



# Pulp and paper from vine shoots: Neural fuzzy modeling of ethylene glycol pulping

L. Jiménez \*, V. Angulo, A. Rodríguez, R. Sánchez, A. Ferrer

Chemical Engineering Department, Campus of Rabanales, C-3, University of Córdoba, Spain

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## ABSTRACT

The influence of operational variables in the pulping of vine shoots by use of ethylene glycol [viz. temperature (155–185 °C), cooking time (30–90 min) and ethylene glycol concentration (50–70% v/v)] on the properties of the resulting pulp (viz. yield, kappa number and viscosity) and paper sheets (breaking length, stretch, burst index, tear index and brightness) was studied.

A central composite factorial design was used in conjunction with the software ANFIS Edit Matlab 6.5 to develop fuzzy neural model that reproduced the experimental results of the dependent variables with errors less than 5%. The model is therefore effective with a view to simulating the ethylene glycol pulping process.

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## 1. Introduction

The production of cellulose pulp generates large amounts of wastewater in the cooking of the raw materials. Such wastewater is especially polluting when some sulphur-containing reagent is used (e.g., in the sulphite and kraft pulping processes). Although the earliest cellulose pulping processes based on sulphur-free organic solvents (viz. organosolv processes) were developed long ago, and in 1980' been implemented on the pilot plant scale and applied in the industry (Neves and Neves, 1998; Muurinen, 2000; López et al., 2006; Rodríguez and Jiménez, 2008), largely as a result of the scarcity of effective alternatives to conventional processes and the need to respond to the new economic and, especially, environmental challenges.

The greatest shortcoming of organosolv processes is that they usually require high pressures, which calls for special equipment and raises operating costs. This makes the use of solvents with a high boiling point (e.g., ethylene glycol) a potentially interesting alternative.

Among 92–95% of the raw materials used to obtain paper consists of hardwood or softwood, even though a number of non-wood materials have been shown to provide excellent fibre for speciality paper and are the sole viable raw materials available for this purpose in some geographic areas. Also, non-wood materials may help alleviate the growing scarcity of forest wood materials (Atchinson, 1998; Jiménez et al., 2006, 2008). These alternative materials in-

clude agricultural residues, which are highly abundant in Spain (particularly in Andalucía and Castilla-La Mancha). For example, Spain produces roughly one million tons of residual vine shoots each year; at a 50% yield, their processing would provide an amount of pulp equivalent to about 30% of the spanish total yearly pulp output (Angulo and García, 2005; Jiménez et al., 2006, 2008).

Current pulp production figures fall short of market demands, which are growing specially fast in developing countries and, to a lesser extent, in some developed countries. This requires establishing new processing plants which, however, should be economical to set up and maintain, and environmentally benign; also, they should provide quality products by efficiently using alternative raw materials (Atchinson, 1998; Angulo and García, 2005; Jiménez et al., 2006, 2008). All these requirements can be met by using organosolv processes with non-wood raw materials such as vine shoots.

Since fairly recently, pulping processes have been subjected to factor design testing in order to develop polynomial models capable of predicting the properties of the pulp and paper sheets they provide as a function of the operating variables with a view to their optimization (Gilarranz et al., 1998; Díaz et al., 2004; Jiménez et al., 2000, 2001, 2002, 2004). Neural fuzzy models, which have proved successfully with non-linear systems (Tay and Zhang, 1999; Perendeci et al., 2004), have not yet been employed in this context, however.

In this work, we examined the influence of the operating variables of the pulping of vine shoots with ethylene glycol (viz. cooking temperature and time, and ethylene glycol concentration) on the properties of the resulting pulp [viz. yield, kappa number, viscosity and drainability (as the Shopper-Riegler index)] and paper sheets (breaking length, stretch, burst index, tear index

\* Corresponding author. Tel.: +34 957 218658; fax: +34 957 218625.  
E-mail address: [iq1jiall@uco.es](mailto:iq1jiall@uco.es) (L. Jiménez).

and brightness) by using a factorial design and the software ANFIS Edit Matlab 6.5.

## 2. Experimental

### 2.1. Vine shoots

Following air-drying and deleafing, vine shoots were chipped on a semi-industrial wood chipper and the 5–10 mm fraction was isolated by sieving. The vine shoots contained 67.14% holocellulose, 41.14%,  $\alpha$ -cellulose, 20.27% lignin, 3.49% ash and 4.87% ethanol-benzene extractable by dry matter weight.

