INFLUENCE OF RETANNING AND FATLIQUORING PROCESSES ON THE CHARACTERISTICS OF GOATSKINS

by
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ABSTRACT

The effect of three different offers of both vegetable retanning agent (0, 3 and 6 %) and fatliquoring agent (0, 5 and 10 %) on the physical properties of skins was assessed.

A standard process was applied to the left halves of goatskins from Nigeria, whereas the right halves were subjected to the corresponding variables. The variation in the results of each studied property was evaluated.

The statistical analysis of the results obtained in the experimental plan was carried out by means of the of the Analysis of Variance (ANOVA), through the Statgraphics Plus Program. The best results of softness and physical resistances were obtained with the highest fatliquoring agent offer (10 %). On the other hand, the effect of vegetable retanning agent offer depends on the physical property considered.

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INTRODUCTION

Skin structure is without doubt the factor that has the greatest effect on the physical properties of leather[3]. According to Mozesky y col. [14], even small differences in chemical composition, such as the decorine deficit, give rise to an important tensile strength decrease.

It is well known that there is a perfect symmetry along the backbone on both sides of a hide[1), (6] and that the direction of the tension lines on each side[1)-(9] has an important effect on both tensile strength and tear resistance. Moreover, the values of these two properties are topographically distributed so that the tensile strength of samples cut parallel to the backbone are a 40 % higher than those cut perpendicularly. In addition, tensile strength, which is higher in areas close to the backbone, decreases on the flanks. As far as tear resistance is concerned, the differences between parallel and perpendicular to the backbone samples are not so marked. However, its topographical distribution is completely opposite to that of tensile strength, i.e., tear resistance values are higher in the flanks than in areas close to the backbone.

As regards the effect of the different chemical processes on the physical resistances, the reduction of liming time from 67 to 24 hours gives rise to a decrease in both tensile strength and grain resistance in the Lastometer[5].

In a comparative study between chrome tanned leather and chrome tanned and vegetable retanned leather, Kanagy [19] found values of tear resistance which were 30 % higher for chrome tanned leather; other works[6)-(18] showed that the higher the leather softness, the better the tensile strength.

Bearing in mind the softness effect, studies[6), (15] on drying conditions, humidity and stretching method were carried out; all drying methods that imply a high tension increase the area yield at the expense of a decrease in physical properties.

This work has been divided into four Parts to study the effect of different chemical processes on physical properties, softness, colour and grain firmness. In this Part we evaluate the influence

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of both vegetable retanning and fatliquoring processes on these characteristics. An optimization procedure based on a 32 factorial design has been applied to determine the best working conditions of these two processes applied on goatskins, which is a substrate hardly studied in bibliography.

EXPERIMENTAL

Materials and Procedure

Wet-blue goatskins from Nigeria of 1 mm thickness and 720 g/skin weight were used.

The skins were divided along the backbone. The left halves were subjected to a standard process in which a 2 % vegetable retanning agent (VEGRET) offer and a 6 % fatliquoring agent (FATLQ) offer were applied. The same process was applied to the right sides but included varying offers of both vegetable retanning agent (0 %, 3 % and 6 %) and fatliquoring agent (0 %, 5 % and 10 %). The offers are based on wet-blue weight shaved to 1 mm. The processes are shown in figure 1.

All the treatments were carried out in pilot plant drums equipped with automatic controls of speed and temperature.

The experiments were programmed in accordance with a 3^2 factorial design[17] for two variables at three levels. Table I shows the nine experiments required by this experimental design. The design assumes that the experimental values are modelled by a series of quadratic equations as follows:

$$Y = b_0 + b_1x_1 + b_2x_2 + b_{11}x_1^2 + b_{22}x_2^2 + b_{12}x_1x_2$$

and it is statistically designed to find such a relationship, i.e., to determine the b-values (regression coefficients). The experimental design allowed us to obtain clear estimates for main effects, quadratic effects and variable interaction. The statistical analysis of the results obtained in the experimental plan was carried out by means of the Analysis of Variance (ANOVA), through the Statgraphics Plus Program. The ANOVA partitions the variability into separate pieces for each of the effects, by comparing the mean square against an estimate of the experimental error obtained through the three degrees of freedom of the residual sum of squares because no replicates were done. The significance of the effects was given by the Snedecor-F parameter with one and three degrees of freedom.

