Chemi-Mechanical Pulping of Rice Straw (I) Liquid-Phase Cooking*

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[Summary]

The main objective of this study is to explore the feasibility of usingan abundant agricultural residue in Taiwan, rice straw, as raw material in chemi-mechanical pulping to produce pulps of adequate quality. The experimental work involved liquid-phase cooking of the straw materials, followed by refining and evaluation of pulp properties.

The parameters which affected the cooking outcomes were analyzed using a factorial experimental design consisting of 3 variables at 2 levels, and the correlations between the dependent and independent variables were thus established. Liquid phase cooking using 10% total sulfur dioxide and a liquor pH of 10.5 at 150℃ for 22.5 min gave a pulp with about 66% yield and when beaten to 200 mL CSF, produced handsheets of adequate strength properties.

Key words: rice straw (*Oriza sativa* L.), chemi-mechanical pulping, liquid phase pulping.

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稻草化學機械法製漿(I)液態蒸煮之研究

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摘 要

本計畫之主要目標係探討台灣產量頗大，惟利用量不大的農業殘留物質，稻草，以化學機械法製造具適當性質紙漿之可行性。試驗包括稻草之液態蒸煮，繼以鍊漿；並評估紙漿性質。影響蒸煮結果之參數均以一含三變數二階層之階乘試驗設計分析以建立應變數與自變數間之關係。以10%總二氧化硫量及pH 10.5之蒸煮液於150℃液態蒸煮22.5 min，紙漿得率約66%。游離度調整爲200 mL CSF之漿料具有良好之手抄紙強度。

關鍵詞：稻草、化學機械法製漿、液態蒸煮。

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INTRODUCTION

Consumption of papers and boards in a society generally keeps pace with the living standard. Statistics suggest that the per capita paper consumption in Taiwan has markedly increased; it now exceeds 213 kg per annum and ranks 7th in the world. This corresponds to an apparent total consumption of 4.55 million metric tons. However, domestic pulp production, a mere 0.3 million tons, is far from meeting the demands for paper and board (TPIA, 1996). Therefore, Taiwan relies heavily on imported pulp to make up the shortfall. A major part of the problem is due to raw material shortages, and so promoting the use of locally produced non-wood fiber sources offers hope for lessening such reliance and lifts some restrictions on domestic pulp production. At the same time, a more environmentally benign pulping process could be developed for use with such raw material.

In order to rectify the raw material shortage, some projects have been undertaken such as the planting of fast-growing trees and the utilization of recycled papers (Ku, 1988; 1989). Nevertheless, the long-term prospect for fibrous raw material supply is cloudy and uncertain because: 1) the environmental movement has attempted to curtail tree cutting and reduce future log supply from SE Asian sources; and 2) although wastepaper accounts for about 75% of the total fiber sources in Taiwan, a large portion of these imports, particularly those from the United States and Canada, may very well dry up as the governments of these countries impose severe laws demanding that a substantial quantity of recycled fibers must be used for paper furnish. For example, in California, the law requires that newsprint furnish has to incorporate 90% recycled fibers by the year 2000. This apparently bodes ill for the future fiber supply for us. Faced with this frustrating situation, active exploration of new sources of paper fiber is essential.

A potential partial solution lies with the use of nderutilized non-wood fibers and the incorporation of advanced pulping technology. As the paper industry in Taiwan already possesses the knowledge and techniques for pulping certain non-wood fibers, such as bagasse and bamboo, it is worthwhile to expand the sphere to include other agricultural residues.

Vast quantities of agricultural residues are available in Taiwan, notably rice straw that can be harvested at least twice a year and which has an annual production on the order of 2.3 million metric tons (TPAAF, 1992). Therefore, it is imperative to investigate how vast amounts of this renewable resource can be used in papermaking and thus adequately maximize its return.

Pulping of rice straw has been conventionally conducted using soda, sulfate, lime, or lime-soda processes. These processes generally yield less than 45% pulp. In the late 1980s, there was a surge of interest in chemi-mechanical (CM) pulping of wood (Ayroud, 1985), and this interest naturally extended also to rice straw in an attempt to produce high-yield pulps (El-Tarabousi and Hurter, 1986, Ray *et al.*, 1991). Indeed, the CM pulping of rice straw can be economically implemented, provided a proper pulping technique is used. If pulping capacity can be increased through the use of high-yield processes while maintaining good pulp properties, rice straw pulp may well have the potential of replacing a substantial portion of wood pulps as a furnish component for newsprint. This would boost the future use of rice straw tremendously.

