^2$ | 0 | 0 | 6.28 | 3.47 |
| 11 | 1 | 0 | $-r$ | 0 | 0 | 0 | 0 | 0 | $r^2$ | 0 | 4.96 | 2.45 |
| 12 | 1 | 0 | $r$ | 0 | 0 | 0 | 0 | 0 | $r^2$ | 0 | 4.41 | 4.13 |
| 13 | 1 | 0 | 0 | $-r$ | 0 | 0 | 0 | 0 | 0 | $r^2$ | 4.46 | 3.41 |
| 14 | 1 | 0 | 0 | $r$ | 0 | 0 | 0 | 0 | 0 | $r^2$ | 7.56 | 3.49 |
| 15 | 1 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 4.59 | 3.39 |
| 16 | 1 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 5.32 | 3.43 |
| 17 | 1 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 4.56 | 3.51 |
| 18 | 1 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 4.37 | 3.34 |
| 19 | 1 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 4.7 | 3.38 |
| 20 | 1 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 4.25 | 3.49 |

**TABLE III**
**Regression Equation for Each Index for One-Bath Processing**

Equation model: $Y = b_0 + b_1X_1 + b_2X_2 + b_3X_3 + b_1b_2X_1X_2 + b_1b_3X_1X_3 + b_2b_3X_2X_3 + b_2^1X_1^2 + b_2^2X_2^2 + b_3^2X_3^2$

| Evaluation index | $b_0$ | $b_1$ | $b_2$ | $b_3$ | $b_1b_2$ | $b_1b_3$ | $b_2b_3$ | $b_1^2$ | $b_2^2$ | $b_3^2$ |
|---|---|---|---|---|---|---|---|---|---|---|
| $y$: percent of residual gum content (%) | 4.6383 | 0.2191 | −0.0721 | 0.0845 | −0.2975 | −0.1425 | 0.1325 | 0.0802 | −0.0241 | 0.4444 |
| $y'$: the residual lignin content (%) | 3.4230 | −0.0897 | −0.0257 | −0.0370 | 0.0325 | 0.0175 | 0.0175 | 0.0043 | −0.0099 | 0.0114 |

## THE ADDITIVE—$MgSO_4$

To avoid oxidative degradation of the cellulose, a small amount of additive like $MgSO_4$ is added, which is proven to be a most efficient protecting agent.

One opinion shows that $MgSO_4$ can improve the stability of $H_2O_2$ to alkali. In the alkali solution, $Mg(OH)_2$ can be yielded to disperse in the medium as a colloid. Having positive charges, it is easy to absorb nucleophilic $HOO^-$, preventing $HOO^-$ from reacting with $H_2O_2$ to form free radicals and thereby restraining the decomposition of $H_2O_2$. If a little heavy metal ion exists in the solution, as the form of coordinate ion with negatron, they are also easily absorbed by $Mg(OH)_2$ to lose the catalysis.

Another explanation describes that $Mg^{2+}$ can react with oxycellulose having carbonyl to form a complex with relative stability reducing the cleavage of cellulosidic linkage to protect the cellulose directly. The reaction equation is shown in Figure 5.

## THE OPTIMUM DESIGN OF THE TECHNOLOGY PARAMETERS

### Experimental

Hemp comes from Anhui Province of China. With pretreatment, hemp is immersed in 1 mL/L $H_2SO_4$ at 40–50°C for 60 min, maintaining a bath ratio of 1 : 20. The fibers are then washed with 70°C water several times until the pH is 7. Alkali-$H_2O_2$ one-bath treatment is carried out at 100°C with a bath ratio of 1 : 20 + 0.1% $MgSO_4$ + 2.7 g/L stabilizer of $H_2O_2$, followed by treatment to help remove the residual gums efficiently. The fibers are then washed with sulfuric acid (1 mL/L, Taian Qingqing Hemp Textile Ltd., Taian, China) for 5 min to neutralize the residual NaOH.

NaOH and $H_2O_2$ concentrations and the treating time are considered to be the main technological parameters of this one-bath process. Therefore, when the experiments are arranged through the universal rotatable experiment composite design, they are chosen as the variables, their levels are shown in Table I. The residual gum content and residual lignin content of hemp fibers are chosen as the evaluation indexes.

### Regression analysis

The results of experiments for each evaluation index under the corresponding experimental combination are shown in Table II. The results of regression analysis based on the data in Tables I and II are summarized in Tables III, IV, and V.

