

DRYING OF LEATHER WITH MICROWAVES

Abstract

The characteristics involved in microwave drying of leather were studied at atmospheric pressure and under vacuum. The temperature of the leather during drying was varied over a wide range by varying the energy input, by increasing the air flow in microwave-assisted hot air drying, and by increasing the vacuum. Experimental data are presented, describing temperature buildup and drying rates as functions of moisture content and drying time. Differences in physical properties and area yields at different drying conditions are discussed as well.

Introduction

The most popular method of drying leather is convective removal of moisture into a hot, humid (but unsaturated) air stream at atmospheric pressure. This is accompanied by a considerable shrinkage in area. Tacking, toggling, and pasting are resorted to as means of preserving area. Recently, vacuum drying has been introduced primarily for drying of aniline leathers because it avoids pasting and yet yields a flat grain surface and a satisfactory area. In both hot-air drying and vacuum drying, the heat required to evaporate the water is applied superficially. This results in a large temperature gradient and non-uniform moisture distribution in the leather during and at the end of the drying process with the surface being much hotter and drier than the center. The most recent addition to the technology is microwave drying, a form of electronic heating that is often called volume heating to distinguish it from the other methods that apply heat superficially. While this method of drying may provide a more uniform distribution of heat and moisture⁽¹⁾, it appears to have at least two serious drawbacks: a potential to cause heat damage⁽²⁾, and a high cost⁽³⁾. Since information on microwave drying of leather is rather meager, this research was conducted to explore the potential of this drying method in the manufacture of leather.

Experimental

DRYING EQUIPMENT

Microwave drying was carried out in a continuous microwave conveyor drying oven manufactured by Cober Electronics, Inc., equipped with a controlled hot-air heating system and an industrial microwave generator (Model S6F-J) capable of delivering up to 6KW power at 2450 MHz.

To permit microwave drying under vacuum, the conveyor belt was kept stationary. As

