Early Detection of Looseness in Bovine Hides Using Ultrasonic Imaging

by

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Abstract

The processing of bovine hides to leather results in a significant proportion of defective leather known as loose leather. It has not previously been possible to recognize hides that may produce loose leather. Hides were processed through to leather with samples retained at the pickle, wet blue and crust leather stages with material that resulted in loose leather compared with that resulting in tight leather, using ultrasonic imaging. The loose precursor is characterized by a lower density of material in the mid grain layer. The looseness is quantified by amplitude differences in ultrasound line scans or cross-sectional area scans between loose leather and tight leather with 2-4 times the amount of low intensity area in loose leather at all three process stages. This enables detection of hides that will result in loose leather and may enable unsuitable hides to be diverted to other process streams to save substantial processing costs.

Introduction

Leather produced from bovine hides may exhibit a defect known as looseness. This defect is present in 5-10% of finished bovine leather and results in a greatly reduced value for the leather and therefore a loss of revenue to the industry. At present there is no way to identify unprocessed hides that may result in loose leather nor is it yet possible to predict which animals are likely to produce loose leather. Many unsuitable hides are therefore processed to leather and subsequently rejected or downgraded at the crust leather stage.

Looseness is a term used in the leather industry to describe an undesirable characteristic of leather with an excessive tendency to exhibit wrinkles or creases in a finished product. Looseness describes a coarse (bad) "break" as defined in the ASTM standard [2] "…where a few coarse wrinkles are formed on bending the grain to form a concave surface may indicate that the grain layer is separating from the corium or main stratum"

In loose leather the grain layer tends to be separated into sheet like structures and there are larger gaps between the collagen fibers than in tight leather [3]. The presence of sufficient elastin in the grain layer has been suggested as being necessary to prevent loosenes [4]. It has been shown that looseness is a structural defect that results from a poor connection within or between layers particularly in the lower grain region or grain-corium boundary. This is manifested in less closely packed collagen fibrils and results from a high degree of alignment of collagen fibrils within the loose leather. The more loosely packed and weakly bonded grain of the loose leather, in comparison to tight leather, becomes separated during folding grain-in resulting in the symptoms of poor break or looseness[1].

Ultrasonic imaging has been shown to be able to clearly identify looseness in the leather with a layer of lower intensity of reflected signal in the grin. [1]. Ultrasonic images of looseness in leather look similar to that of aged or sun damaged skin, which also shows a low intensity of reflected signal in the demis. [5]. The technique can also be used for investigation of skin disease, for example in identification of tumors and inflammatory diseas. [6,7].

While looseness is defined for leather, it may be that the structural characteristics that result in looseness are present in the unprocessed hides and could be identified in the hides or in early stages of the tanning process. The looseness test, that of folding leather grain-in to observe the size of the creases, is not able to detect looseness precursors in unprocessed hides or early stages of leather processing. However, if looseness in leather results from structural characteristics of the hide, then it should be possible to identify these structural characteristics by other means. Here we attempt to identify looseness prior to the crust leather stage using ultrasonic imaging.

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EXPERIMENTAL

Sample Selection

A range of hides were selected from the lime-splitting machine at Tasman Tanning Ltd (New Zealand) with the intention to collect some that might turn out to be loose and some that might be tight. There was no way to accurately determine which hides would turn out loose, however some that appeared to have more draw were chosen as being likely candidates. The selection that was hoped to contain some loose and some tight hides was subsequently processed in the LASRA pilot tannery as detailed below.

Leather Preparation

The hide used for measurements in this study was obtained part-processed from a production run of hides at a local tannery. The hides weighed around 35 kg and have been green fleshed and processed using a conventional recipe. This recipe is based on sodium sulfide, sodium hydrosulfide and lime to both depilate and open-up the structure to allow the removal on non-collagenous proteins in drums of 4 m diameter by 3.8 m wide, which are loaded with approximately 8500 kg of hides at a tim. The Rrotationalkept atprovided a sample acceleratio of 0.017 ms^{-2} gduring the liming stageand .0716 gms^{-2} during washing. Hides are taken from the processing drums after 8 h liming. The lime splitter operated at a splitting thickness of 3.5-4.0 mm. Hides were observed passing through the splitter, and hides selected at this stage which displayed more "draw" across the shoulder and flanks than others in the batch. The selected hides were returned to LASRA for onward processing in the LASRA pilot tannery to leather using a conventional recipe for shoe leather production. The next day the horsed-up leather was sammedand sett to achieve a moisture content g kg^{-1} and toggle dried at 40°C on a tunnel drier until dry in all regions. The crust leathers were conditioned heavily with water on the flesh-side and held in this condition for 24 h. The next day moisture measurements were taken to ensure a moisture content of 14 g kg^{-1} in all areas prior to Molissa staking on settings of 4 and 5.