### 2.2. Analysis of the raw material, pulp and paper

Vine shoots were characterized as follows: holocellulose by the Wise et al. method (1946); and  $\alpha$ -cellulose, lignin, ash and ethanol-benzene extractable according to TAPPI 203 os-61, TAPPI 222, TAPPI 211 and TAPPI 204 (Tappi Standards, 2007). The viscosity, kappa number and drainability (Shopper–Riegler index) of the pulp were determined according to ISO 5351/1, UNE 57-034 and UNE 57-039 (Normas UNE, 2007), respectively. Pulp yield was determined by weighing. The breaking length and stretch, burst index, tear index and brightness of paper sheets were determined according to UNE-57-028, UNE-57-058, UNE-57033 y UNE-57-062 (Normas UNE, 2007), respectively.

### 2.3. Pulping

Pulp was obtained by using a 15 L batch cylindrical reactor that was heated by means of a electrical wires and linked through a rotary axle – to ensure proper agitation – to a control unit including a motor actuating the reactor and the required instruments for measurement and control of the pressure and temperature.

The raw material was cooked in the reactor. Next, the cooked material was defiberized in a wet disintegrator at 1200rpm for 30min and the screenings were separated by sieving through a screen of 0.16 mesh size. The pulp obtained was beaten in a Sprout–Bauer refiner.

Paper sheets were prepared with an ENJO-F-39.71 sheet machine according to the UNE-57-042 standard.

### 2.4. Experimental design

The 2<sup>n</sup> factorial design used (Montgomery, 1991), for three independent variables, consisted of a central one point (central experiment, in the centre of a cube) and 14 additional points (additional experiments lying at the cube vertices -8- and side centres -6-).

The variation of pulp properties as a function of the operational variables of the pulping process can be predicted by using the following expression (Jang et al., 1997):

$$Ye = \frac{\sum_{l=1}^m y_l \left[ \prod_{i=1}^n \mu_{F_i}^l(x_i, \theta_i^l) \right]}{\sum_{l=1}^m \left[ \prod_{i=1}^n \mu_{F_i}^l(x_i, \theta_i^l) \right]} \quad (1)$$

where  $Ye$  is the estimated value of the property to be modelled,  $m$  the number of fuzzy rules applied,  $n$  that of independent variables,  $y^l$  the defuzzifier of a fuzzy rule and  $\mu_{F_i}^l(x_i, \theta_i^l)$  the membership function of the independent variables within its range.

With three operational variables (temperature  $-T$ -, time  $-t$ - and ethylene glycol concentration  $-E$ -), the use of a singleton defuzzifier (a constant parameter,  $c_l$  in Eq. (2)) and a linear membership function for the independent variables (Eq. (3)) allowed Eq. (1) to be simplified to

$$Ye = \frac{\sum_{l=1}^8 c_l \left[ \prod_{i=1}^3 x_i \right]}{\sum_{l=1}^8 \left[ \prod_{i=1}^3 x_i \right]} \quad (2)$$

where

$$x_i = 1 - \frac{1}{x_{\text{high}} - x_{\text{low}}} (x - x_{\text{low}}) \quad x_i = \frac{1}{x_{\text{high}} - x_{\text{low}}} (x - x_{\text{low}}) \quad (3)$$

$x_i$  denoting the values (low  $-x_1$ - and high  $-x_2$ -) of the linear membership function of  $T$ ,  $t$  and  $E$ ;  $x_{\text{high}}$  and  $x_{\text{low}}$  the extreme values of the variable; and  $x$  the value of  $T$ ,  $t$  or  $E$ .

With three independent variables one can establish the following eight fuzzy rules according to the extreme (high and low) values of such variables

R1 : low  $T$ , low  $t$  and low  $E$

R2 : low  $T$ , low  $t$  and high  $E$

...

R7 : low  $T$ , high  $t$  and high  $E$

R8 : high  $T$ , high  $t$  and high  $E$

**Table 1**

Values of operational variables and experimental values of the properties of pulp and paper sheets obtained by ethylene glycol pulping of vine shoots

Temperature (°C), time (min) and ethylene glycol (%)	YI, (%)	KN	VI, (mL/g)	BL, (m)	ST, (%)	BI, (kN/g)	TI, (mN m <sup>2</sup> /g)	BR, (%)
170, 60, 60	49.14	165.6	446.4	1775	1.51	1.09	0.94	14.89
185, 90, 70	41.06	141.1	527.4	1667	1.87	1.03	0.88	15.09
155, 90, 70	48.41	166.2	394.7	1750	1.85	1.22	1.14	16.44
185, 90, 50	44.01	159.5	510.0	1502	1.53	1.00	0.62	14.19
155, 90, 50	49.61	162.7	388.8	1687	1.31	1.11	0.92	15.96
185, 30, 70	47.94	164.2	502.9	1898	1.48	0.98	1.00	14.26
155, 30, 70	54.01	170.6	358.5	2078	1.35	1.11	1.18	16.18
185, 30, 50	49.48	171.6	483.3	1560	1.40	0.97	0.63	13.10
155, 30, 50	56.17	179.0	355.9	1840	1.24	1.07	0.94	15.30
170, 90, 60	44.41	154.0	494.1	1529	1.69	1.12	0.94	15.57
170, 30, 60	52.52	167.0	430.2	1941	1.36	0.98	0.96	14.82
170, 60, 70	49.55	157.7	458.3	1863	1.57	1.17	1.05	14.96
170, 60, 50	50.13	169.6	439.1	1697	1.46	0.99	0.75	14.18
185, 60, 60	47.16	158.7	501.1	1615	1.65	1.02	0.79	13.38
155, 60, 60	52.79	168.0	366.4	1849	1.41	1.13	1.07	15.66

