

# Kraft pulping of olive wood trimmings: Influence of process variables

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**N**ONWOOD RAW MATERIALS account for 5–7% of worldwide pulp production. They are an excellent source of fibers for specialty grades of paper, and in some areas of the world, they are the only source of fibrous raw materials. Moreover, nonwood plants are effective substitutes for the ever-diminishing supply of forest wood. Nonwood pulp can be used alone or mixed with pulp from wood or recycled paper to produce paper, cardboard, and other lignocellulosic derivatives.

World production of nonwood pulps has grown much more rapidly than that of pulp from wood in the last two decades (by a factor of about two in Latin America and three in Africa and the Middle East). This trend is expected to continue in response to rising demand for pulp and paper products in highly populated countries that possess vast agricultural and rapid-growth plant resources (1).

Agricultural residues abound in Spain, particularly in Andalusia (southern Spain). The principal agricultural residues in Spain (olive wood trimmings, cereal straw, sunflower stalks, vine shoots, and cotton plant stalks) are produced in amounts exceeding  $16 \times 10^9$  kg/year, of which Andalusia contributes roughly 32.5% (2). With a yield of 40–50%, these residues could sustain production of paper and paperboard at a level four times greater than current production in Spain (3).

Spain generates the world's largest amount of olive wood trim-

ming, nearly  $2 \times 10^9$  kg/year (3). This raw material is highly concentrated in Andalusia, which produces more than 60% of the national figure. The wood trimmings have no specific application to date. Rather, they are typically eliminated by burning them in the field, a practice that is the root of various problems (pollution, pests, fires) that considerably increase the costs of olive growing.

Spain imported  $2.4 \times 10^9$  kg of paper and board and more than  $10^6$  cubic meters of wood in 1996. Consequently, Spanish papermakers are searching for alternative raw materials, including new eucalyptus plantations and wood trimmings from olive trees.

Only Camacho *et al.* (4, 5) appear to have examined olive wood trimmings as a raw material for pulp making. They studied the kinetics of the kraft process as applied to this material, but they did not investigate the influence of the operating conditions on the characteristics of the resulting pulp.

Pulping processes for various wood and nonwood plants are commonly analyzed using mathematical models aimed at deriving equations for estimating pulp quality in terms of process variables and at determining the most suitable operating conditions in each case. Most such models are based on delignification kinetics (specifically, on predicting to what extent delignification is bound to occur). Few, however, consider the effect of process variables on pulp quality and yield (6).

So far, few researchers have used

## ABSTRACT

A central composite design was used to investigate the influence of kraft pulping conditions (time, temperature, active alkali concentration, sulfidity, and liquid/solid ratio) on pulps from olive wood trimmings. A second-order polynomial equation consisting of these five process variables was used to develop equations that predict the response of seven key pulp properties (yield, holocellulose,  $\alpha$ -cellulose, lignin, brightness, breaking length, and burst index) over the experimental range of cooking conditions. Predictions for yield, brightness, holocellulose, and  $\alpha$ -cellulose were within 10% of the experimental measurements, while the error for predicted lignin content was below 20%. Predictions for breaking length and burst index of the pulp handsheets were within 25% of experimental values. The optimum operating conditions involve pulping at 190°C for 38 min using an alkali concentration of 10%, a sulfidity of 23.9%, and a liquid/solid ratio of 8. These conditions also produced handsheets with the highest breaking length and burst index. Regardless of the operating conditions, the pulp can and should be refined to increase the strength properties of the resulting pulp handsheets.

## Application:

A case study in the use of statistical methods to identify optimum pulping conditions and pulp properties for underutilized fiber resources.

factor design to analyze pulping processes. This technique can be used to develop empirical models involving several independent variables. The resultant model can then be used to evaluate pulp properties (e.g., chemical composition, yield, kappa number, degree of polymerization) from various plant types using different pulping methods (6–11).

These empirical models are preferable to theoretical models, which are too complex when more than two independent variables are involved.

In this work, we used a central composite design to examine the influence of kraft pulping variables (temperature, time, active alkali concentration, sulfidity, and liquid/solid ratio) on pulp properties (yield, brightness, holocellulose,  $\alpha$ -cellulose, lignin, breaking length, and burst index) obtained from olive wood trimmings.

## EXPERIMENTAL

### Raw materials

The olive wood trimmings contained 61.47% holocellulose, 35.67%  $\alpha$ -cellulose, and 19.71% lignin by dry weight. The material—free of young stalks, branches, and leaves—was sun-dried and ground to pieces 10–20 mm in length. The bark was not stripped off because it was very thin and difficult to remove.

### Characterization of raw materials, pulps, and handsheets

The raw wood materials and the resultant pulps and pulp handsheets were characterized using the following test methods:

- Holocellulose: method of Wise *et al.* (12)
- $\alpha$ -cellulose: TAPPI T 203 os-61
- Lignin: TAPPI T 222
- Brightness: UNE 57062
- Breaking length: UNE 57054
- Burst index: UNE 57058-74

### Pulping method and handsheet formation

Pulping was carried out in a 15-L batch cylindrical reactor that was heated by means of electrical resistance. The reactor was linked through a rotary axle to a control unit including a motor to actuate the reactor and instruments for measuring and controlling pressure and temperature. The cooked material was ground in a wet disintegrator, and uncooked residues were separated by sieving through a screen

with a mesh of 1 mm.

Pulp handsheets were produced in a former (ENJO-F-39.71) according to established procedures (UNE 57042-74).

## EXPERIMENTAL DESIGN

The central composite design uses a series of points (experiments) around a central point (central experiment), along with several additional points (additional experiments) to estimate the first- and second-order interaction terms of a polynomial. This design is useful because every parameter in the mathematical model can be estimated from a fairly small number of experiments (13).

