MATHEMATICAL MODEL OF RAW HIDE CURING WITH BRINE

by

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ABSTRACT

The most common method of preserving raw hides is brine curing with sodium chloride. However, this process has three important disadvantages: first, the length of time that it takes, which is a minimum of 18 hours; second, the insufficient degree of curing reached in some hides due to an overload and possibly the low efficiency of the brine raceway; and finally, the environmental impact associated with the discharge of large quantities of electrolytes in the soaking step. Our long term goal is to address all three issues. Initially, we have carried out a study of the salt uptake and its diffusion mechanism in order to attempt a reduction in the curing time. A continuous reaction mathematical model of a closed one dimensional system that describes the diffusion of sodium chloride in the hide during the curing process was chosen in the search for the optimum brine curing conditions such as the optimum brine concentration and percent float. The effect of these two parameters on the values of transport coefficient λ was reported. Brine diffusion into the hide was tracked by measurement of the chloride concentration of the residual brine solution. In addition, a piece of hide was cured with a fluorescently labeled brine solution and analyzed by means of epifluorescent microscopy for direct visualization of the sodium location within the hide.

RESUMEN

El método más común para preservar pieles crudas es el curado con salmuera. Sin embargo, este proceso presenta tres desventajas: en primer lugar, el tiempo que requiere, un mínimo de 18 horas; en segundo lugar, el insuficiente nivel de curado alcanzado en algunas pieles debido a la sobrecarga y baja eficiencia del tanque de curado; y finalmente, el impacto medioambiental asociado con la descarga de grandes cantidades de electrolitos en la operación de remojo. Nuestro objetivo a largo plazo es resolver los mencionados problemas. Inicialmente, estudiamos la absorción de sal y su mecanismo de difusión a fin de reducir el tiempo de curado. Un modelo matemático de reacción continua de un sistema unidimensional cerrado fue escogido para describir la difusión de cloruro de sodio en la piel durante el proceso de curado a fin de buscar las óptimas condiciones de curado, tales como la concentración óptima de salmuera y el porcentaje óptimo de baño. El efecto de estos dos parámetros en los valores del coeficiente de transporte λ son presentados. La difusión de sal en la piel fue monitorizada mediante la determinación de concentración de cloruros en la solución residual de salmuera. Además, una muestra de piel fue curada con una solución de salmuera etiquetada con una sustancia fluorescente y analizada mediante microscopía epifluorescente para la visualización directa del sodio en la piel.

INTRODUCTION

Raw hides and skins are ~ 60-70% water and ~ 25-30% protein. In this form the hide is susceptible to bacterial activity within hours after being removed from the carcass. The autolytic degradation of skins/hides is assumed to be due to a combined action of tissue enzymes and bacteria, the latter requiring moisture to be viable.[1] Curing is the process that provides an environment in which bacteria cannot survive. Several curing agents have been reported in the literature, e.g., potassium chloride,[2] silica gel,[3,4] boric acid,[5] and herbal-based products.[6] Common salt, in spite of its inherent impact on the environment and the large amount required, is the most popular and inexpensive material used to preserve hides and skins. A suitable improved method would yield savings in salt, shipping and effluent treatment costs as well as a diminished environmental impact. Mathematical modeling has been

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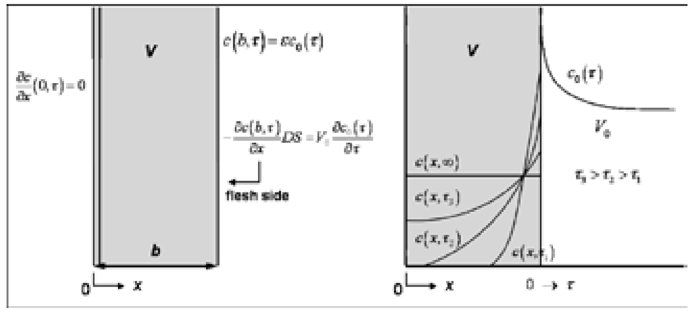

Figure 1: Mathematical model of the curing process of a raw hide.

reported to be a powerful tool in the optimization of processes such as soaking of salted cattle hides.[7,8] Curing has been modeled in substrates such as cheese[9,10] and meat[11], but has not been studied for the particular case of raw hides.