**Table 2**

Values of the constants  $c_l$  in the neural fuzzy model for the pulp properties and paper sheets properties

Rule	T (°C), t (min), E (%)	YI, (%)	KN	VI, (mL/ g)	BL, (m)	ST, (%)	BI, (kN/ g)	TI, (mN m <sup>2</sup> / g)	BR, (%)
1	155, 30, 50	56.39	178.4	354.6	1870	1.25	1.08	0.93	15.26
2	155, 30, 70	54.50	169.1	358.9	2101	1.33	1.11	1.16	16.15
3	155, 90, 50	49.43	162.3	393.2	1674	1.33	1.11	0.92	15.96
4	155, 90, 70	48.50	164.9	400.6	1730	1.84	1.22	1.13	16.43
5	185, 30, 50	49.88	171.2	480.1	1580	1.43	0.98	0.94	13.06
6	185, 30, 70	48.57	163.0	500.6	1911	1.48	0.98	0.99	14.24
7	185, 90, 50	43.97	159.3	512.9	1478	1.57	1.00	0.63	14.21
8	185, 90, 70	41.28	140.1	531.3	1636	1.88	1.02	0.88	15.12
9	155, 60, 50	–	–	–	–	–	–	–	15.17
10	155, 60, 70	–	–	–	–	–	–	–	15.82
11	185, 60, 50	–	–	–	–	–	–	–	13.16
12	185, 60, 70	–	–	–	–	–	–	–	13.85
9	170, 30, 50	–	–	–	–	–	0.90	–	–
10	170, 30, 70	–	–	–	–	–	1.09	–	–
11	170, 90, 50	–	–	–	–	–	1.04	–	–
12	170, 90, 70	–	–	–	–	–	1.25	–	–
R <sup>2</sup>		0.99	0.99	0.98	0.98	0.99	0.99	0.99	0.99
L							6.36		12.74

With a Gaussian membership function with three levels (low, medium and high) for one of the variables and a linear membership function with two levels (low and high) for the other two, Eq. (2) would include 12 terms in the numerator and 12 in the denominator. The Gaussian membership function would be of the form

$$X_i = \exp \left( -0.5 \left( \frac{x - x_c}{L} \right)^2 \right) L \quad (4)$$

where  $x$  is the absolute value of the variable concerned;  $x_c$  its minimum, central or maximum value; and  $L$  the width of its Gaussian distribution.

**Table 3**

Values of the dependent variables as estimated with the neural fuzzy models and deviations from their experimental counterparts (in brackets)

