The Quality of Leather Estimated from Airborne Ultrasonic Testing of Hides

by

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Abstract

High-quality hides are paramount for competitiveness in both domestic and export markets. Currently, hides are visually inspected and ranked for quality and sale price, which is not always reliable when hair is present on the hides. Advanced technologies are needed to nondestructively and accurately characterize the quality of hides and enable one to estimate the qualitative and mechanical properties of leather. We were the first to carry out research for airborne ultrasonic (AU) methods to nondestructively characterize the quality of hides. The developed nondestructive method is based on measuring the AU waves transmitted through the hide samples. Research results demonstrated that the average amplitude distribution received from the ultrasonic wave transmitted through the hide samples yielded the best correlation with the AU test variables: gain, speed, and frequency. Observations showed AU parameters derived from the average distribution values for amplitude (AMPa) and time of flight (TOFa) have a correlation with the quality of leather. This study demonstrated that the fullness, overall characteristic, tensile strength, stiffness, elongation, and toughness of leather could be nondestructively estimated by the ultrasonic quantities obtained from AU testing of hides.

Introduction

Cattle hides are the highest value byproduct of the beef industry. High quality, clean, and well-preserved hides are important for competitiveness in both domestic and export markets. Hides currently, however, are visually inspected and ranked for quality and sale price. Hides cannot be effectively sorted at the earliest stage of processing, because when hair is present, the visual inspection is not reliable for evaluating the quality of hides. Development of an objective and nondestructive method to accurately characterize the quality of hides is necessary and indispensable. Efforts have been made to develop new technologies to provide a quality estimation prior to the leather making process.[1-6]

It has been reported that with appropriate Airborne Ultrasonic (AU) equipment and methods, it is possible to perform sensitive inspections for defects such as voids, cracks, and disbonds in a wide variety of products such as lumber and composites.[7-12] The optimization for resolution or penetration can be achieved by selecting a correct range of AU frequencies for testing. The timber industry was among the first that employed AU commercially. Applications include: (1) inspection of internal decay, voids, and cracking (2) detection of delamination and cracking in composite or processed wood products, and (3) assessment of wood quality by measuring sound transmission velocity. In addition, composite materials, particularly within the aerospace industry, have been a primary area of application for AU methods.[8,13] We were the first to develop AU methods to detect leather defects and characterize its quality. In the previous reports, we presented that AU testing without direct contact with samples offers a great potential for the nondestructive evaluation of the quality of leather.[1-2] As a non-contact technique, AU has shown great potential to be an ideal characterization method for large leather or hides.

The AU sensor is designed for dynamic measurements and offers several key advantages in automation applications. In general, airborne ultrasonic testing involves pulsing ultrasonic signals at the material and measuring the reflected amplitude of those signals emanating from the material. The amplitude of ultrasonic signals reflected at the surface of a planar material (such as films, sheets, fabrics, and leather or hides) is a function of the material's surface morphological variations. Therefore defects, such as scars, insect bites, or knife cuts should be able to be detected because they will change the intensity of the AU signal reflecting from the surface of the material. Our studies indicated that AU testing could reveal the presence of defects or other physical discontinuity in leather, which could affect the leather quality.[2-4] Leather quality such as tensile strength and stiffness showed a good correlation with the AU data, implying those properties could be estimated from AU testing.

Our current AU testing involves pulsing ultrasonic waves and measuring the amplitude of those waves transmitted through the

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Manuscript received September 20, 2019, accepted for publication November 7, 2019.

material.[3-6] By using the through-transmission mode, we observed more useful information can be extracted from the AU scan than the reflective mode, particularly for hides which are covered by hair.

Our previous studies indicated that the key for success in AU testing for soft materials such as hides and leather is to use AU transducers with low frequencies, which leads to effective transmission of ultrasonic waves through the samples.[3-5,14] The variations in the AU quantities, such as amplitude (AMP) and time of flight (TOF) were color coded into C-scan images to reveal the location and shape of the defects or some other physical discontinuity existing in the samples. The research was recently carried out to optimize AU methods for hides and leather inspections and to study the effects of transducer frequency, thickness of leather and AU gain on the resultant AU amplitude received, which was then color-coded into a C-scan image.[5] Observations showed the 100 kHz transducer works well for crust leather. This study also showed the AMP and TOF are strongly affected by the sample thickness and instrument gain applied to the AU tests. Moreover, in a recent experiment, we found that the distribution of Time of Flight (TOF) is a better AU parameter to correlate the mechanical properties of leather.[6] Observations showed tensile strength, stiffness, fracture energy, and grain break of leather could be measured nondestructively by using the AU methods described in an earlier report.[6] These results will be instrumental in finalizing the development of AU technology for characterizing the quality of hides.