From the crust leather, samples showing looseness were taken from the shoulder region, just above the axilla, with tight samples cut from adjacent areas outside of the affected region. The results presented here are from only one hide. However, other leather samples were analyzed which provided results consistent with those reported here.

Looseness Evaluation

Looseness was evaluated using the standard break test method [2], which involves folding the leather grain side in, and quantifying the wrinkles. Samples were considered tight if they gave a break of 2 or less and loose if they had a break of 4 or more.

Ultrasonic Imaging

Ultrasonic images of the leather were recorded with a DermaScan C USB instrument (Cortex Technologies, Denmark). A 20 MHz 2D-scanning head was used to carry out scanning at 6-8 frames per second over a distance of 12.1 mm in 224 steps. The scanning head contains an internal water chamber to minimize attenuation of the acoustic signal. To further improve image quality, scanning was carried out under water once the leather had been soaked in water for at least 24 h. The sound velocity in leather was calibrated from the time of signal reflection and found to be 2561 (σ = 99) m/s. The bandwidth is 0.75, which resulted in an axial resolution of 97 μm (60 μm at 1580 m/s). The lateral resolution is around 150 μm. The focal point sits at a depth of 13 mm from the ultrasonic transducer face, which allows for the distance from the transducer (through the water-filled chamber) to the barrier membrane and through the water film on the leather, such that the focal point falls approximately within the leather sample. The transducer gain level and gain profile were used to adjust the amplification of the signal. Setting the amplification correctly reduces attenuation, allowing for better signal penetration and image quality. The 20 MHz probe transmits ultrasound with a peak intensity of 0.19 W/mm^2. A custom gain profile was created for use on leather with a minimum intensity of 21 dB at the leather grain surface, increasing to a maximum intensity of 42 dB at the corium.

The ultrasonic data can be displayed in what is conventionally called an A-scan or a B-scan. An A-scan is a line scan that represents depth information from one point on the surface of the leather; a B-Scan is a two dimensional image that represents a cross-sectional area of leather (and is composed of a large number of A-scans, but displayed using color for intensity).

Scanning Electron Microscopy

Small samples were fixed for 8 h at room temperature in a modified Karnovsky's fixative, containing 30 g kg^{-1} glutaraldehyde, 20 g kg^{-1} formaldehyde in phosphate buffer (0.1M, pH 7.2) then washed in phosphate buffer (0.1M, pH 7.2) followed by dehydration in a graded ethanol series. The samples were finally washed for 1 h in pure ethanol and critical-point dried in liquid CO_2 and pure ethanol (intermediary), using the Polaron E3000 series II critical point drying apparatus. The samples were mounted on to aluminum stubs and sputter-coated with gold (Baltec SCD 050 sputter coater) and viewed in the FEI Quanta 200 Environmental Scanning Electron Microscope (SEM) at an accelerating voltage of 20 kV.

Results

Sample Selection

A selection of samples of loose and tight leather were successfully made with materials from the pickle and wet blue stages for each of these retained for testing. The crust leather resulting from these had a break of 2 for the tight leather and 5 for the loose leather.

Ultrasonic Images

The ultrasonic imaging of different stages during the processing of leather was able to clearly show differences between the loose and tight leather at each stage of the process (Figure 1). The images display the intensity of the reflected ultrasonic signal. Reflection occurs at interfaces between areas of high and low density so that the signal can be a measure of the number of these interfaces. Therefore an area of very uniform (at the scale of the wavelength of the ultrasound) high density or very uniform low density should both show as black or green on these images. However, leather is not uniform at the scale of the ultrasonic wavelength used so that regions of a high density of collagen fibers contain many interfaces and display as high intensity color scale (white) while regions of low density show as black or green.

In the tight crust leather the grain is uniformly dener thant the corium and grades gradually to less dense material. In contrast, in loose crust leather the top surface of the grain looks dense (high intensity ultrasonic reflections) but there is a region about 0.5 mm below the top of the grain that is less dense (lower intensity ultrasonic reflections) followed by a denser region below this layer towards the corium. In the tight wet blue material there is a gradation of density from higher density towards the top of the grain. In the loose wet blue there is a more uniform density through the leather. In the tight pickle the hide has dense top grain surface with a gradation of decreasing density deeper into the hide, whereas in the loose pickle there is a low density layer at the grain surface with a more dense region about 0.5 mm below the grain.