The aim of this work is to develop and verify a mathematical model that describes the diffusion of sodium chloride in the hide during the curing process. Upon its verification, the model is applied in the search of the optimum values of process variables such as brine concentration, float percentage and curing time.

THEORY

We propose a continuous reaction model to describe the diffusion of sodium chloride from the bath containing brine solution to the surface of the solid phase (hide). The model assumes that salt will further diffuse into the hide's inner volume where it will form a non-stationary concentration field (Figure 1). It also assumes that diffusion takes place only into the flesh side and that hide parameters such as thickness, surface and properties of both hair and flesh sides will remain constant throughout the whole process.

Curing can be considered as a counter diffusion in which sodium chloride soaks into the skin as water simultaneously washes out. Equation (1) describes a non-stationary one dimensional concentration field inside the inner volume of the solid phase, defined by Fick's second law. Boundary condition (1a) assumes that sodium chloride diffuses into the hide from the flesh side only. Terms are defined at the end of the paper.

$$\frac{\partial c}{\partial \tau}(x,\tau)=D\cdot\frac{\partial^2 c}{\partial x^2}(x,\tau) \qquad 0<x<b \qquad \tau>0 \tag{1}$$

$$\frac{\partial c}{\partial x}(0,\tau)=0 \tag{1a}$$

Equation (2) corresponds to a mass balance of a closed system in which salt flow at the hide surface is equal to accumulation speed of sodium chloride in the bath. Equations (2a) and (2b) are the initial boundary conditions (τ = 0). They assume a null initial content of sodium chloride in the fresh hide and a constant initial value of brine concentration in the bath respectively.

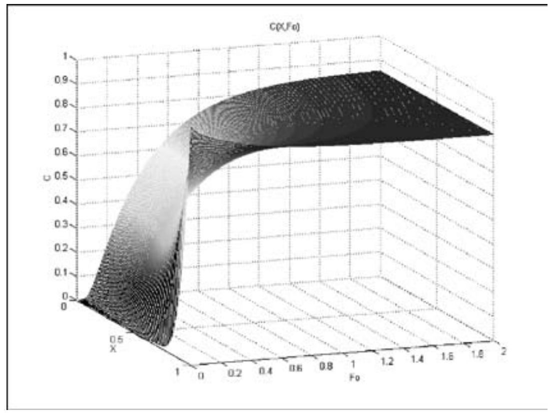

Figure 2: Dimensionless sodium chloride concentration field within the hide during the curing process.

$$-S\cdot D\cdot\frac{\partial c}{\partial x}(b,\tau)=V_0\cdot\frac{\partial c_0}{\partial \tau}(\tau) \tag{2}$$

$$c(x,0)=0 \tag{2a}$$

$$c_0(0)=c_{0p} \tag{2b}$$

Equation (3) is valid under an ideal mass transfer from the bulk solution to the surface of the solid phase.

$$c(b,\tau)=\varepsilon\cdot c_0(\tau) \tag{3}$$

The introduction of dimensionless parameters (equations 4a to 4e) has been demonstrated to be a useful tool in the model development.

$$C=\frac{c}{\varepsilon\cdot c_{0p}} \tag{4a}$$

$$C_0=\frac{c_0}{c_{0p}} \tag{4b}$$

$$X=\frac{x}{b} \tag{4c}$$

$$F_o=\frac{D\cdot\tau}{b^2} \tag{4d}$$

$$Na=\frac{V_0}{V} \tag{4e}$$

The dimensionless time, also called Fourier's number [F_0], assesses the proximity of the process to the equilibrium, i.e. equilibrium is reached when $F_0 \to \infty$. The dimensionless soaking number [Na] expresses the ratio between the volumes of liquid and solid phases. The replacement of the dimensionless parameters into the previous model leads to a new dimensionless model (Eq. (5a) to (5f)).