The design used includes three different parameters:

1.  $k$ , the number of variables
2.  $p$ , a constant where  $p = 0$  at  $k < 5$  and  $p = 1$  at  $k > 5$
3.  $n_c$ , the number of central points

These parameters yield three point groups (14):

1.  $2^{k-p}$  factorial design points
2.  $2 \times k$  star points
3.  $n_c$ , the central point

Thus the total number of observations for  $k$  factors is given by Eq. 1.

$$n = 2^{k-p} + 2k + n_c \quad (1)$$

However, the number of observations can be reduced by using a half-replicate of the factor design (when  $p = 1$ ), with

$$x_k = \sum_{i=1}^{k-1} X_i$$

as the defining relation for the factor design (14).

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

$$Z = a_0 + \sum_{i=1}^3 b_i X_{ni} + \sum_{i=1}^3 c_i X_{ni}^2 + \sum_{i=1, j=1}^3 d_{ij} X_{ni} X_{nj} \quad (2)$$

for  $i < j$  and where:

$Z$  = value for a given dependent variable (one of the seven measured

pulp properties)

$X_n$  = normalized value for a given independent variable (one of the five pulping conditions)

$X_n$  is normalized from -1 to +1 by using Eq. 3.

$$X_n = 2 \frac{X - \bar{X}}{X_{\max} - X_{\min}} \quad (3)$$

Normalizing  $X_n$  facilitates direct comparison of the coefficients and visualization of the effects of the individual independent variables on the response variable. This also increases the accuracy of the estimated regression coefficients, since it reduces interrelationships between linear and quadratic terms (13).

## RESULTS AND DISCUSSION

### Experimental variables

We considered five independent variables for the pulping process:

$T$  = temperature, °C

$t$  = time, min

$A$  = active alkali concentration, %

$S$  = sulfidity, %

$R$  = liquid/solid ratio

The seven dependent variables for pulp properties were:

$Y$  = yield, %

$H$  = holocellulose content, %

$\alpha C$  =  $\alpha$ -cellulose content, %

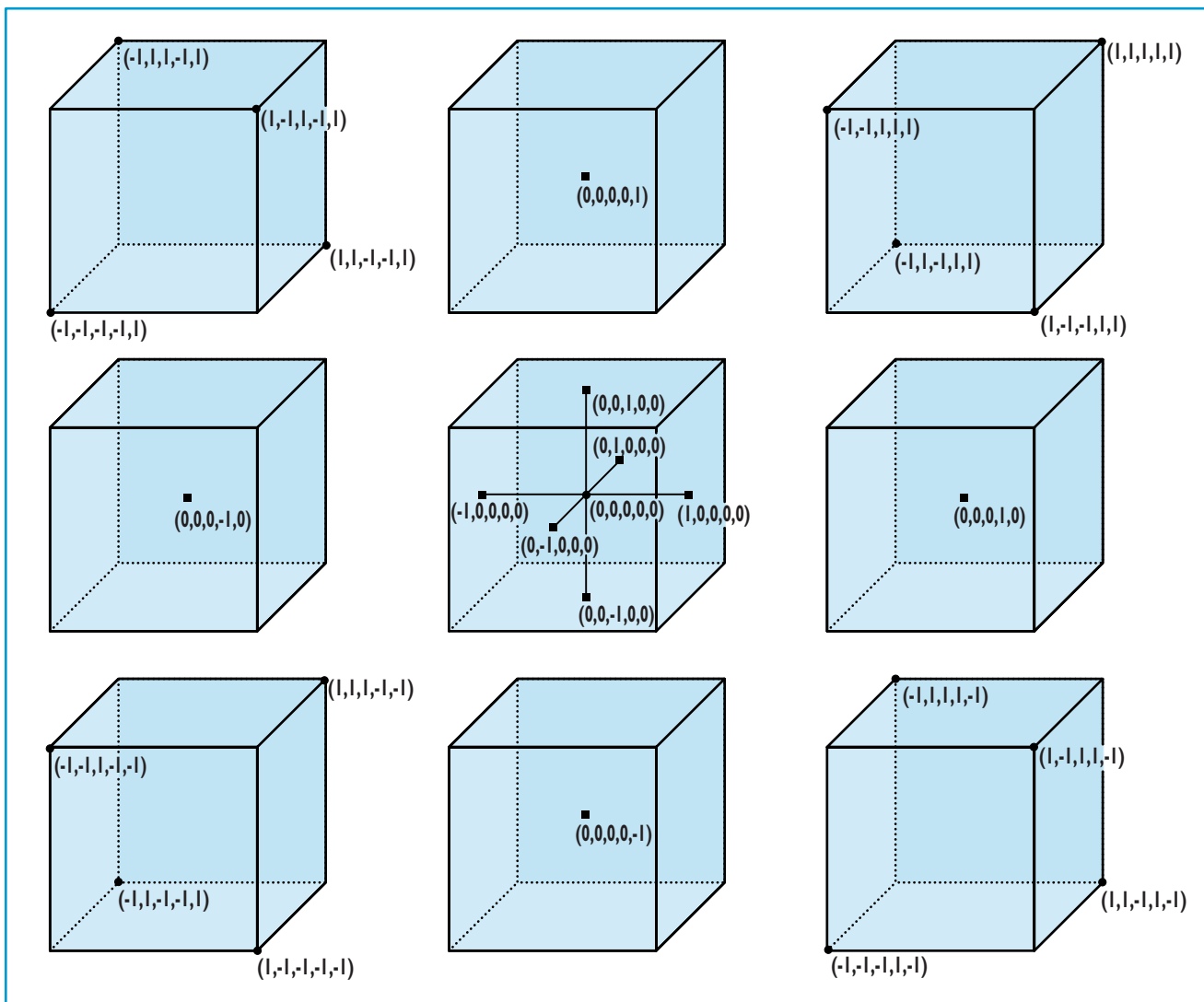
$L$  = lignin content, %

$B$  = brightness, %

$BL$  = breaking length, m

$BI$  = burst index, kN/g

Application of Eq. 1 reveals that fitting the experimental results to the second-order polynomial of Eq. 2 requires 27 experiments. These experiments are shown as data points in Fig. 1, together with the corresponding normalized values for the independent variables. Table I shows the normalized independent variables for the 27 experiments along with the experimental results obtained for each set of conditions.