From the ultrasonic images it is possible to measure the proportion of the leather that consists of lower density regions. To do this, the "B-scan measure" software feature of the Dermascan was used. A region of interest was selected, and for this a rectangular band 0.378 mm thick was chosen containing the grain or the top part of the grain. The proportion of pixels that were in the intensity range 0-20 (where the total range is 0-255 so that 20 represents 7.8%), under the measurement conditions used for all the samples, was measured (Figure 2).

The portion of the grain that gives low intensity ultrasonic reflections, representing regions of low density, can be quantified as a percentage of the total area selected. From these measurements the differences between tight and loose leather can be represented as a ratio of this low density region Table I1). The loose leather contains 2–4 times the amount of low density area than the tight leather and this is at least as apparent in the pickle and wet blue stages of processing as it is in the crust leather. These differences are statistically significant.

An alternative method of analyzing the ultrasonic data is by using A-scans (single point depth scans). By averaging a series of A-scans to give an average composition of a volume of leather the differences between tight and loose leather are easily seen (Figure 3) with a marked dip in the reflected ultrasonic intensity in the loose leather below the grain surface. The tight leather does not have a comparable drop in intensity in this region.

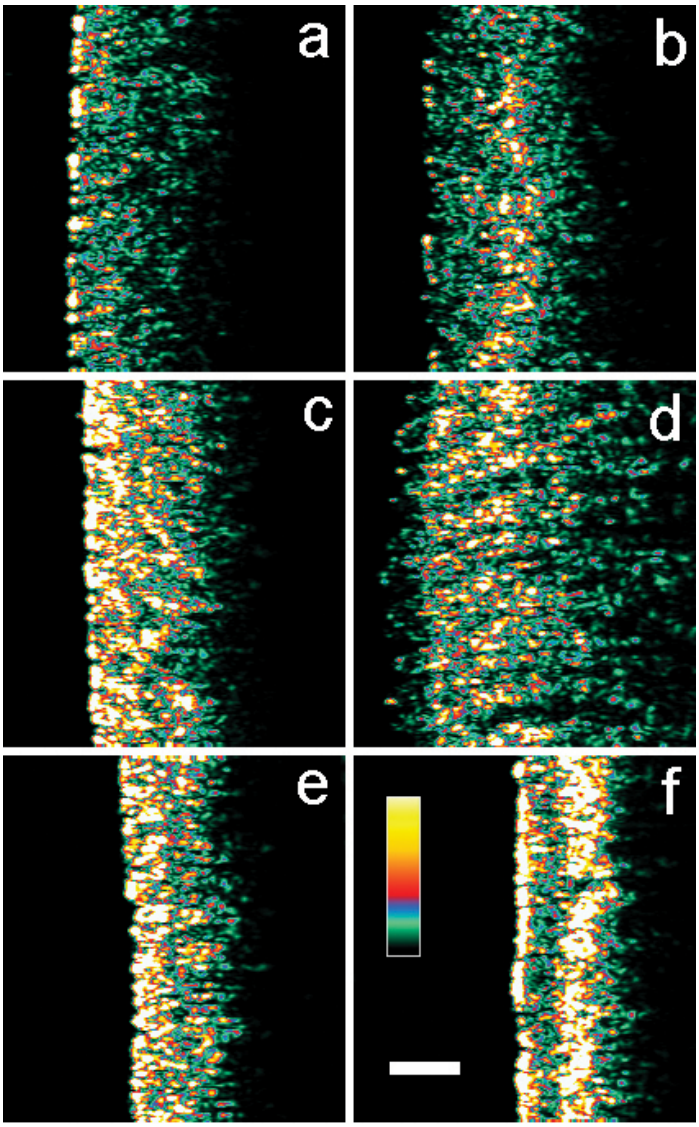

Figure. 1. Ultrasonic images (B-scans) of **A,** tight pickle, **B,** loose pickle, **C,** tight wet blue, **D,** loose wet blue, **E,** tight leather, **F,** loose leather. The grain is on the left, corium on the right. Scale bar is 0.5 mm. The color scale represents signal intensity from black (minimum intensity) to white (maximum intensity).