MICROWAVE DRYING

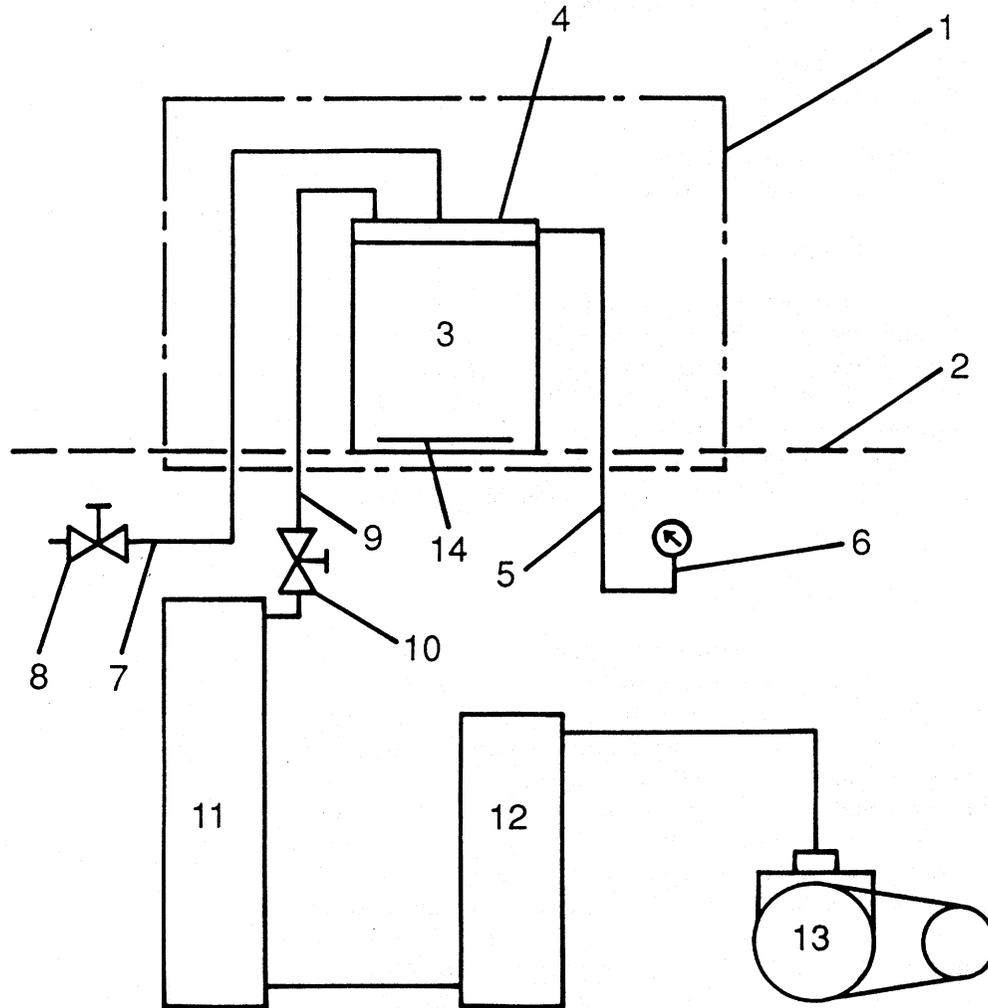


FIG. 1. — Continuous microwave conveyor drying oven adapted to vacuum drying. (1) Processing tunnel, (2) conveyor belt, (3) vacuum chamber (desiccator), (4) vacuum chamber lid with tubulation: hose (5) to manometer (6), hose (7) to off/on valve (8), and hose (9) to valve (10) which is connected first to a vacuum tank (11), then to a cold trap (12) and a vacuum pump (13). Leather sample (14) placed on a desiccator plate.

shown in Fig. 1, a vacuum-type Pyrex brand desiccator with an inside diameter of 25 cm was installed in the middle of the processing tunnel. This desiccator had a lid with three openings with hoses that led outside the vacuum oven. One hose was connected to a manometer, a second hose led to an off/on valve used to break the vacuum in the desiccator, and a third hose was connected to a valve, then to a 50 gal. vacuum tank, which in turn was connected to a cold trap and then to a vacuum pump. The vacuum tank was kept under vacuum at all times during the experiment to permit quick evacuation of the desiccator by opening the valve between the tank and the desiccator. To make weight measurements during drying, the microwave power input was interrupted, the vacuum in the desiccator was broken, and the sample was removed for weighing.

Microwave drying under vacuum was also carried out utilizing a large fritted glass filter funnel 15 cm in diameter made of Pyrex. Instead of filter paper, discs made from leather were placed inside the funnel. These discs were covered with cellophane before vacuum and microwave energy were applied for drying. This simulated the conditions encountered during conventional vacuum drying.

Conventional hot-air drying of leather was carried out in a type TYI, through-circulation, batch, hot-air drier made by the National Drying Machinery Co.

TEMPERATURE MEASUREMENT

During microwave drying, the temperature inside the leather was measured with a Luxtron Fluoroptic Thermometer Model 1000 A/S using a LIA-4 temperature probe made of plastic-clad optical fiber. During vacuum drying, the temperature probe was introduced into the desiccator through the same valve that was used to break the vacuum. To ensure full vacuum, the hole was closed with a putty-like sealing compound for vacuum systems.

Materials

Unless otherwise stated, most of the experiments were conducted on discs of chrome tanned leather 2.3 mm in thickness, 20.5 cm in diameter, and weighing approximately 100 gm. The sole leather used was 5.7 mm in thickness and was vegetable tanned.

Results and Discussion

EFFECT OF TEMPERATURE

Hot-air drying induces temperatures inside the leather that are intermediate between the wet-bulb temperature and the dry-bulb temperature of the air. As long as there is free moisture present on the surface, the temperature in the leather is equal to the wet-bulb temperature. As drying proceeds, the temperature increases and becomes almost equal to the dry-bulb temperature at the end of the drying operation. Fortunately, collagen does not denature readily at lower moisture contents⁽⁴⁾ when the temperature rises, although crosslinking and other chemical reactions have been demonstrated to take place at an accelerated rate at low moistures and high temperatures.

During microwave drying, the situation is just the opposite with the temperature quickly rising⁽⁵⁾ and remaining high as long as there is free moisture present in the leather, since this moisture can absorb microwave energy by molecular friction. Consequently, there is a definite danger from overheating during the initial stages of drying.

Line A of Fig. 2 shows that even at a power input as low as 2 KW, the temperature of the leather can readily rise to 100°C. In this experiment, the leather was fully wet, and cooling by convection drying was minimized with the sample being placed inside a desiccator that was open to the atmosphere only through a small hole in the lid.

Line B of Fig. 2 shows that the temperature does not rise as fast when air is blown over the leather during microwave drying. Several factors contribute to this reduction in temperature rise. As temperature in the leather increases, more water vaporizes and cools the leather. At the same time, the microwave power dissipation decreases due to a drop in the dielectric loss or the so-called imaginary part of the relative dielectric constant, ϵ_i ^(6,7). Power dissipation (or energy conversion) and the numerical value of ϵ_i drop even more when the drying enters the hygroscopic moisture range⁽⁸⁾ in which the bound moisture no longer exerts its full vapor pressure.

