Physical Strength Improvement of *Eucalyptus* Alkaline Hydrogen Peroxide Mechanical Pulp by Low-temperature Plasma Treatment

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Low-temperature plasma treatment technology is an efficient and environmentally friendly surface treatment technology that has been extensively studied for the surface chemical modification to pulp fibers. In this study, *Eucalyptus* alkaline peroxide mechanical pulp (APMP) fibers were modified using a low-temperature plasma generator. The tensile index of the fibers after low-temperature plasma treatment under different conditions was measured and analyzed to evaluate the relationship between the plasma treatment conditions and the physical strength improvement of APMP. It was revealed that factors such as gas source (oxygen, argon, and nitrogen gases), discharge power, vacuum level, and modification time affected the physical strength properties of APMP. In addition, the change in carboxyl group content in the pulp fibers after low-temperature plasma treatment was measured using the Headspace Gas Chromatography (HS-GC) method. The carboxyl content in the fiber increased remarkedly after low-temperature plasma treatment, which was beneficial for improving the physical strength properties of paper made from the APMP.

Keywords: Carboxyl content;Low-temperature plasma treatment; APMP; Tensile index;

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INTRODUCTION

Alkaline peroxide mechanical pulp (APMP) is an important high-yield pulp that allows for better utilization of natural resources. APMP offers various advantages including high yield and low energy consumption, and applications of APMP in the production of packaging paper as a substitute for chemical pulps have attracted great interest (Boeva-Spiridonova *et al.* 2006). While most research has focused on the improvement of APMP bleaching, fewer studies have looked at improving the strength properties of pulp and paper products (Liu *et al.* 2012). To solve these issues, many different methods have been developed to improve the physical properties of APMP fibers. For example, the residual lignin on the fiber surface seriously affects the binding strength between fibers, and pretreatment of pulp with laccase can achieve better tensile strengths by reducing lignin content of the APMP fibers' surface (Cadena *et al.* 2010).

Low-temperature plasma treatment is a well-known fiber modification technology that is environmentally friendly, has low energy consumption, and causes less damage to the fibers. Moreover, it is easy to control the area and degree of modification. Most importantly, plasma treatment can significantly improve interface performance without compromising the structure of the material. The electrons collide with gas molecules when

they are driven from the negative electrode to the positive electrode under the influence of electrostatic force, leading to ionization of the gas molecules. When the activated species within the low-temperature plasma strike the materials' surfaces, new free radicals form on those surfaces. These free radicals significantly change the surface properties.

Many researchers have studied fiber modification with low-temperature plasma technology. For example, low-temperature plasma treatment technology was applied to the treatment of fibers and textiles (Kan *et al.* 2017). After plasma treatment, the hydrophilicity and adhesive property of the fibers were greatly improved (Chef *et al.* 1996). Vander Wielen *et al.* (2005) showed that the physical properties of KP (kraft pulp) papers were significantly enhanced by applying low-temperature plasma treatment before paper-making (Vander Wielen *et al.* 2005). After modifying the surface, the paper's wet strength and stiffness were improved. Zanini *et al.* (2005) used cold Ar plasma treatments to modify the structure of the lignocellulosic fibers. The chemical structure of lignin was heavily modified by the plasma treatment. The amount of both phenolic and secondary alcoholic groups decreased in both CTMP and kraft fibers, and then rebuilt new structures. Finally, the treatment caused a substantial reduction of lignin content on the surface. This indicates that low-temperature plasma treatment can improve the binding strength between different fibers by removing lignin from the fiber surface. Although it is widely accepted that low-temperature plasma treatment can improve the dry and wet strength of the fiber, how the magnitude of the low-temperature plasma treatment conditions influence the strength is still not clear.

In this work, we used low-temperature plasma treatment to modify APMP fibers in order to improve their physical strength properties. To explore the relationship between the effects of modification and plasma conditions, different kinds of gases, levels of vacuum, and treatment times were considered in the experiment. In addition, the content of carboxyl groups on the fibers before and after plasma treatment were detected using gas chromatography, which was further used to explain the enhanced strength of the paper after plasma treatment.