CM pulping of wheat straw and reed has also been carried out, involving advanced vapor-phase cooking with spent impregnation liquor recycling. Auspicious results have been reported for these materials (Tang and Chen, 1990, 1991; Chen *et al.*, 1992: Lo *et al.*, 1993). Based on those results, it is reasonable to expect that such a process can be applied to rice straw as well. Nevertheless, as each species may exhibit specific characteristics that respond differently to pulping conditions, work needs to be done to find the optimal cooking conditions. This effort, in turn, provides useful data to the industry. The present study is striving toward this objective.

MATERIALS AND METHODS

Materials

Two lots of rice straw (*Oriza sativa* L.) were procured separately from 2 villages in Changhwa County, one from Yen-Pu (YP) and the other from Tien-Chung (TC). Both lots were collected immediately after harvest. Straws were nearly air dried when harvested. The air-dried straw was manually chopped into sections of 3-5 cm in length, followed by vibrate screening to remove the chaff, fines, and dust. Clean straw segments were then stored in plastic bags until cooking.

Technical grades of sodium metabisulfite ($Na_2S_2O_5$) and sodium hydroxide (NaOH) were used for preparing the cooking liquors.

Analysis of straw

Intact straw was used for chemical analysis. The leaves of straw samples were manually separated from stems. Each part, as well as the whole straw was ground into fragments. The straw meal portion passing a 40-mesh screen but retained on a 60-mesh screen was collected. The materials were tested in accordance with TAPPI standard methods for 1) cold water and hot water solubles; 2) 1% NaOH solubles; 3) dichloromethane extractables; 4) Klason lignin content; 5) ash content; and 6) metals content in acid-soluble ash as determined by atomic absorption spectroscopy.

Fiber dimensions were measured with a microscope following maceration. Maceration treatment of the straw used a maceration solution containing equal volumes of glacial acetic acid and 35% hydrogen peroxide. The treatment was carried out at boiling temperature with reflux. It took about 6 h to ensure good fiber separation.

Liquid-phase cooking

A laboratory digester system (M/K model 409) was used to cook the straw samples. The M/K cooking system consisted of 2 identical digesters of 6.5 L capacity each. Each digester was equipped with a liquor circulation pump, an external heater, a temperature control circuit, and a liquor sampling valve. The liquor sampling valve was located at the bottom of the digester and connected to a circulating cold water condenser to cool down the withdrawn liquor to ambient temperature, thus reventing any change in liquor composition due to evaporation.

Fresh cooking liquor was prepared by dissolving chemicals in water. Prior to each cooking, the pH of the liquor was adjusted to the desired value with a concentrated solution of NaOH (ca. 30%).

Like most non-wood plants, rice straw has a bulky structure and the digester could hold at most about 425 g (o.d.) of straw. A high liquor to straw ratio (w/w) of 7.5 was thus employed to ensure a good impregnation and liquor circulation. The cooking conditions were established based on this consideration and according to the literature as follows.

Straw weight (o.d.)	425 g
Liquor/straw ratio (w/w)	7.5:1
Impregnation temperature	100℃
Impregnation time	30 min
Time to impreg. temperature (100℃)	20 min

The cooking variables were: total sulfur dioxide charge; liquor pH; maximum cooking temperature; and time at temperature.

During each cook, chopped straw was tightly packed into the digester, and fresh cooking liquor was then poured in. A small screen basket containing 25 g o.d. straw was placed in the middle of the digester, its contents served to determine the yield. The system was heated up to 100℃ and kept at this temperature for 30 min to ensure that the air-dried straw was well impregnated with liquor. Then the system was heated further to a predetermined cooking temperature and maintained there for a prechosen duration. At the end of the cook, spent liquor was quickly withdrawn, and the straw was immediately taken out of the digesters and soaked in cold water. After washing with tap water, the straw was subjected to defibration and refining.

