

## WATER ABSORPTION PROPERTIES OF VARIOUS TYPES OF LEATHER\*

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### ABSTRACT

The water absorption properties of various types of side leathers were studied in order to evaluate the effect of factors such as glutaraldehyde retannage, alkenyl succinic acid (ASA) lubrication, and water-repellent treatments. Data are presented on the absorption of water vapor by leather exposed for prolonged periods of time at relative humidities of 20, 50, 76, and 91 percent. There were only slight differences in water vapor absorption of the various leathers, indicating that retannage with glutaraldehyde, treatment with water-repellent agents, or lubrication with alkenyl succinic acid were minor factors in affecting this property of the leather. The uptake of liquid water on static immersion was lower for the repellent leathers than for the nonrepellent leathers. Previous results indicating that glutaraldehyde retannage improved the efficiency of the water-repellent treatments were confirmed. The application of water-repellent materials did not reduce the water vapor permeability of these leathers to any great extent.

### INTRODUCTION

The water binding properties of leathers have in the past few years been the subject of various publications. Kanagy (1, 2) made a study on the absorption and desorption of water vapor by collagen and leather at various humidities and temperatures over an extended time period. Recently Seligsberger (3) determined the water vapor absorptive capacity of leather and synthetic shoe materials at various temperatures, and Kennedy *et al.* (4), in a comparison made between boots with permeable uppers and boots with impermeable uppers, showed that significant differences in water vapor uptake existed between these two types. Happich *et al.* (5) presented data on the water vapor absorption and desorption of shearlings tanned with various combinations of chrome and glutaraldehyde, and compared these properties with those of synthetic substitute materials. Of

particular interest were the studies of Grassman and Zeschitz (6) and Heide-mann (7), who pointed out that leather impregnated with silicone retained its affinity for water vapor, and its capacity to absorb water vapor showed little difference from that of untreated leather.

In recent studies (8, 9) at this Laboratory some tanning factors were evaluated with regard to their influence on water repellency of certain chrome-tanned leathers when treated with silicon, Scotchgard, and Quilon. In general, it was found that retannage of chrome leather with glutaraldehyde and lubrication with small amounts of an alkenyl succinic acid (ASA) were two factors that greatly improved the efficiency of treatments with these well-known water-repellent agents. In the present paper, the effects of these various tanning factors, namely, retannage with glutaraldehyde, lubrication with an alkenyl succinic acid, and treatment with water-repellents, on the water sorption properties of side upper leather were evaluated. The properties of interest were absorption and desorption of water, in both the vapor and liquid states, and water vapor permeability of the various leathers.

### EXPERIMENTAL

The sides used in the following experiments were commercially tanned, HM weights, obtained in the blue or as finished leathers. The finished sides were: (1) chrome-tanned and given an acrylic finish, (2) chrome-tanned with a ten percent glutaraldehyde retan, and (3) chrome-tanned with a ten percent glutaraldehyde retan and given a silicone water-repellent treatment.

**Leathers Lubricated with ASA.**—For the study on the ASA-lubricated leathers and water-repellent leathers, the sides were obtained from the tanner as chrome-tanned grain splits. Some of these were retanned with ten percent glutaraldehyde, prior to coloring and fatliquoring. The tanned sides were fatliquored with emulsions consisting of one percent alkenyl succinic acid (ASA), ten percent tetrahydrofurfuryl alcohol, and sufficient water to give a 50 percent float, according to our previous procedure (10). The fatliquored sides were then toggled, allowed to air-dry, and returned to a tannery to be sammied and staked. After staking, 12–14 inch strips were taken in the butt section, from the backbone to the flank, for water-repellent treatments.

**Leathers Treated with Water-Repellents.**—For the silicone treatment, the leather strips were dipped in a three percent solution of Dow Corning 1109<sup>‡</sup> resin in perchloroethylene (11). The pickup of silicone was 4.6 percent for the chrome side, and 5.2 percent for the chrome side retanned with ten percent glutaraldehyde. The treatment with Quilon M was effected as recommended by the manufacturer (12), using a 2.7 percent aqueous solution. The uptake of

<sup>‡</sup>Mention of brand or firm names does not constitute an endorsement by the Department of Agriculture over others of a similar nature not mentioned.