EXPERIMENTAL

Materials

APMP fibers were purchased from the Paper Making Branch Office of Nanning Sugar Industry Co., Ltd. They were manufactured from *Eucalyptus* wood and intended for hygienic products. The pulp had a freeness of 750 CSF (mL) and a brightness of 73.3% ISO. They were processed by the method of air-drying. After processing, they were hermetically packaged and stored at 4 °C. Oxygen, argon, and nitrogen gases were produced by Guangxi Guoxin Gas Research Co., Ltd. (China) with a purity of 99.999%.

Air-dried APMP was treated with an HPD-100B sub-atmospheric glow discharge system (Nanjing Suman Electronics Co., Ltd., China) under various atmospheres (oxygen, argon, and nitrogen gases), vacuum levels (pressure ranges from -800 to -1600 Pa), levels of plasma discharge power (55, 70, 85, 100, and 115 W), and modification times (1, 2, 3, 4, and 5 min). The chamber was evacuated and purged twice with the test gas to clean the chamber and the sample before test.

Tensile Index, and Dry and Wet Zero-Span Tensile Index Test

Handsheets with basis weight of 80 g/m^2 were prepared using a standard handsheet former as described in TAPPI Test Method T 205 sp-02 (R2006). All samples were conditioned at 23 °C and 50% RH for 24 h before all tests.

The tensile index was tested using an L&W tensile strength tester (L&W Co., Ltd., Sweden) according to the standard TAPPI T494 om, 2006. The dry zero-span tensile index was tested according to the standard TAPPI T231 cm (2007). The wet zero-span tensile index was tested according to the standard TAPPI T273 pm, 1995. A Z-SPAN 2400 zero-span tensile strength tester (Pulmac International Corporation's production, Williston, Vermont, USA) was used for both dry and wet zero-span tensile index tests. Three measurements were performed on each sample to ensure the reproducibility. Duncan's multiple range test was used to compare all the results for each analysis using the SPSS v17.0. Differences between means were considered significant when the confidence interval is smaller than 0.05.

Detection of APMP fibers' carboxyl groups content

The surface carboxylic acid content of pulp fibers was determined using Headspace Gas Chromatography methods (Chai *et al.* 2001). In brief, hydrochloric acid at a concentration of 0.1 M was used to treat fibers at 25 °C for 1 h. Then, the fiber was washed with distilled water until reaching a pH value of 7. Subsequently, the fibers were dried naturally. Dried pulp samples (0.0300 to 0.0750 g) were placed into a 20-mL test bottle with a height of 75 mm. Four mL of 0.0025 M $NaHCO_3$ and 0.1 M NaCl were placed into the test bottle. The test bottle was sealed immediately after introducing the solutions. Then the HP-7694 automatic headspace sampler and gas chromatograph HP-6890 (Agilent Technologies Co., Ltd. USA) were used for analysis. The gas chromatography operating conditions were as follows: the inner dimension of the capillary columns was 0.153 mm with a length of 30 mm (the type was GS-Q); and the capillary columns' temperature was 30 °C with a nitrogen flow rate of 3.1 mL/min.

RESULTS AND DISCUSSION

Influence of Various Gases on the Physical Strength of Papers

Different kinds of gases used for plasma modification have different efficiencies for surface modification of the fibers. Researchers showed that the content of the polar groups on the fibers' surface was increased after low-temperature plasma treatment (Xiang *et al.* 1995). Simultaneously, the surface free energy was also obviously enhanced. The bonding strength between neighboring fibers was strengthened, which significantly enhanced the surface strength of the papers.

In this work, oxygen, argon, and nitrogen gases were used for the low-temperature plasma treatment of APMP fibers. The low-temperature plasma treatment conditions were as follows: the vacuum level was -800 Pa, the discharge power was 85 W, and the modification time was 3 min. Table 1 summarized the effects of different gases on tensile index.