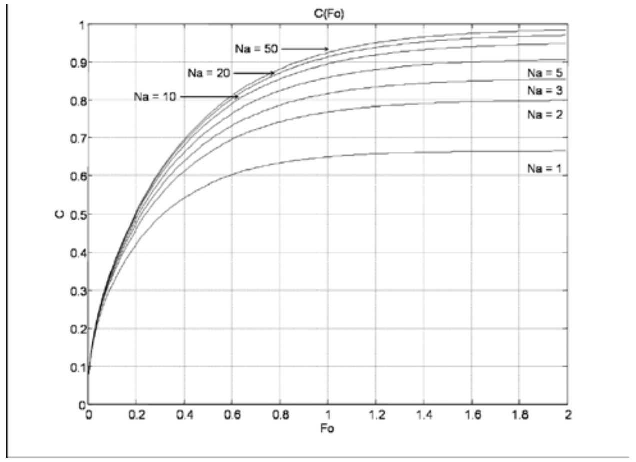

Figure 3: Dimensional sodium chloride concentration field within the hide during the curing process.for various soaking numbers.

$$\frac{\partial C}{\partial F_0}(X,F_0)=\frac{\partial^2 C}{\partial X^2}(X,F_0) \qquad 0<X<1 \qquad F_0>0 \tag{5a}$$

$$C(1,F_0)=C_0(F_0) \tag{5b}$$

$$\frac{\partial C}{\partial X}(1,F_0)=-\frac{Na}{\varepsilon}\cdot\frac{\partial C_0}{\partial F_0}(F_0) \tag{5c}$$

$$\frac{\partial C}{\partial X}(0,F_0)=0 \tag{5d}$$

$$C(X,0)=0 \tag{5e}$$

$$C_0(0)=1 \tag{5f}$$

the analytical solution of which can be obtained by means of Laplace's transformation:

$$C(X,F_o)=\frac{Na}{\varepsilon+Na}+2Na\cdot\sum_{n=1}^{\infty}\frac{\cos(X\cdot g_n)\cdot e^{-F_0\cdot g_n^2}}{\varepsilon\cdot\cos(g_n)-\frac{\varepsilon\cdot\sin(g_n)}{g_n}-g_n\cdot Na\cdot\sin(g_n)} \tag{6}$$

Where g_n are the roots of the transcendent equation (7).

$$tg(g_n)=-\frac{Na\cdot g_n}{\varepsilon} \qquad (g_n>0) \tag{7}$$

The three dimensional concentration field graphic corresponding to Eq. (6) is shown in Figure 2.

Replacing Eq. (7) into Eq. (6) and rearranging terms (for X = 1), we obtain an equation that illustrates the variation of brine concentration with time.

$$C_0(F_o)=\frac{Na}{\varepsilon+Na}+2Na\cdot\sum_{n=1}^{\infty}\frac{e^{-F_0\cdot g_n^2}}{\varepsilon+Na+\frac{Na^2\cdot g_n^2}{\varepsilon}} \tag{8}$$

In addition, integration of Eq. (6) leads to Eq. (9) which calculates the optimal time in order to reach a certain content of sodium chloride in the skin, $\overline{C}$ (integral average concentration).

$$\overline{C}(F_o)=\frac{Na}{\varepsilon+Na}-2Na^2\cdot\sum_{n=1}^{\infty}\frac{e^{-F_0\cdot g_n^2}}{\varepsilon^2+Na\cdot\varepsilon+Na^2\cdot g_n^2} \tag{9}$$

Figure 3 shows the curves of the integral average concentration for various values of soaking number.