1. Plot of the coordinates for the experiments included in the experimental design, based on five independent variables and the following relation:  $x_5 = x_1 \times x_2 \times x_3 \times x_4$ . Circles depict the 16 data points ( $2^{5-1}$ ) in the factor design; squares the 10 axial points ( $2 \times 5$ ); and the diamond the central point at 0,0,0,0.

### Independent variables

Initially, we carried out four experiments under the central operating conditions, i.e., 165°C, 60 min, 10% active alkali, 16% sulfidity, and liquid/solid ratio of 6. In all four experiments, the results for the dependent variables differed from the mean values (shown in the first row of Table I) by less than 10%. In preliminary experiments, the values for the independent variables were fixed to obtain pulp yields of 40–50%.

The other 26 experiments of the experimental design provided the results shown in Rows 2–27 of Table I. The independent variables were varied over the ranges 137–193°C, 30–90 min, 5–15% active alkali,

8–24% sulfidity, and 4–8 liquid/solid ratio. The time to operating temperature (10–20 min) was excluded from the pulping time.

### Dependent variables

The experimental data of Table I were substituted into Eq. 2 using a statistical software package (BMDP), resulting in Eqs. 4–10 for the seven dependent variables.

$$Y = 41.91 - 3.66T - 6.70A + 1.41S + 2.74A^2 + 0.85AR \quad (4)$$

$$H = 81.16 + 2.14T + 6.97A - 1.29R + 3.39TA - 1.43SR \quad (5)$$

$$\alpha C = 66.71 + 4.01T + 1.82t + 8.25A - 1.28R + 1.09TA - 1.09SR \quad (6)$$

$$L = 19.92 + 1.07t - 6.22A - 2.33R^2 - 1.13TA + 1.40tR \quad (7)$$

$$B = 20.88 - 4.57T - 1.85t + 3.31A + 5.18A^2 + 2.71TA - 1.31TS - 0.76tR \quad (8)$$

$$BL = 1750.36 + 612.25T + 110.20t + 734.05A + 92.64S - 137.17R - 287.23t^2 + 468.64S^2 - 282.29R^2 + 270.62TA - 123.73AR \quad (9)$$

$$BI = 0.61 + 0.30T + 0.37A + 0.23A^2 + 0.27TA \quad (10)$$

Table II shows Snedecor's  $F$  as well as multiple  $r$ ,  $r^2$ , and adjusted  $r^2$  values for the seven dependent variables defined in Eqs. 4–10. Table III

| Trial No. | PULPING CONDITIONS |    |    |    |    | PULP PROPERTIES |       |               |       |       |       |          |
|-----------|--------------------|----|----|----|----|-----------------|-------|---------------|-------|-------|-------|----------|
|           | T                  | t  | A  | S  | R  | Y, %            | H, %  | $\alpha$ C, % | L, %  | B, %  | BL, m | BI, kN/g |
| 1         | 0                  | 0  | 0  | 0  | 0  | 41.89           | 80.95 | 65.95         | 18.10 | 22.71 | 1640  | 0.65     |
| 2         | 1                  | 1  | 1  | 1  | 1  | 36.92           | 91.97 | 78.77         | 11.81 | 22.73 | 3270  | 1.70     |
| 3         | -1                 | -1 | 1  | 1  | 1  | 45.86           | 78.52 | 63.51         | 12.02 | 34.91 | 1130  | 0.60     |
| 4         | -1                 | 1  | 1  | -1 | 1  | 42.01           | 81.60 | 72.30         | 16.14 | 26.49 | 1120  | 0.50     |
| 5         | 1                  | -1 | 1  | -1 | 1  | 33.80           | 92.42 | 77.54         | 7.03  | 29.63 | 2800  | 1.80     |
| 6         | -1                 | 1  | -1 | 1  | 1  | 56.15           | 75.24 | 54.69         | 23.41 | 28.52 | 530   | 0.45     |
| 7         | 1                  | -1 | -1 | 1  | 1  | 48.35           | 71.33 | 60.60         | 21.25 | 16.95 | 1460  | 0.50     |
| 8         | 1                  | 1  | -1 | -1 | 1  | 46.28           | 69.62 | 59.71         | 28.76 | 15.25 | 1130  | 0.50     |
| 9         | -1                 | -1 | -1 | -1 | 1  | 54.01           | 75.45 | 53.79         | 22.70 | 31.48 | 450   | 0.45     |
| 10        | -1                 | 1  | 1  | 1  | -1 | 42.02           | 87.98 | 73.61         | 9.60  | 33.00 | 1970  | 0.70     |
| 11        | 1                  | -1 | 1  | 1  | -1 | 33.65           | 93.07 | 80.47         | 9.19  | 30.46 | 3490  | 1.70     |
| 12        | 1                  | 1  | 1  | -1 | -1 | 31.37           | 94.46 | 81.67         | 9.07  | 28.46 | 3490  | 1.70     |
| 13        | -1                 | -1 | 1  | -1 | -1 | 40.29           | 77.93 | 66.68         | 14.27 | 29.79 | 1550  | 0.60     |
| 14        | 1                  | 1  | -1 | 1  | -1 | 50.10           | 81.11 | 67.54         | 23.87 | 14.75 | 1580  | 0.60     |
| 15        | -1                 | -1 | -1 | 1  | -1 | 58.47           | 76.50 | 55.05         | 24.34 | 33.49 | 466   | 0.50     |
| 16        | -1                 | 1  | -1 | -1 | -1 | 50.11           | 74.14 | 57.85         | 24.28 | 27.29 | 790   | 0.45     |
| 17        | 1                  | -1 | -1 | -1 | -1 | 48.49           | 71.14 | 58.74         | 24.30 | 17.41 | 960   | 0.40     |
| 18        | 0                  | 0  | 0  | 0  | -1 | 40.23           | 83.43 | 68.14         | 14.91 | 21.81 | 1450  | 0.60     |
| 19        | 0                  | 0  | 0  | 0  | 1  | 42.20           | 80.64 | 65.99         | 19.67 | 20.86 | 1330  | 0.50     |
| 20        | 0                  | 0  | -1 | 0  | 0  | 51.46           | 74.18 | 58.46         | 20.96 | 22.30 | 1020  | 0.40     |
| 21        | 0                  | 0  | 0  | -1 | 0  | 41.41           | 81.73 | 66.86         | 18.73 | 21.11 | 2125  | 0.60     |
| 22        | 0                  | 0  | 0  | 1  | 0  | 41.72           | 80.01 | 67.54         | 18.90 | 18.75 | 2220  | 0.70     |
| 23        | 0                  | -1 | 0  | 0  | 0  | 42.21           | 81.25 | 64.84         | 18.41 | 23.11 | 1140  | 0.50     |
| 24        | 0                  | 1  | 0  | 0  | 0  | 41.19           | 81.38 | 67.54         | 26.20 | 17.27 | 1650  | 0.70     |
| 25        | -1                 | 0  | 0  | 0  | 0  | 45.46           | 79.94 | 63.52         | 20.00 | 28.04 | 1440  | 0.30     |
| 26        | 1                  | 0  | 0  | 0  | 0  | 39.79           | 80.73 | 68.22         | 22.80 | 15.50 | 2270  | 1.00     |
| 27        | 0                  | 0  | 1  | 0  | 0  | 36.98           | 93.85 | 79.71         | 13.63 | 29.44 | 2670  | 1.40     |

