Influence of cooking variables in the organosolv pulping of wheat straw using mixtures of ethanol, acetone, and water

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ABSTRACT: In studying the organosolv pulping of wheat straw with mixtures of ethanol, acetone, and water, the researchers examined the influence of independent variables on the properties of the pulp and of paper sheets made from it. Equations were used to relate the dependent variables to the independent variables. To minimize solvent losses and to increase pulp yield, wheat straw can be cooked at 180ºC for 60 min using a solution of ethanol mixed at 50% with ethanol plus acetone and using a solution of ethanol plus acetone mixed at 35% with a solution of ethanol, acetone, and water. Under these conditions, the properties of the pulp and paper sheets (except for burst index) differ by less than 22% from their optimum levels. Among ethanol–acetone, ethanol, and acetone pulping of wheat straw, acetone pulping produces the best pulp and the best paper sheets. However, it takes a higher concentration of solvent and higher pressure than ethanol–acetone pulping takes.

Application: Acetone and ethanol pulping can be used effectively to produce quality pulp and paper sheets from wheat straw.

Sulfite- and sulfate-based pulping processes produce large amounts of wastewater that are potentially harmful to the environment. One way to circumvent this shortcoming is to remove fiber and lignin with suitable organic solvents. Although such solvents have long been known to be effective, they have not yet been used on an industrial scale [1-10]. Organosolv processes have been applied with varying degrees of success to hardwood and softwood and, to a lesser extent, to nonwood materials such as wheat straw [11-21]. Alternative, nonwood materials are important, however, because a shortage of woody raw materials is leading to gradual deforestation in some areas in the world.

Pulping processes have been studied in the light of various models, with a view to deriving equations for predicting the quality of the resulting pulp in terms of process variables and optimized operating conditions. Most of these models are mathematical designs based on the kinetics of delignification, and they are used to predict the extent to which delignification is likely to occur.

Few researchers, however, have used a factor design to develop empirical models that involve several independent variables in processes for pulping plant materials. Empirical models are preferred over theoretical ones because theoretical models are rather complex when more than two independent variables are involved. Although organosolv pulping has been studied in this respect [15-27], no one has considered the effect of the operating variables on the physical properties of the paper sheets obtained from the pulp.

For this reason, in our study of the organosolv pulping of wheat straw with mixtures of ethanol, acetone, and water, we used a central composite design to examine the influence of independent cooking variables on the pulp yield, on the content of holocellulose, α-cellulose, and lignin, and on various properties of paper sheets obtained. Our purpose was to establish the optimum operating conditions for the process.

MATERIAL AND METHODS

Raw materials

We used sun-dried wheat straw (*Triticum vulgaris*) throughout this study. The composition of sun-dried wheat straw was determined to be 70.30% holocellulose, 17.73% lignin, 6.84% ash, 4.23% ethanol/benzene extractables, and 0.90% others.

Analysis of raw materials, pulps and paper sheets

The starting materials and the products obtained from them were characterized according to the following standard methods:

- Holocellulose: Wise et al. [28]
- α-cellulose: TAPPI 203 0S-61
- Lignin: TAPPI 222
- Ash: TAPPI 211
- Ethanol and benzene extractables: TAPPI 204
- Breaking length and stretch: UNE 57054
- Burst index: UNE 57058-74
- Tear index: UNE 57033
- Brightness: UNE 57062.

Pulping and sheetmaking

We pulped wheat straw using a 15 L cylindrical batch reactor heated by electrical wires. The control unit for this reactor has instruments for monitoring pressure and temperature.

The wheat straw cooked in the reactor was defibered in a wet disintegrator, and the screenings were separated by sieving through a screen of 1 mm mesh. Paper sheets were prepared with an ENJO-F-39.71 sheet machine in accordance with UNE 57042-74.

Experimental design

The tested model uses a series of points around a central point and several additional points, where every point represents an experiment, to estimate the first- and second-order interaction terms of a polynomial. This design meets the general requirement that every parameter in the mathematical model can be estimated from a fairly small number of experiments [29].

