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Acetic acid pulping of wheat straw under atmospheric pressure

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Abstract Atmospheric acetic acid pulping of wheat straw was carried out. Pulping conditions and their effects on pulp properties were investigated in detail, and a comparison between acetic acid (AcOH) pulp and soda-anthraquinone (AQ) pulps of wheat straw was made of the chemical composition, strength, and fiber morphology of the pulps. Wheat straw was successfully pulped and fractionated into pulp (cellulose), acetic acid lignin, and sugars (monosaccharides from hemicellulose), making it easy to utilize them. It was found that among the pulping conditions the dosage of H_2SO_4 as catalyst was the most notable, and the extent and rate of delignification could be controlled by varying the amount of the catalyst. The results also showed that acetic acid pulp was quite different from soda-AQ pulp. About 70% of the ash or 90% of the silica in wheat straw were kept in AcOH pulp. The ash might function as filler and be beneficial to the printability of paper. It was known that many epidermal cells existed in AcOH pulp in bundles or in single cells. These ash-rich nonfiber cells seemed to hinder the bonding between fibers. AcOH pulp had lower strength than soda-AQ pulp, which might result mainly from the chemical damage of fibers caused by acid, not from the depolymerization of cellulose.

Key words Wheat straw · Acetic acid pulp · Pulping conditions · Pulp properties · Soda-anthraquinone pulp

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Introduction

Wheat straw, as an agricultural residue, is generated in a huge quantity worldwide every year. For example, nearly 300 million tons of wheat straw are produced yearly in North America and Europe,[1] two of the major wheat-producing areas. This renewable material has not yet been used as industrial raw material on a large scale, especially in developed regions.

In many countries wheat straw is partly used as animal feed directly or after pretreatment[2–4] to improve digestibility. Wheat straw is also fractionated to hemicellulose, cellulose, and lignin as raw materials for chemicals by various processes, such as explosion,[1] dilute acid hydrolysis,[5] autohydrolysis,[6,7] and an organosolv process.[8,9] Moreover, in countries short of forest resources, such as China, wheat and rice straws have been used extensively as raw material for pulp and paper. In China more than 9 million tons of straw pulp are produced every year, which accounts for about 90% of the world's total straw pulping capacity.[10] To date, straw pulps have been manufactured mainly by soda-anthraquinone (AQ) and neutral or alkaline sulfite processes. The recovery of chemicals from the black liquors of straws is difficult because of the high viscosity, low caloric value, and in particular high silica content of the liquor. As a result, the black liquors are mostly discharged without treatment and have caused serious water pollution.

The acetic acid pulping process has proved to be an effective method for delignifying and fractionating wood.[11–13] By atmospheric acetic acid pulping, wood can be selectively fractionated to pulp, lignin, and hemicellulose (mainly monosaccharides), which makes it easy to utilize them. In a previous paper[14] we reported that rice straw was successfully pulped by aqueous acetic acid that contained a small amount of H_2SO_4 or HCl as catalyst. In the present study we applied the acetic acid process to wheat straw and investigated the pulping conditions and their effect on pulp properties. Acetic acid and soda-AQ pulps were also compared for pulping results, strength, chemical composition, and fiber morphology.

Materials and methods

Wheat straw

Wheat straw (*Triticum vulgare* CV. Horoshiri) was obtained from the experimental farm of Hokkaido University. The straw was air-dried, cut to about 2cm in length, and used for pulping.

Atmospheric acetic acid pulping

As shown in Fig. 1, wheat straw was refluxed (cooked) under atmospheric pressure in aqueous AcOH that contained H_2SO_4 as catalyst. After cooling, crude pulp was filtered and washed three times with aqueous AcOH and then with large quantities of water. The filtrate (waste liquor) and AcOH washings were combined and evaporated to dryness under reduced pressure. The water washings were poured into the residue. Water-insoluble materials (acetic acid lignin) was filtered, washed with water, and then lyophilized. The filtrate and washings were collected and then concentrated under reduced pressure to obtain the water-soluble substances (sugars). The crude pulp was first disintegrated and then subjected to flat-screening on an 8-cut screener. Screened pulp was collected on a 200-mesh screen.