LEFT HALVES (Control)
Offers on Wet-Blue Weight Shaved to 1 mm

Soaking
500% Water at 35° C
0,2% Ethoxylated Fatty Alcohol (A)
0,2% Oxalic Acid
Drum 2 hours. In bath overnight, drumming 2 min each hour. Next day pH =3,8. Run-off and Wash 10 min

Retanning
100% Water at 35°C
4,0% Chromium Salt 33°Sch
Drum 60 min
2,0% Sodium Formate
Drum 30 min.
2% Buffering- Neutralizing Agent (B)
Drum 30 min
pH=4,6;Run-off and Wash 10 min

Dyeing - Fatliquoring
60% Water at 30°C
2% Vegetable retanning agent (VEGRET) (C)
Drum 45 min
3,0% Dye
Drum45 min
100% Water at 60°C
6,0 % Sulphited fish oil (FATLQ) (D)
Drum 60 min
1,5% Formic Acid
Drum 30 min
1,5% Formic Acid
Drum 30 min. Adjust pH = 3,9.
Run-off and Wash 10 min

RIGHT HALVES (Variables)
Offers on Wet-Blue Weight Shaved to 1 mm

Soaking
500% Water at 35° C
0,2% Ethoxylated Fatty Alcohol (A)
0,2% Oxalic Acid
Drum 2 hours. In bath overnight, drumming 2 min each hour. Next day pH =3,8. Run-off and Wash 10 min

Retanning
100% Water at 35°C
4,0% Chromium Salt 33°Sch
Drum 60 min
2,0% Sodium Formate
Drum 30 min.
2% Buffering- Neutralizing Agent (B)
Drum 30 min
pH=4,6;Run-off and Wash 10 min

Dyeing - Fatliqouring
60% Water at 30°C
0 % - 3 % - 6 % Vegetable retanning agent (VEGRET) (C)
Drum 45 min
3,0% Dye
Drum45 min
100% Water at 60°C
0 % - 5 % - 10 % Sulphited fish oil (FATLQ) (D)
Drum 60 min
1,5% Formic Acid
Drum 30 min
1,5% Formic Acid
Drum 30 min. Adjust pH = 3,9.
Run-off and Wash 10 min

Mechanical Operations
Twelve hours of horse resting. Toggle drying at 50 °C. Two hours of conditioning in a chamber at 22°C and 62% RH. (RH in hide: 12%). Staking.

Figure 1.Applied processes

Once processed, the skins underwent assessment of the following properties: softness, colour intensity, tensile strength and elongation at break, tear resistance, grain resistance and grain firmness. Figure 2 shows the sampling diagram for the assessment of both destructive properties (physical resistances and grain firmness) and non-destructive properties (softness and colour).

The effect of the studied variables on each property was assessed by comparing the right half value with the corresponding left half (standard process) value. The variation was calculated by applying formula 1:

$$\text{Property variation } (\Delta, \%) = \frac{\text{Right half value - Left half value X 100}}{\text{Left half value}} \quad (1)$$

For the discussion of results, three graph types, provided by the Statgraphics Plus Program, were used: i) Main Effects; ii) Variable Interaction and iii) Estimated Response Surface. Although the comments derived from these plots were included in the text, only the "Estimated Response Surface" graphs are shown in this paper for the sake of simplicity.

RESULTS AND DISCUSSION

Tables II, III and IV show the average of the measured values, the standard deviation and the variation between the different treatments with respect to the corresponding controls for each property determined by applying the formula 1.

The values of property variation (Δ in %) were used for the statistical analysis of the results, which was carried out with the Statgraphics Plus Program. All the possible linear, quadratic effects and interactions were included in the mathematical model. The non significant variables were excluded from the model to obtain the optimum regression equations. The regression equation coefficients were estimated by means of the least squares procedure whereas the significance levels of each variable as well as the determination coefficient (R^2) of the model were calculated by the variance analysis (ANOVA).

Table V shows the coefficients of the optimum regression equations, significance levels of the variables and the determination coefficient for all the physical and organoleptic properties over which the studied variables exerted an influence.

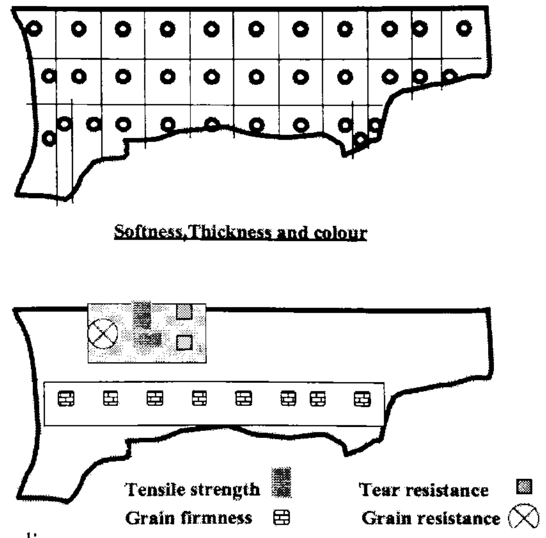


Figure 2. Sampling

TABLE I
Processes of the experimental plan

Right Half Processes	Vegetable retanning agent (%)	Fatliquoring agent(%)
1	0	0
2	3	0
3	6	0
4	0	5
5	3	5
6	6	5
7	0	10
8	3	10
9	6	10

It should be pointed out that in the result discussion below comments are referred to the variation in % of the considered property with respect to the standard process.