In this new study, we have identified the proper parameters for AU testing of raw hides. A statistical experimental design was used to establish the relationship between key test parameters (instrument gain, speed of scanning and ultrasound frequency) and corresponding AU quantities thereby identifying appropriate AU testing methods for hides. We also investigated the relationship between AU quantities of hides, the qualitative and mechanical properties of leather from the AU tested hides. We discovered that when two AU physical quantities were used jointly, they showed a good correlation with the quality and property of leather tanned from the respective hides.

Theory

A large portion of the energy is reflected, and the remaining energy is transmitted, as an ultrasound wave is passed from one medium to another (for example from air to hides). The key parameter that governs the reflected and transmitted relationship is referred to as acoustic impedance $(Z) = (\rho E)^{1/2}$, where ρ = density of medium, E = elastic constant of medium.[15] This square-root relationship shows the acoustic impedance of a material and is governed by the elasticity and density of a material. If one knows the acoustic impedances of the materials (media 1 and 2) on both sides of the boundary, the fraction of the incident wave intensity reflected can be calculated as the acoustic reflection coefficient (R), $R = (Z_1-Z_2)^2 / (Z_1+Z_2)^2$, where: Z_1 = acoustic impedance of medium 1 and Z_2 = acoustic impedance of medium 2.[16] The greater the R value indicates the higher the percentage of energy will be reflected at the interface or boundary between one medium and another. Therefore, it can be expected that the mismatch of acoustic impedance weakens the sound wave transmission. In addition, defects such as voids, insect damage, and brands will change the acoustic reflection coefficient and consequently affect the amplitude of the wave transmitted through the material which will show up in AU images such as either AMP or TOF C-scans.

Experimental

Materials and Methods

Fleshed cattle hides were collected from JBS (Souderton, PA). Twenty-three approximately 30 cm x 30 cm pieces were cut out evenly among the two sides of a heavy steer hide. All hide pieces were sealed in plastic zip-lock bags and stored in a freezer. Before the test, they were transferred to a refrigerator to thaw out overnight. Then the hides were clamped vertically using large binder clips across two parallel bars to minimize any slack in the sample. The hair was left untouched and intact to simulate real life conditions. Scanning was performed on the hides using an AU system. When performing AU testing, AU waves must travel from air, which is a medium with low acoustic impedance, to a medium such as hides and leather with considerably higher acoustic impedance. Therefore selection of the proper AU transducers and frequency are critical to achieving enough penetration of ultrasonic waves in order to extract important information related to the structure and properties of leather such as the number of defects, morphology, strength and softness. Figure 1 shows a photo of an AU testing system which consists of two ultrasonic transducers, a transmitter with a diameter of 38 mm pulsed with a tone burst through a power amplifier (model: NCG50-D38, The Ultran Group, State College, PA), and a receiver (model: NCG50-D38) with the same size as the transmitter connected to a preamplifier. All these components were mounted on a computer-controlled 3-D scanner (UPK-T24, MISTRAS Group,

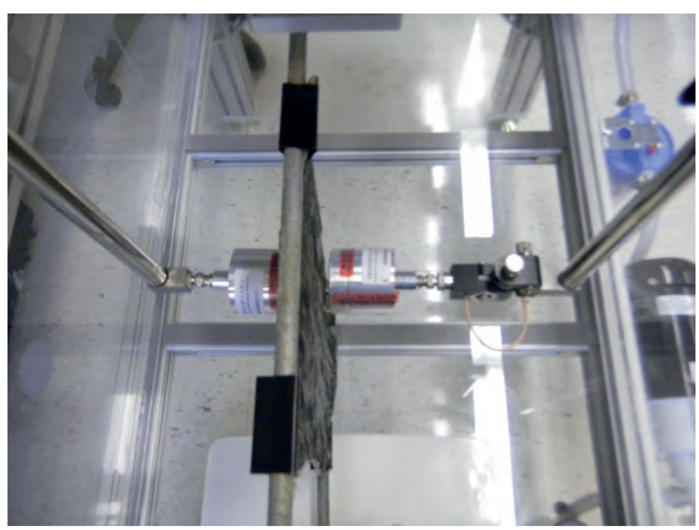

Figure 1. AU testing system

Inc., Princeton Jct., NJ). The software UTWIN (version E1.81, also from MISTRAS Group, Inc.) was used to control the movement of transducer/receiver array over the entire surface of the hide for conducting a C-scan.