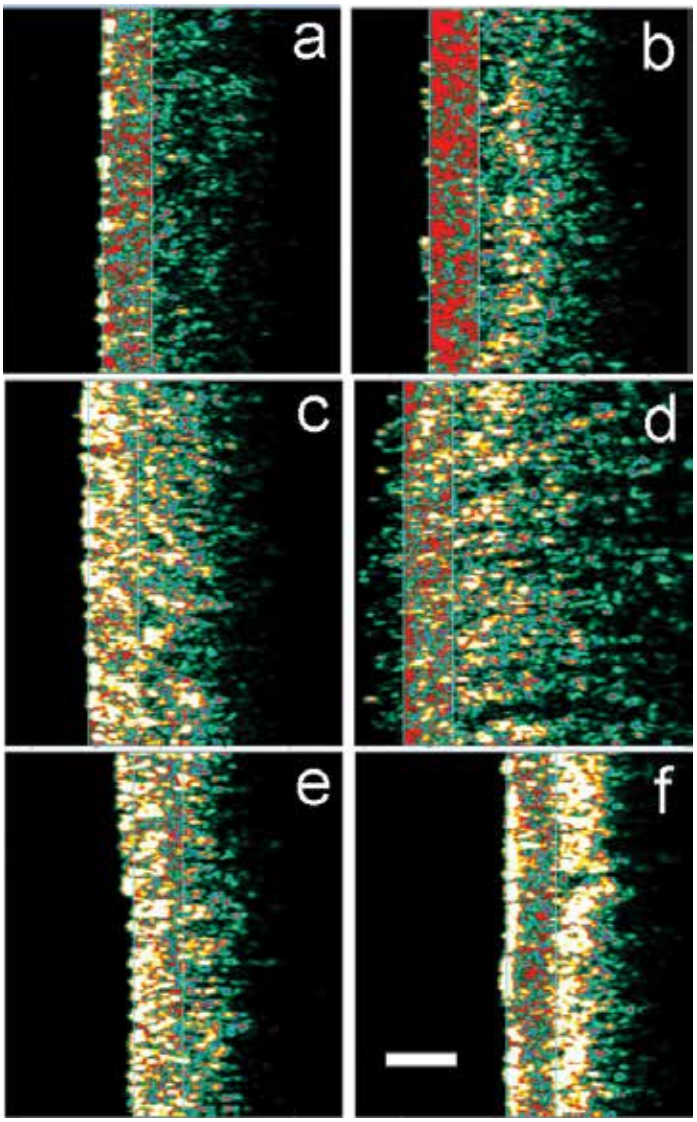

Figure. 2. Display of the portion with intensity below 7.8% of the maximum intensity of reflected ultrasonic signal (in red) for a selected region 0.378 mm wide (defined by the white line) at the grain. **A,** tight pickle, **B,** loose pickle, **C,** tight wet blue, **D,** loose wet blue, **E,** tight leather, **F,** loose leather. These are the same datasets as in Figure. 1.

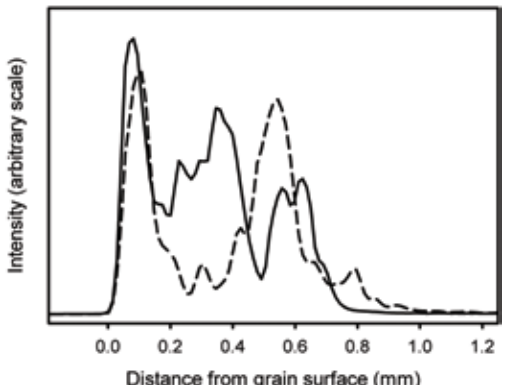

Figure 3. A-scans of tight (solid line) and loose (dashed line) leather. Each plot is an average of 20 scans, representing a total of 1.0 mm movement across the surface of the leather. These are taken from recordings similar to, but not identical to, the images from Figure. 1 and 2, E and F.

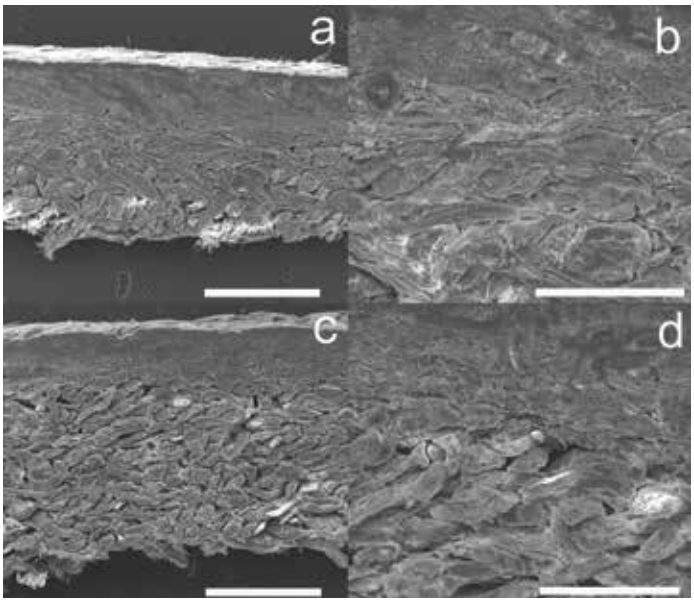

Figure 4. SEM images of cross sections of tight (a, b) and loose (c, d) leather. Grain is at the top. The enlargements (b, d) are near the grain–corium boundary. Scale bar (a, c) 1 mm, (b, d) 400 μm.