Quilon was calculated to be 3.8 percent for the chrome side, and 4.4 percent for the chrome side retanned with ten percent glutaraldehyde. Scotchgard FC-146, which is a 30 percent solution of a chrome complex of a long-chain fluorochemical (13), was applied by dipping in a 2.7 percent aqueous solution, as recommended previously (9). The pickup of Scotchgard was calculated to be 3.7 and 4.0 percent for the chrome side and the chrome side retanned with ten percent glutaraldehyde, respectively. The use of an approximately three percent solution of water-repellent regulated the uptake so that pickup of water-repellent was approximately the same for all three materials.

**Sampling.**—The specimens for the various tests were taken from the bend area of the impregnated leather strips, three inches from the backbone. All other specimens were taken from the "W" position recommended by the official ALCA procedures.

**Water Vapor Permeability.**—The water vapor permeability was determined by ASTM Method E96-66 (14). The specimens, six inches in diameter, were placed grain down over activated silica gel contained in a pyrex glass dish, and tightly sealed. The weight changes were plotted against elapsed time, and a straight line was fitted to the points. The weight change between two points on the slope was taken for the calculation of water vapor permeability.

**Water Vapor Absorption and Desorption.**—Water vapor absorption was determined by noting the weight gain of dry leather specimens (two inches x one inch) placed in large desiccators at  $73^{\circ} \pm 1^{\circ}\text{F}$ . maintained at various humidities, namely, 20, 50, 76, and 91 percent. Relative humidities of 20, 76, and 91 percent were obtained in closed containers from saturated solutions of potassium acetate, sodium acetate trihydrate, and sodium tartrate, respectively. The data at the 50 percent relative humidity were obtained by exposing the specimens in a constant temperature room maintained at  $73^{\circ}\text{F}$ . and 50 percent relative humidity. The samples were weighed after exposures of 0.5, one, two, four, eight, and 24 hours, and every 24 hours thereafter up to 192 hours of exposure. Samples that were to be exposed to 76 and 91 percent relative humidities were first dried in vacuum over phosphorus pentoxide, and then, after the water vapor absorption tests had been completed, the specimens were brought to the dry state in an evacuated oven maintained at  $50^{\circ}\text{C}$ . and in an atmosphere of dry air. In order to detect the slight increases in uptake at the lower humidities, the samples exposed to 20 and 50 percent relative humidities were brought to the dry state in an evacuated oven at  $50^{\circ}\text{C}$ . and then placed in the respective atmospheres.

Moisture desorptions were determined only for the specimens exposed to 91 percent relative humidity. The samples equilibrated at 91 percent relative humidity were placed in a conditioned room maintained at 50 percent relative humidity and  $73^{\circ}\text{F}$ ., and the weight loss at various time intervals was noted.

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**Absorption of Liquid Water.**—The uptake of liquid water of the various leathers was determined at 73°F. by total immersion of the specimens in water under static conditions, according to ASTM Method D1815-62 (15). All surfaces were kept fully submerged, and weighings were made at various time intervals. The water uptake was expressed on a weight basis.

**RESULTS AND DISCUSSION**

The water vapor permeability of water-repellent and nonrepellent leathers is shown in Table I. As expected, for the nonrepellent leathers, the commercial chrome leather with a heavy finish showed the highest resistance to water vapor

**TABLE I**  
**WATER VAPOR PERMEABILITY AND REPELLENCY OF VARIOUS LEATHERS**

Leather	Repellent Treatment	Repellent (%)	Water Vapor Permeability (mg./cm. <sup>2</sup> /hr.)	Water Repellency (flexes*)
Chrome-grain finish (comm.)	none	—	0.70	105
Chrome-glutaraldehyde (comm.)	none	—	1.42	50
Chrome-ASA	none	—	1.76	160
Chrome-glutaraldehyde-ASA	none	—	1.79	172
Chrome-glutaraldehyde (comm.)	silicone	—	1.34	2,300
Chrome-ASA	silicone	4.6	1.68	3,900
Chrome-ASA	Quilon	3.8	1.44	1,000
Chrome-ASA	Scotchgard	3.7	1.56	1,700
Chrome-glutaraldehyde-ASA	silicone	5.2	1.79	52,000
Chrome-glutaraldehyde-ASA	Quilon	4.4	1.44	6,100
Chrome-glutaraldehyde-ASA	Scotchgard	4.0	1.63	12,000

\*Number of flexes in Dow-Corning Leather Tester.

transmission. The other nonrepellent leathers showed permeabilities that were at least twice that of the chrome leather with a finish. As can be noted from the data, lubrication with ASA seemed to produce a leather that was somewhat more permeable to water vapor than the two commercial leathers containing a conventional fatliquor. It is apparent that the most important factor influencing water vapor transmission is the finish coat.

