AN ACOUSTIC EMISSION STUDY OF STAKING AND FATLIQUOR*

by
PAUL KRONICK, ANGELA PAGE AND MICHAEL KOMANOWSKY
Eastern Regional Research Center
U. S. Department of Agriculture, Agricultural Research Service
600 EAST MERMAID LANE
PHILADELPHIA, PA 19118

5962

Abstract

The chemical treatment of leather with fatliquor and the physical treatment of staking produce similar esthetic results. The effects of two such different treatments on the structure of the leather, however, and perhaps various other responses to deformation, are expected to be different. The equivalence of fatliquoring and staking were investigated by comparing the effects of the treatments on the stress-strain curves and the acoustic emission observed during tensile testing. It was found that staking affects only the first 25% of the elongation at break, lowering the initial modulus, while fatliquor affects the whole stress-strain curve. The initial part becomes concave upward instead of downward, and the elongation and stress at failure are greater. Acoustic emission from leather that was merely staked was suppressed only at small elongations, while it was smaller at all elongations from leather that had fatliquor. The results are shown to be consistent with a domain model of deformation. A nondestructive determination of the elongation of leather at tensile failure, based on acoustic emission, is derived from the observations.

Introduction

The temper (stiffness) of leather is set by the addition of oily materials and fatliquors and is adjusted after drying by the mechanical working called staking. Fatliquor can be added to the retanning drum with little effect on productivity, although with considerable increase in the cost; staking requires no additional materials but can be very inefficient, adding production time, labor and capital overhead. Staking can be done to selected areas of a hide and so can make it more uniform than fatliquor, but the two manufacturing steps are evaluated by more or less the same aesthetic results: temper and roundness.

Although their purpose is the same, the effects of two such very different treatments on the structure and properties of the leather would be expected to be different. These differences must be taken into account when attempts are made to lower costs by curtailing the treatments or balancing them against each other. We have undertaken here a comparison of the effects of fatliquor and dry staking on the mechanisms of deformation of leather. To study the mechanisms we monitor the sounds emitted when specimens are stretched during a stress-strain test. The

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* Presented at the 89th Annual Meeting of the American Leather Chemists Association, Boyne Mountain, Michigan, June 14, 1993.

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rationale for this method has been explained earlier[1,2]. Mostly, we are dealing with the adhesions between fibers. The acoustic information considerably expands the interpretation of the stress-strain data, can guide the choice of method used to lower the temper, and can lead to prediction of the elongation at break of a sample without destroying it.

Materials and Methods

Two sides of frozen mature cattlehide were tanned through the blue by the Standard ERRC Process[3]. Each side was then divided evenly between the tail and neck ends, and the tail end of each side was treated, to 5%, with Reilly-Whiteman (Conshohocken, PA) fatliquor X-76-31, a "solvent-type oil," 2% sulfated. Then, one of the sides was dried at constant area at room temperature in still air and was passed through a Molissa staking machine. The other side was dried similarly but without staking.

To obtain acoustic-emission data, a small piezoelectric transducer resonating at 150 kHz (Model R15/C, Physical Acoustics Corp., Princeton, NJ), 10 mm diameter and weighing 20 g, coated with a film of petroleum grease for more efficient acoustic coupling, was clipped against the sample in an Instron tensile tester. Electric signals emanating from this transducer and from the force transducer of the Instron when the sample was stretched were processed with a LOCAN-AT acoustic emission analyzer (Physical Acoustics Corp.), which saved the acoustic and force data on floppy diskettes for later analysis on a VAX-4000 Model 300 computer. Each acoustic pulse from an event in the sample caused a damped oscillation to be emitted by the transducer. The analyzer recorded the arrival time of each oscillation pulse, its amplitude, and its energy. Calculations of forces, acoustic event rates, energies, and pulse-amplitude distributions were carried out with a procedure written in the RS/1 data-analysis program (BBN Software Products Corp., Cambridge, MA), which reproduced the results from Physical Acoustic's LOCAN software. Only pulses giving maximum amplitudes greater than 60 mV from the transducer were counted. The pulse rates were calculated as numbers of pulses counted over sequential elongation intervals of 1% of the elongations at break; the pulse energies were averaged over these intervals by dividing the total energies over these intervals by the corresponding numbers of pulses.