Soda-anthraquinone pulping

Soda-anthraquinone pulping was carried out in an autoclave. Pulping conditions were 15% NaOH and 0.05% AQ on wheat straw, liquor/ straw ratio 6:1, maximum temperature 160°C (1h to the maximum temperature and 2h at the maximum temperature).

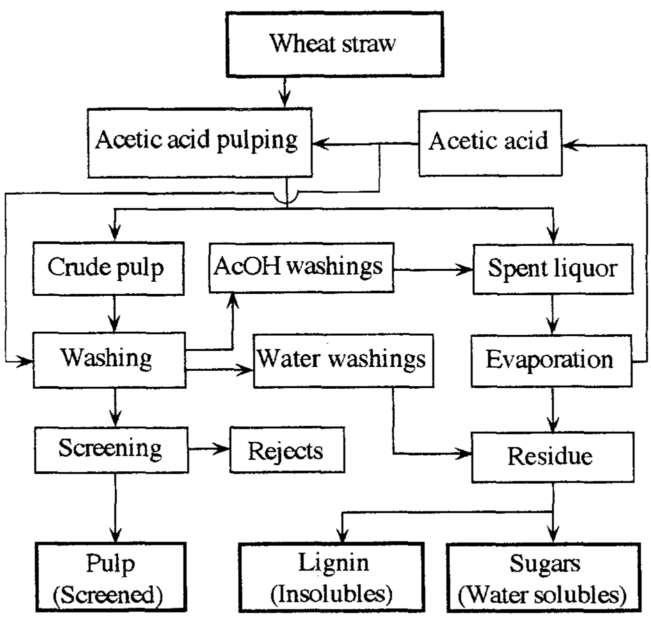


Fig. 1. Atmospheric acetic acid pulping of wheat straw

Elementary chlorine free bleaching

The bleaching was carried out using a sequence of alkaline extraction (E/P), chlorine dioxide bleaching (D), and peroxide bleaching (P). Bleaching conditions were as follows: E/P: 5% NaOH and 1.0% H_2O_2 on pulp, 12% consistency, 80°C, 2h; D: 0.5% ClO_2 on pulp, 12% consistency, pH 4–5, 70°C, 2h; P: 0.5% H_2O_2 on pulp, 12% consistency, pH 11, 70°C, 2h.

Chemical analysis of wheat straw and pulp

Ash and silica were determined using a method described previously.[15] Acetyl groups were estimated with gas chromatography.[16] Acid-soluble lignin was determined according to the method described by Dence.[17] Carbohydrates were determined using the procedure of Borchardt and Piper.[18] Other analyses were performed according to conventional methods.

Results and discussion

Atmospheric acetic acid pulping of wheat straw

Chemical composition of wheat straw

The chemical composition of the wheat straw used in this study is shown in Table 1. The wheat straw had 9.6% ash, and 76% of the ash was silica. The ash content was much higher than that of wood (usually less than 1%) but lower than that of rice straw (16.5%).[15] The percentages of silica in ash of rice and wheat straws were similar (76% and 71%, respectively). It was noted that the wheat straw had higher contents of water and 1% NaOH extractives and a lower content of lignin than wood. The carbohydrates in the wheat straw consisted mainly of glucose and xylose, with a small amount of arabinose, galactose, and mannose.

Table 1. Chemical analysis of wheat straw

Chemical	% (of straw meal)
Ash	9.6
Silica	7.3
Extractives	
Ethanol-benzene	5.3
Cold water	11.8
Hot water	17.5
1% NaOH	46.0
Total lignin	
Klason lignin	16.5
Acid-soluble lignin	2.5
Total carbohydrates	56.9
Rhamnose	0.1
Arabinose	2.9
Xylose	19.9
Mannose	0.8
Galactose	1.7
Glucose	31.5

The wheat straw was pulped by an atmospheric acetic acid process as described in Fig. 1. Pulping results, pulping conditions, and their effects on pulp properties are discussed in detail below.

Pulping time

Figure 2 shows the effect of pulping time on the pulping of wheat straw. When other conditions were fixed and pulping was prolonged, the kappa number decreased gradually and the yield of acetic acid lignin increased slightly, indicating the progress of delignification. At the same time, the pulp yield decreased and the yield of water-soluble substances (sugars from hemicellulose) increased, showing that hydrolysis of polysaccharides was also in progress.