#### I. Pulping conditions (normalized values) and pulp properties obtained from experiments with olive wood trimmings

| Pulp property                     | Equation No. | F      | Multiple r | $r^2$ | Adjusted $r^2$ |
|-----------------------------------|--------------|--------|------------|-------|----------------|
| Yield (Y)                         | Eq. 4        | 114.77 | 0.98       | 0.96  | 0.96           |
| Holocellulose (H)                 | Eq. 5        | 34.28  | 0.94       | 0.89  | 0.86           |
| $\alpha$ -cellulose ( $\alpha$ C) | Eq. 6        | 79.44  | 0.98       | 0.96  | 0.95           |
| Lignin (L)                        | Eq. 7        | 28.06  | 0.93       | 0.87  | 0.84           |
| Brightness (B)                    | Eq. 8        | 60.36  | 0.98       | 0.96  | 0.94           |
| Breaking length (BL)              | Eq. 9        | 61.79  | 0.99       | 0.97  | 0.96           |
| Burst index (BI)                  | Eq. 10       | 203.00 | 0.99       | 0.97  | 0.97           |

#### II. Snedecor's F, multiple r, $r^2$ , and adjusted $r^2$ values for the seven dependent variables defined in Eqs. 4–10

shows the individual Snedecor's  $F$  values for each of the terms in Eqs. 4–10. These results show that the proposed model fits the experimental values for all of the dependent variables quite well.

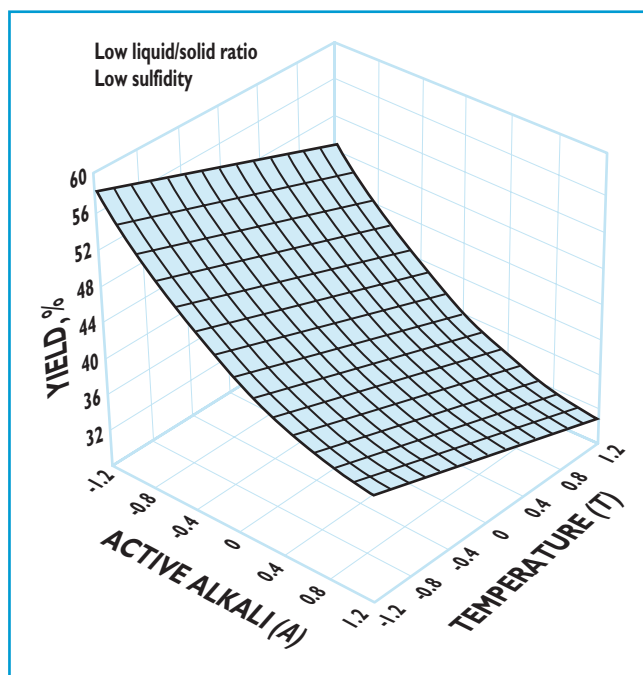
Calculated yield, holocellulose,  $\alpha$ -cellulose, and brightness (Eqs. 4–6 and 8, respectively) differ by less than 10% from their experimental counterparts. The calculated lignin

content (Eq. 7) differs by less than 20%, while the predicted breaking length and burst index (Eqs. 9 and 10, respectively) differ by less than 25% from their respective experimental values.

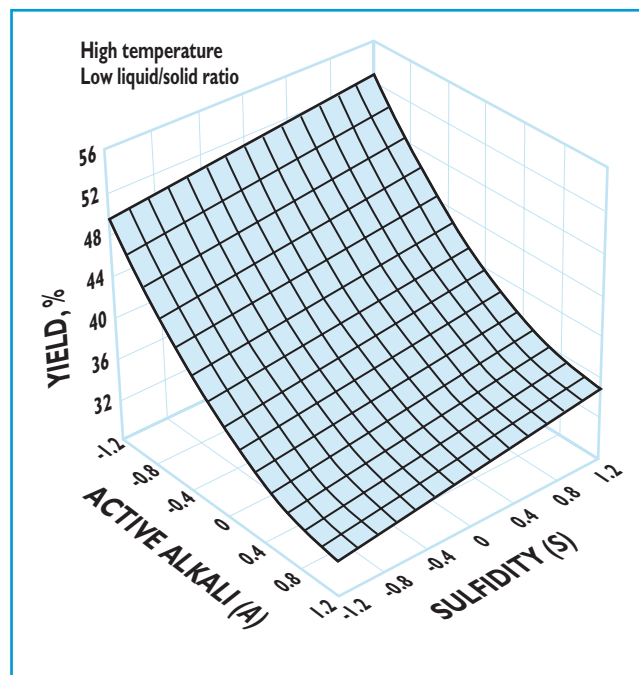
**Yield.** Application of the steepest-descent method (15) over the range of normalized values (-1 to +1) for the independent variables in Eq. 4 reveals that a minimum yield of

32.03% is obtained at high temperature and active alkali concentration (normalized values of +1 for both) and low sulfidity and liquid/solid ratio (-1 for both).