T (°C), t (min), E (%)	YI, (%)	KN	VI, (mL/g)	BL, (m)	ST, (%)	BI, (kN/g)	TI, (mN m <sup>2</sup> /g)	BR, (%)
170, 60, 60	49.06 (0.16)	163.54 (1.4)	441.6 (1.08)	1748 (1.55)	1.51 (0.17)	1.07 (1.99)	0.91 (3.31)	14.57 (2.16)
185, 90, 70	41.28 (0.54)	140.10 (0.71)	531.3 (0.74)	1636 (1.86)	1.88 (0.53)	1.03 (0.31)	0.88 (0.16)	15.05 (0.30)
155, 90, 70	48.50 (0.19)	164.90 (0.78)	400.6 (1.49)	1730 (1.14)	1.84 (0.65)	1.22 (0.11)	1.13 (0.88)	16.39 (0.28)
185, 90, 50	43.97 (0.09)	159.30 (0.13)	512.9 (0.57)	1478 (1.60)	1.57 (2.68)	1.00 (0.35)	0.63 (1.61)	14.62 (3.02)
155, 90, 50	49.43 (0.36)	162.30 (0.25)	393.2 (1.13)	1674 (0.77)	1.33 (1.45)	1.11 (0.10)	0.92 (0.55)	15.91 (0.29)
185, 30, 70	48.57 (1.31)	163.00 (0.73)	500.6 (0.46)	1911 (0.68)	1.48 (0.14)	0.98 (0.33)	0.99 (1.37)	14.22 (0.30)
155, 30, 70	54.50 (0.91)	169.10 (0.88)	358.9 (0.11)	2101 (1.11)	1.33 (1.70)	1.11 (0.10)	1.16 (1.44)	16.13 (0.30)
185, 30, 50	49.83 (0.71)	171.20 (0.23)	480.1 (0.66)	1580 (1.28)	1.43 (2.14)	0.97 (0.34)	0.64 (0.92)	13.07 (0.26)
155, 30, 50	56.39 (0.39)	178.40 (0.34)	354.8 (0.31)	1870 (1.63)	1.25 (0.64)	1.07 (0.07)	0.93 (1.09)	15.26 (0.29)
170, 90, 60	45.80 (3.12)	156.65 (1.72)	459.5 (7.00)	1630 (6.57)	1.66 (2.10)	1.14 (1.63)	0.89 (5.42)	15.49 (0.49)
170, 30, 60	52.32 (0.38)	170.43 (2.05)	423.6 (1.53)	1866 (3.89)	1.37 (0.79)	1.00 (1.86)	0.93 (3.26)	14.67 (1.03)
170, 60, 70	48.21 (2.70)	159.28 (1.00)	447.9 (2.28)	1845 (0.99)	1.63 (3.87)	1.16 (0.99)	1.04 (0.93)	14.91 (0.35)
170, 60, 50	49.91 (0.45)	176.80 (1.06)	435.3 (0.88)	1651 (2.74)	1.40 (4.49)	0.98 (1.20)	0.78 (3.68)	14.23 (0.35)
185, 60, 60	45.91 (2.64)	158.40 (0.19)	506.2 (1.02)	1651 (2.25)	1.59 (3.65)	1.00 (2.13)	0.78 (0.84)	13.59 (1.58)
155, 60, 60	52.21 (1.11)	168.68 (0.40)	376.9 (2.86)	1844 (0.28)	1.44 (1.81)	1.13 (0.32)	1.03 (3.33)	15.55 (0.73)

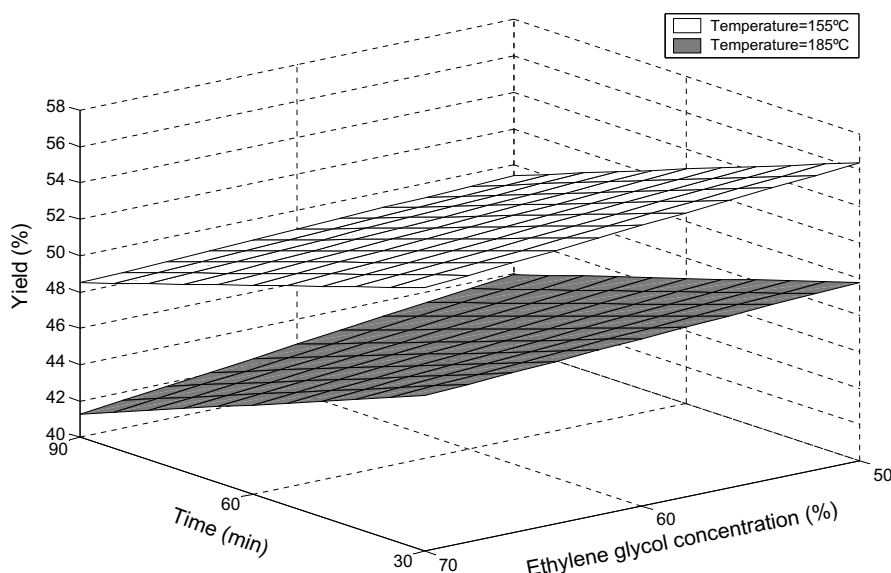
T, t and E, temperature, time and ethylene glycol concentration; YI, yield; KN, kappa number; VI, viscosity; BL, braking length; ST, stretch; BI, burst index; TI, tear index; BR, brightness.

**Table 4**

Experiments used to validate the neural fuzzy models for the pulping of vine shoots with ethylene glycol