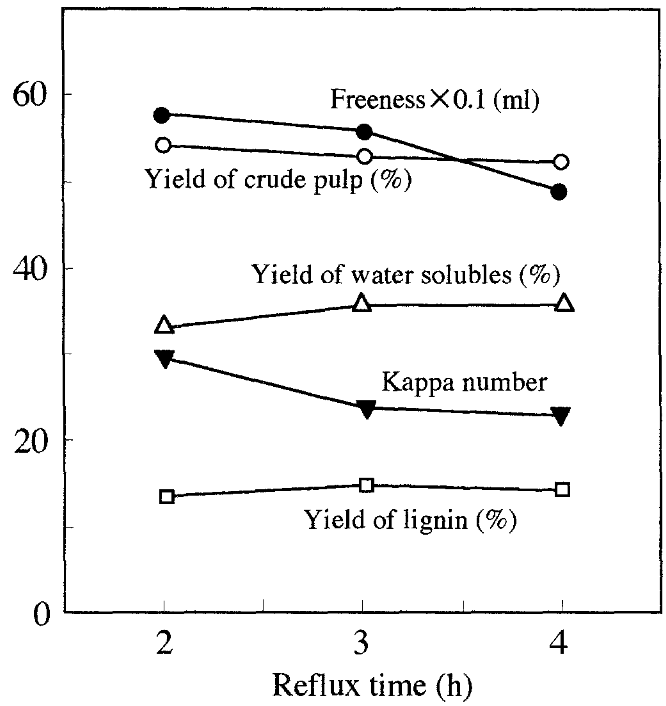


Fig. 2. Effect of reflux time on pulping. Other conditions: 3% H_2SO_4 on wheat straw, liquor/straw (LS) ratio 10, and 90% AcOH

Liquor/straw ratio

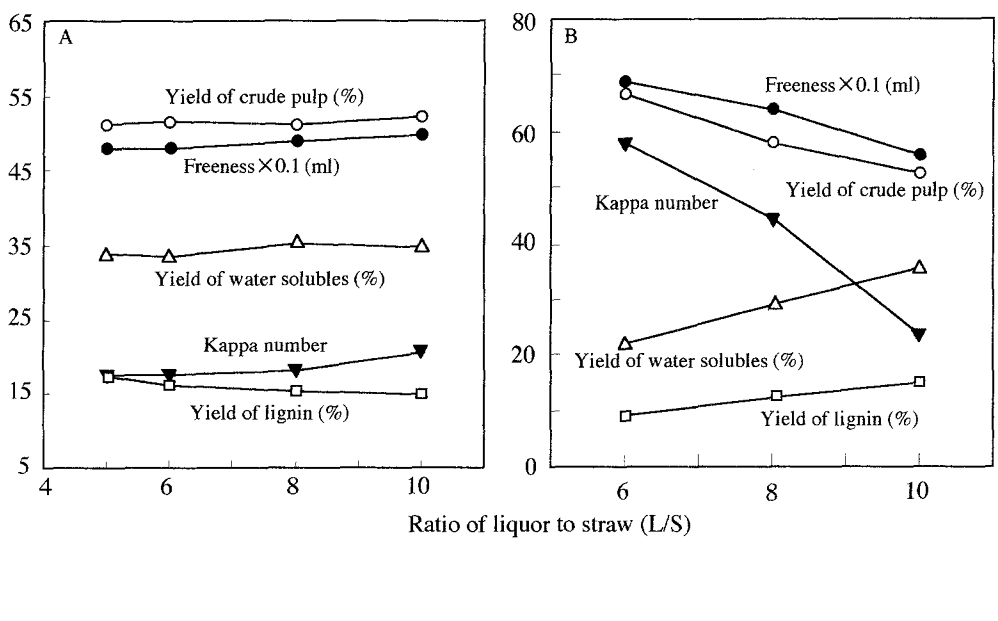
The effect of the liquor/straw (L/S) ratio on pulping is shown in Fig. 3. L/S was not an independent variable. Its effect was related to the dosage of H_2SO_4 as catalyst. As shown in Fig. 3A, when the addition of H_2SO_4 was fixed (3.5% on wheat straw) the kappa number decreased with the reduction of L/S, demonstrating the promotion of delignification by increased concentration of H_2SO_4 in AcOH. Quite different from rice straw, wheat straw could be pulped successfully even at an L/S as low as 5:1. In the case of rice straw, an L/S more than 10:1 was needed for successful pulping.[15] High silica content might make rice straw difficult to soften and thus unable to be pulped successfully at a low L/S.