Suppose we have four independent variables, which are temperature T, cooking time t, the ratio E/D of ethanol (E) to

PROPERTY	VARIABLE IN EQUATIONS	SNEDECOR'S F	r^2	HIGHEST p VALUE
Yield	YI	141.7	0.97	0.01
Holocellulose	HO	33.6	0.87	0.06
α-cellulose	αC	152.2	0.97	0.02
Lignin	LI	108.4	0.94	0.01
Breaking length	BL	122.5	0.84	1×10^{-10}
Stretch	ST	9.9	0.47	0.06
Burst index	BI	44.4	0.95	0.01
Tear index	TI	89.9	0.89	3×10^{-8}
Brightness	BR	4.0	0.21	0.05

I. Statistical figures of merit of the equations.

ethanol plus acetone (*D*), and the ratio *D/L* of ethanol plus acetone (*D*) to a solution of ethanol, acetone, and water (*L*). The total number of observations (or experiments) required is found from the following equation [30]:

$$N = 2^k + 2*k + 1 \qquad (1)$$

where k denotes the number of independent variables. Solving this equation for $k = 4$, we find that N is equal to 25 experiments.

Experimental data were fitted to the following second-order polynomial:

$$Z = a_o + \sum_{i=1}^{k} b_i X_{ni} + \sum_{i=1}^{k} c_i X_{ni}^2 + \sum_{i=1;\, j=1}^{k} d_{ij} X_{ni} X_{nj} \quad (i < j) \qquad (2)$$

where Z denotes the response variables, X_T, X_t, $X_{E/D}$, and $X_{D/L}$ denote the normalized values of T, t, E/D, and D/L, and a_o, b_i, c_i, and d_{ij} are constants.

In each case, the response variable was yield (YI), holocellulose (HO), α-cellulose (αC), lignin (LI), breaking length (BL), stretch (ST), burst index (BI), tear index (TI), or brightness (BR).

The values of the independent variables were normalized from –1 to +1 by using Eq. 3 to make it easier to compare the coefficients and visualize the effects of the individual independent variables on the response variable:

$$X_n = 2\,\frac{X - \overline{X}}{X_{max} - X_{min}} \qquad (3)$$

where
X_n = normalized value of T, t, E/D, or D/L;
X = absolute experimental value of the variable
$\overline{X}$ = mean of all experimental values for the variable
X_{max} = maximum value of the variable
X_{min} = minimum value of the variable.

The normalization also provides more accurate estimates of the regression coefficients because it reduces interrelationships between linear and quadratic terms [29].

The operating conditions used in each of the 25 experiments and the corresponding normalized values for the independent variables are presented in the **Appendix**.

RESULTS AND DISCUSSION

We conducted a set of four preliminary experiments under the central operating conditions. These conditions meant pulping for 90 min at 160ºC using ethanol mixed at 60% with a solution of ethanol and acetone (E/D = 60%) and using a solution of ethanol and acetone mixed at 55% with a solution of ethanol, acetone, and water (D/L = 55%). In determining the dependent variables, we obtained experimental results that differed from the mean values by less than 5%-10%. For the operating variables, we varied the temperature from 140ºC to 180ºC, the cooking time from 60 min to 120 min, the ratio *E/D* from 25% to 75%, and the ratio *D/L* from 35% to 75%. In all tests, we used a constant liquid/solid ratio of 40 to ensure thorough mixing in the reactor.

We used the BMDP software suite [30] to perform a multiple linear regression analysis involving all terms in Eq. 2 except those with Snedecor's *F* values less than 4, which were discarded using the stepwise method [31]. The following equations were obtained:

$$YI = 64.07 - 14.57\,X_T - 3.42\,X_T^2 + 3.13\,X_{(D/L)} + 2.40\,X_T X_{(D/L)} \qquad (4)$$

$$HO = 79.25 + 2.65\,X_T + 4.61\,X_T^2 - 1.09\,X_{(D/L)} + 1.11\,X_t X_{(D/L)} \qquad (5)$$

$$\alpha C = 59.30 + 12.05\,X_T + 1.27\,X_t - 2.97\,X_{(D/L)} - 2.22\,X_T X_{(D/L)} \qquad (6)$$

$$LI = 11.66 - 4.37\,X_T + 0.81\,X_{(D/L)} - 0.73\,X_T X_t \qquad (7)$$

$$BL = 1837 + 1385\,X_T \qquad (8)$$

$$ST = 0.92 + 0.13\,X_T + 0.12\,X_T^2 \qquad (9)$$

$$BI = 0.95 + 0.54\,X_T + 0.17\,X_t + 0.30\,X_T^2 - 0.27\,X_t^2 - 0.38\,X_{(E/D)}^2 + 0.10\,X_T X_{(D/L)} + 0.20\,X_t X_{(D/L)} \qquad (10)$$

$$TI = 3.88 + 0.98\,X_T - 1.56\,X_T^2 \qquad (11)$$

$$BR = 27.77 + 1.44\,X_T^2 - 1.27\,X_t^2 \qquad (12)$$

where
YI = yield, %
HO = holocellulose, %
αC = α-cellulose, %
LI = lignin, %
BL = breaking length, m
ST = stretch, %
BI = burst index, kN/g
TI = tear index, mN • m^2/g
BR = brightness, %.