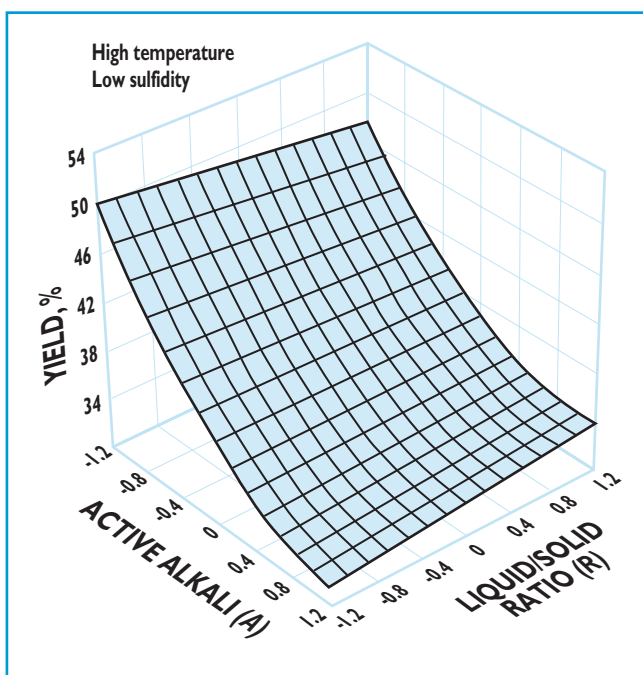
The response of pulp yield to changes in the independent variables over the range studied can be estimated by examining Figs. 2–4 and applying Eq. 4. If the variables that are to remain constant are fixed at the values that produce the lowest yield (+1 for temperature and active alkali and -1 for sulfidity and liquid/solid ratio), then the greatest increase in yield is obtained by altering the active alkali concentration (15.1 points), followed by temperature (7.32-point increase), sulfidity (2.82-point increase), and the liquid/solid ratio (1.7-point increase). Clearly, the yield is much more sensitive to changes in active alkali and temperature than it is to variations in sulfidity or the liquid/solid ratio.



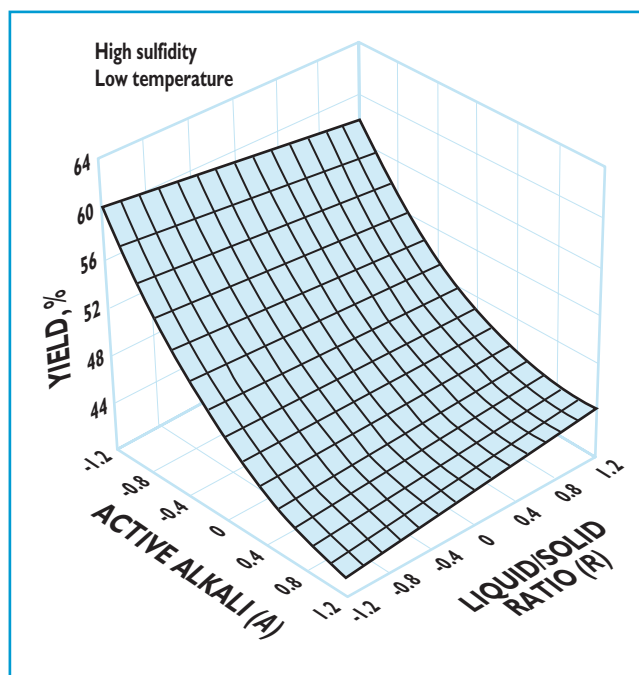
2. Variation of pulp yield with active alkali concentration and temperature at low liquid/solid ratio and low sulfidity



3. Variation of pulp yield with active alkali concentration and sulfidity at low liquid/solid ratio and high temperature.



4. Variation of pulp yield with active alkali concentration and liquid/solid ratio at low sulfidity and high temperature



5. Variation of pulp yield with active alkali concentration and liquid/solid ratio at high sulfidity and low temperature

Application of the steepest-ascent method (15) reveals that the highest yield (57.27%) is obtained at a low temperature, active alkali concentration, and liquid/solid ratio (-1 values for all) and a high sulfidity (+1), as seen in Fig. 5. Under these conditions, changes in yield with temperature, active alkali, sulfidity,

and the liquid/solid ratio are the same as before (i.e., when using appropriate values of the variables to obtain a low yield). Consequently, the yield will be only 4.9% lower than its maximum value (57.27%) if a low sulfidity is used.

**Holocellulose.** The highest holocellulose content of 96.38% is ob-

tained at a high temperature, active alkali, and sulfidity (+1 for all) and a low liquid/solid ratio (-1). From Fig. 6 (and other similar graphs where the holocellulose content was found to vary with the active alkali concentration and the liquid/solid ratio or sulfidity) and application of Eq. 5, it follows that the greatest changes in