Determination of Diffusion Coefficients

The value of effective diffusion coefficient of sodium chloride in the hide can be evaluated from experimental data. Crank[12] suggested an equation for the diffusion coefficients study at short times:

$$C_0(\tau)=\frac{c_{0P}-c_0(\tau)}{c_{0P}-c_0(\infty)}=\frac{2}{\sqrt{\pi}}\cdot\frac{1+Na}{Na}\cdot\sqrt{\lambda\cdot\tau} \tag{10}$$

From the mass balance

$$c_{0p}\cdot V_0=c_{0\infty}\cdot V_0+\varepsilon\cdot c_{0\infty}\cdot V \tag{11}$$

We get

$$c_{0\infty}=\frac{c_{0P}\cdot Na}{Na+\varepsilon} \tag{12}$$

Transport parameter λ is defined as a ratio of the effective diffusion coefficient D' to pore half length (a) square.

$$\lambda=\frac{D}{b^2}=\frac{D'}{a^2} \tag{13}$$

when the factor for the tortuosity of the pores:

$$\xi=\frac{a}{b} \tag{14}$$

λ is an important value from an engineering point of view since it includes two phenomena not considered in the presented model, which are the transport of water from the hide to the bath and the interaction between sodium chloride and water counter flows during the curing.

EXPERIMENTAL

Materials

Fresh cow hides were purchased from a local abattoir. They were soaked for 2 h (with surfactant) and then fleshed. Approximately 6 × 10 in (15 × 25 cm) pieces were cut and stored at -20°C. They were thawed at 4°C just before use. Food grade sodium chloride of purity minimum 99.82% was obtained from US Salt Corporation (Watkins Glen, NY). All other chemicals were reagent grade and used as received.

Methods

Thawed hide was cut into square pieces of approximately 4 × 4 in (10 × 10 cm) with an average weight of ~ 100 g. They were transferred to a Dose drum (Model PFI 300-34, Dose

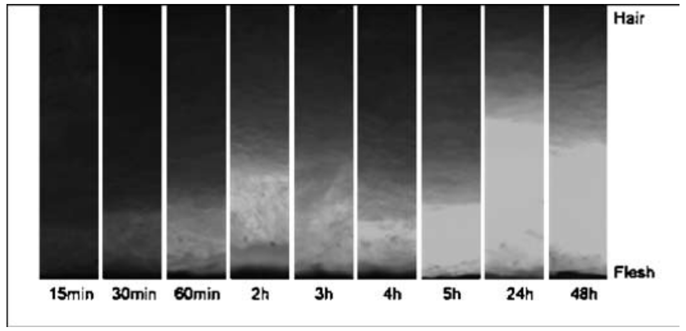

Figure 4: Epifluorescent microscopic images of a cross section of a hide at different stages of curing. The hide was cured with 30% (w/v) labeled sodium chloride solution.

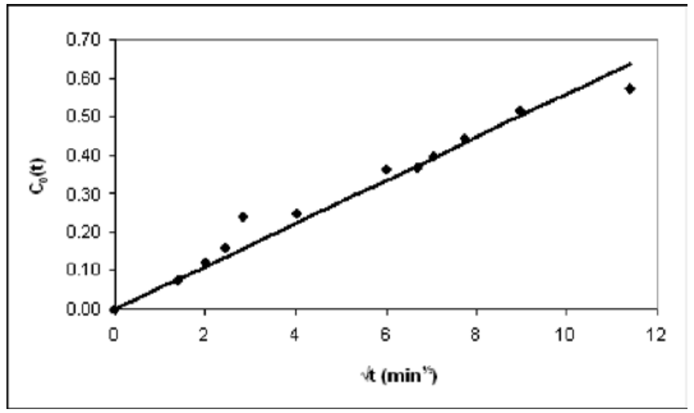

Figure 5: Determination of transport parameter λ from experimental data. The graph corresponds to c0p = 30% (w/v) and Na = 3.