If the concentration of H_2SO_4 in AcOH was fixed, as shown in Fig. 3B, good pulping could not be achieved at a low L/S. For example, the kappa number was as high as 58.0 at an L/S of 6:1 because the amount of H_2SO_4 on wheat straw became too small to meet the need of delignification.

Catalyst

Figure 4 shows that the dosage of H_2SO_4 as catalyst was a notable factor influencing the pulping results. When the dosage increased from 2.5% to 4.0% (on straw), the kappa number dropped sharply from 42.4 to 14.1, indicating promotion of delignification. In contrast, the sugar yield increased by only a small margin, demonstrating the selectivity of H_2SO_4. At least, the increment of hydrolysis of carbohydrate was less marked than the improved delignification when more H_2SO_4 was added. This finding suggested that the rapid delignification of wheat straw

Fig. 3. Effect of concentration and dosage of H_2SO_4 on pulping (90% AcOH) under different L/S ratios. **A** H_2SO_4 3.5% on straw, 2h. **B** H_2SO_4 0.3% in AcOH, 3h



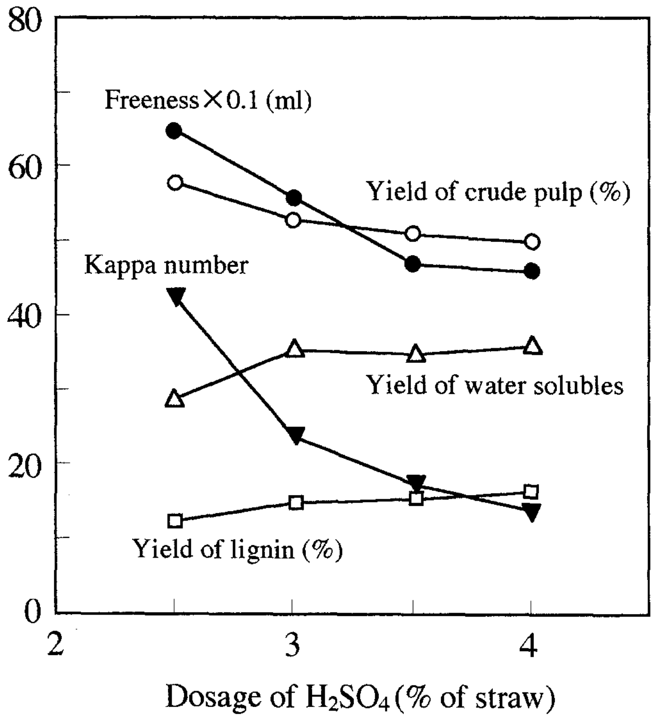


Fig. 4. Effect of H_2SO_4 dosage on pulping. Other conditions: L/S 10:1, 3h, and 90% AcOH

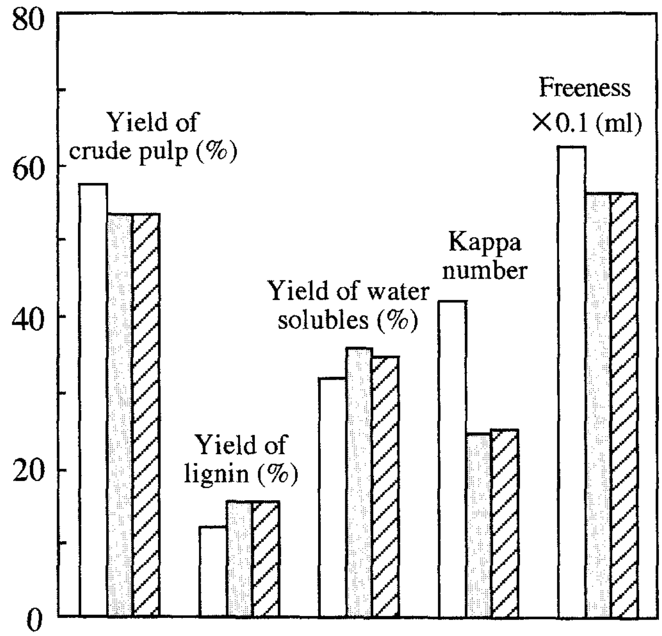


Fig. 5. Effect of AcOH concentration on pulping. Other conditions: 3% H_2SO_4 on wheat straw, L/S 10:1. In each group of bars: *first open bars*, 80% AcOH; *second open bars*, 90% AcOH; *hatched bars*, 95% AcOH

might be achieved by increasing the catalyst to a certain extent.