| Pulp property                     | Equation | Terms in each equation | Snedecor's F for terms in each equation |
|-----------------------------------|----------|------------------------|-----------------------------------------|
| Yield (Y)                         | Eq. 4    | T                      | 120.63                                  |
|                                   |          | A                      | 403.08                                  |
|                                   |          | S                      | 18.22                                   |
|                                   |          | A <sup>2</sup>         | 22.48                                   |
|                                   |          | A·R                    | 5.83                                    |
| Holocellulose (H)                 | Eq. 5    | T                      | 12.02                                   |
|                                   |          | A                      | 127.80                                  |
|                                   |          | R                      | 4.41                                    |
|                                   |          | T·A                    | 27.00                                   |
|                                   |          | S·R                    | 4.89                                    |
| $\alpha$ -cellulose ( $\alpha$ C) | Eq. 6    | T                      | 84.80                                   |
|                                   |          | t                      | 17.46                                   |
|                                   |          | A                      | 357.41                                  |
|                                   |          | R                      | 8.65                                    |
|                                   |          | T·A                    | 5.51                                    |
| Lignin (L)                        | Eq. 7    | S·R                    | 5.61                                    |
|                                   |          | t                      | 3.68                                    |
|                                   |          | A                      | 123.91                                  |
|                                   |          | R <sup>2</sup>         | 5.77                                    |
|                                   |          | T·A                    | 3.61                                    |
| Brightness (B)                    | Eq. 8    | t·R                    | 5.60                                    |
|                                   |          | T                      | 169.42                                  |
|                                   |          | t                      | 27.62                                   |
|                                   |          | A                      | 88.65                                   |
|                                   |          | A <sup>2</sup>         | 72.32                                   |
| Breaking length (BL)              | Eq. 9    | T·A                    | 53.13                                   |
|                                   |          | T·S                    | 12.61                                   |
|                                   |          | t·R                    | 4.21                                    |
|                                   |          | T                      | 215.80                                  |
|                                   |          | t                      | 6.98                                    |
| Burst index (BI)                  | Eq. 10   | A                      | 309.45                                  |
|                                   |          | S                      | 5.01                                    |
|                                   |          | R                      | 10.84                                   |
|                                   |          | t <sup>2</sup>         | 7.39                                    |
|                                   |          | S <sup>2</sup>         | 20.13                                   |
|                                   |          | R <sup>2</sup>         | 7.08                                    |
|                                   |          | T·A                    | 37.50                                   |
|                                   |          | A·R                    | 7.84                                    |
|                                   |          | T                      | 238.70                                  |
|                                   |          | A                      | 371.65                                  |
|                                   |          | A <sup>2</sup>         | 48.91                                   |
|                                   |          | T·A                    | 180.20                                  |

### III. Snedecor's F values for the terms in Eqs. 4–10

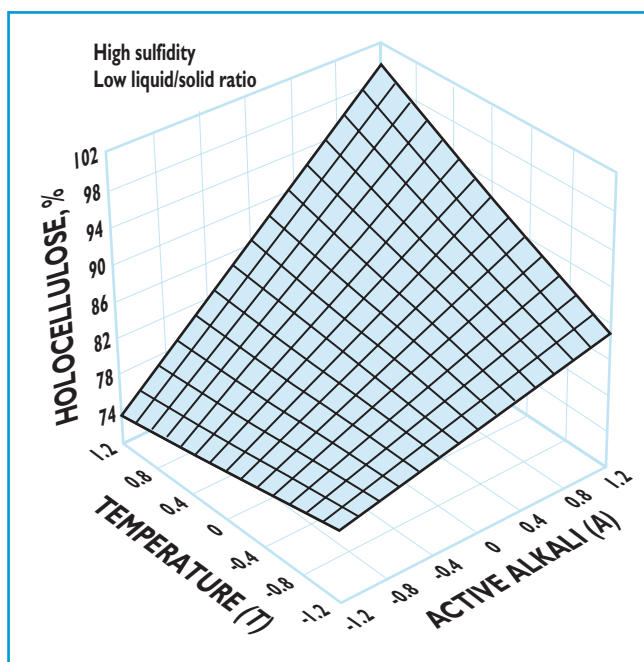
holocellulose content are obtained by altering the active alkali concentration (20.72 points), while the smallest changes occur by varying the sulfidity (2.86 points), with temperature and liquid/solid ratio having intermediate effects (11.06 points and 5.44 points, respectively). Consequently, the holocellu-

lose content at a low sulfidity is only 3.0% less than its maximum value. The decrease is 11.5% if a low temperature is used.

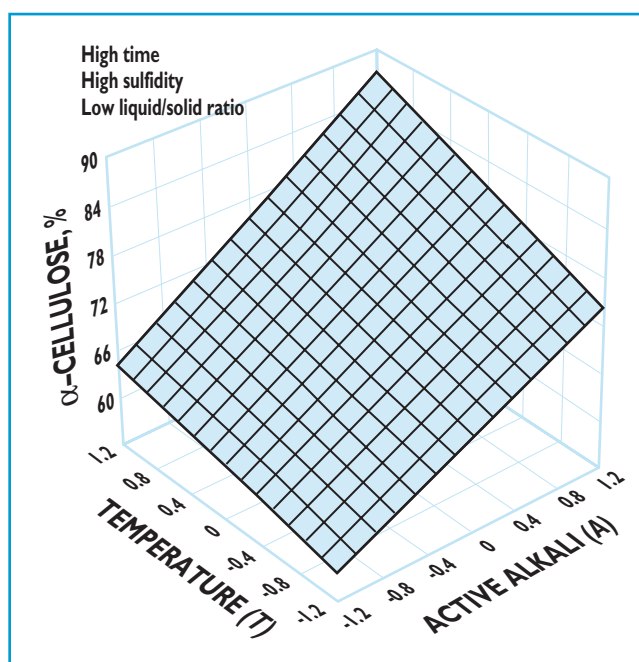
**$\alpha$ -cellulose.** The highest  $\alpha$ -cellulose content of 84.25% is obtained at high temperature, active alkali, and sulfidity (+1 for all), a long time (+1), and a low liquid/solid ratio (-1).

Under these conditions, from Fig. 7 (and other similar graphs where the  $\alpha$ -cellulose content was found to vary with the active alkali concentration and the liquid/solid ratio, pulping time, or sulfidity) and Eq. 6, it is clear that the  $\alpha$ -cellulose content is more sensitive to variations in the active alkali concentration (18.68 points) and temperature (10.2 points) than it is to changes in the liquid/solid ratio (4.74 points), pulping time (3.64 points), and sulfidity (2.18 points). Thus using a low sulfidity reduces  $\alpha$ -cellulose content only slightly, from its maximum value of 84.25% to 82.07%. On the other hand, using a short pulping time reduces the  $\alpha$ -cellulose content to 80.61% (3.64 points lower than the optimum value), and operating at a low sulfidity and a short pulping time leads to a content 6.9% less than the maximum. The convenience of operating under these conditions must be weighed to select the best possible compromise between a shortened process time and reduced sulfidity on the one hand, and a decreased holocellulose content and consequent lower quality in the resulting pulp.