The energies of the pulses, estimated by the "ring-down" method[2], are proportional to the average areas underneath the rectified pulses, so are determined by the pulse amplitudes and the pulse durations. Since the pulses are highly damped in leather, the durations are always short and increase with the pulse amplitudes. Therefore the energies are correlated with the pulse amplitudes.

The tensile tests were carried out at 75° C, 80% relative humidity with an elongation speed of 20%/min on six "dog-bone" samples, 100 mm × 20 mm × 1.5 mm, from each material.

Results

Untreated Leather

The stress-strain curve of untreated leather from the center of the hide, cut perpendicular or parallel to the backbone, has a relatively steep slope the first time that the leather is stretched. The curve is concave downward for the first 4% of elongation, and then apparently becomes linear until shortly before failure (Fig. 1) at an average elongation of 34% (Table I). We have shown previously[1] that the linear portion does not indicate linear elasticity; rather, it marks a subtle inflection where the curve becomes concave upward and a new mechanism of deformation appears. Over this quasi-linear part of the stress strain curve the rate of acoustic emission is constant to the failure point, as the mutually adhering fibers are pulled apart. After

TABLE I

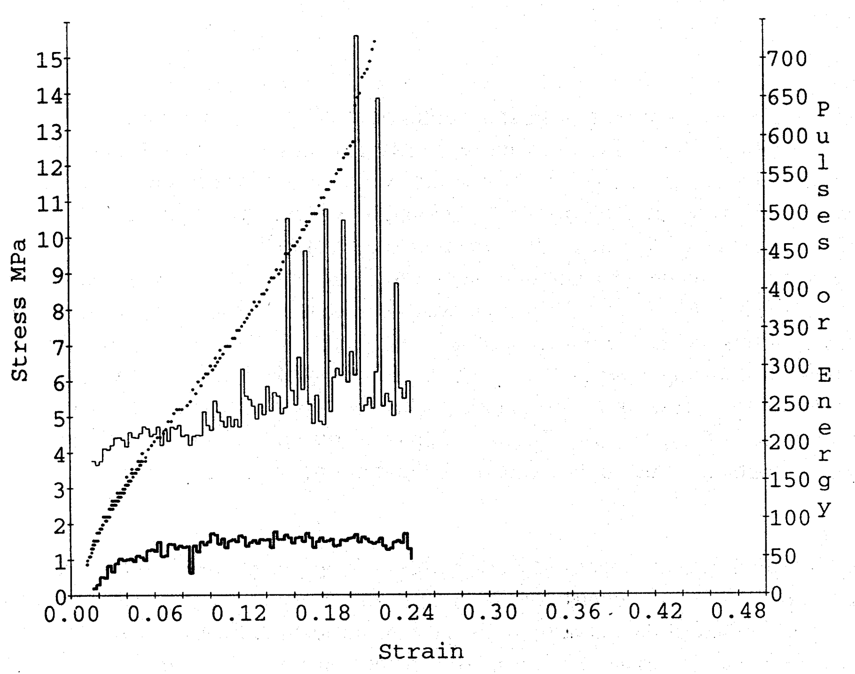
Effect of staking and fatliquor on break elongation and on the onset of frequently emitted acoustic pulses

Treatment			
Staking	Fatliquor	Break elongation	(AE Onset)/ (Break elongation)
-	-	34 ± 6%	0.70 ± 0.04*
+	-	34 ± 10%	0.72 ± 0.07
-	+	46 ± 8%	0.75 ± 0.04
+	+	53 ± 9%	0.71 ± 0.06

*Tests where late AE onset was present (see text).

the point of inflection, however, large-energy pulses are counted, as shown by the energy curve in Fig. 1. The onset of these high-energy pulses is at 15% elongation, or 62% of the elongation at break. We have shown previously that these large pulses are due to breaking fibers[1].