Defibration and refining

Cooked straw was first defibered in a 12" Sprout-

Waldron disc refiner equipped with a rotor and a stationary disc, using a gap of 0.25 mm between the 2 plates. After defibration, the coarse pulp was thoroughly washed in a centrifugal extractor until the discharged water was clear. The pulp was further refined at 2% consistency to several freeness levels with a blender. The use of a blender instead of a PFI mill was justified because: 1) a blender was far more convenient and rapid to operate than a PFI mill; and 2) the suitability of using a blender for CM pulp refining has been well demonstrated (Shaw, 1984; Tang, 1992).

Pulp evaluation

Handsheet forming and evaluation of pulp properties (including bulk, tensile strength, tear index, burst index and brightness) were carried out according to TAPPI Standard Test Methods. Pulps were evaluated at 3 or 4 freeness levels; the results presented in this report refer to those interpolated to 200 mL CSF.

RESULTS AND DISCUSSION

Characteristics of rice straw

Both rice straw source materials used in this study consisted of approximately 75% leaves and 25% stems. The results of chemical analyses are summarized in Table 1.

Table 1 suggests that, in contrast with wood, a rather high proportion of the rice straw is soluble in cold water, amounting to 13.5% and 15% for the YP and TC samples, respectively. Also, one-fourth of the YP and one-third of the TC stems could be extracted with hot water. Since stems constituted only about 25% of the plant, and hot-water solubility of the leaf was less than 14%, the overall whole straw hot-water solublility dropped to 16.5% in the YP and 17.6% in the TC samples. These values suggest that the ceiling of pulping yield would be less than 85%, even through a purely mechanical pulping process. Knowing the high solubility of rice straw in water, it is not surprising to find that one-half of the plant mass can be dissolved in a 1% NaOH solution, conforming to the low pulp yield through the traditional chemical pulping processes.

Lignin content was higher in the TC straw than in the YP straw. There were higher contents of lignin in leaves than in stems.

Ash content of rice straw is much higher than any known fibrous plants. As shown in Table 1, ash content was generally higher in leaves than in stems; and the YP samples had more ash than the TC samples. As to the acid-insoluble ash, *i.e.*, mostly silicates, the contents in YP and TC straws were similar, accounting for 7.6 and 7.1%, respectively.

Table 2 shows the fiber dimensions. The average fiber length of rice straw was about 0.93 mm, with a minimum of approximately 0.5 mm and a maximum of 2.5 mm. Though this fiber is much shorter than that of softwoods, its high slenderness ratio of about 145 could be advantageous in sheet formation and smoothness.

A number of metal contents were determined from the acid-soluble ash and are reported in Table 3. There were very little heavy metals, most were alkaline and alkaline earth metals, *i.e.*, sodium, calcium, magnesium, and potassium. The latter was the most abundant, besides silica, amounting to 2.5% of the o.d. weight.

Liquid phase cooking

A series of liquid phase cookings were carried out with varying liquor pH, total sulfur dioxide charge, temperature, and time. The cooking conditions and pulp yields are presented in Table 4.

In the blank cook, L-0, the straw was cooked without any chemicals and the yield of 80.4% suggests that at 150℃, about 20% of the straw would be dissolved in water, thus setting a cap in yield. The cooks L-1, L-2, and L-3 with 10% total sulfur dioxide were conducted at different liquor pH, to test the effect of pH on cooking yield. In conformance with other studies (Tang and Chen, 1990; Chen *et al.*, 1992; Lo *et al.*, 1993), delignification of straw material was favored in neutral and alkaline media. A change of pH from 3.5 to 7 caused roughly a 10% loss in yield; a further increase of alkalinity to pH 12 lowered yield another

Table 1. Chemical analysis of the rice straw

Source	Yen-Pu (YP)			Tien-Chung (TC)		
% O.d. straw	Leaf	Stem	Whole	Leaf	Stem	Whole
Cold-water solubles	11.67	22.48	13.46	9.15	31.78	14.96
Hot-water solubles	13.59	26.36	16.48	11.59	33.61	17.58
1% NaOH extractives	48.30	50.30	50.10	46.80	54.40	50.10
DCM[1])extractives	3.28	2.14	2.72	3.66	1.68	3.01
Klason lignin (based on DCM-extracted samples)						
Ash included	19.40	11.25	17.04	22.77	15.93	20.36
Ash corrected	12.67	8.86	11.46	15. 61	13.12	15.11
Acid-soluble lignin	2.78	2.12	2.61	2.44	2.04	2.31
Ash content						
Total ash	13.43	9.82	13.20	11.72	8.99	11.51
Acid-insoluble	6.31	—	7.65	8.91	3.66	7.09

[1]) DCM: Dichloromethane.