Table 1. The Influence of Various Gases on Tensile Index

Samples	Tensile Index ($N \cdot m \cdot g^{-1}$)	Zero-span Tensile Index ($N \cdot m \cdot g^{-1}$)	Wet Zero-span Tensile Index ($N \cdot m \cdot g^{-1}$)	% Increase in tensile index		
				Tensile Index	Zero-span Tensile Index	Wet Zero-span Tensile Index
Unmodified fibers	6.48	68.10	42.79	0	0	0
O_2-modified fibers	8.50	81.69	64.31	31.17	19.96	50.29
N_2-modified fibers	7.90	77.38	60.30	21.91	13.63	40.92
Ar-modified fibers	7.69	74.62	53.75	18.67	9.57	25.61

As can be seen from Table 1, the tensile index of three samples was enhanced after the fibers were modified. The treatment efficiency using oxygen and nitrogen was better than argon. This may be attributed to the fact that oxygen or nitrogen are the reactant gases. Oxygen or nitrogen atoms in low-temperature plasma are very effective in changing the surface of the fibers because of the formation of oxygen or nitrogen functional groups at the fiber surface through the reaction between active species from the plasma and the fiber surface atoms (Navik *et al.* 2015). In contrast, argon is an inactive gas; its plasma cannot introduce new functional groups to the surface even if modification and reorganisation of the chemical groups already present at the surface can occur (Øiseth *et al.* 2002). As a result, change of fiber tensile index was relatively small compared to oxygen or nitrogen plasma treatment. For both the tensile index and the zero-span tensile index, the O_2-modified fibers showed the best results. Oxygen plasma induced the surface activation from the process of such as excited molecules, atomic oxygen, ozone, molecular ions, and the relevant atomic ions which may behave as active sources to the formation of new hydrophilic carboxylic and hydroxyl derivatives on fiber surface. On the other hand, O_2-plasma treatment might increase polar component surface energy and possibly surface roughness, along with a small increase in wettability which contribute to the swelling of fiber surface. The formation of those interactive layers on the cellulose surface may result in strong fiber interactions (Mahlberg *et al.* 1998; Vander Wielen *et al.* 2006). The tensile index of the oxygen plasma-treated fibers increased from 6.48 to 8.50 Nm/g, and the zero-span tensile index was enhanced from 68.10 to 81.69 Nm/g, in addition to the improved wet zero-span tensile index.

Effect of Discharge Power of Low-temperature Plasma on the Physical Strength of Papers

Previous literature has already shown that the power level of low-temperature plasma treatment has a great impact on the effects of surface modification. Bai (2002) used low-temperature plasma treatment to modify the polyester fiber and evaluated treatment performance by the decrement of fiber quality. It was found that the decrement of the fiber

quality was increased linearly when discharge power was raised. Ren *et al.* (2002) discovered that the extent of loss of fiber strength has a direct relation to the etching interaction during the oxygen plasma treatment of silk fibers (Ren *et al.* 2002). These results indicate that the higher the discharge power, the more energy the particle absorbs, and in turn the surface of the fibers is bombarded more intensively, giving rise to serious etching of the fibers.

In this research, the treatment effects under different discharge powers (55, 70, 85, 100, and 115 W) was studied. The detailed experiment conditions were as follows: oxygen was used as gas source, the reaction chamber pressure (vacuum level) was -800 Pa, the treatment time was 3 min, and all other conditions remained unchanged.