The constant pulse rate over the linear part of the linear stress strain curve until final failure, even with the additional large pulses, means that the rate of fiber breakage must be small. The failure mode is therefore probably slipping apart of adherent fibers. These features are similar in the samples cut parallel to the backbone.



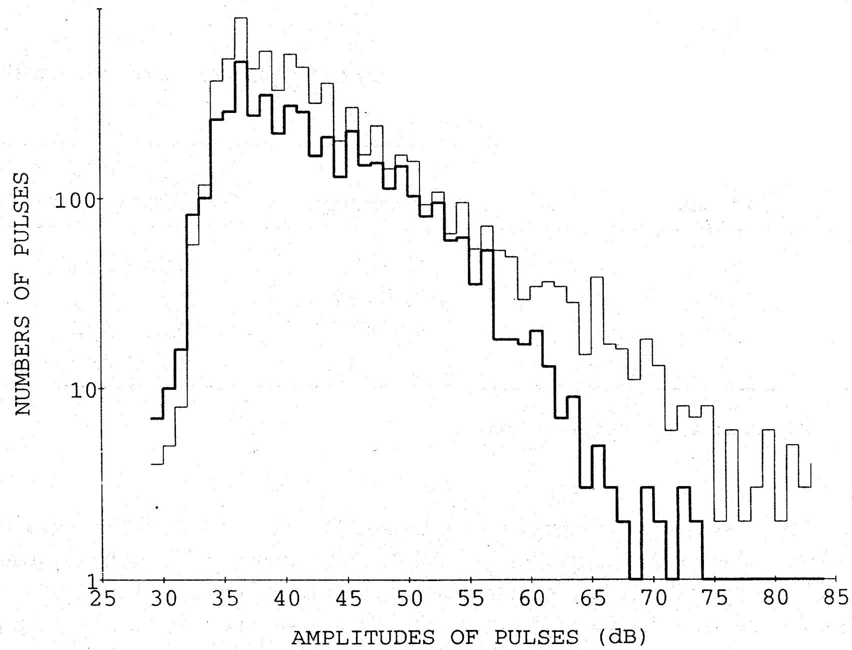


FIG. 2. — Frequency distributions of the pulse amplitudes from two kinds of leather. Thin solid line: staked without fatliquor; bold solid line: staked with fatliquor.

Staked Leather

Staking the leather causes changes in its mechanical and acoustic behavior at small elongations, below 10%. The stress rises more gently and more linearly (less downward concavity) (Fig. 3). The pulse rate also rises linearly, with no downward concavity, and not becoming constant until the elongation of 10%. At elongations beyond 10% until failure at 34% elongation (Table I), the behavior is like that of the untreated material.

The energy curve is unchanged by staking. Again, high energies are observed near failure, accompanied by a slightly rising pulse rate that was occasionally observed also in unstaked samples. The onset of this increase in pulse rate and energy is at 20% elongation, or 74% of the elongation at break. The average energy rises with the pulse rate; here, apparently, many fibers are breaking instead of a few sporadic breaks. That is, fiber breakage is an important contribution to the mode of failure, in addition to slippage. This conclusion is confirmed by the elevated numbers of high amplitude pulses shown by the thin line of Fig. 2.

Fatliquor

The addition of fatliquor to the unstaked leather, like staking, reduces the initial slope of the stress-strain curve, even making it concave upward (Fig. 4). The rate of accompanying acoustic pulses is lower, over the part of the curve up to the point of inflection, than when the leather was staked, but with fatliquor it begins to rise at the inflection until the sample fails at 46% elongation (Table I).