Dependent variable	Experiment 16 ( $T = 175^\circ\text{C}$ ; $t = 75$ min; $E = 60\%$ )			Experiment 17 ( $T = 160^\circ\text{C}$ ; $t = 75$ min; $E = 70\%$ )			Experiment 18 ( $T = 160^\circ\text{C}$ ; $t = 75$ min; $E = 60\%$ )		
	Experimental value	Estimated value	Deviation, (%)	Experimental value	Estimated value	Deviation, (%)	Experimental value	Estimated value	Deviation, (%)
YI, (%)	47.81	46.37	3.00	48.94	48.85	0.18	51.38	49.53	3.60
KN	160.4	158.1	1.45	164.9	162.6	1.39	168.5	164.1	2.60
VI, (mL/g)	469.8	471.74	3.05	410.9	412.4	0.37	353.9	408.1	2.93
BL, (m)	1732	1660	4.13	1846	1803	2.32	1765	1745	1.13
ST, (%)	1.52	1.61	5.81	1.69	1.72	1.78	1.55	1.53	1.29
BI, (kN/g)	1.08	1.08	0.22	1.15	1.19	3.90	1.19	1.14	4.55
TI, (mN m <sup>2</sup> /g)	0.87	0.86	1.15	1.12	1.10	1.79	1.01	0.99	1.98
BR, (%)	14.61	14.75	0.96	15.38	15.85	3.07	14.90	15.57	4.51

T, t and E, temperature, time and ethylene glycol concentration; YI, yield; KN, kappa number; VI, viscosity; BL, braking length; ST, stretch; BI, burst index; TI, tear index; BR, brightness.



**Fig. 1.** Variation of yield with time and ethylene glycol concentration at extreme temperatures.

The parameters and constants in the previous equation were estimated by using the ANFIS (Adaptive Neural Fuzzy Inference System) Edit tool in the Matlab 6.5 software suite.

### 3. Results and discussion

Table 1 shows the experimental values of the properties for the pulps and the paper sheets obtained from it. All differed by less than 3–10% from the mean values obtained in three separate determinations of each dependent variable.

Table 2 gives the values of parameters  $c_i$  in the neural fuzzy equations for the yield, kappa number and viscosity of the pulps and breaking length, stretch and tear index of the paper sheets as obtained with a linear membership function at two levels (low and high) for the three operational variables.

Table 2 lists the values of parameters  $c_i$  in the neural fuzzy modelling equation for the burst index of paper sheets as obtained by using a Gaussian membership function at three levels (low, medium and high) for the variable temperature and a linear membership function at two levels (low and high) for the other two. Finally, the membership functions used for the brightness were of the Gaussian type with the variable cooking time and of the linear type with the temperature and ethylene glycol concentration.

Table 3 shows the values of the dependent variables as estimated by the models, as well as their differences from their experimental counterparts. As can be seen, the differences were all small, so the model can be used for accurate predictions in the pulping of vine shoots with ethylene glycol.

The proposed models were validated in three pulping experiments. Table 4 shows the operating conditions used and the pulp properties obtained, in addition to the calculated values of the dependent variables as estimated with the fuzzy models and the

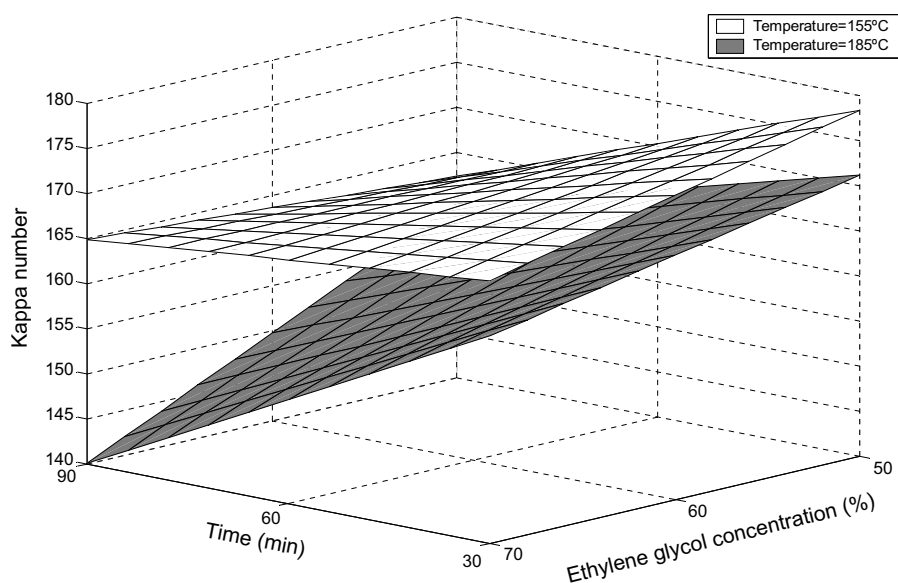


Fig. 2. Variation of kappa number with time and ethylene glycol concentration at extreme temperatures.