Scanning Electron Microscopy

SEM was used to confirm the loose and tight internal structure of the crust leather used in this work (Figure 4). It is clear from these images that the tight leather has a compact structure in both the grain and corium including at the grain-corium boundary (Figure 4 a, b) whereas the loose leather has fiber bundles that in the corium are not tightly packed together (Figure 4 c, d).

Discussion and Conclusions

Looseness in leather is a structural defect that results from a poor connection within or between leather layers, particularly in the lower grain region or the grain-corium boundary. This is manifested as less closely packed collagen fibrils and is due to highly aligned collagen fibrils within the loose leather. . The more loosely packed and weakly bonded grain of the loose leather, in comparison to tight leather, becomes separated during folding, resulting in the symptoms of poor break or looseness

The ultrasonic imaging on hides and wet blue show that there are differences between loose and tight precursors present in these stages. These differences can be identified relatively easily even at the pickle stage. At the pickle stage the differences in structure between the loose and tight precursor hides is rather similar to the differences in the final crust leather with a low density region in the loose precursor about 0.3 mm below the grain surface. This indicates that looseness in leather is a direct result of the structure of the hide rather than being formed by some deficiency of processing. At the

wet blue stage, which is an intermediate process stage between raw hide and leather there is still a distinctive difference between tight and loose precursors. However this difference is manifested not by a gap below the grain surface but by differences in the density distribution. The tight precursor wet blue shows a higher density at the grain surface with the density gradually decreasing with depth while the loose precursor shows a lower density at the grain surface and a fairly uniform density throughout the rest of the material. The wet blue is a stage where a lot of hides are traded and therefore identification of the propensity to looseness at this stage may also have commercial utility.

We have shown that this tendency of loose leather to have a region of lower density below the surface of the grain can be quantified by measuring the proportion of low density material from the ultrasonic images. What is perhaps surprising is that this feature is apparent even in the wet blue and pickle stages of the processed hide, with loose pickle and wet blue containing 2–6 times the amount of low density area than the tight pickle or wet blue, with these differences of the same order as in the crust leather. There is no published evidence that it is possible for a leather maker to identify looseness visually in the pickle or wet blue, but the features that lead to looseness, namely a less dense layer below the grain surface, which results from a high alignment of the collagen fibrils, [1] are present in these stage

From this investigation into looseness through the leather process it is clear that the tendency for looseness is an inherent property of the hide. At the pickle stage the density profile through a hide follows a similar pattern to that in crust leather and the differences between tight and loose hides are preserved. In wet blue the hide is swollen so that the appearance of the ultrasound images is rather different to the pickle and leather, however the quantification of low density regions reveals a similar structural difference between loose and tight as in the other stages.

The underlying cause of this low density region has been shown previously to be at least partly attributable to the higher alignment of collagen fibrils in the grain region of loose leather, leading to poorer interlayer connections of the collagen fiber structure, and therefore a tendency to come apart in this region [1] even though this may result in greater strength in the leather. [1,80].

Ultrasonic imaging is a non-destructive technique so this method could be used for hide selection, with those hides that have inherent looseness being diverted to other lower value process streams. This could save around 5% of processing costs in a typical New Zealand tannery for bovine leather and probably similar amounts in other countries.

TABLE I
Percentage of the area in the selected region that has low intensity ultrasonic reflection (0-20 / 255). Averages are from the analysis of 8–13 images.

Sample type	Tight % area (σ)	Loose % area (σ)	Ratio loose/ tight	t-test for difference t-stat, P
Pickle	20.6 (4.1)	38.0 (13)	1.8	-4.6, <0.0001
Wet blue	3.4 (1.0)	12.6 (2.2)	3.7	-12, <0.0001
Leather	5.3 (1.4)	9.8 (2.7)	1.9	-4.7, 0.0001

We have not investigated whether these tests could be conducted while the hide is still on the living animal, and this would be an interesting avenue for further investigation.

This work also does not answer the question of why some animals have hide that results in loose leather. This could be due, for example, to breed, sex, age, condition, or maybe even sun exposure. This is the subject of another study that is running in parallel to the work reported here.

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