Table 2. Fiber dimensions of straw samples

Source	Yen-Pu (YP)			Tien-Chung (TC)		
	Leaf	Stem	Whole	Leaf	Stem	Whole
Length, L (mm)	0.95	0.97	0.95	0.93	0.91	0.93
Width, W (μ m)	6.68	6.55	6.59	6.32	6.26	6.38
Slenderness, L/W	142	148	144	144	144	146

Table 3. Acid-soluble metals in rice straw samples

Metal oxide	K	Na	Ca	Mg	Fe	Mn
TC whole straw						
Soluble ash = 4.81%						
% of soluble ash	56.1	5.69	6.74	6.95	UD[1])	0.94
% of straw	2.70	0.27	0.31	0.33	UD	.045
YP whole straw						
Soluble ash = 4.84%						
% of soluble ash	51.2	3.62	10.2	8.65	UD	0.46
% of straw	2.48	0.18	0.49	0.42	UD	0.022
YP straw stem						
Soluble ash = 6.66%						
% of soluble ash	67.9	0.6	2.42	5.17	.005	0.22
% of straw	4.52	0.04	0.16	0.34	UD	.015

[1]) UD: Undetectable.

1%.Using liquor at pH 7 or 12 apparently made little difference in yield. However, pulp obtained at pH 7 displayed very slow drainage. Cooking in an alkaline medium was therefore adopted for further studies Increases in total sulfur dioxide charge, liquor pH, and temperature all led to lower yields. Both the YP and TC samples showed similar responses to the cooking variables..

Analysis method

The main effects of each variable, as well as the interactive effects among the variables, on pulp yield and properties were studied with a 2^3 factorial design (3 variables at 2 levels). Based on the experimental results, the intervals of the variables were chosen as follows: 1) total sulfur dioxide: 8 and 12%; 2) liquor pH: 9 and 12; and 3) time at maximum cooking temperature: 15 and 30 min. The fixed conditions were cooking temperature of 150℃; and time for impregnation temperature to maximum temperature of 20 min. Fig. 1 shows the

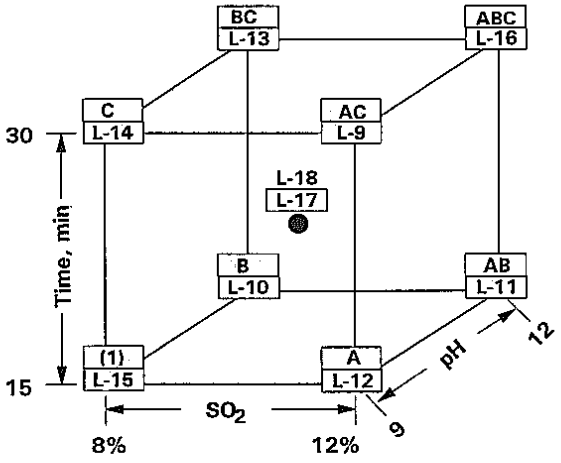


Fig. 1. Randomized layout of the 3-variable at 2-level factorial design, with A: sulfur dioxide charge; B: pH; and C: cooking time.

factorial experiments schematically. Table 5 summarizes the operation conditions and cooking results, and Table 6 gives the physical properties of the resulting pulps. The results are also presented in Figs. 2 to 5 in a format corresponding to the experimental design.

The method of ANOVA (analysis of variance) (Montgomery, 1984) was used to examine the effects of the 3 cooking variables. Only the parameters which resulted in a F-value corresponding to 10% significance level were retained to establish the correlations. The values of the independent variables, as appearing in the correlations, are values normalized toward averages of zero and can be converted to the actual values by the following equations:

x_1 (sulfur dioxide charge) = (% SO_2 - 10)/2 (%)

x_2 (liquor pH)= (pH - 10.5)/1.5

x_3 (cooking time at-temp.) = (Time - 22.5)/7.5 (min)

Thus, the normalized values can be either positive or negative, depending on the levels of the treatments with respect to the central point in the experimental design. The constant in the correlation equation reflects the result of an experiment with the central point conditions. The coefficient of each term in the equation represents the magnitude of the effect of this term on the response.