In the water vapor permeability data for the water-repellent leathers, also given in Table I, one leather was a glutaraldehyde-retanned side treated with silicone and produced commercially. The remainder were experimental leathers lubricated with ASA, with or without a glutaraldehyde retannage, and treated with silicone, Quilon, or Scotchgard. Again, lubrication with ASA seems to result in a leather with slightly more permeability to water vapor, and retannage with glutaraldehyde appears to have little influence on water vapor transmission. Interestingly enough, the treatment of the leathers with water-repellents did not

reduce water vapor permeability to any great extent. The treatments with silicone, Quilon, or Scotchgard did not differ greatly from one another in regard to water vapor permeability. These values are only about five to 15 percent lower than those shown for the nonrepellent leathers.

As was demonstrated in previous work, retannage with glutaraldehyde markedly improved the water repellency of the leathers after treatment with silicone, Quilon, or Scotchgard (8,9). This is evident in the high flex values of the last three leathers shown in Table I, which ranged from 6,000 to 52,000, compared to the 1,000 to 4,000 range for the straight chrome leathers in the dynamic flex tester.

The absorption of liquid water by nonrepellent leathers upon total static immersion in water is presented in Figure 1. Both the commercial chrome and the chrome-glutaraldehyde retanned leathers absorbed water rapidly in the first few

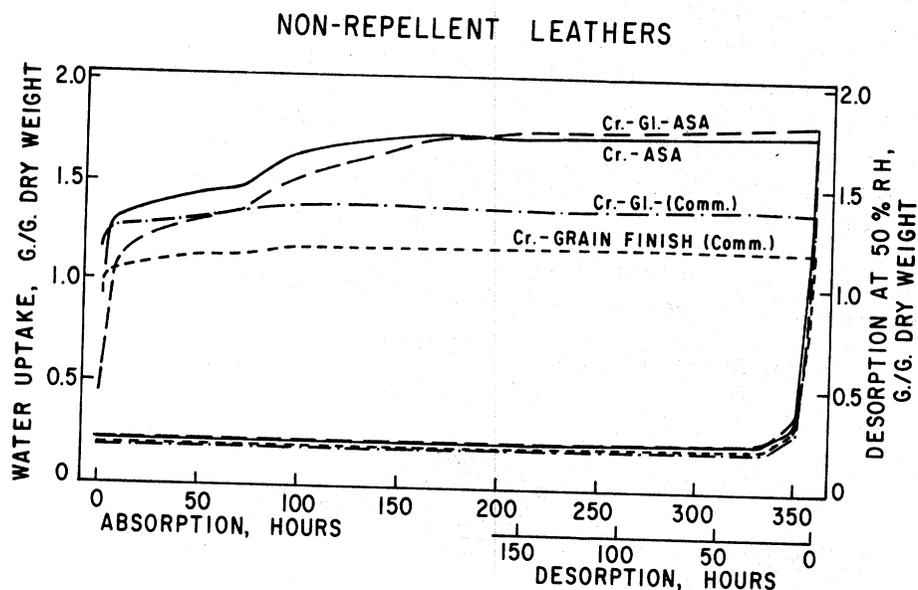


FIGURE 1.—Uptake of liquid water and loss of water by drying at 50 percent relative humidity at 73°F. by nonrepellent leathers. Cr. refers to chrome-tanned, Gl. to glutaraldehyde-retanned, and ASA to alkenyl succinic acid-lubricated leathers.

hours, and then leveled off after 72 hours' immersion. The experimental leathers, which were lubricated with ASA, absorbed water less rapidly than the commercial leathers up to 72 hours. However, water absorption gradually continued and leveled off at a water uptake higher than that of the commercial leathers. The ASA-lubricated leathers were still buoyant or showed an apparent specific gravity less than that of water at the 72-hour period, and had to be kept im-

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mersed by means of glass rods. After 72 hours, however, the ASA-lubricated leathers were no longer buoyant for the duration of the immersion period. This observation on the buoyancy of the ASA-lubricated leather was observed and reported by Von Fuchs (16), and may be a factor in the enhancement of water-repellent treatments by ASA lubrication. Equilibrium water absorption values were about 25 percent higher for the ASA-lubricated leathers, compared to the commercially fatliquored chrome leathers. On comparison of the various curves, ASA lubrication appeared to be a more important factor than glutaraldehyde retannage, as far as water uptake was concerned.