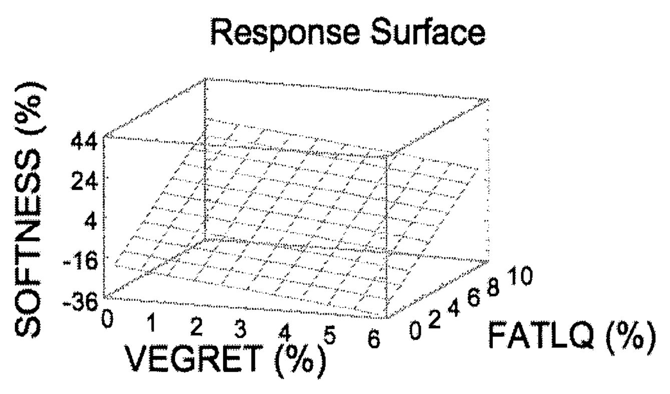
Softness

This was measured with the Softness Tester in accordance with the IUP-36 Standard. Eleven determinations in each area of the half skin were carried out, which resulted in a total of thirty three determinations.

Formula 1 was applied to compare the right half values with those of the left halves (control).

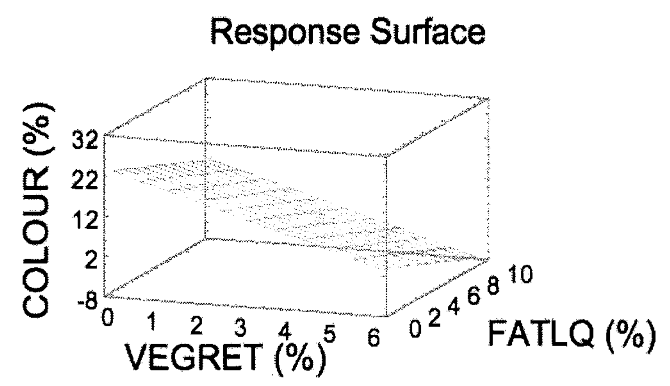
As shown in table V the determination coefficient (R^2) for this property was 84.8 %. This Table also reveals that the fatliquoring agent offer was the only variable which exerted a significant influence on softness (significance level 1 %).

Softness decreased from +2 % to -13 % for increasing offers of the retanning agent. On the other hand, softness strongly increased from -28 % to +17 % when fatliquoring offer varied from 0 % to 10%.



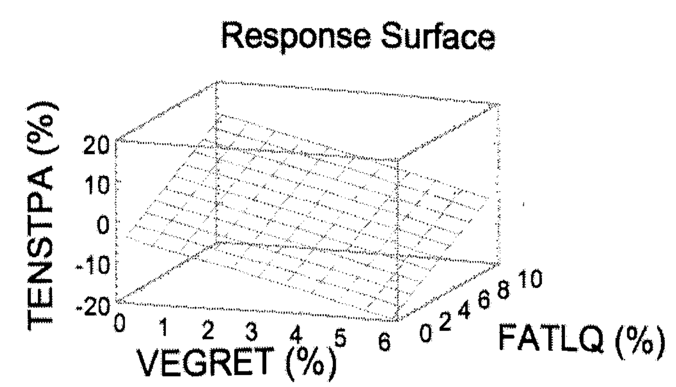
VEGRET: Vegetable Retanning Agent; **FATLQ:** Fatliquoring Agent

Figure 3. Response surface for softness as a function of vegetable retanning agent and fatliquoring agent offers



VEGRET: Vegetable Retanning Agent; **FATLQ:** Fatliquoring Agent

Figure 4. Response surface for colour intensity as a function of vegetable retanning agent and fatliquoring agent offers



VEGRET: Vegetable Retanning Agent; **FATLQ:** Fatliquoring Agent
TENSTPA: Tensile Strength for parallel test samples

Figure 5. Response surface for tensile strength for parallel test samples as a function of vegetable retanning agent and fatliquoring agent offers

The highest softness values corresponded to a vegetable retanning agent offer of 0 % and a 10 % offer of the fatliquoring agent as observed in figure 3.

Colour intensity

Colour intensity L* was measured in thirty three points of each half skin by using a reflexion spectrophotometer.

Values of the right halves were compared with those of the left halves by means of formula 1. Table V shows that for colour intensity the determination coefficient (R^2) obtained was 96.9%. Both variables, vegetable retanning agent offer and fatliquoring agent offer, exerted considerable influence on colour intensity (significance level 0.1 %).

Given that high luminosity (L*) values correspond to values of lower intensity, we changed the sign in the graph so that "colour" denomination correspond to intensity.