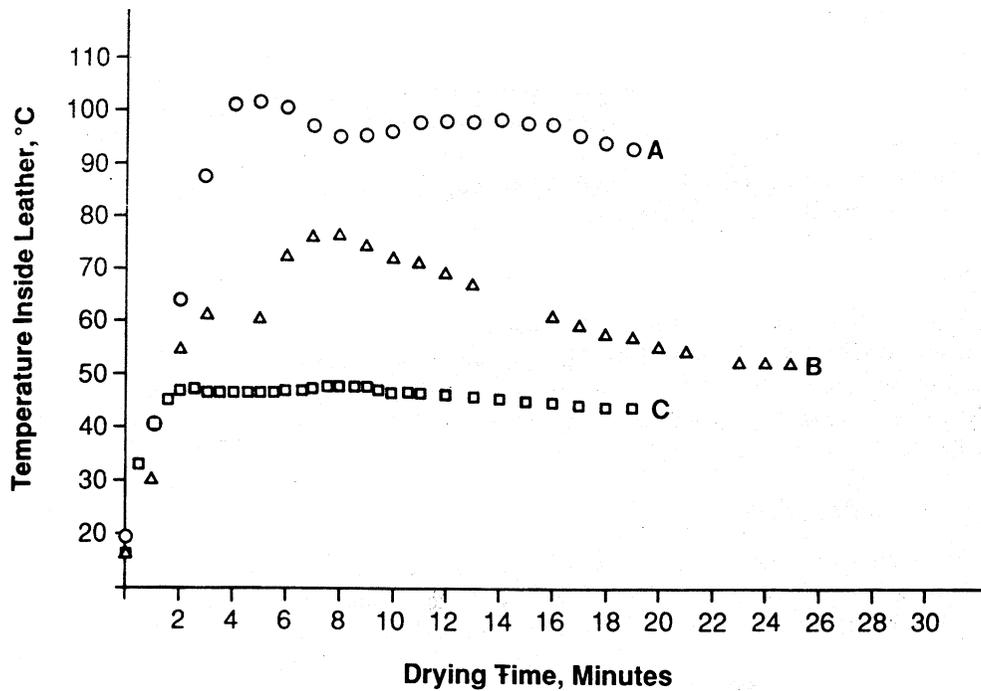


FIG. 2. — Temperature buildup inside leather during microwave vacuum drying of chrome tanned leather: Line A - 1 atm, 2 KW, no air flow; Line B - 1 atm, 2 KW, with air flow; Line C - 711 mm Hg vacuum, 2 KW. Frequency = 2450 MHz.