There are various AU quantities that can be displayed as a function of time or position for one AU scanning result. The velocity, amplitude (AMP) and duration of ultrasonic waves (Time of Flight, TOF) measured by the receiver and changed with the material properties of the samples. The C-scan is very commonly used in AU testing in which the transmitted AU pulses are captured and the amplitudes of the transmitted pulses are mapped using pseudo color from the maximum amplitude in gate 1 or gate 2 set on the A-scan.[6] The A-scan presents the waveform of the received signal and gate 1 is set to the first waveform and gate 2 is set to the second waveform. C-scan is an automated two-dimensional scanning system in AU testing which provides a planar-type view of the location and size of the test specimen. The resolution of the C-scans generated was set to 0.5 mm in both the scanning and indexing directions. The amplitude and TOF of the transmitted signals at regular intervals were recorded, color-coded and mapped into an image file as shown in Figure 2. The proper conversion of C-scan images to numeric data is a critical step to realize the quantitative representation of hides. Based on our previous research,[4,6] gate 1 representing the direct transmission through the material and gate 2 showing the thickness reflection of waveform bouncing through the sample were set to collect data at designed intervals. All the data recorded in the UTWIN software under designed testing conditions were screened to eliminate the data set containing null values. The effective data were further analyzed through the statistical software Minitab-19 (Minitab LLC, State College, PA).

Experimental Design

The Box and Hunter's central composite rotational design was used to establish the relationship between variables and responses.[17-18] Three important factors of AU testing including gain (X_1), speed (X_2) and frequency (X_3) were studied for their effects on the resultant AU physical quantities (such as AMP and TOF). In order to simplify the regression equations, the variables X_i were coded from original values (X_i'). The coded values of X_1, X_2 and X_3 were obtained by the following equations: $X_1 = (X_1'-10)/10$, $X_2 = (X_2'-30)/10$, $X_3 = (X_3'-50)/20$. All the experimental conditions and coded values were listed in Table 1, including 9 center points. Based on different responses under testing conditions, regression models were calculated containing variables presented as their linear, quadratic and bifactorial cross products. Meanwhile, statistical analyses were performed to identify the strongest relationship between variables and responses.

After AU measurement, the hide pieces were taken off the clamps and stamped using a custom-made number die (Durable Mecco, Franklin Park, IL) with the size of the numbers being 1.27 cm x 1.27 cm. Five numbers were hand-stamped into the hides with 4 of the numbers in a square pattern approximately 6 cm apart from the center, and 1 number stamped directly in the middle. The stamped hide pieces (named marked hides hereafter) were placed back on the clamps and retested using the same conditions. After AU testing, the hide pieces were placed back into the freezer until all of the hide pieces were tested according to the experimental plan as shown in Table I. The hides pieces were then tanned and retanned using the methods reported previously.[19-20]

Table I

The design of experimental conditions

Run	Coded values			Actual values		
	X_1 Gain	X_2 Speed	X_3 Frequency	X_1' Gain (dB)	X_2' Speed (mm/min)	X_3' Frequency (kHz)
1	-1	-1	-1	0	20	30
2	-1	-1	1	0	20	70
3	-1	1	-1	0	40	30
4	-1	1	1	0	40	70
5	1	-1	-1	20	20	30
6	1	-1	1	20	20	70
7	1	1	-1	20	40	30
8	1	1	1	20	40	70
9	-1.682	0	0	-6.82	30	50
10	1.682	0	0	26.82	30	50
11	0	-1.682	0	10	13.18	50
12	0	1.682	0	10	46.82	50
13	0	0	-1.682	10	30	16.36
14	0	0	1.682	10	30	83.64
15	0	0	0	10	30	50
16	0	0	0	10	30	50
17	0	0	0	10	30	50
18	0	0	0	10	30	50
19	0	0	0	10	30	50
20	0	0	0	10	30	50
21	0	0	0	10	30	50
22	0	0	0	10	30	50
23	0	0	0	10	30	50

Quality Assessments

The samples were rewet and staked twice before being evaluated by an experienced tanning professional for the qualitative leather properties: handle, fullness, grain (break), color and overall characteristic. Among them, handle is the sensation or judgment by feeling for certain physical properties of leather through touch with the fingers and hands for the flexibility and smoothness of the leather. Fullness is the feeling of compressed leather in the hand. A full leather fills the palm with the force to bounce back, while a flat leather has more of a cardboard effect. Grain break is the way to characterize the wrinkles formed on the surface of leather when bent grain inward. In general, the finer the wrinkles the better the quality of the tested leather. A scoring system from 1 to 5 was used for each property, where 1 is the worst while 5 is the best. Based on the scores of these four properties, an overall characteristic was also determined and reported on a scale of 1 to 5. In addition, the relationship between AU quantities on hides and subjective scores from the leathers was explored.