Colour intensity strongly decreased from +17 % to -2 % for increasing offers of the vegetable retanning agent. Likewise, the intensity also diminished from +13 % to +3 % for higher offers of the fatliquoring agent.

The highest colour intensity values corresponded to an offer of 0 % of both the vegetable retanning agent and the fatliquoring agent as observed in figure 4.

Tensile strength and percentage of elongation at break

Tensile strength and percentage of elongation at break were determined in accordance with the IUP-6 Standard for test samples cut perpendicular and parallel to the backbone.

a) Tensile strength

a-1) Parallel test samples

The determination coefficient (R^2) obtained for this property was 70.8 % as shown in Table V. The significance of the influence exerted by both variables on the tensile strength for parallel test samples was small (significance level 5 %).

Tensile strength strongly decreased from +4 % to -11 % for increasing offers of the vegetable retanning agent; however, the tensile strength considerably increased from -12 % to +5 % for higher offers of the fatliquoring agent.

The highest tensile strength values corresponded to an offer of 0 % of the vegetable retanning agent and 10 % of the fatliquoring agent as observed in figure 5.

a-2) Perpendicular test samples

Table V shows that a determination coefficient value (R^2) of 64.3 % was obtained. As in the parallel test samples, the significance level of the influence exerted by both variables on this property for perpendicular test samples was small (10 % for the offer of vegetable retanning agent and 5 % for the offer of fatliquoring agent).

TABLE II

Average of the measured values, standard deviation and property variation for treatments 1 - 3

Property	Units	Treatment 7			Treatment 8			Treatment 9		
		Right half (variable)	Left half (control)	Δ (%) (formula 1)	Right half (variable)	Left half (control)	Δ (%) (formula 1)	Right half (variable)	Left half (control)	Δ (%) (formula 1)
Softness										
	mm	4,56±0,28	6,80±0,37	- 33	4,58±0,27	5,96±0,35	- 23	4,12±0,26	6,07±0,34	- 32
Colour Intensity										
	L*	78,0±3,8	62,4±2,9	25	68,6±3.6	61,3±3,0	12	64,6±3,0	61,9±2,8	4.5
Tensile Strength										
Parallel	N/mm^2	26,05±1,09	26,00±0,96	0,2	19,05±0,72	25,40±1,02	- 25	24,30±0,97	28,00±1,06	- 13
Perpend.	N/mm^2	21,00±0,86	21,00±0,78	0	19,93±0,78	22,40±0,94	-11	17,80±0,68	21,30±0,75	-16,2
Elong. at break										
Parallel	%	19,0±1,2	24,0±1,6	- 21	24,0±1,7	27,0±1,9	- 11	18,0±1,2	28,0±1,8	- 35
Perpend.	%	49,0±3,2	63,0±4,4	- 22	46,0±3,2	60,0±4,0	- 24	49,0±3,2	64,0±4,3	- 23
Tear resistance										
Parallel	N	25,5±0,8	51,0±1,8	- 50	31,3±1,5	52,3±2,2	- 40	23,7±0,9	48,0±1,6	- 52
Perpend.	N	43,2±1,5	57,6±2,2	- 25	44,6±2,0	58,0±2,3	- 23	47,9±1,5	56,4±1,7	- 15
Grain Resistance										
	mm	5,37±0,11	8,27±0,15	- 35	6,97±0,13	8,45±0,15	-17,5	5,30±0,10	8,38±0,13	- 37
Grain firmness										
	(0-10)	4,2±0,18	4,0±0,16	5	5,5±0,22	5,0±0,21	10	7,15±0,31	5,5±0,23	30

TABLE III

Average of the measured values, standard deviation and property variation for treatments 4 - 6

Property	Units	Treatment 4			Treatment 5			Treatment 6		
		Right half (variable)	Left half (control)	Δ (%) (formula 1)	Right half (variable)	Left half (control)	Δ (%) (formula 1)	Right half (variable)	Left half (control)	Δ (%) (formula 1)
Softness										
	mm	6,87±0,38	6,58±0,38	4,5	7,11±0,39	7,01±0,39	1,5	6,03±0,34	6,94±0,37	- 13
Colour Intensity										
	L*	73,9±3,7	62,8±3,0	17,7	67,1±3,1	63,0±2,9	6,6	59,8±3,0	62,7±3,1	- 4,5
Tensile Strength										
Parallel	N/mm^2	28,90±1,13	27,70±1,14	4,4	30,90±1,05	30,00±1,11	3	22,50±0,83	26,70±0,99	-15,4
Perpend.	N/mm^2	21,80±0,81	19,80±0,79	10,4	25,80±0,90	21,90±0,79	18	23,90±0,86	20,70±0,72	15,4
Elong. at break										
Parallel	%	26,0±1,7	25,0±1,7	3,7	32,0±2,3	26,0±1,9	22	29,0±1,9	23,0±1,6	26
Perpend.	%	83,0±5,6	67,0±4,6	24	56,0±3,9	66,0±4,8	-15,3	49,0±3,3	59,0±4,1	- 17
Tear resistance										
Parallel	N	30,0±1,3	52,8±2,0	- 43	47,7±2,0	49,4±2,0	- 3,4	51,6±2,1	53,2±1,9	-3
Perpend.	N	34,2±1,3	55,2±1,9	- 38	59,2±2,5	58,7±2,3	0,8	60,5±2,6	59,4±2,3	2
Grain Resistance										
	mm	7,95±0,14	7,80±0,16	2	8,04±0,16	8,00±0,15	0,5	7,02±0,11	7,80±0,13	- 10
Grain firmness										
	(0-10)	3,86±0,18	4,0±0,19	- 3,3	5,02±0,23	5,2±0,23	- 3,3	5,28±0,26	4,8±0,23	10