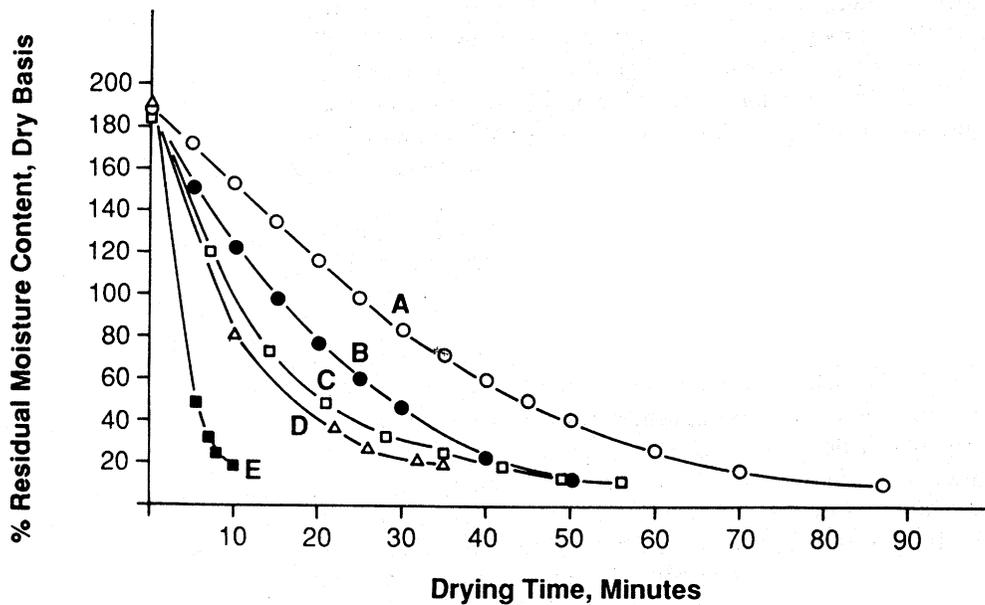


FIG. 3. — Drying of chrome-tanned leather. With hot air: Line A (at 43°C dry bulb temperature and 22 per cent relative humidity); Line B (at 55°C dry bulb temperature and 13 per cent relative humidity). With microwaves (2450 MHz): Line C (1 atm, 2 KW), Line D (724 mm Hg vacuum, 2 KW); Line E (724 mm Hg vacuum, 5 KW). Thickness = 2.3 mm.

But, as shown by line C, the smallest temperature increase by far is encountered by conducting the microwave drying under vacuum, although the experimentally measured temperature of 48°C is 10°C higher than the boiling point of water at the experimentally determined vacuum of 28 in. Hg (711 mm Hg).

DRYING TIME

The drying times are compared in Fig. 3 for two conventional hot-air drying conditions (lines A and B) and three microwave drying conditions at different pressures (lines C, D, and E). The slow drying rate observed at 43°C by conventional drying (line A) is seen to increase considerably as the temperature is raised to 55°C (line B), approaching microwave drying at atmospheric pressure with a power input of 2 KW (line C). It takes approximately the same amount of energy to vaporize water regardless of the pressure of the surroundings; consequently, microwave drying under vacuum (line D) takes place almost at the same rate as microwave drying at atmospheric pressure (line C) as long as the power input is the same (2 KW). Of course, when the power was more than doubled (to 5 KW), the drying time decreased by about the same factor as shown by line E. It should be noted, however, that increasing the power to 5 KW was permissible under vacuum only. At atmospheric pressure, the temperature rise often caused denaturation in small spots (even in chrome tanned leather) as the shrink temperature was exceeded.

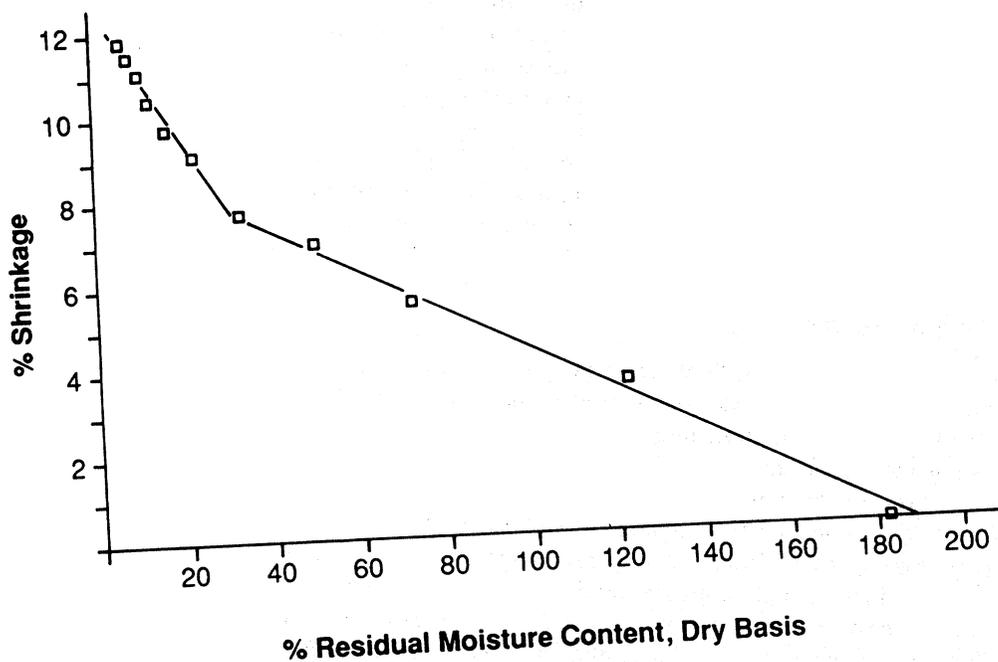


FIG. 4. — Shrinkage of chrome-tanned leather during microwave drying. Leather thickness = 2.3 mm. Power input = 2 KW at 2450 MHz and 1 atm pressure.