Cooking yield

Liquid phase cooking of the straw resulted in yields from 60 to 72%, depending on the cooking conditions. As the following equation suggests, an increase in any of the independent variable values will result in a lowering of pulp yield (Fig. 2).

Yield, % = $66.1 - 2.33(x_1) - 1.57(x_2) - 2.23(x_3) + 1.6(x_1)(x_3)$ [1]

with a correlation coefficient R = 0.98. Eq. [1] indicates a more pronounced effect caused by either the

Table 4. Liquid-phase cooking of straw samples

Exp. no.:	L-0	L-1	L-2	L-3	L-4	L-5	L-6	L-7	L-8
Source:	YP	YP	YP	YP	YP	YP	TC	TC	TC
Total SO_2									
Initial vs. straw, %	0	10	10	10	12	8	12	8	10
Residual vs. initial, %		81			68.3	59.9	54.1	59.1	61.9
Residual vs. straw, %					8.2	4.8	6.5	4.7	6.1
pH: Initial		3.5	7.0	12.0	12.0	9.0	12.0	9.0	10.5
Final		5.1	6.4	7.1	7.7	6.8	7.1	6.8	6.9
Temp.: Impregnation, ℃	100	100	100	100	100	100	100	100	100
Maximal, ℃	150	150	150	150	160	155	160	150	155
Time: Impregnation, min	30	30	30	30	30	30	30	30	30
Cooking, min	20	20	20	20	30	20	30	20	25
Yield, %	80.4	76.8	66.9	65.8	62.1	72.8	57.4	71.9	65.2
Ash content: % of pulp				11.2	14.4	12.5			
% of straw				7.26	8.97	9.10			

Note : Fixed conditions for all the experiments were: 425 g of strawper cook (25 g for yield determinations); and liquor to straw ratio of 7.5. L- : liquid-phase cooking; YP: Yen-pu; TC: Tien-chung.

Table 5. Liquid-phase cooking of rice straw following a 3-variable at 2-level factorial design

Exp. no.:	L-9	L-10	L-11	L-12	L-13	L-14	L-15	L-16	L-17	L-18
Source:	TC	TC	TC	TC	TC	TC	TC	TC	TC	TC
Total SO_2										
Initial *vs.* straw, %	12	8	12	12	8	8	8	12	10	10
Residual *vs.* initial, %	65.9	60.7	67.8	62.6	59.1	53.8	60.3	57.6	58.7	58.7
Residual *vs.* straw, %	7.90	4.85	8.14	9.90	4.73	4.31	4.82	6.91	5.87	5.87
pH: Initial	9.0	12.0	12.0	9.0	12.0	9.0	9.0	12.0	10.5	10.5
Final	7.1	7.1	7.3	7.2	7.4	6.7	6.7	7.3	6.9	6.9
Time: Impregnation, min	30	30	30	30	30	30	30	30	30	30
Cooking, min	30	15	15	15	30	30	30	15	22.5	22.5
Yield, %	64.8	71.6	62.5	67.3	63.7	66.6	72.2	60.7	65.6	66.1

Note: The fixed conditions for all the experiments were: 425 g of straw percook; liquor to straw ratio was 7.5; impregnation temp. of 100℃ andmaximal cooking temp. of 150℃.
L- : liquid-phase cooking; TC: Tien-chung

Table 6. Pulp properties of liquid-phase cooked rice straw followinga 3-variable at 2-level factorial design