**TABLE IV**
**Results of Significance Tests of Regression Equations**

| Evaluation index | Percent of residual gum content | Residual lignin content |
|---|---|---|
| Fit test | $10.326254 < F_{0.01}(5,5) = 11.0$ good | $10.326254 < F_{0.01}(5,5) = 11.0$ good |
| Significance test | $51.673523 > F_{0.01}(9,10) = 4.49$ significant | $132.425761 > F_{0.01}(9,10) = 4.49$ significant |

**TABLE V**
**Results of Significance Tests of Regression Coefficients**

| Evaluation index | | Percent of residual gum content | Residual lignin content |
|---|---|---|---|
| Regression | $b_0$ | $10.3951 > t_{0.001}(10) = 4.587$ | $21.6037 > t_{0.001}(10) = 4.587$ |
| | $b_1$ | $3.0425 > t_{0.1}(10) = 1.812$ | $4.6051 > t_{0.01}(10) = 3.169$ |
| | $b_2$ | 0.2941 | $4.9730 > t_{0.001}(10) = 4.587$ |
| | $b_3$ | 1.3572 | $11.6528 > t_{0.001}(10) = 4.587$ |
| Coefficients | $b_1b_2$ | 0.3876 | 1.0953 |
| | $b_1b_3$ | $2.9634 > t_{0.1}(10) = 1.812$ | $8.0535 > t_{0.001}(10) = 4.587$ |
| | $b_2b_3$ | $2.4163 > t_{0.05}(10) = 2.228$ | $6.287 > t_{0.001}(10) = 4.587$ |
| | $b_1^2$ | $3.1981 > t_{0.01}(10) = 3.169$ | $10.6042 > t_{0.001}(10) = 4.587$ |
| | $b_2^2$ | $2.3904 > t_{0.05}(10) = 2.228$ | 1.2567 |
| | $b_3^2$ | $2.5093 > t_{0.05}(10) = 2.228$ | $3.6235 > t_{0.01}(10) = 3.169$ |

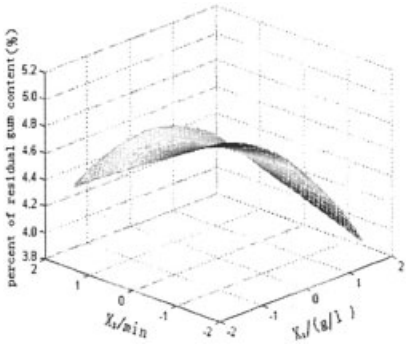



**Figure 6** Effects of NaOH concentration and treating time on residual gum content.

## RESULTS AND DISCUSSION

Based on the valid regression equations shown in Table III, three-dimensional pictures concerning the corresponding evaluation indexes have been plotted. These pictures provide information on changes of the indexes with percent of residual gum content and residual lignin content.

### Percent of residual gum content

As can be observed in Figures 6-8, when NaOH concentration increases, there is an evident tendency for the residual gum content to be reduced. It means that NaOH concentration has a significant effect on residual gum level. Within a certain range, the percent of the residual gum content dramatically decreases with the increase of $H_2O_2$ concentration. However, when the $H_2O_2$ concentration exceeds a certain range, the percent of residual gum content has a tendency to increase. It shows that a higher $H_2O_2$ concentration is unnecessary for removing lignin (as shown in Figs. 7 and 8). Figures 6 and 8 show that a long treating time can reduce the gum content, but this result is no longer significant compared with NaOH concentration. When it exceeds a certain value, there is only a minor effect on the residual gum content.

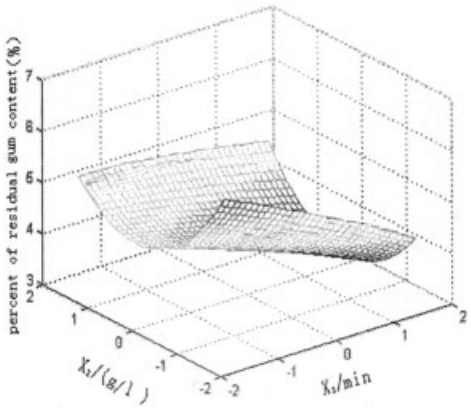



**Figure 8** Effects of $H_2O_2$ concentration and treating time on residual gum content.

### Residual lignin content

As shown in Figures 9-11, the residual lignin content has a tendency to decrease with the increase of NaOH