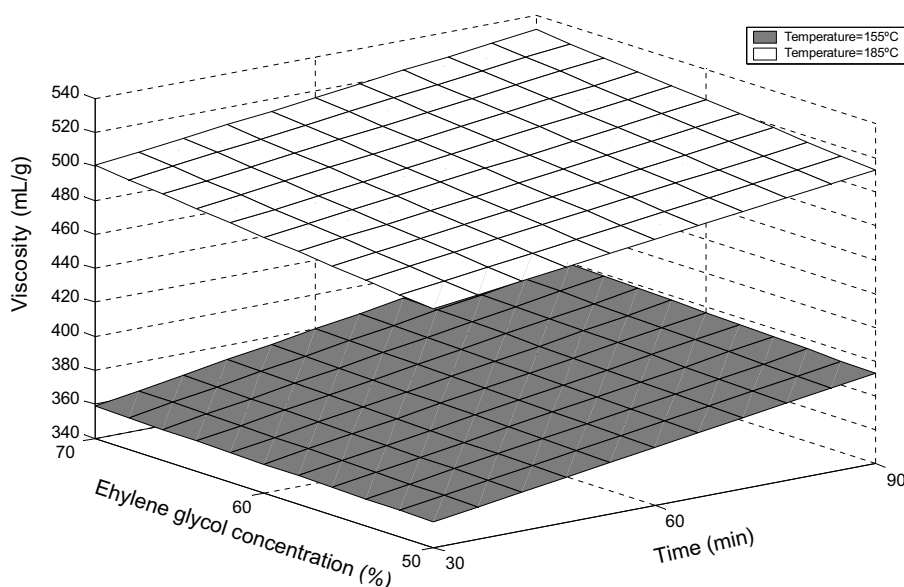


Fig. 3. Variation of viscosity with time and ethylene glycol concentration at extreme temperatures.

errors in their predictions. As can be seen, the predicted values departed very little from their experimental counterparts which confirm the accuracy of the proposed fuzzy model.

Based on the  $R^2$  values obtained (Table 2), the proposed neural fuzzy models can be used to accurately predict pulp properties as a function of the operating conditions used.

Figs. 1–8 show two response surfaces obtained at high and low values of one operational variable on constancy of the other two. In these figures, easily it is possible to be found the values of the different dependent variables for extreme values of temperature (155°C and 185°C) and whatever of the values of the time and ethylene glycol concentration. For example, in Fig. 1 it is observed that yield oscillates between 56.4% when temperature, time and ethylene glycol concentration are low (155°C, 30min and 50%, respec-

tively) until 41.3% for high value of the operation variables (185°C, 90min and 70%).

The neural fuzzy models used involve roughly the same number of parameters as second-order polynomial models (Gilarranz et al., 1998; Díaz et al., 2004; Jiménez et al., 2000, 2001, 2002, 2004). This allows simple neural fuzzy models to be employed with experiments involving only 15 tests – the typical number with polynomial models – (Gilarranz et al., 1998; Díaz et al., 2004; Jiménez et al., 2000, 2001, 2002, 2004).

On the other hand, neural fuzzy models provide a physical interpretation of the constants ( $c_i$  parameters) as these represent the average value of each property (dependent variable) under the conditions defined by the specific fuzzy rule. Thus, the average pulp yield obtained with a low temperature, time and ethylene gly-

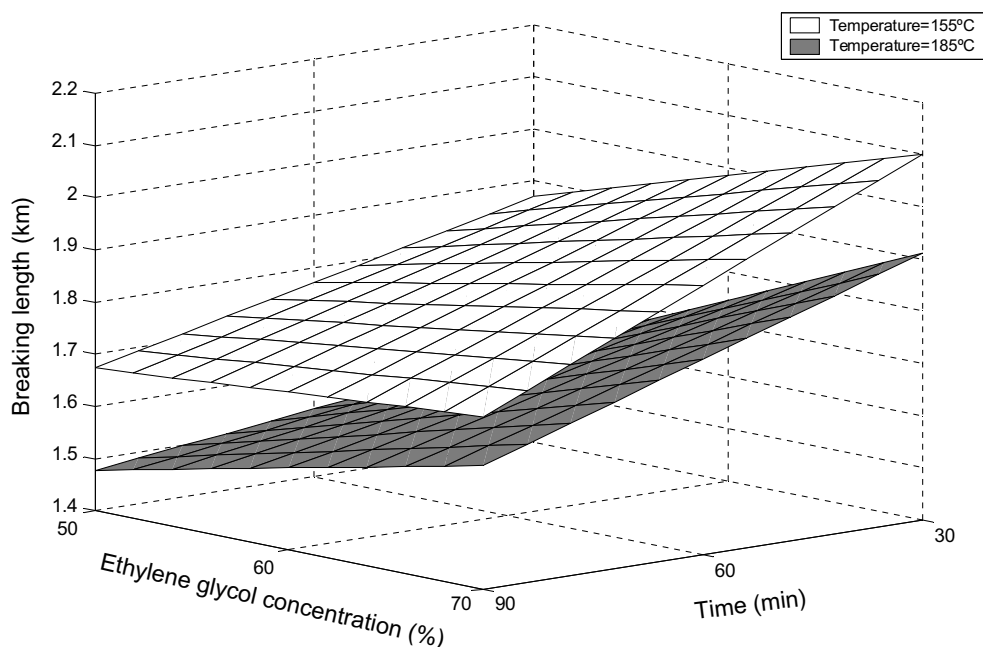


Fig. 4. Variation of breaking length with time and ethylene glycol concentration at extreme temperatures.