Mechanical Property Tests

Leather samples were also tested for mechanical properties. An Insight-5 test frame and Testworks-4 data acquisition software (MTS Systems Corp., Minneapolis, MN) were used throughout this work. Tensile strength, Young's modulus, elongation and fracture energy (toughness) were tested as described in a previous paper.[6] The samples were cut parallel to the backbone with 1x 10 cm in shape. Tested at 25.4 cm/min with a 5 cm grip or gage length. All samples were preconditioned in an environmental chamber at 23 ± 2°C and 50 ± 4 % RH for at least 24 h (ASTM D1610).

Results and discussion

C-scan images of unmarked hides (Figure 2a, Figure 2c) and marked hides (Figure 2b, Figure 2d) are demonstrated in Figure 2. AMP average distribution values (AMPa) were recorded in the UTWIN software, which represent the percentage of waves that reached the maximum amplitude from gate 1. The TOF average distribution values (TOFa) were recorded also from gate1. The average distribution values (AMPa and TOFa) and other AU data were calculated by the UTWIN software.

Through analyzing the response surface design by Minitab, a full quadratic regression equation was obtained with corresponding analytical values. First, R-squared values were compared among all models. R-squared is a statistical measure of how close the data are to the fitted regression line. After the examination of various AU quantities, the average amplitude (AMPa) showed to be the most sensitive response to the change in the three important test variables (gain X_1, speed X_2, and frequency X_3). The R-squared for the derived quadratic model is 0.97 (Table II) which is the highest among all other equations.

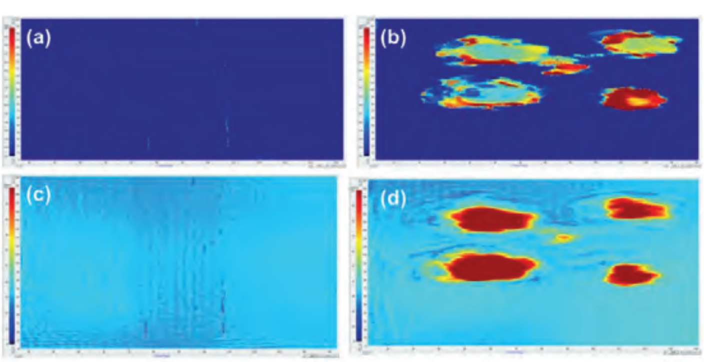

Figure 2. C-scan images of AMP for (a) non-marked hide, (b) marked and C-scan images of TOF for (c) non-marked hide, (d) marked hide

Table II

R-squared values of established models

Model	R-sq	R-sq(adj)	R-sq(pred)
Full quadratic model	97.28%	95.41%	83.21%
Adjusted model	97.16%	96.53%	94.35%

*R-Sq (adj): Adjusted R-squared; R-sq(pred): Predicted R-squared

The corresponding regression model is expressed as follows:

$$Y=27.77+26.32X_1-0.04X_2-19.47X_3+8.46X_1^2-0.97X_2^2-0.14X_3^2-0.75X_1X_2-16.4X_1X_3-0.63X_2X_3 \quad (1)$$

In general, the higher the R-squared, the better the model fits the data. However, R-squared may not reflect the whole story, and there are other analytical values that need to be considered for a good fit. As seen in the analysis of variable table (Table III), the p-value of "lack of fit" is 0.017, which rejects the null hypothesis that the model fits the data well at a significance level (α) of 0.05. In other words, one may conclude that the model does not adequately reflect the whole picture of data. Based on p-values, some terms in the equation are not meaningful since their high p-values (>0.05) indicate the acceptance of the null hypothesis that the coefficient has no effect on the model equation. For example the p-value of speed is 0.981. It is strong evidence that as a variable, speed may not be a significant

Table III

Analysis of variance of the full quadratic model

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Model	9	17955.1	1995.01	51.76	0.000
Linear	3	14640.0	4880.01	126.60	0.000
Gain	1	9460.5	9460.50	245.43	0.000
Speed	1	0.0	0.02	0.00	0.981
Frequency	1	5179.5	5179.52	134.37	0.000
Square	3	1156.2	385.41	10.00	0.001
Gain*Gain	1	1139.0	1138.98	29.55	0.000
Speed*Speed	1	14.8	14.81	0.38	0.546
Frequency*Frequency	1	0.3	0.29	0.01	0.932
2-Way Interaction	3	2158.8	719.60	18.67	0.000
Gain*Speed	1	4.5	4.45	0.12	0.739
Gain*Frequency	1	2151.2	2151.22	55.81	0.000
Speed*Frequency	1	3.1	3.14	0.08	0.780
Error	13	501.1	38.55		
Lack-of-Fit	5	389.2	77.85	5.57	0.017
Pure Error	8	111.9	13.98		
Total	22	18456.2			