Exp. no	Bulk cm^3/g	Breaking length km	Burst index $kPa \cdot m^2/g$	Tear index $mN \cdot m^2/g$	Brightness %, ISO
L-9	1.95	5.54	3.36	7.21	26.5
L-10	2.02	4.55	2.61	7.57	24.7
L-11	1.89	5.61	4.18	6.31	25.8
L-12	2.07	4.28	3.60	8.58	23.4
L-13	1.82	5.28	3.88	6.59	27.7
L-14	1.80	4.16	2.69	7.18	24.5
L-15	2.16	4.12	3.11	8.53	23.1
L-16	1.79	5.96	3.95	6.30	28.8
L-17	1.87	5.39	3.20	7.21	25.5
L-18	1.94	5.20	3.32	7.69	25.5

total sulfur dioxide charge or the cooking time than by the liquor pH. An interaction between sulfur dioxide charge and cooking time also has a notable influence on pulp yield as expressed by the last term of the equation

Pulp properties

In general, a shift of cooking variables from low to high levels could result in improvements of the pulp strength properties, with the exception of the tear index (Fig. 3), sheet conformation (in terms of bulk, Fig. 4) and pulp brightness (Fig. 4). After being beaten to 200 mL CSF, the pulps obtained were generally comparable with high-yield straw pulps of another study (Ren *et al.*, 1988).

The strength properties, i.e., tensile breaking length, BL; tear index, TI; and burst index, BI; and the cooking variables can be related through the following equations:

$BL, in\ km = 5.01+0.41(x_1) + 0.41(x_2) + 0.3(x_3) - 0.2(x_1)(x_3);$
$R = 0.96$ [2]

$TI, in\ mN \cdot m^2/g = 7.31 - 0.18(x_1) - 0.59(x_2) - 0.46(x_3) + 0.22(x_2)(x_3);$ $R = 0.93$ [3]

$BI, in\ kPa \cdot m^2/g = 3.39+0.35(x_1)+ 0.23(x_2) - 0.17(x_1)(x_3)+ 0.21(x_2)(x_3);$ $R = 0.90$ [4]

From Eqs. 2-4, it is obvious that the breaking length would be improved by augmenting any one of the 3 cooking variables; while the tear index would be reduced correspondingly, a well-known relationship between these two strength properties. Eq. 4 shows that there is no 1st-order term of x_3, suggesting that cooking time has no direct bearing or significant main effect on bursting strength. However, its interaction with charge and pH would have certain effects on this property.

As for pulp bulk, the total sulfur dioxide charge appears to have no main effect but manifests its effect by interacting with either the liquor pH or the cooking time:

$Bulk, cm^3/g = 1.93 - 0.058(x_2) - 0.098(x_3) - 0.029(x_1)(x_2) + 0.043(x_1)(x_3);$ $R = 0.95$ [5]

All pulps obtained displayed a dark brown color, hence a low level of brightness as shown in Fig. 4. There

is a report of rice straw soda pulps with yields of 40.4 to 44.4% having brightness in the range of 31 to 33.4% GE (Kuo and Shen, 1992). The low brightness values might be attributed to the high residual lignin content in pulps with high content of chromophoric groups. The following equation indicates the relationship between brightness and cooking variables, showing that cooking time has the most pronounced effect.

Brightness, % = $25.6 + 0.56(x_1) + 1.19(x_2) + 1.31(x_3)$;
$R = 0.96$ [6]

Spent liquor

Fig. 5 provides information concerning spent liquor pH and residual sulfur dioxide. As a result of the hydrolysis of carbohydrates and the formation of lignosulfonic acid, liquor pH dropped to about 7 in all cases. As for the residual sulfur dioxide, it is interesting to note that about 60% of the initial charge remained in the spent liquor for most of the experiment, except in the case where both the initial sulfur dioxide charge and liquor pH were low and the cooking time was long. In other words, if cookings are to be conducted under