Table I shows the F and r^2 values for these equations, along with the highest p values. These equations reproduce the experimental results for the different dependent variables, as a function of the independent ones, with errors smaller than those shown in **Table IIA**. (The error is the percent discrepancy that arises in estimating the dependent variables with respect to the experimental values.)

Table IIA also shows the optimum (greatest or smallest) values for the dependent variables as obtained using the steepest ascent (or descent) method [32], as well as the values of the independent variables needed to achieve them. Table IIB also shows the variation of the dependent variables with changes in each independent variable from –1 to +1 (with all other independent variables held constant) required to obtain the optimum levels for the dependent variables. In Table IIB, variations are expressed in units of the dependent variables and as percentages relative to the optimum values.

As Table IIB shows, a high temperature is needed to obtain pulp and paper sheets with the best possible values in

DEPENDENT VARIABLES	ERROR, %	OPTIMUM VALUE	NORMALIZED VALUES OF THE INDEPENDENT VARIABLES LEADING TO OPTIMUM VALUES OF THE DEPENDENT VARIABLE				NEAR OPTIMUM VALUE	VARIATION, %
			X_T	X_t	$X_{(E/D)}$	$X_{(D/L)}$		
Yield, %	10	75.95*	-1	—	—	+1	74.95	1.96
		40.55**	+1	—	—	-1	45.55	46.61
Holocellulose, %	4	88.71*	+1	-1	—	-1	88.71	—
α-cellulose, %	5	77.81*	+1	+1	—	-1	75.27	3.26
Lignin, %	15	6.75**	+1	+1	—	-1	6.75	—
							8.21	21.63
Breaking length, m	30	3222*	+1	—	—	—	3222	—
Stretch, %	25	1.17*	+1	—	—	—	1.17	—
Burst index, kN/g	30	2.02*	+1	+0.69	0	+1	2.02	—
							1.25	38.12
							1.07	47.03
Tear index, mN m²/g	25	4.04*	+0.34	—	—	—	3.40	15.84
Brightness, %	10	29.21*	-1 or +1	0	—	—	27.9	4.35

*Minimum value. **Maximum value.

IIA. Optimum (highest or lowest) values of the dependent variables and variations with changes in the independent variables in the ethanol-acetone pulping of wheat straw.

DEPENDENT VARIABLE	TEMPERATURE, *T*	COOKING TIME, *t*	*E/D*	*D/L*
Yield, %	24.33 (32.05%)	—	—	1.46 (1.96%)
	33.94 (45.56%)	—	—	11.06 (27.27%)
Holocellulose, %	9.11 (10.27%)	2.22 (2.50%)	—	4.40 (4.96%)
α-cellulose, %	28.54 (36.68%)	2.54 (3.26%)	—	10.38 (13.34%)
Lignin, %	10.20 (60.18%)	1.46 (21.63%)	—	1.62 (24.00%)
Breaking length, m	2770 (88.97%)	—	—	—
Stretch, %	0.28 (23.93%)	—	—	—
Burst index, kN/g	1.28 (63.37%)	0.77 (38.12%)	0.38 (18.81%)	0.48 (23.76%)
Tear index, mN·m²/g	2.60 (64.36%)	—	—	—
Brightness, %	1.27 (4.35%)	1.44 (4.93%)	—	—

*Percent of optimum value.

IIB. Maximum changes in the dependent variables with changes in the independent variables (from -1 to +1).

the dependent variables (with the exception of yield and tear index). Also, a long pulping time ensures a high α-cellulose content and a low lignin content. On the other hand, medium-to-long pulping leads to high burst index, a medium pulping time leads to increased brightness, and a short pulping time gives a high holocellulose content. The ratio of ethanol to (ethanol + acetone) influences the burst index, which is optimum at intermediate values of the ratio. Finally, the ratio of (ethanol + acetone) to (ethanol + acetone + water) should be kept low to obtain high holocellulose content, high α-cellulose content, and low lignin content. However, this ratio should be kept high if an elevated burst index is desired.