Loss of water on drying the wet nonrepellent leathers at 50 percent relative humidity and 73°F. is also shown in Figure 1. Equilibrium was attained within 24 hours, and very little difference was noted at this period for all four leathers. However, because of the higher uptake of water by the ASA-lubricated leather, it dried at a more rapid rate than conventionally fatliquored leather.

The curves for the uptake of liquid water for various water-repellent leathers, including one commercial silicone-treated leather, are shown in Figure 2. The commercial repellent leather showed much lower static water absorption than the experimental ASA-lubricated leather impregnated with either silicone or Quilon. Of course, the silicone content of the leathers, which was about five percent for the experimental leathers, but was not known for the commercial leather, would be an important factor in regard to this property. All the silicone-impregnated leathers showed the same type of curve, differing only in the amount

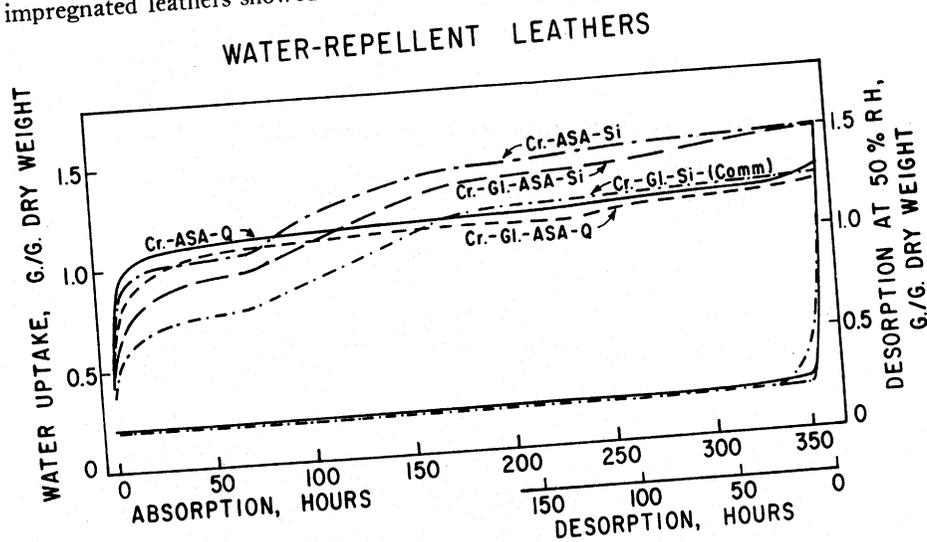


FIGURE 2.—Uptake of liquid water and loss of water by drying at 50 percent relative humidity at 73°F. by water-repellent leathers. Cr. refers to chrome-tanned, Gl. to glutaraldehyde-retanned, ASA to alkenyl succinic acid-lubricated leathers, Si to silicone-treated leather, and Q to Quilon-treated leather.

of water absorbed. The curves all rise uniformly to the 72-hour absorption period, and then rise sharply to equilibrium. This break in the curves correlated with the buoyancy of the leathers, which was lost after 72 hours. On the other hand, the Quilon-treated leathers remain buoyant throughout the immersion period, and this can be observed by the uniformity of the curves for the chrome and chrome-glutaraldehyde ASA-lubricated leathers. The chrome stock that had been made water-repellent with either silicone or Quilon gave higher water absorption values throughout the immersion time period than the corresponding glutaraldehyde-retanned leathers. The leathers treated with Scotchgard were not plotted, since they practically coincided with the curves for the Quilon-treated leathers, with only minor differences in liquid water absorption. As was expected, water-repellency appears to be the most important factor regarding water uptake.