Maschinenbau GmbH, Lichtenau, Germany), and tumbled at 6 rpm with brine solution for varying time intervals after which they were pulled out of the drum, hand-squeezed to wipe excess water, sealed in plastic bags and placed in the refrigerator. A fraction of the residual brine solution was also collected at different time intervals. Two sets of experiments were carried out: a constant 300% (v/v) float (volume of brine solution/ volume of hide) at different initial salt concentration levels (20%, 25%, 30% and 35% (w/v), which correspond to 64, 80, 96 and 100°SAL, respectively) and a constant 30% (w/v) initial salt concentration (weight of NaCl/volume of solution) at different float percentages (300%, 500%, 750% and 1000% v/v). Density of hide was assumed to be ~ 1g/cm^3.

Analyses

Chloride concentration determination

Chloride concentration was determined by classical Mohr titration.[13] Residual brine samples were diluted (1:100 v/v) in nano pure water prior to titration. All samples were run in triplicate.

Fluorescence Imaging

CoroNa™ Green Sodium Indicator fluorescent dye (Invitrogen, Carlsbad, CA) was used as a probe of sodium ions diffusing into raw hide from brine solution. A piece of raw hide of approximately 1 × 1 in (2.5 × 2.5 cm) was immersed in a beaker containing 500% v/v float of a 30% w/v NaCl solution and 5μM of the fluorescent dye and gently agitated. A 2-3 mm wide slice of the sample was excised manually with a stainless steel razor blade (cutting from flesh surface toward the grain) at regular intervals of incubation time, then mounted onto Petri dishes for imaging, using a Leica MZ FLIII stereomicroscope (Leica Microsystems, Bannockburn, IL, USA) equipped for epifluorescence and with a model DC200 color charge couple device camera system at 2.5x magnification. Samples were irradiated with blue (470/40 nm) and UV (360/40 nm) light, and images of the fluorescence were acquired at 0.1 (blue) and 0.44 (UV) seconds of exposure time. Control samples, immersed in 500% v/v nano-pure water and 5μM dye, were examined under the same conditions of concentration and time to assess the penetration of the fluorescent dye in the absence of salt, and a blank sample was also examined to evaluate possible autofluorescence of the untreated raw hide.

RESULTS AND DISCUSSION

Results and Discussion Epifluorescence Microscopy

The diffusion of labeled sodium ions into the cross section of hide samples was followed by means of epi-fluorescent microscopy. In Figure 4, increased fluorescence indicating diffusion of salt started on the flesh side and gradually moved toward the hair side. The lack of fluorescence development at the hair side validated the mathematical assumption described by Eq. (1a); this can be attributed to the existence of a thin protective barrier of sebaceous oil.[14] The series of images demonstrate the advance of fluorescence due to sodium ions into the cross sections of hide as well as increases in fluorescence intensity throughout curing time. Signal saturation in the area near the flesh side, denoted by a very bright fluorescence, was observed after 5 h of curing. The apparent retrograde movement of the labeled sodium between 2 h and 5 h may be due to the shrinking of the hide caused by dehydration. Surprisingly, sodium ions did not seem to reach the hair side even after 48 h of curing. This could be due to many factors. In order to determine if the penetrability of the dye is a technical factor, a sample of hide incubated in an aqueous solution of dye for 24 hours was examined under the microscope and then transferred to a beaker with concentrated brine solution for 24 more hours. The dye did not fluoresce in the uncured sample but showed a high fluorescence after being cured for 24 hours (graphs not shown). However, fluorescence was absent or undetectable in the upper part of the corium, leading to the possibility that the dye may not penetrate into the tightly-woven and dense structure of the corium, possibly due to its size (MW = 586 Da). The use of scanning electron microscopy with energy dispersive X-Ray spectroscopy (SEM-EDS) and elemental mapping to measure the amount and location of salt in a brine-cured hide is an alternative method to fluorescence imaging and this approach is planned.

Determination of Diffusion Coefficients

The diffusion of salt in the hide was evaluated by means of the transport coefficient λ, which can be calculated from the slope of the straight line that results from plotting $C_0(t)$ versus square root of time (Eq. 10). Taking into account early published results[12] and the accuracy of our measurements, the linear dependence holds approximately as far as to the value of $C_0(t) = 0.6$. Figure 5 depicts this correlation for the particular case of c_{0p} = 30% (w/v) and Na = 3.