As the table also shows, temperature is the independent variable that most strongly influences all dependent variables except brightness.

The relative influence of the different independent variables on each dependent variable is also apparent from **Figs. 1-3**, which illustrate the situation for the burst index. (Burst index is the dependent variable related to the largest number of independent variables).

Table IIA also compares the optimum values for the dependent variables with other, slightly different values in the next to the last column. These values were obtained by using more economically favorable values of those independent variables that influence the dependent variables less markedly. The last column shows the most suitable differences (percentages) from the optimum values for the dependent variables.

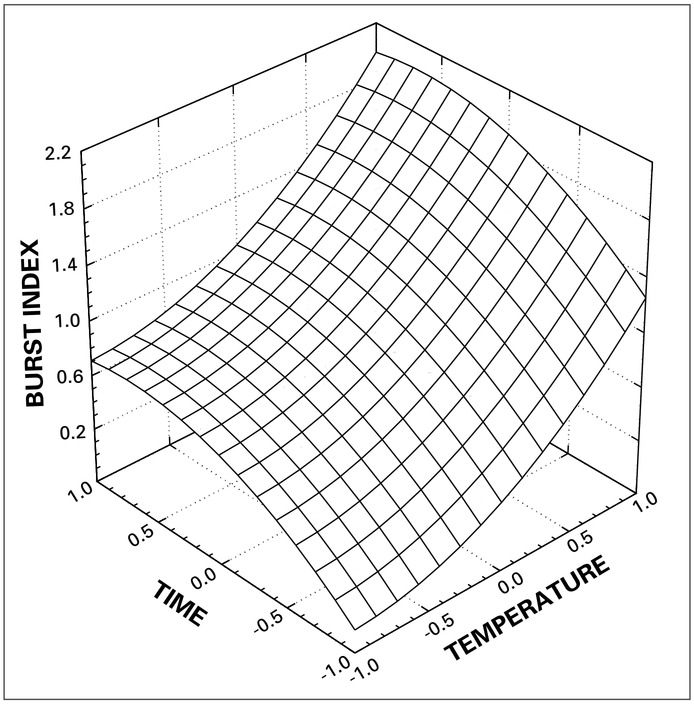
As the table shows, using a short processing time and a low ratio of *D/L*, in addition to a medium ratio of *E/D* and a high temperature, provides values of the dependent variables that differ little (less than 22%) from the optimum values (with the exception of burst index). In view of these results, it is advisable to operate under these conditions in order to save solvents and increase pulp yield, even though the burst index for the paper sheets is slightly reduced as a result.

CONCLUSIONS

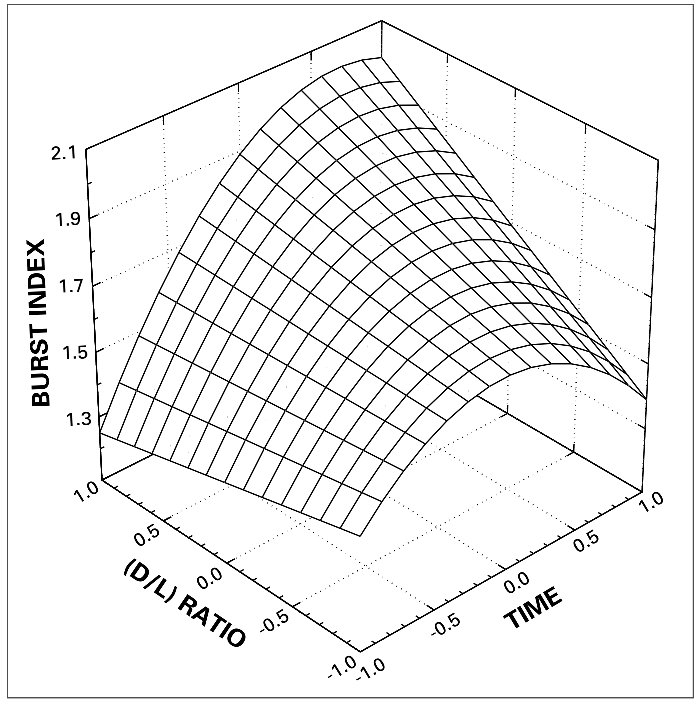
Both ethanol pulping [17, 33] and acetone pulping [18, 33] of wheat straw are best conducted at a high temperature (180ºC), using a short processing time (60 min) and a low concentration of organic solvent (ethanol or acetone, 40%). Under these conditions, most dependent variables exhibit values that differ by less than 10%-20% from their optimum levels. Furthermore, the amount of solvent needed is greatly reduced, and the yield is substantially increased.