The pulse energies of the fatliquored leather are nearly constant over the whole elongation. The scatter of the data is great because the pulse rates from which they are averaged are small,

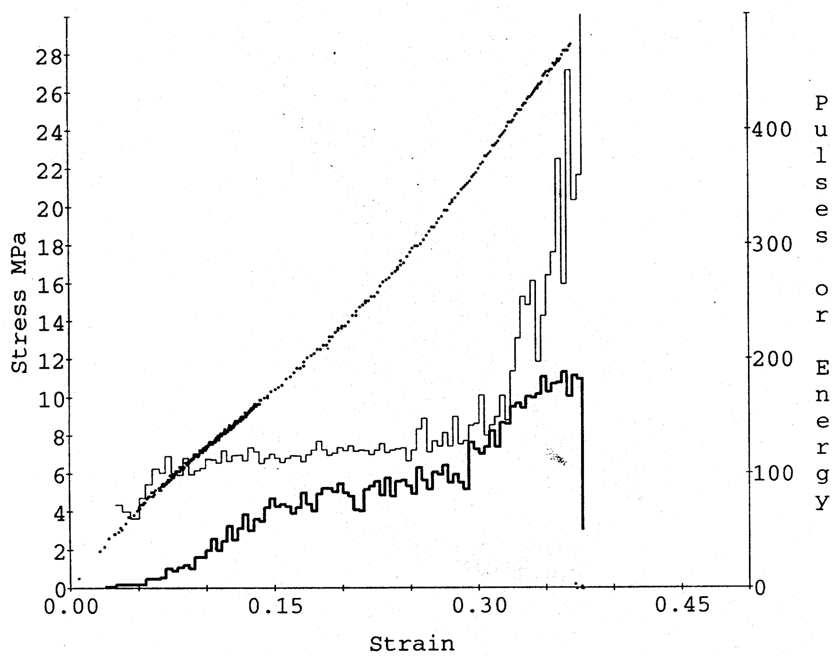


FIG. 3. — Acoustic emission during elongation of leather that had been staked without fatliquor. Thin line: energy per pulse, averaged over an elongation interval of 1% of the elongation at break; thick line: number of pulses on this elongation interval; scatter plot: stress vs strain.

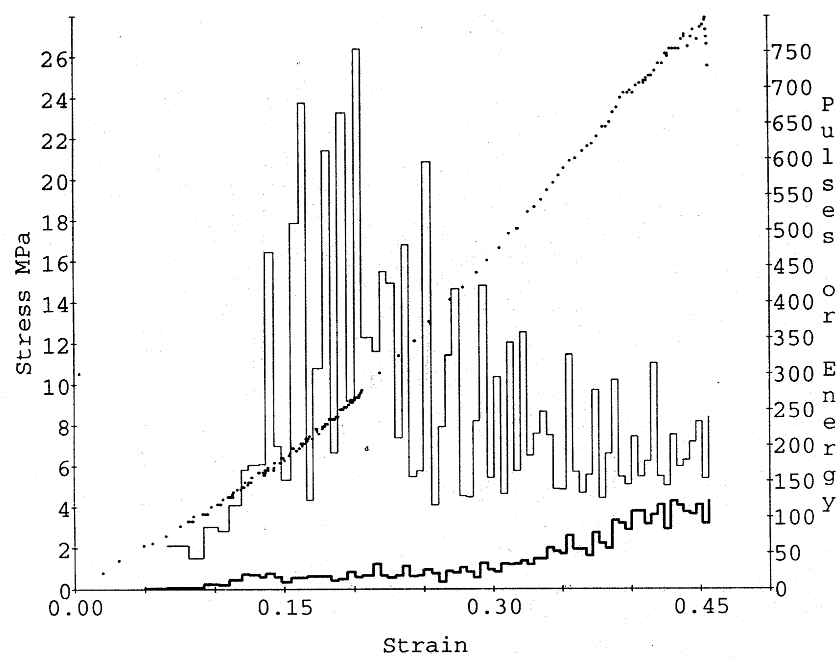


FIG. 4. — Acoustic emission during elongation of leather that had been dried from fatliquor, but not staked. Thin line: energy per pulse, averaged over an elongation interval of 1% of the elongation at break; thick line: number of pulses on this elongation interval; scatter plot: stress vs. strain.