Table 2. The Effect of Discharge Power on Tensile Index (O_2 as gas source)

Power (W)	Tensile Index ($N \cdot m \cdot g^{-1}$)	Zero-span Tensile Index ($N \cdot m \cdot g^{-1}$)	Wet Zero-span Tensile Index ($N \cdot m \cdot g^{-1}$)	% Increase in tensile index		
				Tensile Index	Zero-span Tensile Index	Wet Zero-span Tensile Index
0 (Unmodified)	6.48	68.10	42.79	0	0	0
55	7.17	75.71	58.69	10.48	11.17	37.44
70	7.76	76.71	59.70	19.75	12.64	39.81
85	8.50	81.69	64.31	31.17	19.95	50.60
100	8.09	78.83	60.73	24.84	15.75	42.22
115	7.15	75.45	59.30	10.33	10.79	38.87

As can be seen from Table 2, the tensile index of papers gradually increased as the discharge power increased. When the tensile index of papers reached the highest value (8.50 Nm/g at 85 W), it started to decrease as the discharge power increased. Similarly, the zero-span tensile index and wet zero-span tensile index also reached their maximum values (81.69 and 64.31 Nm/g) at 85 W. When the power was higher than 85 W, the modified paper's tensile index and zero-span tensile index decreased promptly. This observation suggests that low levels of treatment contributed to the increase in fiber-fiber bonding. This can be explained by the fact that increasing the discharge power will result in an increase of the amount of reactive plasma species. The presence of more plasma species can lead to increased fiber wettability due to a more intense bombardment on the fiber surface (Indubarkar *et al.* 2010). As a result, more surface carboxylic acid groups are formed on fiber surfaces, which enhances the fiber-fiber bonding strength because of an improved absorbency and swelling performance of each fiber. However, as levels of treatment dosages are further increased, the fiber swelling decreases (Vander Wielen and Ragauskas 2003). This may be induced by the over-etching of the fibers when the discharge power is very high. The over-etching greatly weakened the binding force between the adjacent fibers. Thus, during the process of low-temperature plasma treatment of APMP fibers, an appropriate power level should be chosen. In this study, the optimal discharge power was about 85 W.

Influence of Vacuum Pressure during Low-temperature Plasma Treatment on the Physical Strength of Papers

The extent of surface dielectric barrier discharge plasma is affected by the operating pressure and normally a low discharge pressure leads to a high intensity of plasma generation. It was assumed that decreasing the pressure results in a larger ionization level and a wider plasma region but it also reduces the gas density and thus the momentum transfers by collision (Benard *et al.* 2008). This study evaluated the effects of treatment under the condition of different vacuum levels ranging from -800 to -1600 Pa, with 200-Pa intervals. Oxygen was used as the gas source. The discharge power was 85 W. All samples were treated for 2 min. Table 3 summarizes the tensile index evolution as a function of the vacuum level.

Table 3. The Influence of Vacuum on Tensile Index

Vacuum	Tensile Index ($N \cdot m \cdot g^{-1}$)	Zero-span Tensile Index (N·m·g−1)	Wet Zero-span Tensile Index (N·m·g−1)	% Increase in tensile index		
				Tensile Index	Zero-span Tensile Index	Wet Zero-span Tensile Index
0 (Unmodified)	6.48	68.10	42.79	0	0	0
-800	8.15	75.92	59.93	25.77	11.48	40.06
-1000	8.41	82.08	62.39	29.78	20.53	45.81
-1200	8.34	79.02	61.04	28.70	16.04	42.65
-1400	8.27	76.19	59.83	27.62	11.88	39.82
-1600	7.98	74.55	53.50	23.15	9.47	25.03

As can be seen in Table 3, compared with the untreated fiber, the tensile index of the plasma-treated paper was greatly increased when the vacuum level was varied between -800 and -1600 Pa. It was observed that the tensile index was first increased and then decreased as the vacuum level increased, with a threshold of the vacuum level at -1000 Pa. It's likely that when the operating pressure is higher than -1000 Pa, the plasma collided with the surface of fibers more frequently. The frequent bombardment generated more cracks on the fiber surfaces, resulting in more surface etching and generation of carboxylic groups on the fibers, which contributed to the maximal tensile index of the paper (Wu *et al.* 2013). However, as the pressure further decreases, the electron collision frequency decreases and eventually weaken physical or chemical changes of fiber surface, which leads to the decrease of tensile index while the level of vacuum was larger than -1000 Pa. Thus, a maximal fibers tensile index reached at vacuum level of -1000 Pa.

