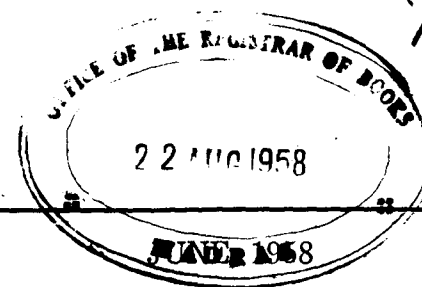


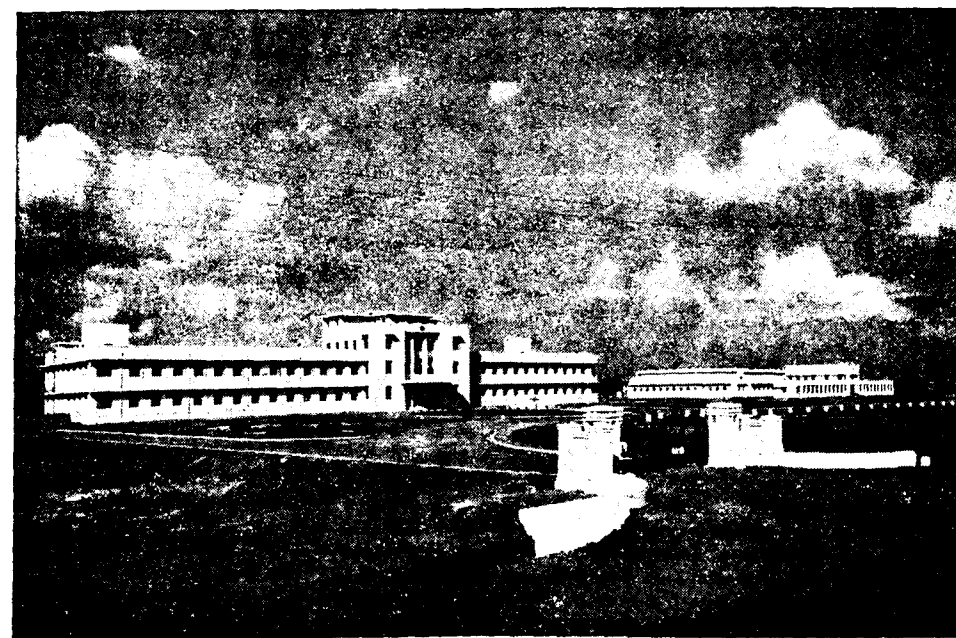
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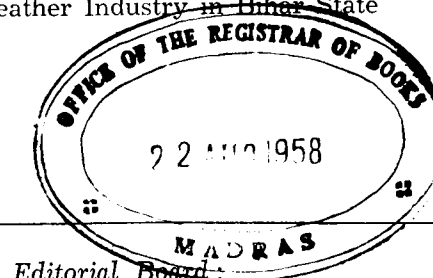
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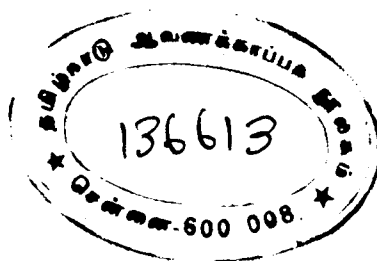
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**PHYSICO-CHEMICAL INVESTIGATIONS OF
TANNIN FRACTIONS OBTAINED FROM
FRESH BABUL BARK**

BY

Y. NAYUDAMMA, (Miss) A. NAGASIROMANI, (Miss)
P. V. RAJALAKSHMI, K. N. SHAMA SASTRY and
D. RAMASWAMY

Central Leather Research Institute, Madras.

SUMMARY

Babul bark has been fractionated using several solvents and the fractions so obtained have been acetylated and methylated. The fractions and their derivatives have been analysed using chromatographic, electrophoretic, spectrophotometric, polarographic, and nonaqueous and photometric titration techniques. On the basis of the results babul appears to be mixture of hydrolysable and condensed tannins; differentiation of the various acidic groups could be effected to some extent.