SHRINKAGE DURING MICROWAVE DRYING

The shrinkage experienced by leather when it is dried with microwaves while unrestrained is shown in Fig. 4. It appears to shrink just a little bit more than during conventional

hot-air drying. The reason for this slightly increased shrinkage rate is the fact that hot-air drying induces a steep moisture gradient between the center and the surface of the leather. During the first part of the drying cycle, the center, being still very wet, resists shrinkage and prevents the already dry surface from normal shrinking. At a later stage of drying, the somewhat stretched but very dry surface, in turn, prevents the center from experiencing normal shrinkage. During microwave drying, the moisture gradient is relatively small and shrinkage occurs simultaneously at all depths with a minimum amount of restraint. It is most interesting that there is a sudden increase in shrinkage rate at about 32% moisture content at which, according to the literature, there is no more free moisture present in collagen fibers. It is also noteworthy that, unlike during air drying, there is an increase in shrinkage rate at that moisture content rather than a decrease.

PHYSICAL PROPERTIES

Aside from the minor differences in the degree of shrinkage, microwave drying at atmospheric pressure yields similar results to those of conventional hot air drying, and microwave drying under vacuum yields results similar to those obtained by vacuum drying. For example, placing a disc of leather inside a fritted glass filter funnel and pulling vacuum followed by microwave drying gave the same restraint and yielded the same result as spreading the leather on a steel plate during vacuum drying or the same effect as pasting during air drying. This procedure imparted a firmer feel to the leather, increased the area, and improved the grain. The surface of the grain was shiny and smooth, as it is after vacuum drying. During microwave drying, evaporation takes place mostly inside the leather; nevertheless, there is still a problem arising from migration of the tanning materials and especially oils. Discoloration stemming from this problem was easily overcome by making the moisture move toward the flesh side by placing the leather on a glass surface with the flesh side up during unrestrained drying. When dried without restraining, microwave-dried leather is somewhat stretchier, most probably because it had shrunk just a little bit more during drying.

MOISTURE DISTRIBUTION DURING DRYING

There is a great difference in moisture content between the center and the surface of the leather during air drying. The constant-rate drying period ends at the first critical point when the surface is already at the hygroscopic moisture content for leather (which is about 60 per cent on solids basis) while the center is still very wet. A second critical point is reached when the center is at the hygroscopic moisture content while the surface is already at the moisture content corresponding to equilibrium with the moisture in the drying air. As can be seen from Fig. 5, the moisture distribution is different when using microwave drying. To augment the difference in moisture distribution, microwave drying experiments at atmospheric pressure were conducted on three layers of leather discs with a total thickness of 6.9 mm and a diameter of 23 cm. The leather discs were dried for 20, 40, and 55 minutes using 2 KW of power. After each drying period, the batch was removed from the drier, and the three discs were quickly separated. Each of the discs was then cut into 3 concentric bands or rings and a small center disc. The moisture content was then determined for each of these rings and the center disc. The initial moisture content in the leather was 185 per cent on dry basis. Further inspection of this figure shows that after 20 minutes of drying, more water was removed from the top layer than from the bottom layer, and more moisture was lost from the outermost ring than from the inner