*DF: Degree of freedom; Adj SS: Adjusted sums of squares; Adj MS: Adjusted mean squares

Table IV

Analysis of variance of the adjusted model

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Model	4	17932.4	4483.10	154.06	0.000
Linear	2	14640.0	7320.01	251.55	0.000
Gain	1	9460.5	9460.50	325.11	0.000
Frequency	1	5179.5	5179.52	177.99	0.000
Square	1	1141.2	1141.16	39.22	0.000
Gain*Gain	1	1141.2	1141.16	39.22	0.000
2-Way Interaction	1	2151.2	2151.22	73.93	0.000
Gain*Frequency	1	2151.2	2151.22	73.93	0.000
Error	18	523.8	29.10		
Lack-of-Fit	10	411.9	41.19	2.95	0.070
Pure Error	8	111.9	13.98		
Total	22	18456.2			

*DF: Degree of freedom; Adj SS: Adjusted sums of squares;
Adj MS: Adjusted mean squares

factor in the equation. Therefore, a more concise equation (equation 2) was obtained after removing insignificant terms. Its corresponding variance analysis was shown in Table IV. From the table, we could find that the p-value of Lack of Fit is 0.07 which is larger than the significance level (α) of 0.05, so it is possible to conclude that this adjusted model fits the data well. Additionally, both adjusted R-squared and predicted R-squared (Table II) improved compared with the full quadratic model.

$$Y=27.77 + 26.32 X_1 -19.47 X_3 + 8.46 X_1^2 -16.4 X_1X_2 \quad (2)$$

According to Equation 2, a 3-D plot of the response surface of amplitude average distribution (AMPa) value as a function of speed and gain was shown in Figure 3 (a). It is demonstrated that AMPa has a significant increase with the increase of gain. Moreover, Figure 3 (b) shows a 3-D plot of the response surface of AMPa as a function of AU frequency and speed. We notice there is a negative correlation between AMPa and frequency. With the reduction of frequency during the test the average amplitude dramatically increases. From these two plots the effects of speed on amplitude are very limited,

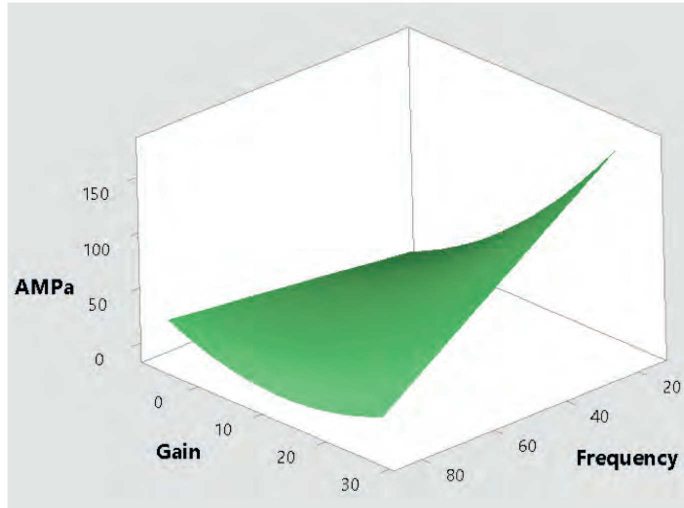

Figure 4. 3-D regression plot of AMPa as a function of gain and frequency

which confirms the statistical analysis that speed is not a meaningful term in the equation.

Moreover, the relationship among AMPa, frequency and gain is shown in Figure 4. It is evident that there is no linear increase or decrease trend between each factor and response. Frequency and gain may interact with each other to affect the response.

After determining the condition to obtain the most sensitive response under testing variables we also explored the relationship between AU physical quantities measured from hides and subjective qualitative evaluation scores of the corresponding leather after tanning. In this study, we have used the Pearson correlation to examine the strength and direction of the linear relationship between each pair of variables. The highest Pearson correlation coefficient for AMPa was found with fullness, which is 0.66. It represents a positive relationship between them. In other words, when the AMPa increases the fullness score also increases. The fullness of leather is closely related to its inner structure. The fiber bundles remain intact and well-distributed throughout the structure in the leather and retain their elasticity which may also contribute to the easier transmission of ultrasonic waves. Therefore, waves with higher amplitude were collected by the receiver. The p-value is 0.053,