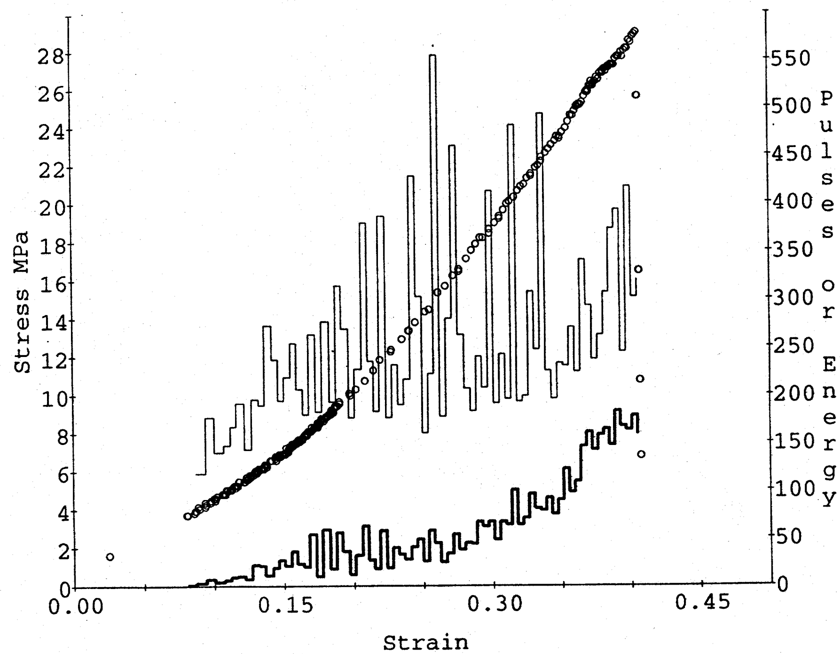


FIG. 5. — Acoustic emission during elongation of leather that had been treated with both fatliquor and staking. Thin line: energy per pulse; averaged over an elongation interval of 1% of the elongation at break; thick line: number of pulses on this elongation interval; scatter plot: stress vs strain.