We suspected that wheat straw may need more H_2SO_4 than wood for satisfactory pulping because the ash in the wheat straw might consume some of the H_2SO_4. However, when we treated wheat straw and wood chips with AcOH containing H_2SO_4 and then detected the change of H_2SO_4, no evidence was found that the ash in wheat straw consumed H_2SO_4.

Acetic acid concentration

Figure 5 shows the pulping results of wheat straw with AcOH at various concentrations (80%, 90%, 95%). Compared with 90% and 95% AcOH, 80% AcOH seemed to have less ability to delignify wheat straw. The pulp obtained with 80% AcOH had a larger kappa number, higher pulp yield, and lower yields of lignin and sugars than were obtained with 90% and 95% AcOH. No obvious difference was found between 90% and 95% AcOH. From an economical point of view, 90% AcOH might be more advantageous.

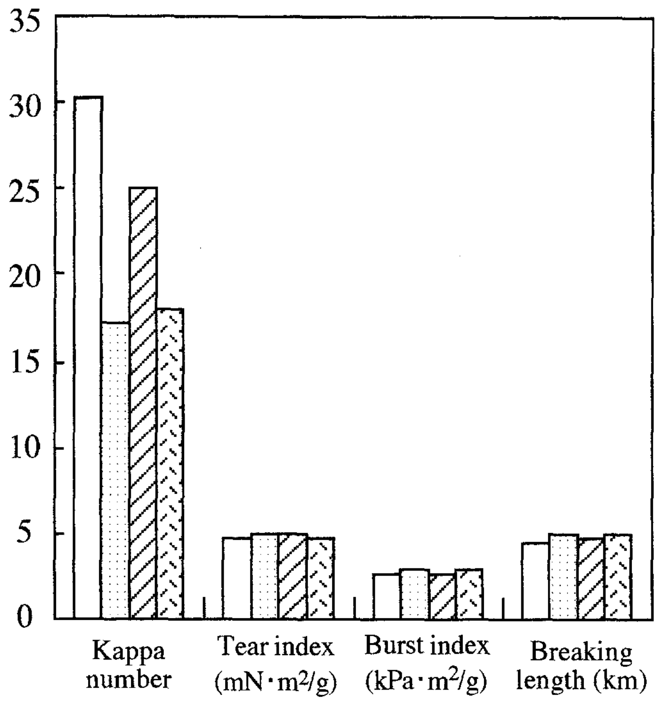


Fig. 6. Strength properties of AcOH pulps of wheat straw prepared under various conditions

Effect of pulping conditions on pulp properties

In Fig. 6 we examined the influence of pulping conditions on the strength properties of pulp. The results further demonstrated that delignification was improved and rapid delignification could be achieved with increasing catalyst without an obvious negative effect on pulp strength. For example, under the conditions of 90% AcOH, L/S 6:1, and 5h, as H_2SO_4 increased by 0.5 percentage points from 2.5% to 3.0% the kappa number dropped from 30.4 to 17.3, and the strength of the pulp obtained was improved by extensive delignification. Moreover, we prepared two pulps with roughly the same kappa number using different conditions.

One was prepared with 3.0% H_2SO_4 and 5h and another with 3.5% H_2SO_4 and 2h. It was found that the two pulps had almost the same strength properties.

Extent of delignification

To investigate the relation between the extent of delignification and the physical properties of acetic acid pulp from wheat straw, we prepared a series of pulps with kappa numbers from 13.2 to 42.9 under various pulping conditions. As shown in Fig. 7, with the progress of delignification, the density, burst index, and breaking length of the pulps increased gradually, indicating improved bonding between fibers due to the removal of lignin. In contrast, the tear index decreased slightly, showing the weak strength of the fibers themselves, which might result from the so-called chemical cuttings on a part of the fibers, as shown in Fig. 8B. The sharp drop of freeness at a low kappa number might be also caused by such chemical cutting, although the improvement of hydrophilicity by delignification also lowered the freeness.