TABLE I
Transport Coefficient λ for Various Conditions of Initial Brine Concentration (c_{0p}) and Soaking Number (Na)

Na = 3			C_{0p} = 30% (w/v)		
C_{0p} (% w/v)	λ 10^5 (s^{-1})	R^2	Na	λ 10^5 (s^{-1})	R^2
20	4.2	0.835	3	5.3	0.949
25	3.8	0.921	5	9.0	0.887
30	5.3	0.949	7.5	4.1	0.905
35	10.7	0.776[a]	10	8.6	0.876

[a] R^2 < critical value for $\alpha = 0.05$.

As seen in Table 1, all λ values, except from that of c_{0p}=35% (w/v), are on the order of 10^{-5} s^{-1}. These results are of the same order of magnitude as those reported in the mathematical model of soaking[15], which suggests that the diffusion of salt does not significantly differ between curing and soaking. A numerical value of λ for c_{0p}=35% (w/v) may not be reliable, because the brine was initially supersaturated and the model was developed for homogeneous solutions solely. Note than a saturated brine solution holds 31.7g of salt in 100 ml of solution, (c_{0n}) at 25°C.[16]

A comparison of the individual values of λ is not simple, since they may be affected by some of the following factors: 1. The thickness of the hide, which may vary throughout the process and exerts a strong effect on the value of λ, as seen in Eq. (13). 2. The pore length, which varies among the hides and is hardly measurable. 3. The dry matter content of the hide, the variation of which may extensively modify λ. In fact, λ may drop up to two orders of magnitude between a wet and a dry hide.[15,17] 4. The temperature, which affects the diffusion rate and was sometimes difficult to keep at 25°C during the process. 5. The influence of the error in the measurement, i.e. the difficulty to measure a small chloride concentration diminution with the Mohr method despite the very low coefficient of variation (CV) found for this technique (< 1%).

Even so, one can draw the conclusion on the effect that both c_{0p} and Na exert on the values of λ. Increasing values of c_{0p} yielded larger values of λ as a consequence of an increasing gradient concentration between the solid phase and the solution, which is in accordance with Fick's second law of diffusion. This fact corroborates a general practice applied in curing raceways, where solid salt is periodically added to the brine solution to keep it close to saturation (≥ 97 °SAL). The float percentage also exerts a remarkable effect on the values of transport parameter λ. Larger floats yielded faster diffusion of salt into the hide, even though that effect became less significant for Na > 5. That experimental observation corroborates the common practice in tanneries, which operate at Na ~ 4 even though the generally accepted rule requires a Na ≥ 5 in order for hides to receive a proper cure.[18] In addition, a large float will help maintain an almost constant salt concentration. The outstandingly low value of λ obtained for Na = 7.5 may be due to factors inherent to the hide, e.g., poor fleshing, which slows down salt penetration, agglutination of the fibers and content of dry matter.

Determination of Optimum Brine Curing Conditions

An 85% salt saturation of the water remaining in the hide was established as a minimum standard in order to attain a proper degree of cure.[19] One can calculate the theoretical minimum soaking number needed to attain this saturation percentage in the equilibrium, that is, at infinite time, and without further additions of salt into the solution. From Eq. (8), if $\tau \rightarrow \infty$ then $F_o \rightarrow \infty$; thus the second summand can be neglected, giving:

$$C_0 = \frac{c_0}{c_{op}} = \frac{Na}{\varepsilon + Na} \tag{15}$$

Replacing $c = \varepsilon \cdot c_0$ and rearranging for Na,

$$Na = \frac{\varepsilon \cdot c}{\varepsilon \cdot c_{0p} - c} \tag{16}$$

Using $c = 0.85 \cdot \varepsilon \cdot c_{0n}$, a porosity of ε = 0.5 and c_{0p} = 30 % (w/v), and assuming that all pores are filled up with brine solution, a soaking number of 4.4 is needed to reach 85% saturation. Solving Eq. (16) for c_{0p} = 20 and 25% (w/v), negative values of Na are obtained, indicating the unfeasibility to attain 85% saturation. On the other hand, the minimum soaking number would drop to 2.8 if the cure was started out with a saturated brine solution (31.7 % (w/v), or 100 °SAL). Notice that these values depend on the porosity of the hide, which varies from one to another and within itself.[20] Therefore, slightly different values of minimum Na's would be obtained using another value of porosity.