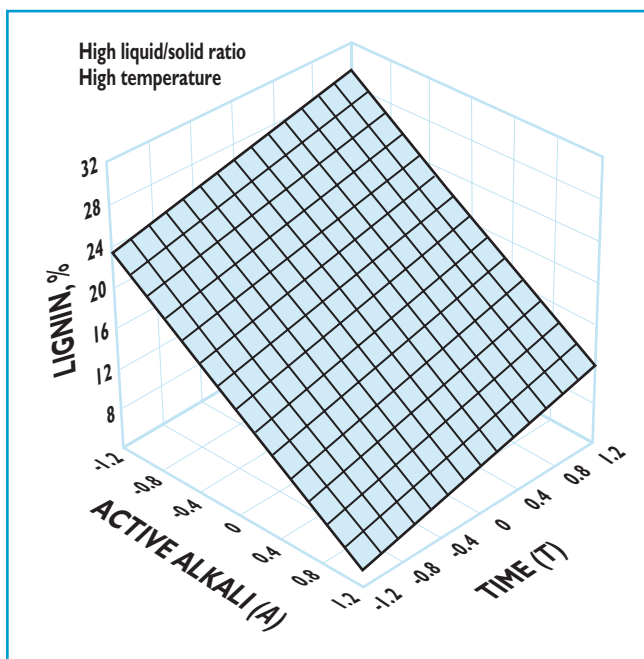
**Lignin.** The lowest lignin content of 7.77% is obtained at a high temperature, active alkali, and liquid/solid ratio (+1 for all) and a short pulping time (-1). Under these conditions, from Fig. 8 (and other similar graphs where the lignin content was found to vary with the active alkali concentration and the liquid/solid ratio or temperature) and Eq. 7, it is clear that the lignin content is more sensitive to changes in active alkali (14.7 points) than to variations in the pulping temperature (2.26 points), with the pulping time and liquid/solid ratio having intermediate effects (4.94 and 3.94 points, respectively). Pulping at low temperature increases the lignin content by 2.26 points, or an increase of 29.1% relative to its minimum value.



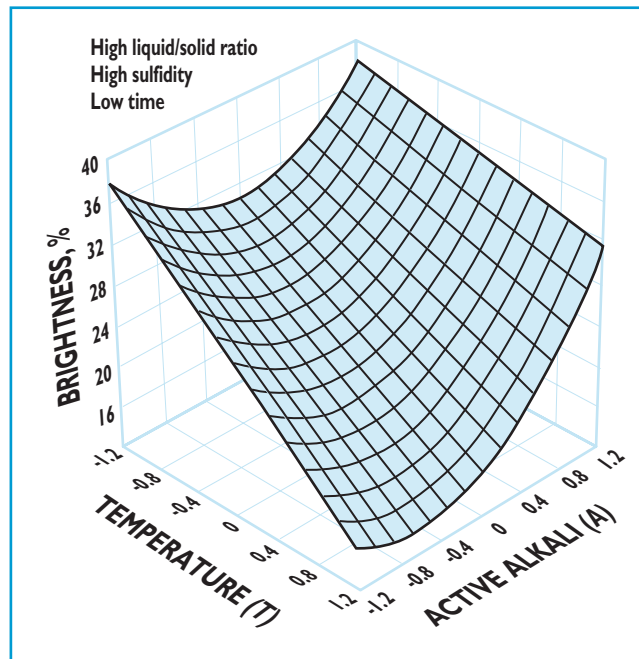
6. Variation of holocellulose with active alkali concentration and temperature at high sulfidity and low liquid/solid ratio



7. Variation of  $\alpha$ -cellulose with active alkali concentration and temperature at high sulfidity, long pulping time, and low liquid/solid ratio



8. Variation of lignin with active alkali concentration and pulping time at high temperature and high liquid/solid ratio

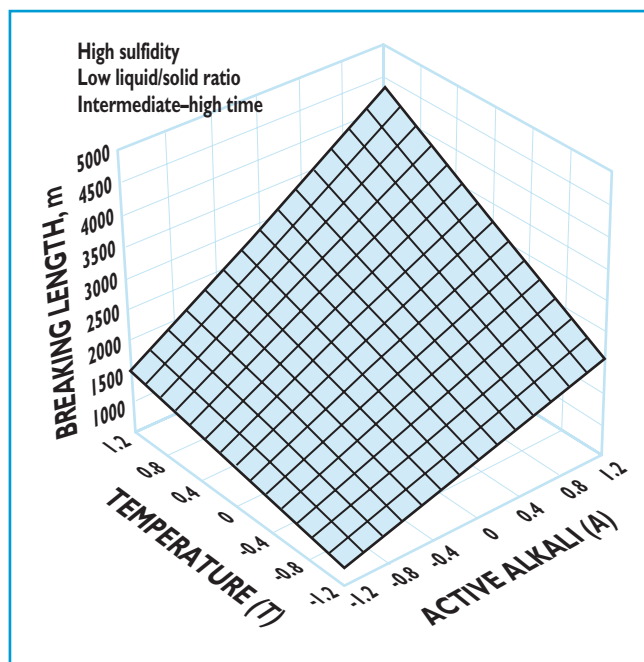


9. Variation of brightness with temperature and active alkali concentration at short pulping time, high sulfidity, and high liquid/solid ratio