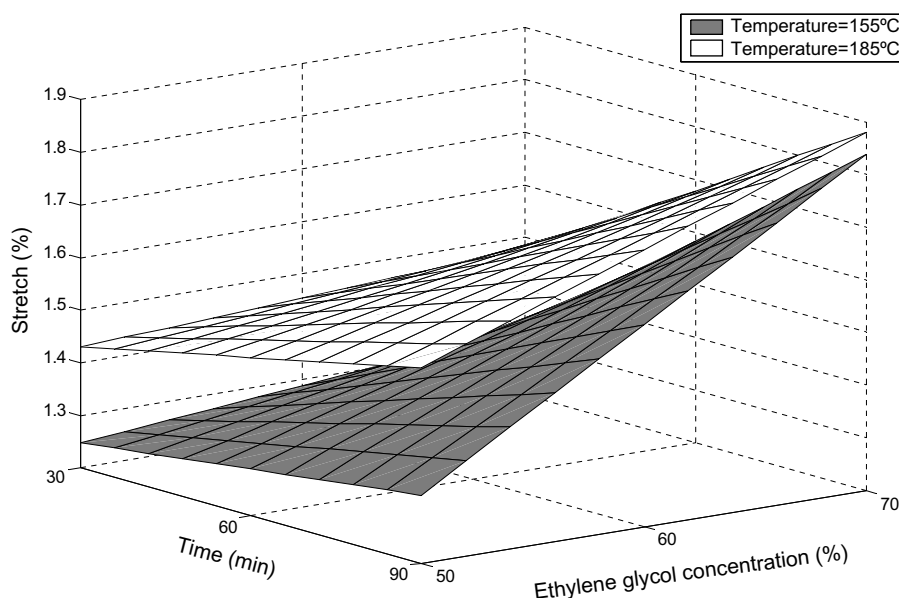


Fig. 5. Variation of stretch with time and ethylene glycol concentration at extreme temperatures.



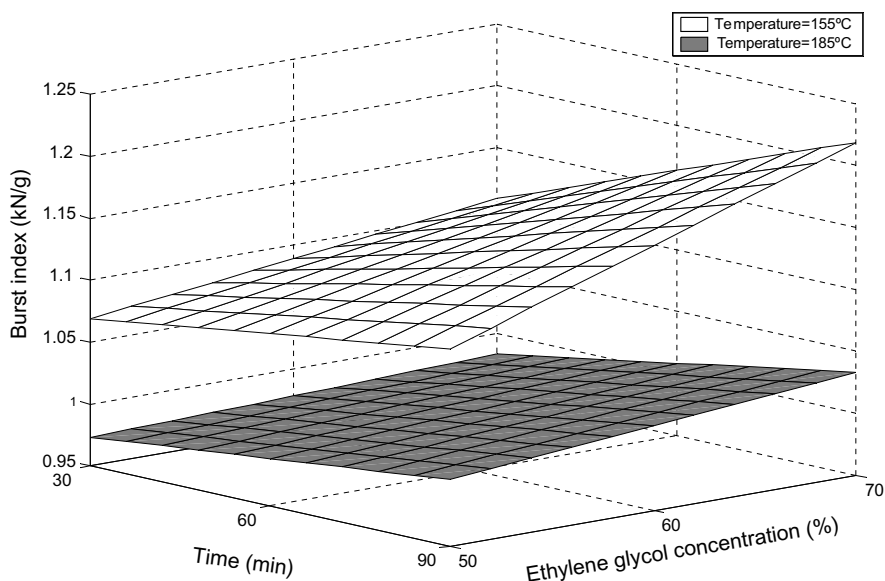


Fig. 6. Variation of burst index with time and ethylene glycol concentration at extreme temperatures.