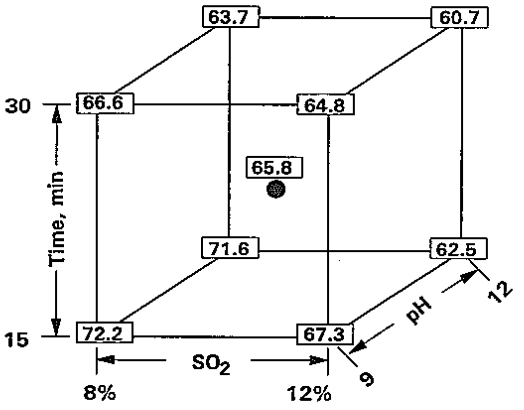
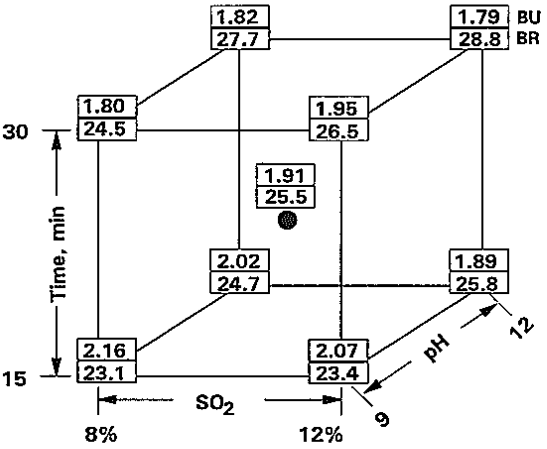
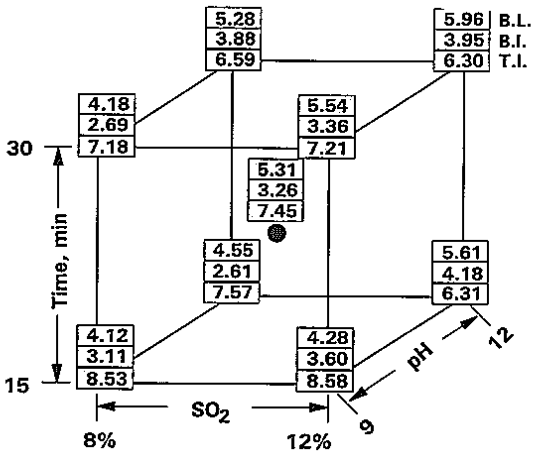


Fig. 2. Yields of rice straw pulps following liquid-phase cookings.



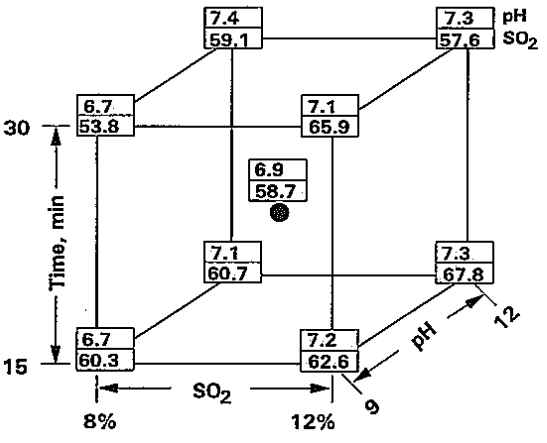
BU: bulk, cm^3/g
BR: brightness, % ISO

Fig. 4. Bulk and brightness of rice straw pulps following liquid-phase cookings.



B.L.: breaking length, km
B.I.: burst index, $kPa.m^2/g$
T.I.: tear index, $mN.m^2/g$

Fig. 3. Stregnth properties of rice straw pulps following liquid-phase cookings.



pH : pH after cooking
SO_2: % of initial charge

Fig. 5. Residual sulfur dioxide charges and pH of spent liquor of liquid-phase cookings.

optimal conditions, only about 40% of the initially charged total sulfur dioxide would be consumed during delignification, rendering the recycling of the spent cooking liquor economically significant.

CONCLUSIONS

The study results demonstrate the feasibility of using rice straw to produce chemi-mechanical pulp of acceptable quality with yields ranging from 60 to 70%. The advantage of the high slenderness ratio of rice straw fibers can produce paper with improved comformation and surface smoothness.

With liquid-phase cooking at 150℃ for 22.5 min, using a liquor pH of 10.5 and 10% total sulfur dioxide charge, the pulp yield was about 66%; and at 200 mLCSF, the handsheets from the pulp possess a bulk of 1.91 cm^3/g; breaking length of 5.31 km; bursting index of 3.26 $kPa \cdot m^2/g$; tear index of 7.45 $mN \cdot m^2/g$; and brightness of 25.5% ISO.

Since the potassium content in rice straw is only about 2.5%, it is not adviseable to burn the straw in the field for fertilizer also creating a severe air pollution problem. At a yield of 60% or more, rice straw should be utilized in papermaking instead.

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