Loss of water on drying of the wet leathers at 50 percent relative humidity and at a constant temperature of  $73^{\circ} \pm 1^{\circ}\text{F}$ . was rapid, as is shown by the desorption curves in Figure 2, and equilibrium was reached in 24 hours or less. The

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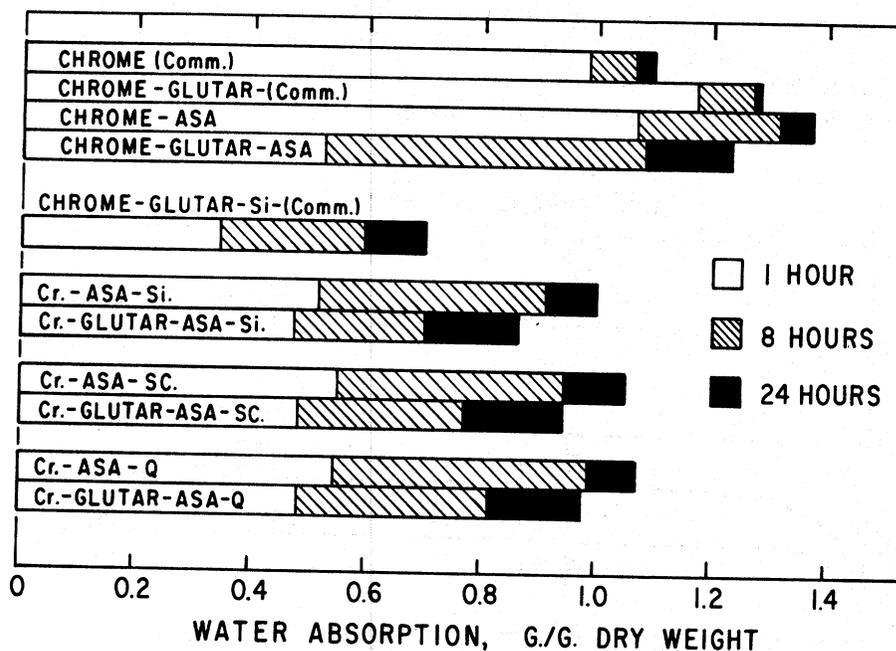


FIGURE 3.—Amount of water absorbed by repellent and nonrepellent leathers after one, eight and 24 hours of static immersion. Cr. refers to chrome-tanned, Glutar to glutaraldehyde-retanned, ASA to alkenyl succinic acid-lubricated leathers, Si to silicone-treated leather, Q to Quilon-treated leather, and SC. to Scotchgard-treated leather.

silicone-treated leathers dried somewhat more rapidly than the others. The water uptake of the leathers when immersed for short periods of time more nearly simulates actual use conditions, and in order to demonstrate this behavior uptakes at one, eight and 24 hours were plotted as bar graphs and are shown in Figure 3.

In the case of the commercial nonrepellent leathers, retannage with glutaraldehyde seemed to increase the water uptake slightly. In the case of the ASA-lubricated leathers, retannage with glutaraldehyde appeared to give a slightly lower uptake of water. This seemed true for all three periods of immersion. As was expected, treatment with water-repellent agents was by far the most important factor, reducing uptake of water in one hour to about half that of the untreated leather. Strangely enough, the chrome leather retanned with glutaraldehyde and lubricated with ASA showed almost as little pickup of water in one hour as these same leathers did after treatment with water repellents. However, after eight hours of immersion, uptake of water was comparable to that of the other non-repellent leathers.

The water vapor absorption curves for a commercial chrome leather at various relative humidities are shown in Figure 4. The curves are all of the same type, rising sharply up to 24 hours, then slowly attaining equilibrium. Absorption of water vapor increased with increasing relative humidity, as previous investigators have already demonstrated with many types of leathers. The amount of moisture absorbed increased with increasing relative humidity, but this was not a linear