TABLE IV
Average of the measured values, standard deviation and property variation for treatments 7 - 9

Property	Units	Treatment 7 Right half (variable)	Treatment 7 Left half (control)	Treatment 7 Δ (%) (formula 1)	Treatment 8 Right half (variable)	Treatment 8 Left half (control)	Treatment 8 Δ (%) (formula 1)	Treatment 9 Right half (variable)	Treatment 9 Left half (control)	Treatment 9 Δ (%) (formula 1)
Softness										
	mm	9,10±0,47	6,70±0,35	36	6,06±0,33	5,95±0,36	2	6,67±0,35	6,09±0,37	9,6
Colour Intensity										
	L*	67,5±3,2	60,7±2,8	11,3	62,8±3,2	61,4±3,0	2,3	59,2±2,9	62,0±2,9	- 4,5
Tensile Strength										
Parallel	N/mm²	33,00±1,30	29,40±1,12	12,5	28,68±1,18	28,60±1,12	0,3	29,30±1,03	29,70±1,10	- 1,2
Perpend.	N/mm²	29,80±1,19	19,50±0,72	53	30,50±1,22	23,0±0,94	33	19,10±0,71	21,70±0,82	- 11,6
Elong. at break										
Parallel	%	32,0±2,2	28,0±1,8	15,3	33,0±2.1	29,0±2,0	13,8	26,0±1,8	23,0±1,5	13
Perpend.	%	80,0±5,4	65,0±4,1	24	69,0±4,8	62,0±4,4	11,6	58,0±4,2	63,0±4,3	- 7,7
Tear resistance										
Parallel	N	51,2±2,2	47,5±1,8	7,9	64,7±2,5	51,8±2,2	25	79,3±2,8	48,4±2,0	64
Perpend.	N	68,9±2,8	57,4±2,1	20	70,2±2,6	56,2±2,5	25	71,5±2,8	55,0±2,4	30
Grain Resistance										
	mm	9,39±0,16	8,70±0,16	8	9,90±0,21	8,90±0,17	11,3	7,80±0,13	7,90±0,14	- 1,5
Grain firmness										
	(0-10)	5,31±0,20	5,5±0,21	- 3,3	5,47±0,27	5,3±0,27	3,3	4,53±0,19	4,7±0,19	- 3,6

TABLE V
Regression Coefficients, Determination Coefficients and Significance Levels of the Variables for the Physical and Organoleptic Properties

Effect	Softness	Colour	Tensile Strength Parallel	Tensile Strength Perpend.	Elongation at Break Parallel	Elongation at Break Perpend.	Tear Resistance Parallel	Tear Resistance Perpend.	Grain Resistance	Grain Firmness
Constant	- 20.72	22.97	-4.25	5.81	- 16.21	- 9.36	- 51.48	- 35.58	- 29.37	0.42
Veg Ret Ag.: V_1	- 2.38	-3.25^a	-2.59^c	-4.21^d	0.33	-4.09^c	0.39^c	3.33^a	5.59	4.22^c
Fatl. Ag.: V_2	4.52^b	-1.08^a	1.65^c	3.39^c	3.64^c	3.23^c	5.06^a	4.6^b	7.36^a	-0.36^c
V_1^2	-	-	-	-	-	-	-	-	-1.15^c	-
$V_1 x V_2$	-	-	-	-	-	-	0.97^c	-	-	-0.42^d
V_2^2	-	-	-	-	-	-	-	-	-0.38^d	-
R^2	84.8	96.9	70.8	64.3	57.3	77.5	94.9	80.4	96.1	82.9
R^2 adj.	79.7	95.9	61.1	52.4	43.0	70.0	91.9	73.9	92.2	72.6

Significance levels: [a]0.1 %; [b]1 %; [c]5 %, [d]10

Tensile strength strongly decreased from +23 % to -3 % for increasing offers of the vegetable retanning agent. By contrast, the tensile strength considerably increased from -7 % to +27 % when the offer of the fatliquoring agent varied from 0 % to 10 %.