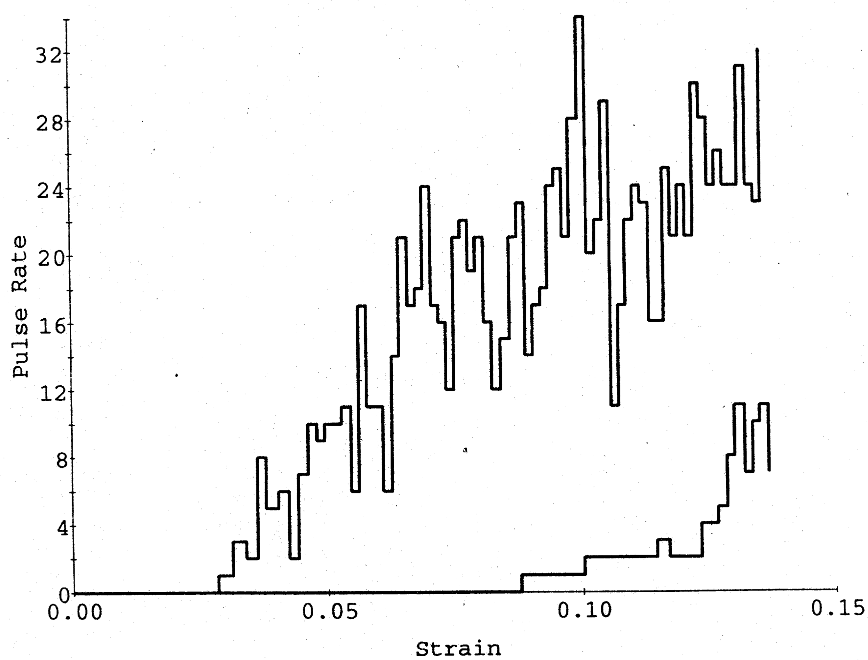


FIG. 6. — Rates of emission during a 13% elongation of sheepskin that had been air-dried from fatliquor but not staked. Upper curve: first elongation; lower curve: second elongation, after retraction from the first at zero stress over a period of three months.

with relatively large variability, but it seems to become gradually smaller as the pulse rate rises and the sample approaches failure.

Staking and Fatliquoring

If, in addition to treatment with fatliquor, the leather is then staked, the additional effect on the behavior of the leather was but subtle. In samples taken from the middle of the hides the acoustic emission almost vanished at elongation below 8% if the leather had been staked, as shown in the pulse rate curve of Fig. 5. This effect of staking is more apparent in samples taken from the butt than from the middle. The rising pulse rate starting at about 0.7 of the elongation at break is common to all three *treated* leathers (Table I). On the other hand, the leather with the combination treatment (fatliquor and staking), although like that which had been only fatliquored, was mechanically different from that with only the staking treatment, being much softer and emitting much fewer acoustic pulses at all elongations. As seen in Fig. 2 the amplitudes of the pulses are generally smaller in the fatliquored leather[1], compared with staked leather that had not been staked. The elongation at break was not statistically different from that of unstaked samples with fatliquor.

Discussion

Leather is fatliquored and staked to make it pliable or soft. The result of either treatment can therefore be seen in the corresponding stress-strain curve, of which the initial slope (elastic modulus) is reduced from that of the untreated leather. The softness of leather is determined by its resistance to only small extensions, where the stress-strain curve of untreated material is concave downward. After staking or fatliquor the curves become concave upward. The increasing elastic modulus at larger elongations, however, does not affect how easily the leather bends.

Staking and fatliquor, however, do not yield equivalent results. The main difference is in the smaller amounts of incidents of cohesive failure, seen in the much lower rate of emission of acoustic pulses in fatliquored leather (cf. Fig. 4). Staking also suppresses the acoustic emission, but only at small elongations. This suppression seems to be an example of the "Kaiser effect"[2], which we illustrate with Fig. 6. This figure shows acoustic emission from a sheepskin sample when it was stretched 13% twice, with a three-month period of relaxation between stretches. The sample recovered its original dimensions during the relaxation period. During the first stretch the acoustic pulse rate increased continuously with elongation over the whole interval; on the second, however, the sample was silent over 80% of the elongation interval.

This diminution of acoustic activity after the first time that a material is deformed is very common among many types of plastic, metallic and fibrous materials[2]. The ratio of the stress at which it resumes on the second cycle to the maximum stress of the test, here 80%, is called the "felicity ratio." For metals it is usually 1; for fibrous composites, the amount by which it is less than 1 is a measure of internal damage during the first cycle. In the case of leather it could also mean that interfiber adhesions partially reform while standing.

The difference between the effects of staking and fatliquor can be understood by a consideration of deformation mechanisms. Leather is not a homogeneous material. Parallel bunches of its fibrils are organized into fibers; where these find themselves mutually parallel and close together, they are again organized into cohesive domains. When the leather is stretched, the body yields by the opening of crazes lying between the cohesive fibrous domains. Crazes are crack-like volumes in a failing fibrous body that are filled with fine, pulled-apart fibers. Some of the regions that yield to such cracking have greater cohesive strength than others. As the crosshead of the testing machine moves and the local stress increases beyond the strength of some of these weaker regions, more of them open into cracks, and the sample elongates. As in crazing, the open cracks need not be empty; there might be a few nonadherent fibers spanning them. What is important is that some cracks, either because of their orientation or their intrinsic strengths, open before others.