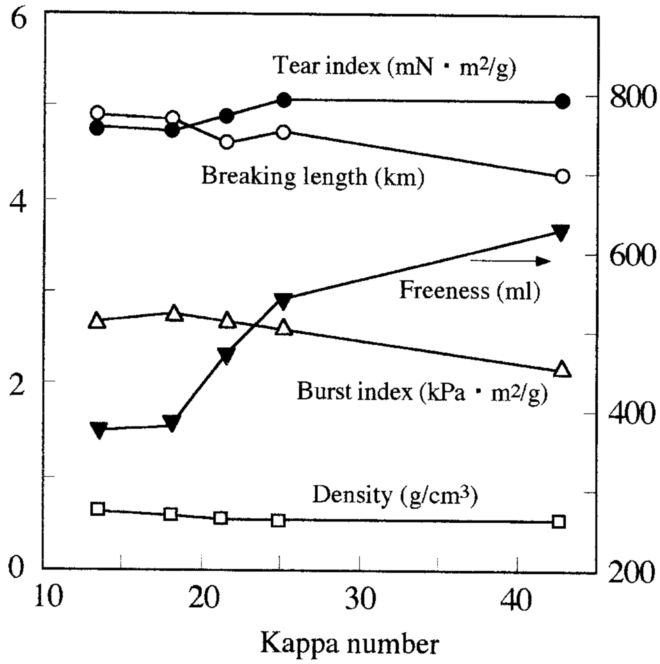


Fig. 7. Relation between extent of delignification and physical properties of acetic acid pulp from wheat straw

Based on the results and discussion above, it can be concluded that AcOH pulp from wheat straw with a kappa number of 15–20 could be prepared in a yield of about 50% under the conditions of 90% AcOH, L/S 6:1, 3%–4% H_2SO_4 on straw, and 3–5h.

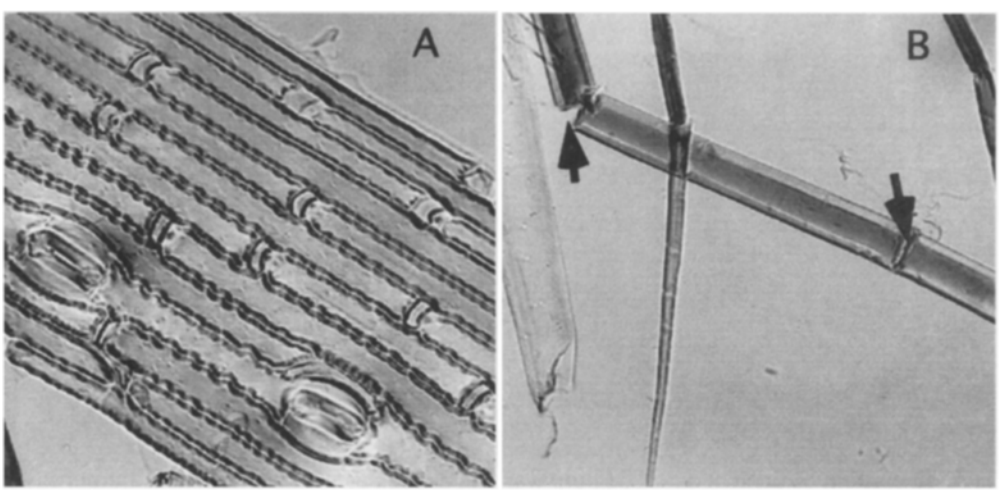
Comparison between acetic acid and soda-AQ pulps

As discussed above, wheat straw could be successfully pulped by the atmospheric acetic acid process, and monosaccharides (mainly from hydrolysis of hemicellulose) and acetic acid lignin were obtained from waste liquor as by-products. With this process it is possible to utilize fully the biomass components of wheat straw. AcOH pulp can be used for paper and cellulose derivatives.[19] Lignin can be converted to valuable products, such as carbon fiber,[20] activated carbon,[21] and adhesives.[22] From monosaccharides we can obtain chemicals, sweeteners, fuel, and polymers.[23] A blemish on an otherwise perfect situation was that the acetic acid pulp from wheat straw had relatively weaker strength, especially tear strength, than other pulps (e.g., soda-AQ pulp). To determine the cause we investigated the chemical composition, strength, bleachability, and fiber morphology of AcOH pulp and compared the resutls with those of soda-AQ pulp.