As in the parallel test samples, the highest tensile strength values corresponded to an offer of 0 % of the vegetable retanning agent and 10 % of the fatliquoring agent as observed in figure 6.

b) Percentage of elongation at break

b-1) Parallel test samples

Table V shows that the determination coefficient (R^2) value obtained for elongation at break was 57.3 %. This Table also shows that the influence exerted by the offer of the vegetable retanning agent was not significant, whereas that exerted by the offer of the fatliquoring agent had a significance level of 5 %.

Elongation at break for parallel test samples slightly increased from +2% to +4 % for increasing offers of the vegetable

retanning agent. Likewise, the elongation at break strongly increased from -15 % to +21 % when the offer of the fatliquoring agent varied from 0 % to 10 %.

The highest elongation at break values corresponded to an offer of 10 % of the fatliquoring agent. However, this remained practically constant for all offers of the vegetable retanning agent as observed in figure 7.

b-2) Perpendicular test samples

A determination coefficient value (R^2) of 77.5 % was obtained as shown in Table V. The significance of the influence exerted by both variables on the elongation at break for perpendicular test samples was small (significance level 5 %).

Elongation at break decreased from +7 % to -18 % for increasing offers of the vegetable retanning agent and strongly increased from -22 % to +11 % when the offer of the fatliquoring agent increased from 0 % to 10 %.

Figure 8 reveals that the highest elongation at break values corresponded to an offer of 0 % of the vegetable retanning agent and an offer of 10 % of the fatliquoring agent.

Therefore, when the elongation at break is considered, there are differences not only in the absolute values for parallel/perpendicular test samples but also in the effect of the vegetable retanning agent offer.

Tear resistance

Tear resistance was determined in accordance with the IUP-8 Standard for test samples cut perpendicular and parallel to the backbone.

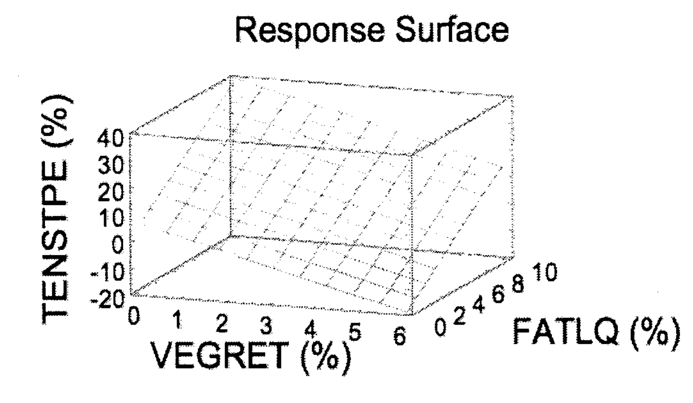
a) Parallel test samples

The determination coefficient (R^2) obtained for this property was 94.9 % as observed in Table V. The influence exerted by the offer of the fatliquoring agent was highly significant (significance level 0.1 %), whereas the significance level of the influence exerted by the vegetable retanning agent was small (5 %).

Tear resistance for parallel to the backbone test samples increased from -26 % to +7 % when offers of the vegetable retanning agent increased from 0 % to 6 %. Likewise, the tear resistance strongly increased from -51 % to +29 % when the offer of the fatliquoring agent also increased.

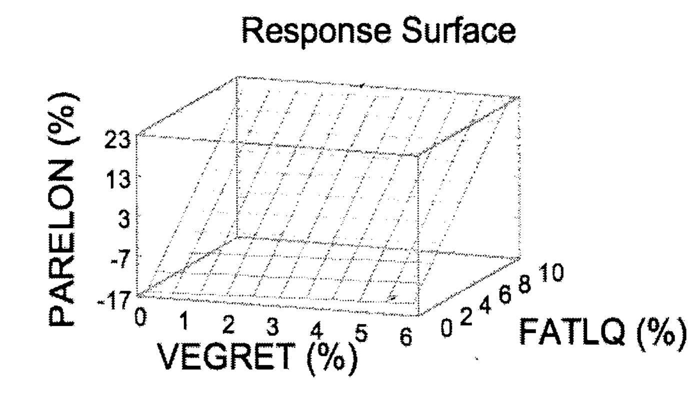
An interaction between both variables, which was significant at 5 % level, was observed for this property. For a fatliquoring agent offer of 0 %, the tear resistance values slightly increased from -52 % to -47 % for increasing offers of the vegetable retanning agent, whereas for a fatliquoring agent offer of 10 % the tear resistance strongly increased from 0 % to +60 % when the offer of the vegetable retanning agent varied from 0 % to 6 %.