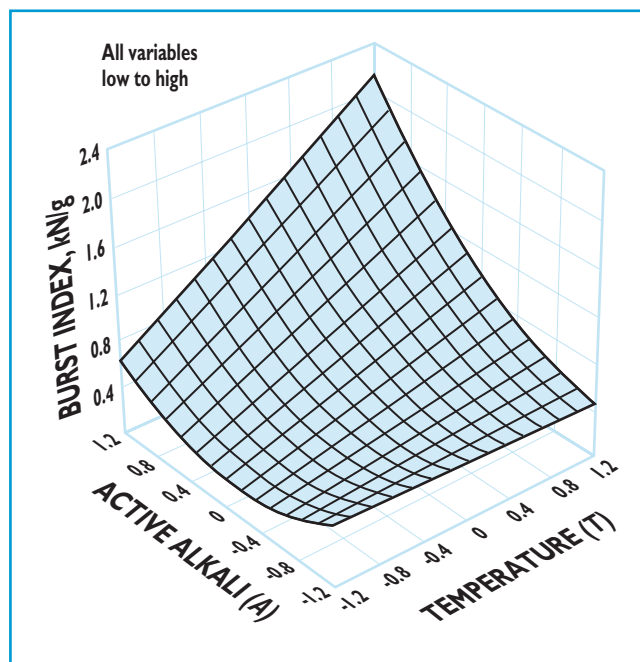
**Brightness.** The maximum brightness of 35.15% is obtained at a low temperature and short time ( $-1$  for both) and a high active alkali concentration, liquid/solid ratio, and sulfidity ( $+1$  for all). Under these conditions, from Fig. 9 (and other similar graphs where the brightness was

found to vary with the temperature and the pulping time, the sulfidity or the liquid/solid ratio) and Eq. 8, it is clear that brightness changes more markedly with temperature (6.34 points), active alkali concentration (5.79 points), and pulping time (5.2 points) than it does with sulfidity

(2.62 points) or the liquid/solid ratio (1.52 points). Consequently, using a low liquid/solid ratio and a low sulfidity decreases brightness by 4.3% and 7.4%, respectively, relative to its peak value of 35.15%. On the other hand, operating at a low sulfidity and a low liquid/solid ratio decreases



10. Variation of breaking length with active alkali concentration and temperature at high sulfidity, low liquid/solid ratio, and moderate-to-long pulping time



11. Variation of burst index with active alkali concentration and temperature at any value of the other variables

|                    | EUCALYPTUS         |                    | OLIVE<br>WOOD<br>TRIMMINGS |
|--------------------|--------------------|--------------------|----------------------------|
|                    | <i>E. globulus</i> | <i>E. rostrata</i> |                            |
| Yield, %           | 53.67              | 54.80              | 36.92                      |
| Breaking length, m | 4757               | 3696               | 3053                       |
| Burst index, kN/g  | 1.97               | 1.56               | 1.72                       |

\* 165°C, 60 min, 10% active alkali, 16% sulfidity, and liquid/solid ratio of 6

#### IV. Pulp yield and handsheet properties for pulps obtained from eucalyptus wood and from olive wood trimmings at central conditions used in this study\*

brightness to 31.01%, which is 11.8% lower than the peak value.

**Breaking length.** The maximum breaking length of 3999 m is obtained at high temperature, active alkali, and sulfidity (+1 for all), a moderate-to-long pulping time (+0.19), and a moderate-to-low liquid/solid ratio (-0.46). Under these conditions, from Fig. 10 (and other similar graphs where the breaking length was found to vary with active alkali and the liquid/solid ratio, sulfidity, or pulping time) and application of Eq. 9, it is clear that the breaking length is more sensitive to changes in active alkali (2123 m) and temperature (1766 m) than it is to variations in

pulping time (408 m), with sulfidity and the liquid/solid ratio having intermediate effects (566 and 603 m, respectively). Thus using a short pulping time decreases the breaking length by 10.2% relative to its maximum value, whereas a low sulfidity reduces it by 14.1%.

**Burst index.** The maximum burst index of 1.78 kN/g is obtained at a high pulping temperature and active alkali concentration (+1 for both). Based on Fig. 11 and Eq. 10, the burst index is more sensitive to changes in active alkali concentration (1.28 kN/g) than it is to variations in the pulping temperature (1.14 kN/g).

#### Optimum pulping conditions

From the results presented here, it follows that pulps with high holocellulose and  $\alpha$ -cellulose contents can be obtained using a high temperature, active alkali concentration, and sulfidity, a long pulping time, and a low liquid/solid ratio. Shortening the pulping time scarcely decreases the  $\alpha$ -cellulose content while decreasing lignin content and increasing brightness.

Assuming that the holocellulose,  $\alpha$ -cellulose, and lignin contents will not vary by more than 10% from their optimum values (96.38%, 84.25%, and 7.77%, respectively), a quadratic programming method can be used to determine the best operating conditions and highest pulp yield. The calculated yield at high temperature (+0.90), active alkali concentration (+1), sulfidity (+0.99), and liquid/solid ratio (+1) and short pulping time (-0.73) was 36.92%. The pulp handsheets obtained under these conditions showed acceptable burst index (1.72 kN/g, which is only 3.2% less than the maximum value) in addition to a brightness (28.43%) and breaking length (3053 m) that

are less than 25% from their optimum values. Regardless of the pulping conditions, the pulp can and should be refined to increase the strength properties of the resulting pulp handsheets.

## CONCLUSIONS

Olive wood trimmings in Spain are a substantial fiber resource, amounting to more than twice the nation's wood imports (3). In addition, they are superior to other agricultural residues such as cereal straw or sunflower stalks, since they can be processed as received at kraft mills.

The results of this study suggest that olive wood trimmings can produce a kraft pulp with acceptably high yields, brightness, holocellulose and  $\alpha$ -cellulose contents, and a low lignin content. The optimum operating conditions involve pulping at

190°C for 38 min using an alkali concentration of 10%, a sulfidity of 23.9%, and a liquid/solid ratio of 8. These are also the most suitable conditions for obtaining pulp handsheets with a high breaking length and burst index. Regardless of the operating conditions, the pulp can and should be refined to increase the strength properties of the resulting pulp handsheets.

Table IV shows pulp yield, breaking length, and burst index for olive wood trimmings and eucalyptus (*E. globulus* and *E. rostrata*) wood pulped at the central operating conditions of this study. The results show that the pulp handsheets from olive wood trimmings had a slightly lower breaking length and an intermediate burst index. Pulp yield for the olive wood was lower than for the eucalyptus.

Kraft pulps from juvenile wood (e.g., olive wood trimmings) have shorter but finer fibers than those from mature wood. This promotes sheet consolidation, giving a denser, smoother, stronger, and more extensible sheet ideally suited for coated grades and low-basis-weight papers (17). TJ

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