Table III shows the values for the properties of the pulp (yield, holocellulose content, α-cellulose content, and lignin content) and of the paper sheets obtained by using a high temperature of 180ºC, a short pulping time of 60 min, a low solvent (ethanol or acetone) concentration of 40%, a medium *E/D* ratio of 50%, and a low *D/L* ratio of 35%.

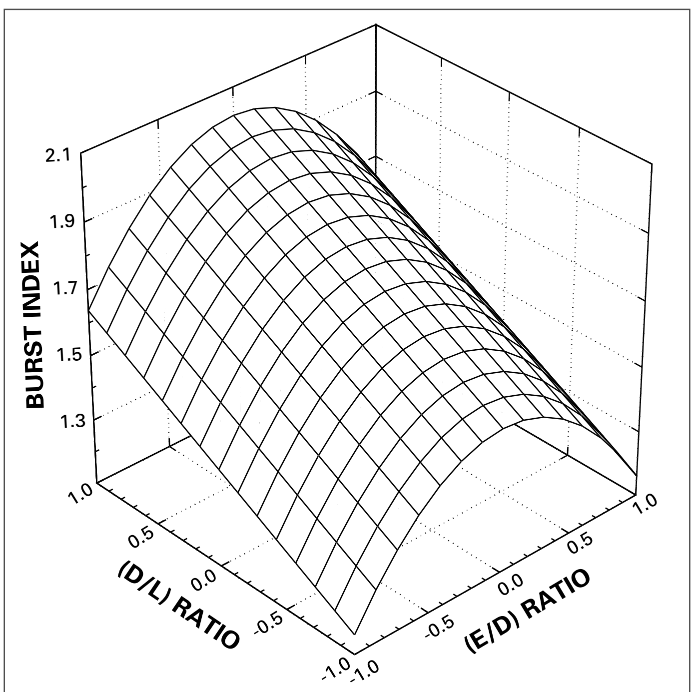
As shown, acetone pulping provides higher yield, breaking length, burst index, and tear index values and also lower lignin contents than do ethanol



1. Variation of the burst index with the temperature and cooking time at a constant E/D ratio of 50% and a constant D/L ratio of 75%.



2. Variation of the burst index with the cooking time and D/L ratio at a constant E/D ratio of 50% and a constant temperature of 180°C.



3. Variation of the burst index with the E/D and D/L ratios at a constant temperature of 180°C and a constant cooking time of 111 min.

pulping and ethanol-acetone pulping.

The yield, holocellulose, and brightness achieved with ethanol-acetone pulping are similar to those obtained with acetone pulping. The values for lignin content and breaking length obtained with ethanol-acetone pulping lie between those obtained with ethanol pulping and acetone pulping. Finally, the stretch, burst index, and tear index are all lower than those obtained by pulping with either solvent alone.

It follows that acetone pulping is the most effective choice, even though it uses a higher solvent concentration than ethanol–acetone pulping (40% vs. 35%) and an also higher pressure (18×10^5 Pa vs. 14×10^5 Pa). **TJ**

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DEPENDENT VARIABLE	ETHANOL PULPING	ACETONE PULPING	ETHANOL-ACETONE PULPING
Yield, %	41.77	45.86	45.55
Holocellulose, %	88.13	88.27	88.71
α-cellulose, %	77.59	77.15	75.27
Lignin, %	9.24	7.77	8.21
Breaking length, m	3000	3456	3222
Stretch, %	1.42	1.42	1.17
Burst index, kN/g	1.27	1.36	1.07
Tear index, $mN\cdot m^2/g$	3.76	3.86	3.40
Brightness, %	28.68	27.71	27.94

III. Comparison of the performance of ethanol, acetone, and ethanol-acetone pulping, each at 180ºC for 60 min with a low solvent concentration (40%), a medium E/D ratio of 50%, and a low D/L ratio of 35%.

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INSIGHTS FROM THE AUTHORS

Wheat straw is an agricultural residue produced in massive amounts in some Spanish regions such as Andalusia. As an alternative raw material for pulping, wheat straw needs to be studied to determine the best pulping conditions to achieve the best sheet properties. We studied the use of ethanol–acetone mixtures for pulping wheat straw and the influence of operating conditions on the properties of the paper sheets.