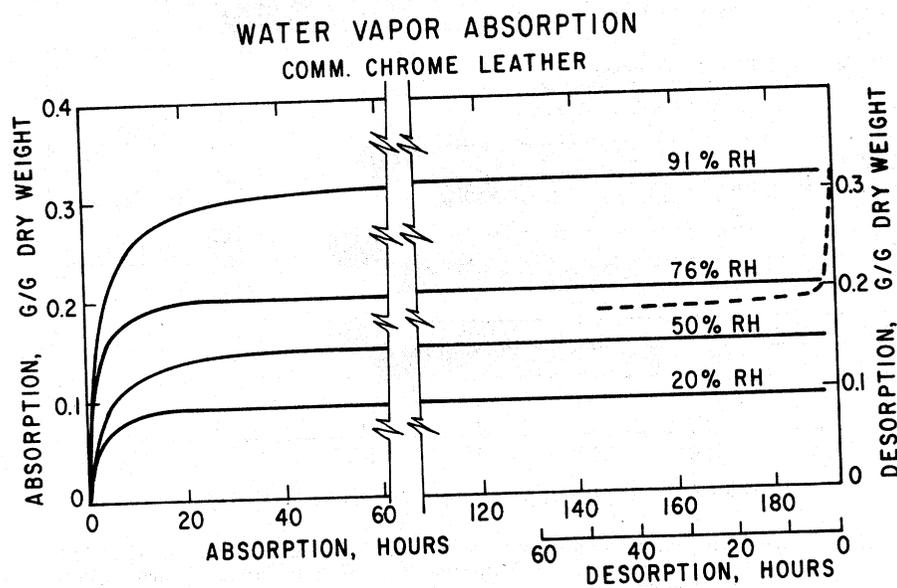


FIGURE 4.—Absorption of water vapor by commercial chrome leather at various relative humidities.

function. Desorption (Figure 4) was measured only for the leather equilibrated at 91 percent relative humidity and then placed in a conditioned room at 50 percent relative humidity at a constant temperature of  $73^{\circ} \pm 1^{\circ}\text{F}$ . Loss of moisture was rapid in the first eight hours, and almost reached equilibrium in this short time interval.

The water vapor absorption curves obtained for a commercial chrome glutaraldehyde-retanned leather at the various relative humidities were identical to, and superimposable on, those of the chrome leather shown in Figure 4. There appeared to be no differences in moisture vapor absorption effected by the glutaraldehyde retannage.

The water vapor absorption curves for a commercial chrome-tanned glutaraldehyde-retanned silicone-treated leather are shown in Figure 5. These curves are very similar to those shown in Figure 4. However, in this leather the water

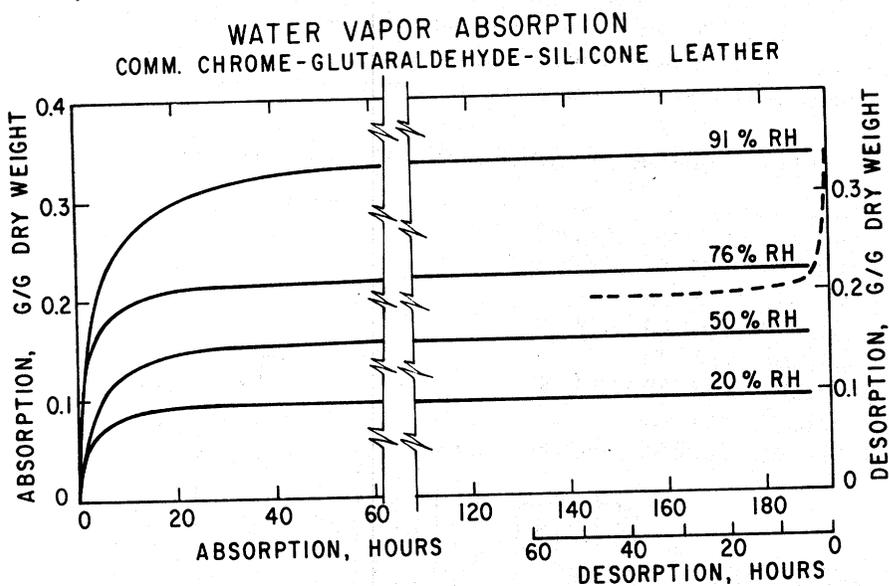


FIGURE 5.—Absorption of water vapor by a commercial chrome glutaraldehyde-retanned silicone-treated leather at various relative humidities.

vapor absorption is slightly higher than that of the nonrepellent leathers. In the 91 percent relative humidity atmosphere, this silicone-treated leather absorbed about ten percent more moisture than the nonrepellent leathers. The increase in moisture absorption was less at the lower humidities, and virtually nil at 20 percent relative humidity.

Desorption of water vapor from the silicone-treated leather (Figure 5) was comparable to that noted for the nonrepellent leathers, and leveled off at a mois-

ture content roughly ten percent higher than that in the case of the nonrepellent leathers.

Curves for the ASA-lubricated leathers impregnated with the other water-repellent materials were not plotted, since they fell along the same curves as those already shown in Figure 5. After 24 hours of exposure at 91 percent relative humidity, the water vapor absorption for these leathers ranged between 0.303 and 0.334 grams of moisture per gram of dry weight. Thus it can be concluded that retannage with glutaraldehyde and treatment with water-repellents did not hinder the water vapor absorption of the leathers.