Effect of Low-temperature Plasma Treatment Time on the Physical Strength of Papers

Plasma treatment time also has a great influence on fiber etching or cross-linking. During the low-temperature oxygen plasma treatment of nylon fibers, it has already been revealed that the etching extent of the fiber increases as the treatment time is prolonged.

However, the tensile strength of fibers is enhanced at the beginning, then reduced, as the treatment time is prolonged (Yip *et al.* 2002).

Here, we explored the effect of different treatment times (1, 2, 3, 4, and 5 min). Other treatment conditions remained unchanged: the gas source was oxygen; the discharge power was 85 W; and the vacuum level was -800 Pa. The plasma treatment time's influence on the tensile index is shown in Table 4.

Table 4. The Influence of Treat Time on Tensile Index

Treatment Time/min	Tensile Index ($N \cdot m \cdot g^{-1}$)	Zero-span Tensile Index (N·m·g−1)	Wet Zero-span Tensile Index (N·m·g−1)	% Increase in tensile index		
				Tensile Index	Zero-span Tensile Index	Wet Zero-span Tensile Index
0	6.78	69.26	40.74	0	0	0
1	7.52	73.50	56.15	10.91	6.11	37.82
2	8.06	75.04	59.29	18.88	8.34	45.55
3	8.41	82.08	62.39	24.04	18.51	53.15
4	7.38	76.90	50.56	8.85	11.03	24.10
5	7.09	72.45	47.34	4,57	4.61	16.20

As can be seen in Table 4, plasma treatment time had an obvious impact on the paper's tensile index. When the treatment time was relatively short, the change in tensile index was obvious. In an appropriate treatment time range (~1 to 3 min), the tensile index was dramatically increased. If the plasma treatment time was too long (> 3 min), the tensile index declined steeply. Therefore, the duration of the plasma treatment is crucial in determining the plasma modification performance. Below a certain treatment time, plasma modification was beneficial for improving the amount of polar groups on the fiber surface, but when the time was too long, it led to the destruction of the crystalline region of the fibers and a reduction in fiber strength. This is also consistent with the previous results obtained by Yip *et al.* (2002).

Content Change of Pulp Carboxyl Groups during Plasma Treatment

Koljonen (2004) found that there is a positive correlation between the tensile strength and the carboxyl group content of paper. Quantitative determination of the carboxyl group content in traditional methods is often performed using acid-base titration (Jayme and Neuschaffer 1955, Samuelson and Wennerblom 1955, Katz *et al.* 1981) or EDTA complex titration (Sobue and Okubo 1956). These methods are complex and time-consuming, and more seriously, the repeatability of experimental determination results is poor, even in the same laboratory (Wilson and Mandel 1961). Headspace gas chromatography has been widely applied for the analysis of volatile components in complex matrix samples (Wilson and Mandel 1961; Drozd and Novák 1979; Namieśnik *et al.* 1990; Kolb 1999; Kolb and Ettre 2006). The most important case is the phase transformation of HS-GC technology introduced by Chai *et al.* (2001). It inversed the carbonate in the sample black liquor into carbon dioxide by acidification, and then the CO_2 entered the gas phase, and eventually determined the signal peak area of the carbon dioxide

with HS-GC technology. By doing this, the carbonate content in the black liquor can be evaluated (Chai *et al.* 2001). On this basis, HS-GC technology can be further applied to determining the carboxyl group content of pulp (Chai *et al.* 2003). In other words, the carboxyl content of pulp can be measured using a liquid reactant that can produce a chemical reaction and release carbon dioxide with solid pulp fiber carboxylic acid.

Some active groups were introduced into the fiber surface after low-temperature plasma treatment. Therefore, the fiber surface produced carboxyl groups when the treatment atmosphere contained oxygen or when the treated fibers were exposed to air. In this study, it can be seen that low-temperature plasma treatment can improve the papers' physical strength through measuring the change in the carboxyl content of pulps before and after low-temperature plasma treatment.