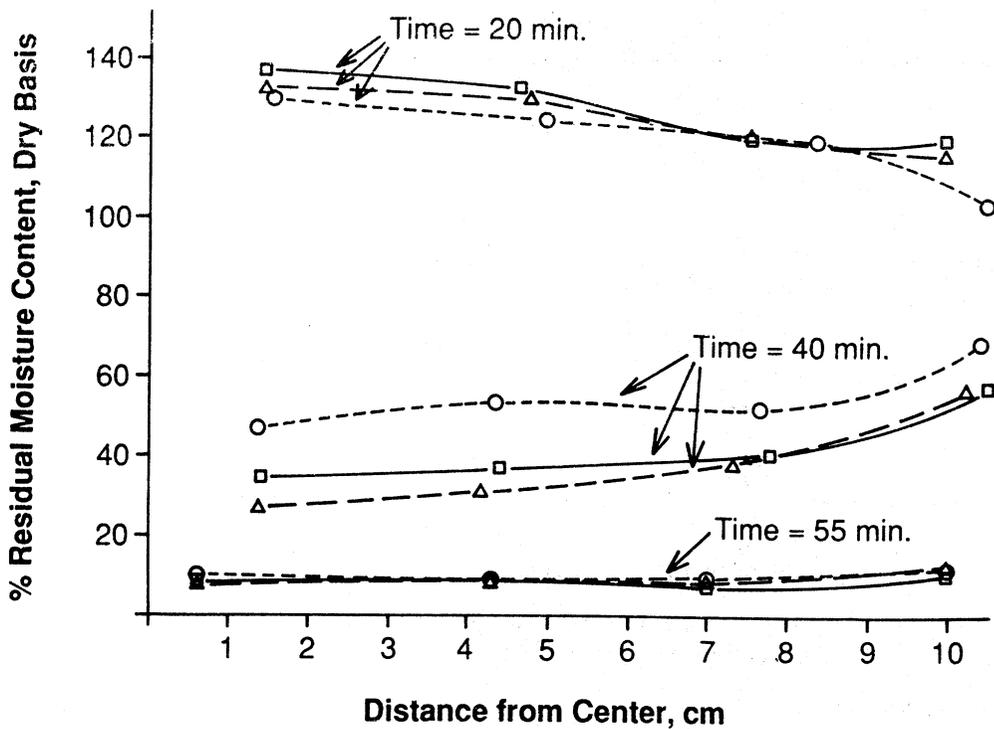


FIG. 5. — Moisture distribution with time during microwave drying of chrome-tanned leather: Leather thickness = 6.9 mm. ○ Top layer, △ Middle layer, □ Bottom layer. Power input = 2 KW. Initial moisture content = 185% dry basis. Pressure = 1 atm.

disc. The dielectric loss, ϵ_i , decreases with decrease in moisture content; therefore, this moisture gradient, although small, resulted in a temperature gradient with the center being at a higher temperature than the peripheries. Consequently, relatively more moisture started to be removed from the center and, therefore, the opposite was found to be true after 40 minutes of drying with the center being drier and the center of the middle layer being the driest. The moisture distribution at this time period indicates that movement of water to the surface proceeds most probably by capillary condensation. While there is practically no difference in moisture content over the whole area of the top layer at the end of drying, i.e., after 55 minutes of drying, there is still a moisture gradient in the bottom layer, with the peripheries containing about 3% more moisture.

To demonstrate the effect of the capillary condensation mechanism of moisture movement in conjunction with a temperature gradient, a disc of chrome-tanned leather was placed between two Pyrex watch glasses and subjected to the same drying conditions of 2 KW power input at 2450 MHz. The result is shown in Fig. 6. The moisture content near the edges is higher than in the center, just the opposite from what it would be during air drying. This can occur only if the vapors traveling from the center recondense nearer the edge. However, if one considers that moisture can escape only from the edges of the leather discs under the conditions of the experiment, the fairly small moisture gradient observed even under the above conditions is a convincing indicator that microwave drying of leather indeed occurs at a relatively uniform moisture distribution.

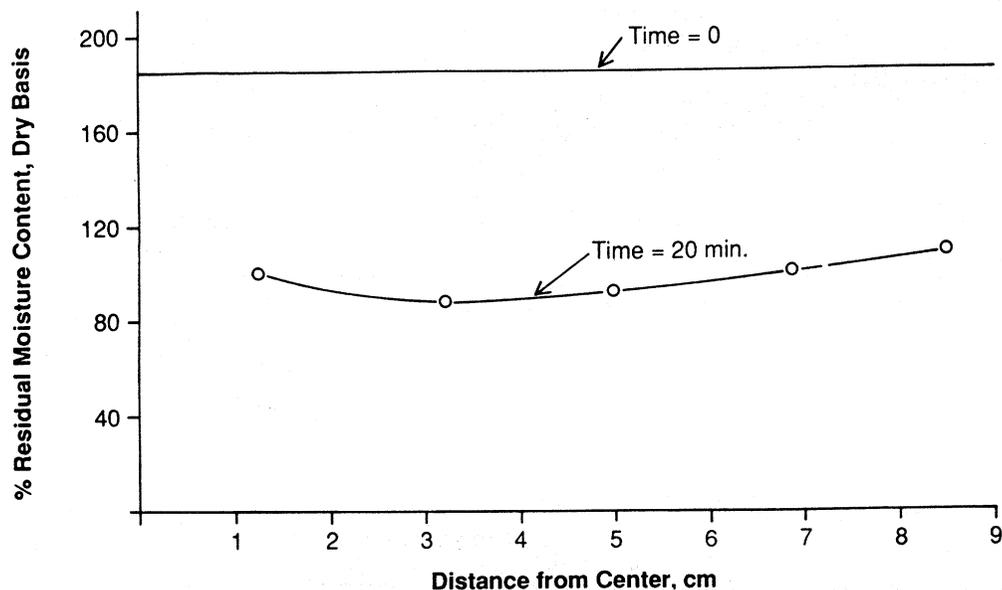


FIG. 6. — Moisture distribution with time during microwave drying of chrome-tanned leather: Power input = 2 KW, Leather thickness = 2.3 mm. Drying only from edge of a 20.5 cm disc of leather.