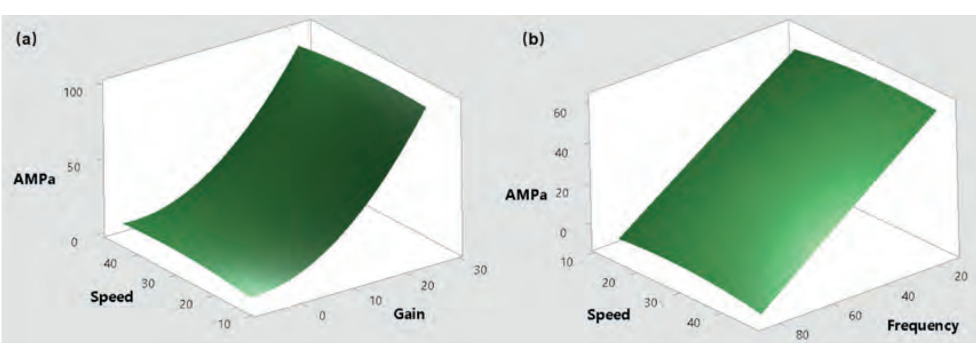

Figure 3. 3-D regression plot of AMPa as a function of (a) gain and speed; (b) frequency and speed

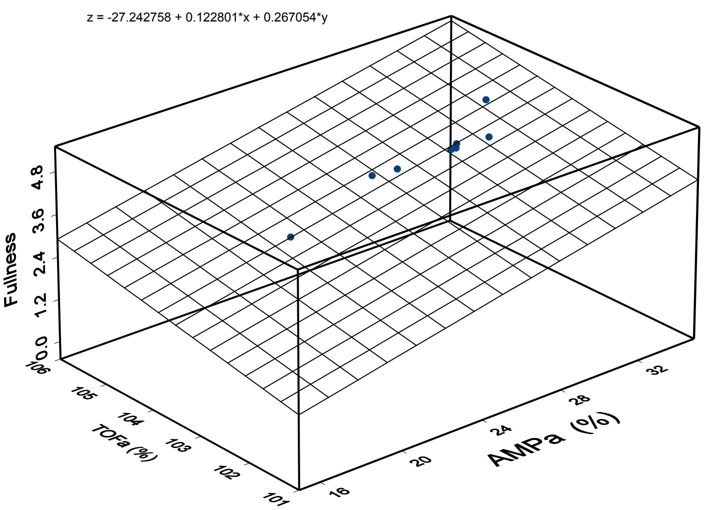

Figure 5. Fullness as a function of TOFa and AMPa

Table V

Pearson correlation matrix among subjective scores and AMPa

	AMPa	Break	Handle	Fullness	Color
Break	*0.099				
	**0.800				
Handle	0.559	0.500			
	0.118	0.170			
Fullness	0.660	0.300	0.750		
	0.053	0.433	0.020		
Color	0.388	0.550	0.500	0.750	
	0.303	0.125	0.170	0.020	
Overall	0.181	0.575	0.812	0.750	0.688
	0.642	0.105	0.008	0.020	0.041

*Pearson correlation, ** P-Value

which is close to the significance level of 0.05. This indicates that the correlation between AMPa and fullness is more significant than other pairs. Moreover, as shown in Figure 5, we have demonstrated there is a good correlation between the degree of fullness and the combination of AMPa and TOFa. On the other hand, in Figure 6, the overall characteristic of leather showed a similar trend as fullness, which implied that fullness is a key factor affecting the value of overall characteristic of leather.

Along with the AMPa, the relationship between different subjective evaluation categories was also assessed in Table V. The correlation coefficient between handle and overall is 0.812, and the p-value is 0.008. It is very obvious that a strong positive correlation exists between handle and overall. The overall characteristic of the leather was determined by combining all of the other subjective qualitative values such as fullness, grain break, color distribution and handle. It is possible to use only the handle score to evaluate the overall quality of leather since it can almost reflect the whole picture.