Here, the change of the carboxyl content in the pulp fibers after low-temperature plasma treatment was measured using the HS-GC method. Dried pulps (0.0300 g) before and after plasma treatment were used for analysis. The results are shown in Fig. 1. The carboxyl content of pulps can be calculated using Eq. 1 (Hou *et al.* 2006),

$$C_B = \frac{f}{\omega \times 0.9}\left(A_T - A_{Air}\right) \quad (1)$$

where C_B is the concentration of fiber's carboxyl (mmol/g), ω is the weight of dried pulp, and A_T is the metrical peak area of GC.

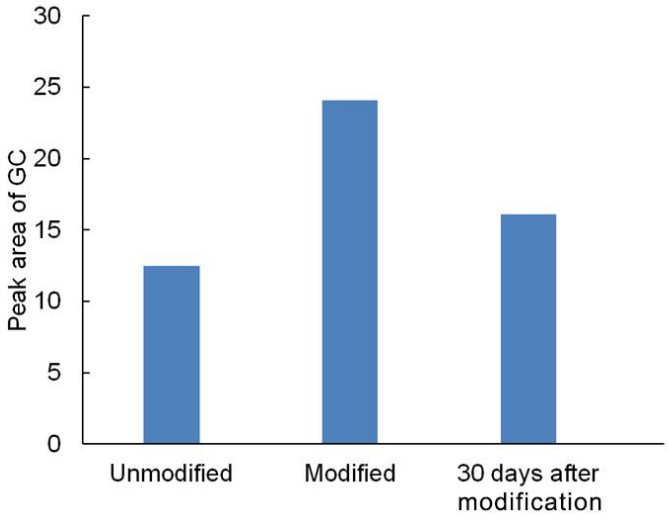

Fig. 1. Change in the carboxyl peak area before and after treatment of the pulp fibers

Figure 1 shows that the carboxyl content of the fiber was increased after low-temperature plasma treatment. The unmodified fibers' peak area of GC was calculated to be approximately 12.24, while the modified fibers' peak area was calculated to be approximately 24.24. The carboxyl content in the pulp fibers can be calculated according to Eq. 1. The unmodified fibers' carboxyl content was determined to be approximately 0.0802 mmol/g. However, the value of modified fibers increased to 0.1659 mmol/g. The carboxyl groups can ionize in water and generate hydrogen ions and carboxylate anions. The large amount of anions on the fiber can adsorb cationic additives. The increased carboxyl content can improve the swelling of the fiber, thus affecting its flexibility and increasing the bonding area between neighboring fibers. With increased carboxyl group

content on the fiber surface, the density of the resulting paper may be higher and the more bonding surface becomes available for other fibers to bond onto, which is considered to be one of the main contributes to the tensile strength of the paper.

CONCLUSIONS

1. Of the three gases used (oxygen, argon, and nitrogen), oxygen had the highest treatment efficiency to create carboxyl-containing groups and improve the tensile strength paper. After modifying the fibers with oxygen, the tensile index was increased from 6.48 to 8.50 Nm/g. Dry and wet zero-span tensile indexes exhibited a similarly increased trend.
2. Discharge power determined the papers' physical strength. When the power was too low, the treatment was not effective. On the other hand, when the power is too high, the etching effect is intensified, causing the papers' physical strength to decline.
3. During low-temperature plasma treatment, too high/long or too low/short vacuum level /time cannot obtain the highest treatment efficiency. When the treatment is either insufficient or too much, the strength of the fibers' surface is only slightly improved compared with that of untreated fibers.
4. The content change of the carboxyl groups after plasma treatment explained that the low-temperature plasma bombarded the surface of the fibers and created more oxygen-containing groups (such as carboxyl groups). Low-temperature plasma treatment induced physical etching and generation of new carboxyl groups on fiber surface, contributing to more bonding areas between the fibers and the dramatic increase of the papers' tensile index and dry and wet zero-span tensile index.

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