DRYING OF VEGETABLE TANNED LEATHER

It was shown above that microwave drying at atmospheric pressure results in an increase in temperature (even at slow drying rates) up to 100°C if there is free moisture present in the leather and if the air velocity and humidity are inadequate to cool the leather sufficiently. With chrome leather, this does not present a problem if the power input is carefully controlled because well-tanned chrome leather has a shrink temperature of about 100°C. There is only tightly bound moisture present in collagen at moisture contents below 34 per cent⁽⁹⁾. Also, microwave energy is not as readily converted into heat in the absence of free water, i.e., when the moisture content is low, because bound water is a poorer microwave absorber than free water⁽⁵⁾. Consequently, there is little danger from overheating of staked leather, especially staked chrome leather. This is not true with fully wet vegetable-tanned leathers with shrinkage temperatures normally ranging between 70-85°C. Application of as little power as 1 KW at 2450 MHz caused denaturation of vegetable-tanned leather and exudation of oils from it even at moisture contents when only bound water was present. This overheating is pronounced when drying sole leather because the usual heating effect, which is due to dipole rotation of water molecules, is enhanced by ionic polarization, which is due to the presence of such ionic salts as Epsom salt. But, whereas microwave drying at atmospheric pressure damaged vegetable tanned leather, microwave drying under vacuum was found to be quite satisfactory.

Line A of Fig. 7 demonstrates the slow drying observed with 5.7 mm thick, vegetable-tanned sole leather when using hot air at 43°C and 22 per cent relative humidity, even though at these conditions the drying time is much shorter than that observed under normal commercial drying conditions for this type of leather. Line B contrasts the short time needed to dry the same sole leather with microwaves under a vacuum of 28.5 in. Hg (724 mm Hg) using only 2 KW of power at 2450 MHz. Although dried in less than 20

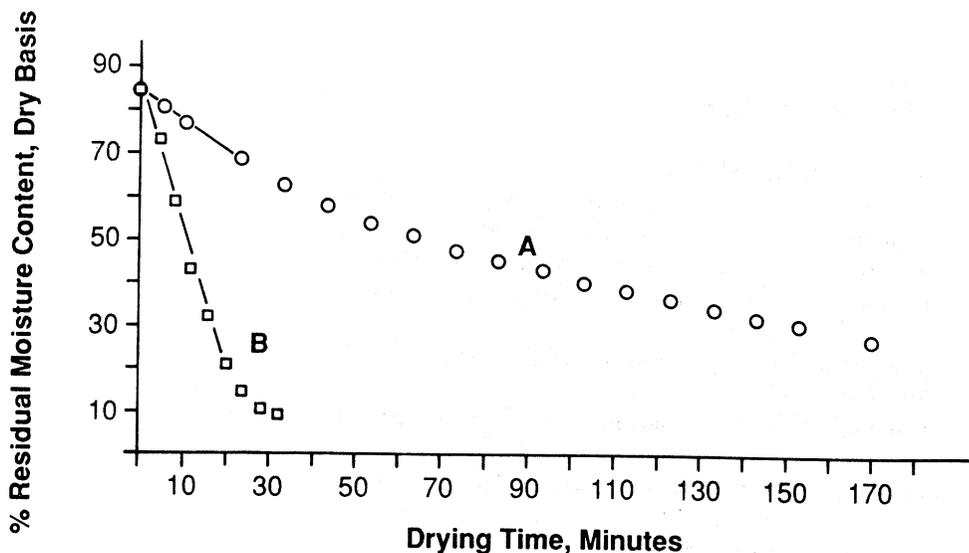


FIG. 7. — Drying of vegetable-tanned sole leather. Line A: (hot air, dry bulb temp. = 43°C, 22 per cent rel. hum.). Line B: (Microwaves, 2 KW at 2450 MHz, 724 mm Hg vacuum). Leather thickness = 5.7 mm.

min., microwave-dried sole leather was indistinguishable from similar leather air-dried at room temperature. No migration of tanning material to the surface was observed, probably because the film of paste—composed of oils, Epsom salt, and other ingredients—prevented it. Also, shrinkage due to drying was minimal (probably less than 1 per cent). It is significant that (according to line B) the constant-rate drying period extends down to at least 20 per cent moisture content by microwave drying under vacuum.