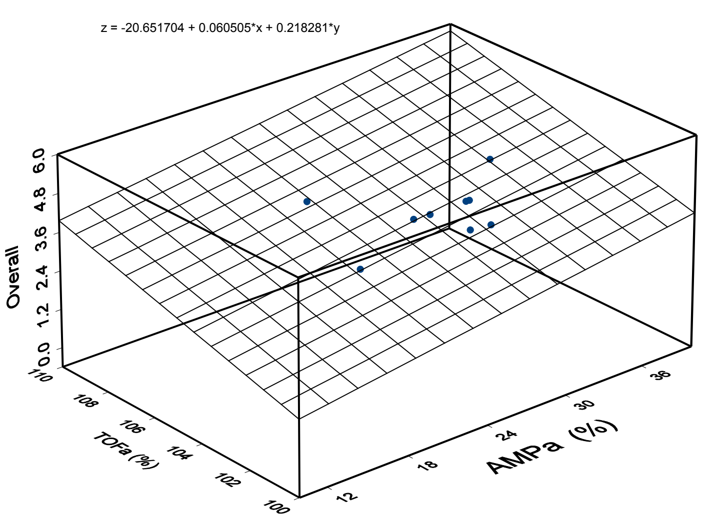

Figure 6. Overall characteristic as a function of TOFa and AMPa

This research also investigated the relationship between AU quantities measured from hides and the mechanical properties of corresponding leather tanned from the same hides. Tensile strength determines the maximum tensile stress the leather can sustain without fracture. Adequate tensile strength is very important in manufacturing leather goods where the leather is often subjected to a tensile force during mechanical stretching or elongation. Moreover, in a variety of end uses, leather goods must be capable of resisting considerable stress without fracture. Figure 7 displays a 3-D regression plot of tensile strength as a function of AMPa and TOFa simultaneously, where the dots are data points. Tensile strength shows a strong positive correlation with TOFa, while it has only a small negative correlation with AMPa. The reason for this difference is related to the leather structural difference. TOFa is a distribution of time of flight which is increased with the transit time of the sound waves through the hide sample. It reflects how "smooth" the sound can travel through the hide sample, which is

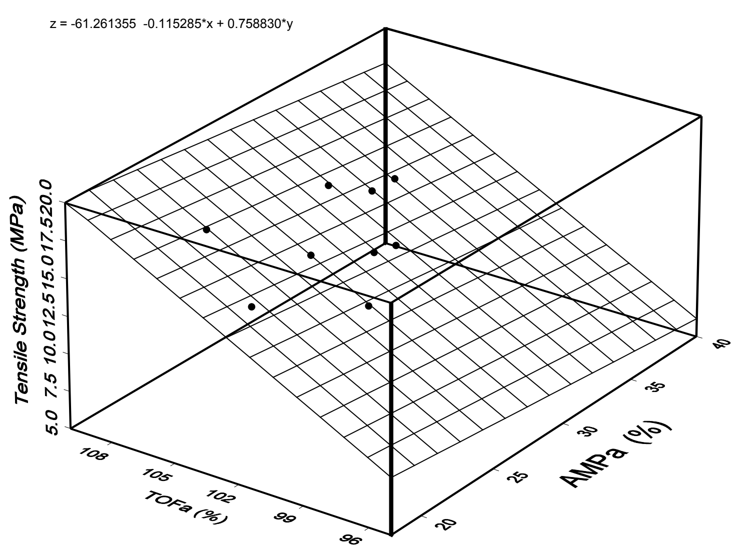

Figure 7. Tensile strength as a function of AMPa and TOFa.

governed by the internal structure of the material. The greater amount of TOF reflects the denser fibrous structure of hides, which results in a longer transmission of ultrasonic waves through the hides therefore it could indicate higher tensile strength values for leather. However, this phenomena may not be applicable to the AMPa, probably because the AMP is more related to the elasticity of the hide fibers. The higher AMP indicates higher rigidity, which has a negative impact on the tensile strength of leather. Importantly, the results demonstrated in Figure 4 imply that the tensile strength of leather could be estimated by the AU nondestructive test method described here by measuring the TOF of the original hides.

Stiffness is one of the most important physical quantities characterizing the mechanical properties of leather. It expresses the resistance of leather subjected to a small tensile deformation. It is commonly known that the higher Young's modulus, the stiffer the material. In the literature, its reciprocal has been named compliance.[21] In various reports, Young's modulus has been linked to the fine structure of leather, such as the degree of fiber orientation[22-23] and fiber adhesion.[24] It also has been associated with leather softness, temper and handle in various reports.[25-27] Figure 8 is a regression plot of Young's modulus as a function of AMPa and TOFa. It shows the Young's modulus negatively correlates with AMPa but has little effect on TOFa.