Babul (*Acacia arabica*) an indigenous tanning material is widely used for sole leather tannage. A systematic investigation of this important and valuable tanstuff does not appear to have been made hitherto. Hence a concerted and detailed study of this material has been undertaken. Using conventional solvent fractionation techniques, several fractions have been obtained from babul bark and the physico chemical characteristics of these and their derivatives have been studied.

Preparation of fractions. Freshly cut babul bark was shaken for half an hour with chloroform to remove chlorophyll. The chlorophyll-free material was next treated with ethyl acetate, two fractions being obtained—one after half an hour and the other after 72 hours. In each case the solvent in the fraction was removed on a steam bath and

the dry residue obtained by evaporation to dryness in vacuum. The dry extract thus obtained was taken up with methyl alcohol, the precipitated gums and mucilageous matters were removed and a dry residue obtained from the solution exactly as above. This solid extract was next taken up with ethyl alcohol to precipitate carbohydrates, and the carbohydrate free ethyl alcohol fraction was converted into the solid extract as above. This extract was Soxhlet extracted with petroleum ether, to remove fats and solvents. The residue was dried. Fraction I was that derived from 72 hours ethyl acetate extraction and Fraction IA from ½ hour extraction with the same solvent. Fraction I was extracted with ethyl ether for 12 hours. Fraction II was that obtained by removing the solvent from the ether soluble portion; the ether insoluble residue was Fraction V. Fraction II was dissolved in a little acetone and then treated with benzene. The precipitate obtained was Fraction III; the acetone benzene soluble part gave after removal of the solvents under reduced pressure Fraction IV.

Fraction IA should contain matter readily extractable with EtOAc. Fraction I contains mostly tannins. Fraction II contains more acidic portions of Fraction I and the less acidic portions are contained in Fraction V. Fraction III contains higher molecular size polyphenols and Fraction IV, smaller size molecules.

Methylated and Acetylated Compounds

Acetylation: In general, the following procedure was adopted for acetylating the fraction: 1 g. of the fraction was dissolved in 10 ml. of pyridine, to which was added 15 ml. of acetic anhydride and kept over, after shaking well, for 48 hours. Next the solution was poured into large excess of water containing a few drops of concentrated hydrochloric acid. The precipitated material was filtered and purified with ethanol or dilute ethanol. Difficulties were experienced in acetylating the fractions completely.

Methylation: Two procedures have been resorted to for methylation—one using dimethyl sulphate, and the other using diazomethane in ether solution, diazomethane being prepared starting from acetamide. However, it was noticed that better results were indicated with the use of diazomethane, for mostly reddish coloured derivatives were obtained with dimethyl sulphate. With dimethyl sulphate, the procedure followed was similar to the one employed for wattle.¹ 2 g. of material under consideration were dissolved in 10 ml. methanol to which were added 4 ml. of dimethyl sulphate and 2 g of KOH in 10 ml. of 50% methanol, the reagents being added alternately in small portions. The mixture was cooled in ice bath during the reaction and left in ice chest overnight. The precipitate was filtered off, washed with water, and purified through ethanol. For methylation with diazomethane the substance was taken up in acetone and treated with an excess of ethereal solution of diazomethane and kept for 48 hrs. The excess of solvents and diazomethane was removed and the substance purified.

The melting and decomposing points obtained with the methylated and acetylated derivatives are given in Table 1.

TABLE 1

Fraction	Acetylated product	Methylated product
I.	Decomposes at 118°	Decomposes at 167°
IA.	Decomposes at 125°	Melts at 117° (Using CH ₂ N ₂)
II.	Melts at 125° (Softening starts at 195°; decomposes at 215-218°)	Melts at 112-117°
III.	Decomposes at 220°	Partly m.p. 105° and " 135° (Using CH ₂ N ₂)
IV.	M.P. at 152°	
V.	Decomposes at 182-184°	Decomposes at 230°

The following techniques were employed for studying the characteristics of the fractions:

Chromatographic Study: Two dimensional chromatograms were prepared with the above fractions, using (i) 10% acetic acid for the first dimension and (ii) a mixture of n-butanol, acetic acid and water, (40:10:50) for the second dimension. When developed with 3% ferric chloride and 0.3% potassium ferricyanide, it was found most of the fractions left a trail, with one or two definite areas. Fraction I gave a trail and some indication of 2-3 fairly defined areas as was the case with Fraction IA. The ether soluble portion, i.e., Fraction II gave very clearly defined chromatograms, wherein it was indicated that they essentially consisted of four main constituents. The benzene precipitated Fraction III showed on chromatogram two well defined areas, thereby indicating partial fractionation of Fraction II. The R_f values for the ether soluble fractions are: 0.75, 0.68, 0.6 and 0.52 respectively.

Electrophoretic Study: The electrophoretic study of the fractions in acetate buffer of pH 4.75 does not provide any clear information except that it gives two spots moving towards the anode in all the fractions thus indicating the anionic nature of the compounds. This may indicate that the activated phenols are ionizable readily, or that the molecule may contain carboxyl groups, or that there may be some free acids carried over in the ethyl acetate fraction.

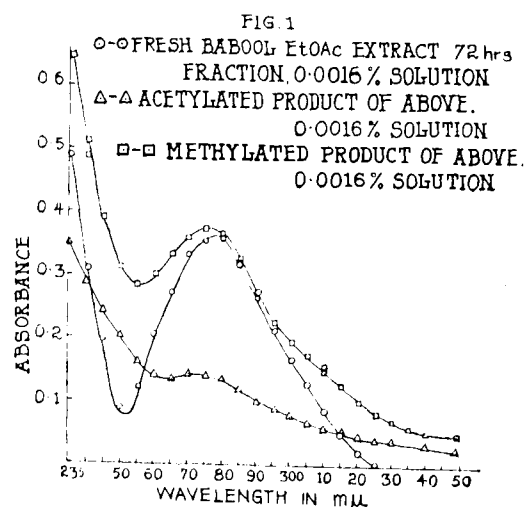
Spectrophotometric Study: (UV. spectra) The absorption behaviour of all the fractions and their derivatives in the UV range was examined with Beckman DU Spectrophotometer using 1 cm. quartz cells and 0.001-0.005% solution in water or dilute methyl alcohol. The spectra with addition of buffer (boric acid-NaOH pH 10.0) and acid added to buffer (pH 1-2) also were taken to study the shifts in maxima and those in 5% aluminium chloride solution to indicate the orthodihydroxy nature of phenolic groups.

TABLE 2
Shifts in peaks with Aluminium chloride and buffer added solutions for different solvent purified fractions
(and their derivatives) of fresh babul

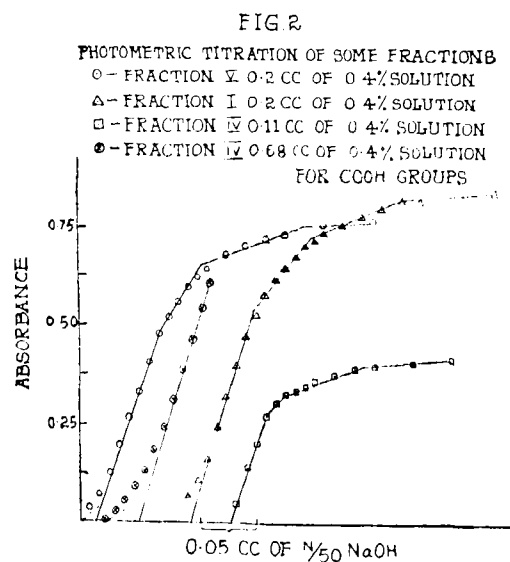
Fraction	AlCl ₃ added solution		Buffer added solution	
	Shift in I peak mμ	Shift in II peak mμ	Shift in I peak mμ	Shift in II peak mμ
I. Fresh Babul EtOAc extract 72 hours	7	34	12	32
Acetyl derivative	Nil	38(a)	11	Nil
Methyl derivative	Nil	Nil	4	Very small inflexion
IA. Fresh Babul EtOAc immediate	8	34	10	28
Acetyl derivative	Nil	38(a)	14	37
Methyl derivative	Nil	Nil	Nil	Nil
II. EtOAc extract ether soluble	7	34	17(b)	39(b)
Acetyl derivative	Not done		310(c)	(315) (c)
III. Ether soluble—benzene precipitated	7	36	inflexion	
Acetyl derivative	7	38(a)	Nil	39(b)
Methyl derivative	Nil	Nil	14	35
IV. Ether soluble—benzene soluble	6	26	Nil	Nil
IV. Ether insoluble—alcohol extracted	8		14(b)	42(b)
Methyl derivative	Nil	32	10	32
Acetyl derivative	—	Nil	—	32(b)
		—	—	(320) (c)