The water vapor absorption of the various leathers upon short-term exposure (eight hours) at the various humidities is shown in the bar graph (Figure 6). The relationship between the various types of leathers can be seen more clearly from this presentation. The water vapor absorption increases with increasing relative humidity. At the higher humidities of 76 and 91 percent, there were only slight differences between the water vapor absorptions of the various water-

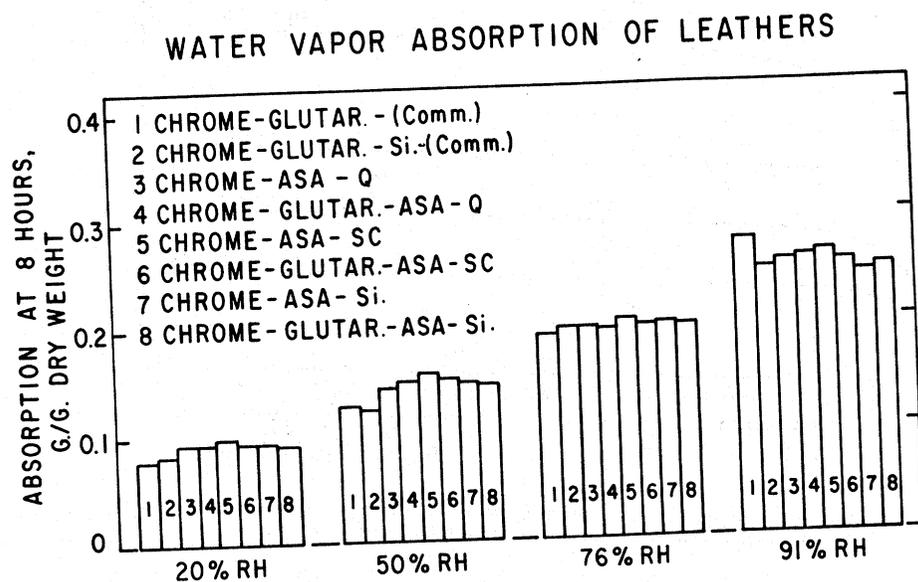


FIGURE 6.—Amount of water vapor absorbed after eight hours by water-repellent and nonrepellent leathers at various relative humidities. Glutar. refers to glutaraldehyde-retanned, ASA to alkenyl succinic acid-lubricated leathers, Si. to silicone-treated leather, Q to Quilon-treated leather, and SC to Scotchgard-treated leather.

repellent leathers. At lower humidities there was, in general, more variation among the repellent leathers, but this was not large. Generally, the highest water vapor absorption was noted in the case of the Scotchgard-treated leathers.

## SUMMARY

Data are presented on the absorption of water vapor of various leathers at relative humidities of 20, 50, 76, and 91 percent. As the relative humidity was increased, so did the absorption of water vapor by the leathers under study. There were only slight differences in water vapor absorption of the various leathers, indicating that retannage with glutaraldehyde and treatment with water-repellent agents (such as silicone, Scotchgard, and Quilon), or lubrication with alkenylsuccinic acid, were minor factors in this property of the leather. The water-repellent leathers generally showed slightly higher water vapor absorption values than the nonrepellent leathers. When immersed in water, the water repellent-treated leathers absorbed less water than nonrepellent leathers. In confirmation of previous results, glutaraldehyde retannage improved the efficiency of the water-repellent treatments. The ASA lubrication increased absorption of liquid water. These lubricated leathers, however, dried more rapidly than similar leathers treated with water-repellent materials. The data also showed that the application of water-repellent materials to ASA-lubricated leather did not drastically reduce the water vapor permeability of the ASA-lubricated leather.

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DISCUSSION

DR. LOLLAR: The discussion on this most interesting paper will be led by Jim Cassel of the National Bureau of Standards.

DR. CASSEL: I'm sorry that Dr. Kanagy, who was the listed discussion leader for this paper, couldn't be here to add some lucid comments to a very fine presentation by Mr. Luvisi, summarizing a terrific amount of work and data in a short time.

I'm sorry especially because Dr. Kanagy has proved to be an expert in this field and certainly could have added some comments that would have been of interest.