Ethanol-acetone mixtures had not been studied before for pulping wheat straw. The greatest challenge we faced was to minimize the time required to reach the operating temperature. We overcame this difficulty by pre-heating the reactor, raw materials, and reagents used.

The process provides paper sheets with good properties in a fairly short cooking time using relatively small amounts of ethanol and acetone. Mills can make use of this research because cooking wheat straw with organic solvents provides pulp of high enough quality.

The next step for us is to replicate our experiments in a pilot plant.

—Luis Jiménez

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Appendix. Operating conditions used in the ethanol–acetone pulping of wheat straw and experimental results obtained in the characterization of the pulp and the resulting paper sheets

X_T	X_t	$X_{(E/D)}$	$X_{(D/L)}$	YI, %	Ho, %	αC, %	LI, %	BL,m	ST,%	BI, kN/g	TI, mNm^2/g	BR, %
+1	+1	+1	+1	46.61	85.94	67.53	9.21	3833	1.41	1.54	3.66	27.37
+1	- 1	+1	+1	54.40	83.03	64.82	10.54	2729	1.17	1.02	3.65	26.85
- 1	+1	+1	+1	74.24	82.72	47.61	16.55	362	0.96	0.32	1.70	27.27
- 1	- 1	+1	+1	74.69	81.97	46.97	16.15	346	0.99	-	1.56	27.76
+1	+1	+1	- 1	40.65	89.07	77.51	6.69	2430	1.14	0.92	2.73	29.81
+1	- 1	+1	- 1	40.18	88.19	77.24	7.23	3033	1.13	1.25	3.40	27.70
- 1	+1	+1	- 1	71.38	79.40	49.34	17.31	469	0.88	0.17	1.68	27.66
- 1	- 1	+1	- 1	72.90	80.25	45.77	16.57	358	0.76	0.13	1.34	29.17
+1	+1	- 1	+1	50.32	88.95	70.61	7.80	3990	1.26	1.62	3.67	28.43
+1	- 1	- 1	+1	53.13	82.67	62.59	10.51	2542	0.94	0.75	2.98	25.05
- 1	+1	- 1	+1	75.51	82.42	47.68	20.60	441	1.12	0.34	1.39	28.20
- 1	- 1	- 1	+1	75.54	82.36	46.22	16.53	419	0.92	0.91	1.21	28.76
+1	+1	- 1	- 1	42.09	83.81	73.71	6.28	2465	0.98	-	2.94	26.24
+1	- 1	- 1	- 1	41.98	88.62	75.76	8.40	2849	1.13	1.05	3.38	28.33
- 1	+1	- 1	- 1	79.86	79.27	48.92	16.29	494	0.74	0.20	1.55	28.38
- 1	- 1	- 1	- 1	74.70	81.23	47.67	16.57	429	0.91	0.14	1.24	28.77
0	+1	0	0	62.79	80.24	60.49	12.44	2535	1.09	1.03	4.30	27.83
0	- 1	0	0	65.69	77.78	53.47	14.88	1529	0.77	0.48	3.14	26.54
+1	0	0	0	45.38	89.27	74.90	7.27	4745	1.39	1.88	4.19	30.59
- 1	0	0	0	78.16	81.24	47.54	16.06	409	0.93	0.76	1.29	29.21
0	0	0	+ 1	71.28	77.93	54.24	13.41	1503	0.72	1.01	4.54	26.41
0	0	0	- 1	55.65	80.38	65.81	11.34	2244	1.04	0.87	3.56	28.16
0	0	+1	0	63.91	78.89	58.08	11.99	2087	1.02	0.72	4.16	27.95
0	0	- 1	0	64.06	79.11	59.31	12.70	1735	0.91	0.56	3.52	28.50
0	0	0	0	65.09	80.43	58.65	13.20	1897	0.90	0.71	3.94	26.44

X_T = Normalized temperature; X_t = Normalized time; $X_{(E/D)}$ = Normalized ethanol/(ethanol+acetone) ratio; $X_{(D/L)}$ = Normalized (ethanol+acetone)/(ethanol+acetone+water) ratio; YI = Yield; HO = Holocellulose; αC = alpha-cellulose; LI = Lignin; BL = Breaking length;; ST = Stretch; BI = Burst index; TI = Tear index; BR = Brightness