Working out the value of c_{0p} from Eq. (16) and using Na = 3, we received a minimum initial brine concentration of 30.8% (w/v) in order to achieve the target saturation level. By means of Eq. (9), the plot of which is depicted in Figure 3, one is able to calculate the curing time needed to reach an 85% salt saturation in the hide. As just mentioned above, this level cannot be achieved for c_{0p} = 20, 25 and 30% (w/v) and Na = 3. However, a time of 4.2 h is obtained for a 35% (w/v) supersaturated brine and Na = 3. The 85% saturation is also achieved in 4.9, 7.7 and 3.4 h if the hide is cured with a brine of c_{0p} = 30% (w/v) and Na = 5, 7.5 and 10, respectively. These values are substantially lower than the 18 hours that usually are required for a full cure in a normal float (Na~5). The calculations of those times contain the parameter λ, and therefore are affected by the same factors mentioned in the previous section. In spite of this, it is interesting to note the decrease of curing time with increasing float percentages, except from the erratic value obtained for Na = 7.5.

CONCLUSIONS

Over 20 millions brine-cured hides were exported by the U.S. in 2006 (U.S. Leather Industry Statistics, 2007). Increasing commodity prices for sodium chloride over the past few years together with issues associated with water pollution set the alarm off in the leather and meatpacking industries. The purpose of research reported in this article was to optimize the brine curing of hides and skins under specific process conditions by means of mathematical modeling. The diffusion of salt into the hide was characterized by the transport coefficient λ, which was found to be in the order of 10^{-5} s^{-1}. The usage of saturated brine as well as large floats (>500%) yielded higher values of λ, therefore higher diffusion rates. From the model it was also possible to determine the minimum float and initial brine concentration needed to attain an 85% salt saturation in the hide. This saturation level was not achieved employing brines of initial concentration of 20 and 25% (w/v) independently of the float percentage used. For 30 and 35% brines, a minimum float of ~ 440% and ~280% was found, respectively. Using a 30% (w/v) brine, the targeted 85% saturation is attained in shorter times as the % float increases, and one may expect the same trend for any other initial brine concentration. The established 85% salt saturation in the hide obviously plays a critical role in the search for optimum conditions of curing, and the need to attain this saturation level for a proper cure will be discussed in our next contribution.

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DEFINITION OF TERMS

c: concentration of sodium chloride in the hide moisture, at a distance *x* from the boundary (τ > 0) [mol m^{-3}]

c_0: concentration of sodium chloride in the bath (τ > 0) [mol m^{-3}]

c_{0n}: concentration of saturated sodium chloride solution at 25°C [mol m^{-3}]

c_{0p}: initial concentration of sodium chloride in the bath (τ = 0) [mol m^{-3}]

$c_{0\infty}$: equilibrium concentration of sodium chloride in the bath [mol m^{-3}]

$\overline{C}$: dimensionless concentration integral average [1]

D: diffusion coefficient of sodium chloride in the hide [m^2 s^{-1}]

D': effective diffusion coefficient of sodium chloride in the hide [m^2 s^{-1}]

S: outer surface of the solid phase (skin) [m^2]

a: pore half length of the skin [m]

b: thickness of cured hide [m]

Na: soaking number [1]

V: volume of skin [m^3]

V_0: volume of brine solution [m^3]

F_0: Fourier number/dimensionless time [1]

X: dimensionless distance [1]

C, C_0: dimensionless concentrations [1]

Greek symbols

τ: time (s)

ε: porosity of solid state [1]

λ: transport coefficient [s^{-1}]

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