Summary and Conclusion

Microwave drying of leather can be used only under carefully controlled conditions to prevent damage by overheating. However, under vacuum conditions (or if only bound water is present in the leather) the abuse to leather caused by microwaves is minimal. Overheating must be avoided, especially when drying non-chrome tanned leathers because of their low shrink temperatures. Microwave drying may have an advantage over air drying because throughout the whole drying cycle the moisture distribution is almost uniform over the area and at all depths; however, this potential advantage has not been fully explored. From the standpoint of area yield, feel, and other physical properties, microwave drying under vacuum gives results similar to those obtained by conventional vacuum drying. Microwave drying under vacuum with proper development of suitable equipment shows the best promise for the drying of thick leathers, especially vegetable-tanned sole leather because it can shorten the drying time to a small fraction of that normally needed to dry this leather.

Acknowledgments

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Discussion

MR. ROBERT GOOD, Pfister and Vogel: Is the chromium molecule in the leather affected by the microwave radiation to excite it and cause heating of the leather?

MR. KOMANOWSKY: Theoretically, all polar molecules should be excited by microwaves, but I have intentionally placed dry leather inside the drier and kept it in there for a very long time while conducting other experiments. I could not detect much increase in temperature inside the chrome-tanned leather. Based on this observation, I believe that chrome in dried leather is not affected.

DR. ESA MANTYSALO (Tampere University of Technology): You have to subject leather material to a high electric field in a microwave cavity. What kind of cavity arrangement and configuration were used?

MR. KOMANOWSKY: At atmospheric pressure, the drying was carried out inside a commercially available tunnel drier provided with an endless conveyor belt. I assume that its cavity was designed to have the proper resonance at the wavelength generated. When placed in different locations in the cavity, leather samples dried at the same rate, indicating a uniform distribution of the electric field. There is a need, however, for additional development of equipment to accomplish efficient vacuum-drying of leather with microwaves. There is no equipment presently available for doing this continuously.

DR. MANTYSALO: I am not sure how you can measure the microwave power in your cavity, if you do not know the mode of configuration.

MR. KOMANOWSKY: The equipment we have been using has an instrument indicating the amount of energy supplied and the amount reflected. Thus there seems to be no problem in calculating the total energy used.

DR. MANTYSALO: I think the power of which you have been speaking is not "effective power".

MR. KOMANOWSKY: True. As a matter of fact, almost 50% of the input power is lost during conversion of the line power into microwaves. There are additional losses which depend on many factors such as drier construction and loading. Of course, you also have losses in conventional hot-air drying equipment where the efficiency is often as low as 20%. To compare the two drying methods on the basis of cost would require a major study.

MR. GEORGE STOCKMAN, Pfister & Vogel Tanning Company: Michael, I have a question. We have heard over the past several years from different sources that microwave drying generally results in an improvement in yield, and yet in your studies you seem to suggest that you had some contraction. Can you shed a little more light on this?

MR. KOMANOWSKY: While the method of introducing heat into the leather and the mechanism governing the moisture removal are different, the capillary forces which cause shrinkage appear to act in the same way during microwave drying as during conventional hot-air drying. As shown in Figure 4, the percent shrinkage observed is similar to values reported in the literature for cowhide leather, although a measurable difference was observed on different pieces of leather from the same hide.

DR. KEN ALEXANDER, British Leather Confederation: While you were looking at microwave drying did you get a chance to compare the degree of fiber sticking with that occurring during air drying? Were there, for example, any effects in promoting softer or stronger leather?

MR. KOMANOWSKY: I was hoping that I would get softer leather and the initial indications showed it to be so, as seen from the longer flat region of the stress-strain curve; when I repeated the experiment, however, with different leather I observed no difference between air drying and vacuum drying. Consequently, I can't answer the question definitively.

MR. DONALD HARRIS, DYNATAN Incorporated: My principal in Germany — DYNAVAC GmbH — has done quite a lot of work trying to develop Microwave Dryers. Having manufactured prototype chamber dryers we found that drying with microwave energy was very expensive. Therefore, we are working on a system that will result in level moisture content ready for staking. We want the tanner to be able to dry from, say, 28% to a positive level moisture content of perhaps 18% to 20% — whatever he wants for staking. We are continuing to direct our efforts toward getting a level moisture content within the leather. Can you comment on this?

MR. KOMANOWSKY: This is one of the reasons I did the research. Based on my findings, I believe it should be possible to build a microwave drier which would dry leather to a predetermined uniform moisture throughout, especially if the desired moisture content is low.

MR. STOCKMAN: Michael, I would like to thank you for an excellent presentation.