Pulping results

As shown in Table 2, when comparing AcOH and soda-AQ pulps at similar kappa numbers, AcOH pulp gave a yield 4 percentage points higher than soda-AQ pulp for both crude and screened pulps. The higher yield resulted from the high retention of ash and the introduction of acetyl groups, as discussed later. Moreover, the freeness of AcOH pulp was

Fig. 8. Bundle of epidermal cells (**A**) and chemically cut fiber (**B**) in acetic acid pulp of wheat straw (A,B; ×200)



lower than that of soda-AQ pulp. It was one reason that many nonfiber cells, such as parenchymal and epidermal cells, remained in AcOH pulp, which were much smaller than fibers and resulted in a low freeness. Another reason might be that a part of the fibers were chemically cut into small segments during the AcOH pulping, as shown in Fig. 8B.

Chemical composition

Table 2 shows that the chemical compositions of AcOH and soda-AQ pulps were quite different. AcOH pulp had an ash content as high as 14.0%, which was almost pure silica, whereas soda-AQ pulp had only 1.7% ash, about 60% of which was silica. In other words, about 68% of ash or 88% of silica in wheat straw remained in AcOH pulp, whereas as much as 93% of ash or 94% of silica was dissolved in waste liquor during soda-AQ pulping, which causes problems in the chemical recovery system, as is well known. The ash that remained in AcOH pulp might be good as a filler and beneficial to the opacity and printability of paper.[24]

Another difference is that AcOH pulp had 3.8% acetyl groups due to partial acetylation of hydroxyl groups in both polysaccharides and lignin. In addition, AcOH pulp had less carbohydrate than soda-AQ pulp due to the higher content of ash and acetyl groups in the former. Furthermore, although glucose and xylose were the main sugars in both pulps, the proportions were different. AcOH pulp had more glucose and less xylose than soda-AQ pulp, implying that the sulfuric acid in AcOH hydrolyzed less cellulose than hemicellulose and had better selectivity than NaOH. The larger amount of arabinose in soda-AQ pulp than in AcOH pulp suggested that the bonding of arabinose was more resistant to alkaline hydrolysis than to acid hydrolysis.

Pulp properties

Table 2 gives the physical properties of unbleached AcOH and soda-AQ pulps at a comparable freeness. It was found that AcOH pulp was much weaker than soda-AQ pulp. Breaking length, tear, and burst indexes of AcOH pulp were only 53%, 64%, and 36%, respectively, of those of soda-AQ pulp. Ash-rich epidermal cells in AcOH pulp could be regarded as a reason for these differences. These nonfiber cells made no contribution to strength and obstructed the bonding between fibers. The second reason might be the chemical damage to fibers caused by acid. Here, the chemical damage to fibers represents the cutting, slashing, or damage to the fibers caused during pulping, not the acidic hydrolysis of cellulose chains, as shown in Fig. 8B.

ECF bleaching

The bleaching results for AcOH and soda-AQ pulps are given in Table 3. By a short ECF sequence, AcOH pulp of wheat straw could be easily bleached to 86% brightness. The ash content of bleached AcOH pulp was still as high as 14.2%, because of which the AcOH pulp had higher opacity than soda-AQ pulp. Although the viscosity of bleached AcOH pulp was lower than that of bleached soda-AQ pulp, the difference was not as large as that in strength, indicating that the low strength of AcOH pulp might result mainly from the acidic damage to the fibers, not from the acidic hydrolysis of cellulose.