The highest tear resistance values corresponded to an offer of 6 % of the vegetable retanning agent and 10 % of the



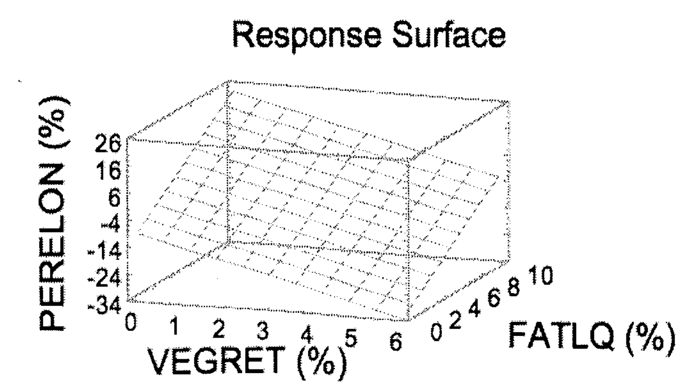
VEGRET: Vegetable Retanning Agent; **FATLQ:** Fatliquoring Agent
TENSTPA: Tensile Strength for perpendicular test samples

Figure 6. Response surface for tensile strength for perpendicular test samples as a function of vegetable retanning agent and fatliquoring agent offers



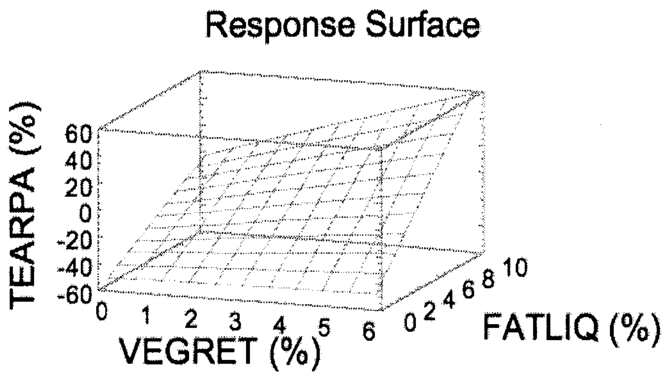
VEGRET: Vegetable Retanning Agent; **FATLQ:** Fatliquoring Agent
PARELON: Elongation at break for parallel test samples

Figure 7. Response surface for elongation at break for parallel test samples as a function of vegetable retanning agent and fatliquoring agent offers



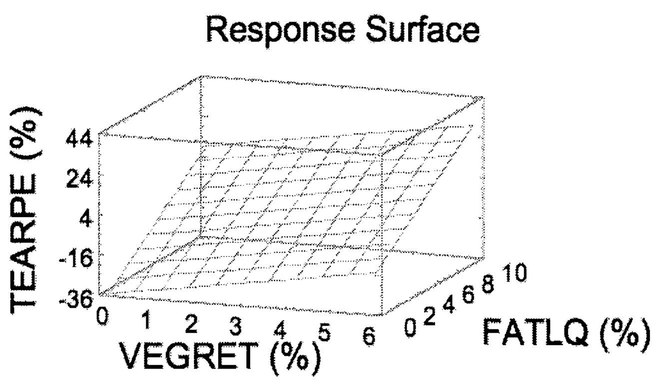
VEGRET: Vegetable Retanning Agent; **FATLQ:** Fatliquoring Agent
PARELON: Elongation at break for perpendicular test samples

Figure 8. Response surface for elongation at break for perpendicular test samples as a function of vegetable retanning agent and fatliquoring agent offers



VEGRET: Vegetable Retanning Agent; **FATLQ:** Fatliquoring Agent
TEARPA: Tear resistance for parallel test samples

Figure 9. Response surface for tear resistance for parallel test samples as a function of vegetable retanning agent and fatliquoring agent offers



VEGRET: Vegetable Retanning Agent; **FATLQ:** Fatliquoring Agent
TEARPA: Tear resistance for perpendicular test samples

Figure 10. Response surface for tear resistance for perpendicular test samples as a function of vegetable retanning agent and fatliquoring agent offers

fatliquoring agent as observed in figure 9.

b) Perpendicular test samples

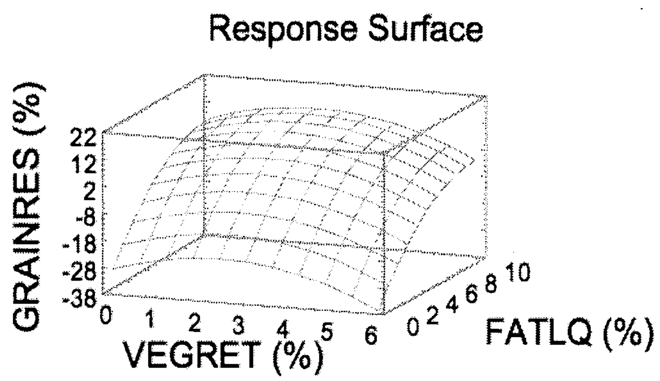
A determination coefficient value (R^2) of 80.4 % was obtained, as shown in Table V. This Table also shows that the influence exerted by the two variables on this property was highly significant (0.1% for the offer of the vegetable retanning agent and 1 % for the offer of the fatliquoring agent).