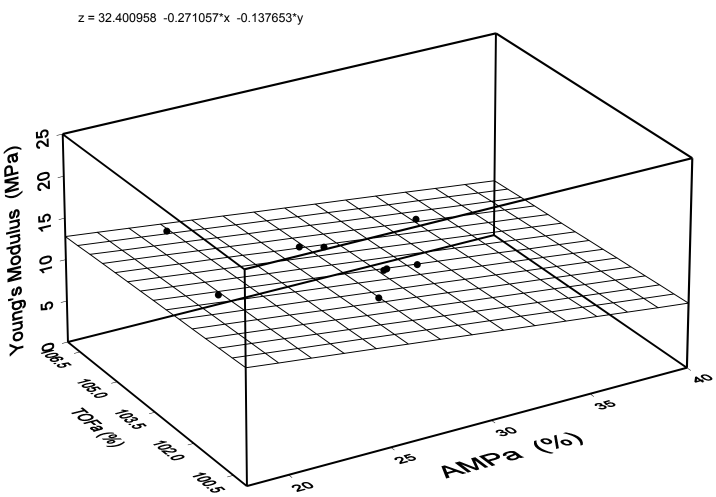

Figure 8. Young's modulus as a function of AMPa and TOFa

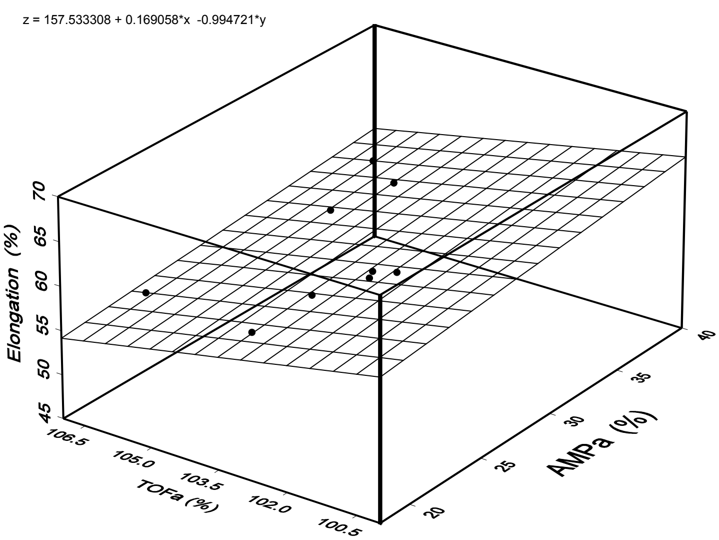

Figure 9. Elongation as a function of AMPa and TOFa

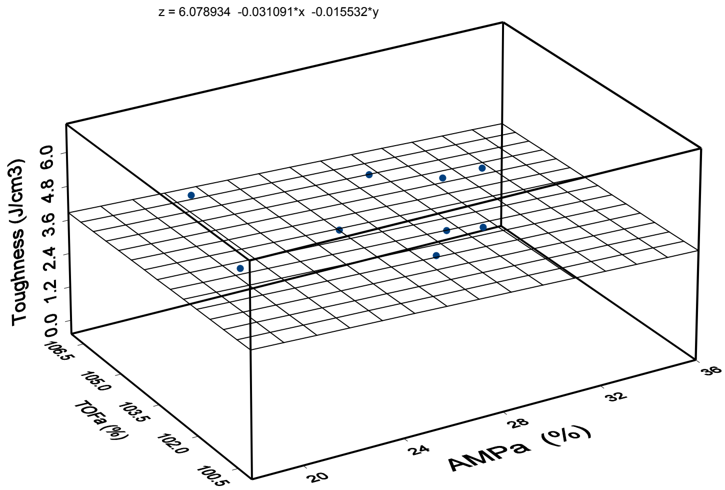

Figure 10. Toughness as a function of AMPa and TOFa.

A 3-D regression plot of elongation as a function of AMPa and TOFa is shown in Figure 9, where the elongation shows little change with AMPa and however decreases significantly with TOFa. Again, this behavior is probably due to the denser fiber structure which is more resistant to an extension when it is subjected to a tensile force.

Toughness (also known as fracture energy in materials science) was determined by measuring the energy required to fracture the leather sample which is the area under the stress-strain curve.[28] As we have reported previously, contrary to tensile strength, the measurement angle shows little effect on the toughness.[28] Good toughness is an important quality requirement for leather or other fibrous materials, which reflects a superior balance of strength and flexibility. Figure 10 displays a 3-D regression plot of toughness as a function of AMPa and TOFa, in which both AU quantities show little effects on toughness. This could be due to the structural factors affecting toughness balanced out in the AMPa and TOFa measurement. Therefore these two AU parameters do not affect fracture energy significantly.

Conclusions

The objective of this research is to develop a nondestructive method using AU to characterize the quality of hides and further to estimate the qualitative and physical properties of leather. This method is based on measuring the AU quantities as ultrasound waves are transmitted through the hide samples. In this investigation, we discovered the amplitude average distribution value (AMPa) yielded the best correlation with the AU testing variables. Observation showed that the fullness, overall characteristic, tensile strength, stiffness, elongation and toughness of leather could be estimated by the AMPa and TOFa quantities tested from corresponding hides. The results derived from this research are instrumental in establishing a quality control/quality assurance method for manufacturing.

Acknowledgement

The authors wish to thank Joe Lee for his evaluation of leather quality.

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