Fiber morphology

Figure 8, a microscopic photograph of AcOH pulp, shows that there were numerous epidermal cells in bundles or as

Table 2. Comparison between AcOH and soda-AQ pulps from wheat straw

Parameter	AcOH pulp	Soda-AQ pulp
Yield of crude pulp (%)	49.7	45.8
Yield of screened pulp (%)	46.5	42.3
Reject (%)	0.4	1.1
Kappa number	13.2	15.4
Freeness (ml)	375	490
Freeness after beating (ml)	255	275
Density (g/cm^3)	0.6	0.8
Breaking length (km)	4.9	9.2
Tear index ($mN \cdot m^2/g$)	4.8	7.5
Burst index ($kPa \cdot m^2/g$)	2.7	7.4
Fold endurance (time)	34	470
Brightness (%)	34.6	22.6
Opacity (%)	92.8	91.6
Ash (% of pulp)	14.0	1.7
Silica (% of ash)	98.7	60.5
Acetyl groups (% of pulp)	3.8	0.0
Total lignin (% of pulp)	4.3	3.7
Klason lignin (% of pulp)	3.6	2.6
Acid-soluble lignin (% of pulp)	0.7	1.1
Carbohydrates (% of pulp)	77.4	93.2
Composition of carbohydrates (%)		
Rhamnose	0.0	0.0
Arabinose	1.0	3.8
Xylose	11.2	27.5
Mannose	0.8	0.5
Galactose	1.0	1.5
Glucose	86.0	66.7

Pulping conditions were as follows. AcOH pulp: 90% AcOH, L/S 8, 4% H_2SO_4, 3h. Soda-AQ pulp: 15% NaOH, 0.05% AQ, L/S 6, 160°C, 3h (1h to 160°C and 2h at 160°C)

Table 3. ECF bleaching of AcOH and soda-AQ pulps of wheat straw

Parameter	AcOH pulp	Soda-AQ pulp
Yield (% of screened pulp)	88.8	89.4
Brightness (%)	86.0	83.6
Opacity (%)	75.7	55.5
Ash (%)	14.2	0.3
Viscosity (mPa · S)	35.4	40.1
Density (g/cm^3)	0.6	0.8
Breaking length (km)	3.9	7.6
Tear index ($mN \cdot m^2/g$)	6.2	8.2
Burst index ($kPa \cdot m^2/g$)	2.5	7.1
Fold endurance (time)	26	937

Bleaching sequence: alkaline extraction (E/P), chlorine disxide bleaching (D), and peroxide bleaching (P). (E/P–D–P). Conditions are given in the materials and methods section
ECF, elementary chlorine free

single cells in AcOH pulp (Fig. 8A). These ash-rich epidermal cells are thought to hinder the bonding between fibers. As shown in Fig. 8B, a part of the fibers in AcOH pulp were chemically cut short or damaged during pulping, which might be the main reason the fibers of AcOH pulp were weak. In contrast, no chemical damage of fibers was found, and there were only a few nonfiber cells (e.g., epidermal cells) in the soda-AQ pulp, as seen microscopically (photographs not shown here).

Conclusions

Wheat straw was successfully pulped by an atmospheric acetic acid process. During the pulping the wheat straw was fractionated into pulp (cellulose), acetic acid lignin, and sugars (monosaccharides from hemicellulose).

The dosage of H_2SO_4 as catalyst was the most notable pulping condition. The extent and rate of delignification could be controlled by varying the amount of the catalyst.

Acetic acid pulp was different from soda-AQ pulp in terms of strength, chemical composition, and fiber morphology. About 70% of the ash or 90% of the silica in wheat straw was kept in AcOH pulp. The ash remaining in the pulp might function as filler and be beneficial to the printability of paper. As seen microscopically, many epidermal cells existed in AcOH pulp in bundles or an single cells. These ash-rich nonfiber cells seemed to hinder bonding between fibers. AcOH pulp had strength properties lower than those of soda-AQ pulp based on the results of microscopic observations and the viscosity of bleached AcOH pulp, which may result mainly from the chemical damage to fibers, not from the depolymerization of cellulose.

AcOH pulp could be easily fullybleached by a short ECF sequence. Bleached pulp had an ash content as high as 14.2% and good opacity.

To overcome the shortcoming (low strength) of AcOH pulp and make full use of its high opacity due to the high content of ash, it may be better to proportion the AcOH pulp and other pulp with high strength, such as kraft pulp, to make paper with satisfactory physical properties. Other than paper, AcOH pulp could be also used as the raw material of cellulose derivatives.

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