Tear resistance increased from -13 % to +7 % for increasing offers of the vegetable retanning agent. This increase was more marked, from -26 % to +20 % when the offer of the fatliquoring agent varied from 0 % to 10 %.

As in the parallel test samples, the highest tear resistance values corresponded to an offer of 6 % of the vegetable retanning agent and 10 % of the fatliquoring agent as shown in figure 10.

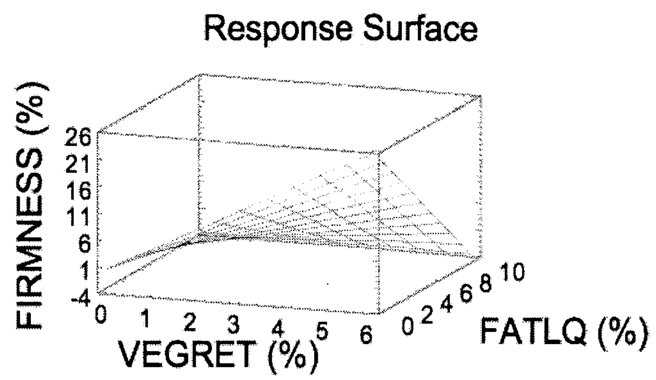
Grain resistance

Grain resistance was determined in accordance with the IUP-9 Standard



VEGRET: Vegetable Retanning Agent; **FATLQ:** Fatliquoring Agent
GRAINRES: Grain Resistance

Figure 11. Response surface for grain resistance as a function of vegetable retanning agent and fatliquoring agent offers



VEGRET: Vegetable Retanning Agent; **FATLQ:** Fatliquoring Agent
FIRMNESS: Grain Resistance

Figure 12. Response surface for grain firmness as a function of vegetable retanning agent and fatliquoring agent offers

A determination coefficient (R^2) value of 96,1 % was obtained as shown in Table V. This Table also shows that the influence exerted by the offer of the vegetable retanning agent was not significant, whereas that exerted by the offer of the fatliquoring agent was highly significant (significance level of 0.1 %).

Grain resistance increased from -3 % to +5 % when the offer of the vegetable retanning agent varied from 0 % to 3%. However, grain resistance decreased for vegetable retanning agent offers from 3 % to 6%. As far as the fatliquoring process is concerned, the grain resistance strongly increased from -23 % to +13 % when the offers of the fatliquoring agent varied from 0 to 10%.

The highest grain resistance values were provided by offers of 3 % and 10 % of the vegetable retanning agent and the fatliquoring agent respectively, as shown in figure 11.

Grain firmness

Grain firmness was determined by using the break/pipiness scale apparatus in accordance with the Satra PM-36 method.

Table V shows that the determination coefficient (R^2) value obtained for elongation at break was 82.9 %. The significance of the influence exerted by both variables on grain firmness was small (significance level 5 %).

Grain firmness increased from -1 % to +11 % for increasing offers of the vegetable retanning agent but considerably decreased from +13 % to -3 % when the offer of the fatliquoring agent varied from 0 % to 10 %.

An interaction between both variables, which was significant at 10 % level, was observed for this property. Grain firmness was constant when the offer of the vegetable retanning agent varied from 0 % to 6 % for an offer of the fatliquoring agent of 10 %. However, grain firmness strongly increased from +1 % to +26 % for a fatliquoring agent offer of 0 % for increasing offers of the vegetable retanning agent.

The highest grain firmness was obtained with offers of 6 % and 0 % of the vegetable retanning agent and the fatliquoring agent respectively as observed in figure 12.

CONCLUSIONS

- The highest softness values corresponded to a vegetable retanning agent offer of 0 % and 10 % of fatliquoring agent.
- Offers of 0 % of both fatliquoring and vegetable retanning agents provided the maximum colour intensity.
- The highest tensile strength values for both parallel and perpendicular test samples corresponded to a 0 % offer of the vegetable retanning agent and to a 10 % offer of the fatliquoring agent.
- Increases in fatliquoring agent offers resulted in enhanced elongation values for both parallel and perpendicular test samples. The maximum elongation for perpendicular test samples corresponded to a vegetable retanning agent offer of 0 %, whereas this variable was not significant for parallel test samples.
- The highest values of tear resistance for both parallel/perpendicular test samples corresponded to a vegetable retanning agent offer of 6 % and to a fatliquoring agent offer of 10 %.
- Offers of 3 % and 10 % of vegetable retanning agent and fatliquoring agent, respectively, resulted in the best grain resistance values.
- The best grain firmness values corresponded to an offer of 6 % of the vegetable retanning agent and to an offer of 0 % of the fatliquoring agent.

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