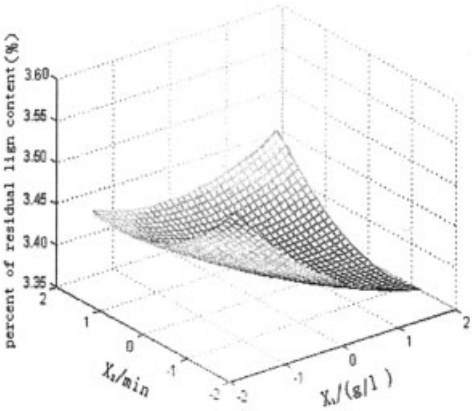



**Figure 9** Effect of NaOH concentration and treating time on residual lignin content.

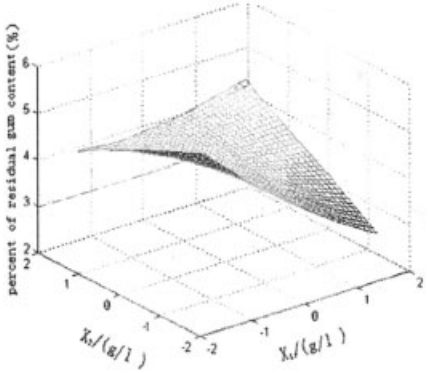



**Figure 7** Effects of NaOH and $H_2O_2$ concentration on residual gum content.

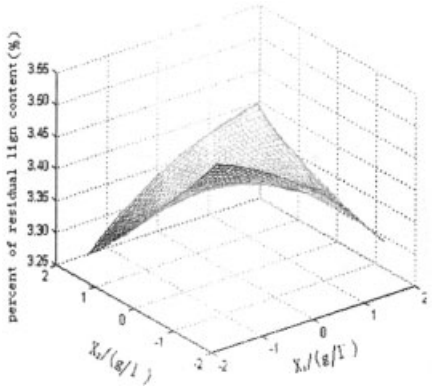



**Figure 10** Effect of NaOH and $H_2O_2$ concentration on residual lignin content.

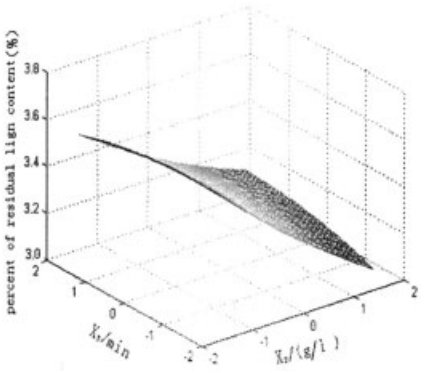



**Figure 11** Effect of $H_2O_2$ concentration and treating time on residual lignin content.

concentration and the treating time. However, with the increase of these two variables, the decreasing tendency of residual lignin content becomes smaller. Although, with the increase of $H_2O_2$ concentration, the residual lignin content dramatically decreases, which signifies that $H_2O_2$ plays a critical role of removing the lignin, NaOH and treating time can assist $H_2O_2$ to remove lignin. Increasing NaOH concentration or prolonging the treating time does not remove the lignin substantially.

### Determination of optimum technological parameters

The valid regression equations in Table III can be used to establish a multicriteria optimization mathematical model. In this study, one regression equation concerning the percent of residual gum content is considered to be objective function, while the other valid regression equation is regarded as constraint functions, in such a manner to establish the optimization model. With the aid of Object Programming Approach and Constrained Random Ray Method, the solution to the model is obtained [i.e., $X_1$ (NaOH concentration) = 10.531 g/L, $X_2$ ($H_2O_2$ concentration) = 9.765 g/L, $X_3$ (treating time) = 127.249 min].

Therefore, NaOH 10.5 g/L, $H_2O_2$ 9.8 g/L, and the processing time of 127 min with 0.1% $MgSO_4$ + 2.7 g/L stabilizer of $H_2O_2$ at 100°C are recommended to be the optimum parameters of alkali-$H_2O_2$ one-bath cooking and bleaching of hemp.

## CONCLUSIONS

Due to the different accessibilities to acid, alkali, and $H_2O_2$ among hemicellulose, lignin, and pectin of hemp, NaOH and $H_2O_2$ can work simultaneously to remove these noncellulosic substances substantially. During alkali-$H_2O_2$ one-bath treatment, alkali not only removes most of the hemicelluloses, pectin, and lignin, but also provides $H_2O_2$ an alkaline environment to optimize the action of $H_2O_2$ and to remove lignin more efficiently. The additive $MgSO_4$ and a stabilizer of $H_2O_2$ are added to prevent cellulose from oxidation. NaOH 10.5 g/L, $H_2O_2$ 9.8 g/L, and the processing time of 127 min with 0.1% $MgSO_4$ + 2.7 g/L stabilizer of $H_2O_2$ at 100°C are recommended to be the optimum parameters; then, a compromise is achieved among the lowest lignin content, the residual gum content, and the best strength and whiteness of hemp fiber.

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