If the sample is stretched less than about 60% of the elongation at break, before fiber breakage is frequent, and then allowed to relax, the cracks close again under the elastic forces of the intact domains (and, perhaps, single fibers still spanning the cracks), but they are no longer internally cohesive. The adhesions between the domains that flank them have been broken. On a following deformation, some cracks will open, more easily than before, because of the lost adhesion between domains. The re-opening should therefore occur without acoustic emission.

The above mechanism of deformation, based on domains, fully explains the changes in the stress-strain curve and acoustic emission of leather due to staking, if it assumed that in this treatment the leather is stretched 10%. This is the elongation at which the slope of the stress-strain curve and the acoustic activity resume their normal values in the staked bovine leather of our experiments.

When fatliquor is applied before the leather is dried, it reaches the fibrils that, on drying, will lie within the cohesive domains, as well as those in the cracks. On drying, then, all the adhesions in the leather are weaker than in the untreated material. This effect, on the total volume of the leather, distinguishes the action of fatliquor from that of staking.

The difference is evident in the stress-strain curve and is confirmed by the acoustic emission. The stress-strain curve is not only concave upward over much of the elongation, but the elongation at break is much greater than in the untreated material (Table I). This is evidently due to the weaker adhesions between fibrils, allowing more cracks to open at smaller stresses, freeing more fibers and allowing them to move about to comply with the imposed deformation. The smaller acoustic pulse rate is another result of the diminished adhesion among fibrils and fibers. The domains can continue to break down, up to strains of about 46%, before fibers finally slip apart and the sample fails: from the acoustic-emission data we see no evidence of a substantial contribution of fiber breakage to the mechanical failure.

This mechanism also provides a reason to carry out both staking and fatliquor treatments. Staking affects the mechanical properties only at small elongations, which define softness. Its effect is enhanced somewhat by the addition of fatliquor, since it increases the number of the weakest cracks. For springiness and resilience, however, the behavior at larger elongations becomes important. For these properties, fatliquor, not staking, becomes determinative.

It is worth noting that, on close inspection, an acceleration of the pulse rate can be discerned as failure is approached among all the classes of leathers examined here, even in some untreated samples (although not the sample of Fig. 1). Table I shows that the onset of this acceleration occurs at about 70% to 75% of the elongation at failure, even though the leathers with fatliquor fail at much greater elongations than those without it. The standard errors within the individually treated classes are smaller than those between classes, so the treatments altered this onset point slightly, but the change is minor. This constant AE-onset-to-break ratio means that during the tensile test supplemented with detection of acoustic emission, failure can be anticipated when the acoustic pulse rate begins to increase. Thus acoustic emission can be used nondestructively to determine the elongation at which a leather will tear. (In the case of the leather described here, this would be 140% of the elongation at AE-onset.)

Conclusions

Staking, applied to bovine leather that has been chrome-tanned and dried with no fatliquor, affects the tensile properties only at small elongations, in the case studied here, the first 10% of the stress-strain curve, or 25% of the elongation at break. Over that elongation interval the elastic modulus is diminished, consistent with softening. Acoustic emission is much smaller over the affected interval.

Fatliquor applied to the same leather affects the whole stress-strain curve. The shape is changed, becoming initially concave upward instead of downward, and the elongation at break is greater, consistent with a tougher, more elastic product. Acoustic emission is diminished at all elongations up to failure.

The behavior is consistent with a domain model of elongation, in which the deformation occurs only at the boundaries between cohesive domains. Staking would cause the formation of cracks between boundaries; fatliquoring, the properties of the domains themselves and where the cracks appear. Thus, acoustic emission spectroscopy, coupled with stress-strain measurements, can give fundamental data for evaluating the performance of fatliquors.

Acoustic emission can serve as a nondestructive test to determine the elongation of leather at tensile failure.

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