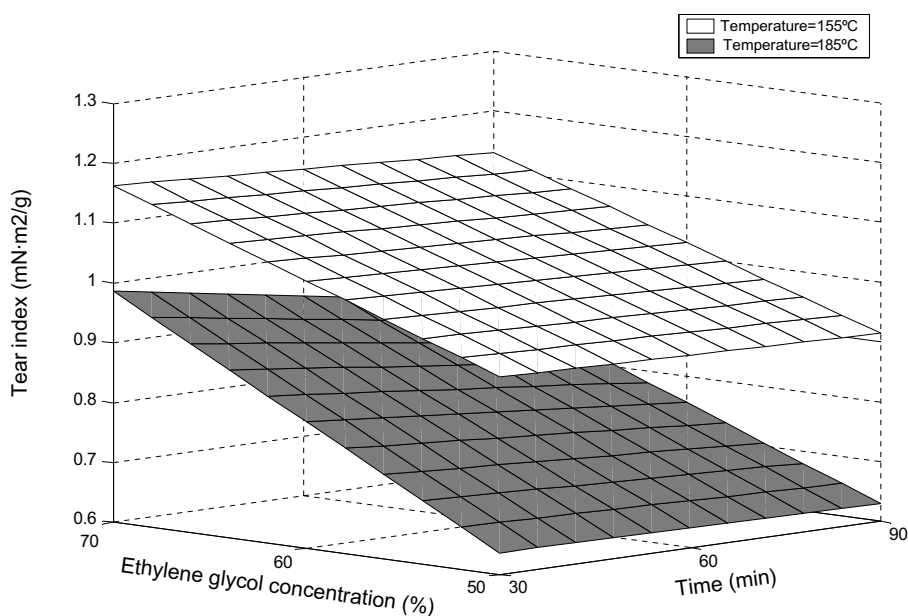


Fig. 7. Variation of tear index with time and ethylene glycol concentration at extreme temperatures.

col concentration was 56.39%, which is almost identical with the parameter value in the equation. This provides an additional advantage over polynomial models (Gilaranz et al., 1998; Díaz et al., 2004; Jiménez et al., 2000, 2001, 2002, 2004).

Also, the neural fuzzy models allow one to assess the influence of each operational variable on pulp properties. Thus, at a low temperature and time, increasing the ethylene glycol concentration would slightly lower the yield (from 56.39% to 54.50%) as per rules 1 and 2 in Table 2 (or row 9 and 7 in Table 3). Also, increasing the pulping time at a low temperature and concentration would decrease the pulp yield from 56.39% to 49.43% as per rules 1 and 3 in Table 2 (or row 9 and 5 in Table 3). This allows one to compare any two rules involving identical levels of two variables and differ-

ing in the third in order to make reliable predictions by determining the influence of the third variable on each pulp property.

#### 4. Conclusions

Applying the experimental results obtained using a factorial design of experiments in the pulping of vine shoots with ethylene glycol, to a fuzzy neural model and using software ANFIS Edit Matlab 6.5, there are obtained equations that reproduce the experimental results of the dependent variables of pulp (viz. yield, kappa number and viscosity) and paper sheets (breaking length, stretch, burst index, tear index and brightness), with errors less than 5%.

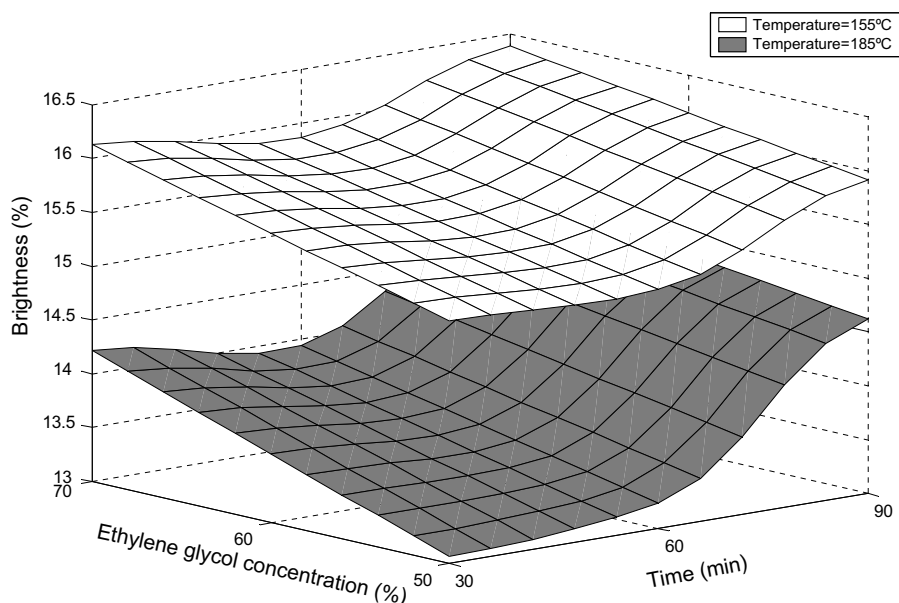


Fig. 8. Variation of brightness with time and ethylene glycol concentration at extreme temperatures.

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