Last week, when I learned that I would be discussing this, I went back to the literature to find out some of the previous work leading up to that presented here today.

In a series of papers presented from the Eastern Regional Laboratory, the first one had to do with developing a procedure by which ASA could be incorporated as a lubricant into chrome leather with a drumming procedure.

I believe the second paper, which followed this, had to do with developing a chrome retannage, using the ASA to show that water-repellency treatments could be more effectively given in leather that had been so pretreated.

This paper today emphasizes that this can be done without loss of water vapor permeability. In discussing this paper with Dr. Kanagy, one of the things that he was concerned about and that, perhaps, should be commented on was this: In the first two slides there is a comparison of nonrepellent leather and repellent leather as to the water vapor permeability. The water vapor permeability of the leather prior to the water-repellency treatment appears to be fairly low already, so that the repellency treatment can't be expected to drop it down significantly. I don't know whether you'd care to comment on that or not, Mr. Luvisi.

MR. LUVISI: The water vapor permeability values as we expressed them are low, owing in part to the units of expression. When calculated in units as specified in the ASTM method, these values are of the order of 1.8 to 5.3. These values are about normal for leather. We were more interested in the relative water vapor permeability of the various leathers than in the absolute values, and were especially interested in the effect of the water-repellent. A significant change in this property would be apparent, as you will note, from the value of the leather with the acrylic finish, which is about one half that of the other leathers.

DR. CASSEL: A second question that also relates to this same area is this. I believe in previous work that you have done you found that you had to go to a much higher silicone treatment in order to obtain a water-repellency on unfinished leather that would guarantee water-repellency in the finished product. I think you went to something like ten percent, but here you went to five percent.

The question I'm raising is, would the situation have been any different had you gone to a much higher silicone content in your water-repellency treatment?

MR. LUVISI: In the paper to which you are referring, a three and a six percent dip were used, which corresponded to about a five to six percent pickup of silicone for the three percent dip and about a ten to 11 percent pickup for the six percent dip.

We found, of course, that the higher the amount of silicone is, the higher flex values would be. With the three percent dip, that is, the five percent pickup, the flex values were on the lower side when compared to the higher pickup. In other words, the higher the silicone uptake, the better the repellency. However, we do not believe this would significantly change the water vapor permeability. We attempted to keep this pickup of repellents at about five percent, because this amount gave adequate water repellency.

We were particularly interested in the water-repellency of these leathers, and when we tested them we found that we were getting the same order of repellency. The Quilon was a little lower than the Scotchgard, and the Scotchgard was a little lower than the silicone.

DR. DONOVAN (Canada Packers): So you are saying you wouldn't necessarily have to shoot so high.

MR. LUVISI: Yes, that is right. A higher percent would give you higher flex values, but what is the highest flex value needed for water-repellent leather? Perhaps 50,000 or even 10,000 flexes would be sufficient.

DR. CASSEL: I might ask just one question for my own information because I don't understand it. Why does the glutaraldehyde-retannage lead to an increased pickup of water-repellent substances?

Of course there has been quite a bit said on this already this morning. There may be a simple answer, such as additional chemical action.

MR. LUVISI: Well, I won't try to state the chemical action. I believe it's just that you get a different temper and a softer and more open leather, and more even distribution of the water-repellent materials throughout the leather that has been glutaraldehyde retanned. We find with the glutaraldehyde retannage we get better water-repellency as compared to the straight chrome tannage.

MR. WEDERBRAND (Rohm and Haas Co.): You mentioned that you use glutaraldehyde retannages, and that you get a more open fiber which appears to take up the water-repellent. Have you done any work at all with various synthetic tanning materials?

MR. LUVISI: No, we haven't. We just worked with glutaraldehyde retannage.

MR. RAY POTTS (Wolverine World Wide): Mr. Luvisi, have you done any

work with any other type of fatliquor than ASA? I mean the new type of synthetic fatliquors.

MR. LUVISI: We just used the ASA fatliquor, since the previous papers were on ASA lubrication and we tried to avoid conventional fatliquors. As Mr. Heit brought out this morning, conventional fatliquors would have some effect on water-repellency.

DR. CASSEL: I think, again, this ties in with the fact that this paper follows previous work, so it was a logical step to take this approach. Any other comments? If not, we would like to thank Mr. Luvisi and his cohorts for a very fine presentation of a very fine paper.

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