(a) Small but measurable inflexions. maxima taking acid added as blank. 1 not be obtained since the derivatives as such did not give a maximum or m.

Typical spectral curves are given in Fig. 1 and the shifts obtained by addition of different agents in Table 2.



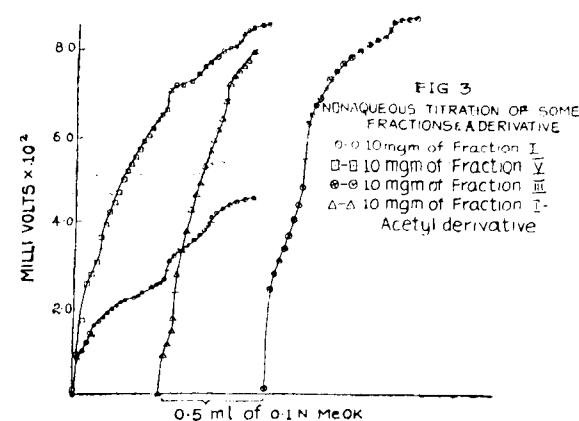
Photometric titration technique: The differentiation in the acidities due to COOH groups and the OH groups activated to various extents is attempted by this technique. The



photometric titration was carried out in a quartz beaker supported in the test tube adaptor assembly in nitrogen atmosphere. The results were plotted according to the method

of Goddu and Hume.² Stirring is provided by a magnetic stirrer and the standard alkali of strength $N/50$ was added from a micro-burette. Figure 2 gives the typical photometric titration curves for some of the fractions. If the concentration is too low (0.0005%-0.001%) for getting correct acidity due to COOH groups, the first part of the curve with higher concentration (0.004 to 0.008%) alone is taken as shown in the figure to give accurate values. The activated and the total ionisable phenolic groups are obtained by change in slope and the considerable flattening respectively of the curve.³

Nonaqueous titration technique: A successful non-aqueous titration technique has been developed⁴ for the differential titration of phenolic groups and for the differentiation of acidities due to COOH group and to the activated and total phenolic groups. Titration was carried out in dimethyl formamide solution with anodically polarized platinum and calomel electrodes in an atmosphere of nitrogen using 0.1N potassium methoxide as titrant. The titration curves for some of the typical fractions and for one derivative are given in Fig. 3.



Polarographic technique: It has been observed⁵ that tanning materials and polyphenolic compounds like catechol, etc., yield on auto-oxidation in alkaline buffer (ammonia—

NH₄Cl of pH 9.1) hydrogen peroxide which gives a cathodic wave at a half wave potential ($E_{1/2}$) of -0.9 to -0.95 volts depending on the anions present. All the fractions gave similar waves with $E_{1/2}$ values ranging from -0.9 to $-0.95V$. The derivatives also were examined polarographically.

Discussion

The absorption spectra curves indicate that for all the fractions there is a characteristic maximum at $278 m\mu$ with minimum occurring at $252 m\mu$. However, with the addition of aluminium chloride or buffer, two peaks appear with shifts of 6 to $8 m\mu$ and 26 to $36 m\mu$ for the former and of 10 to $14 m\mu$ and 28 to $39 m\mu$ for the latter. It is known⁶ that shifts to longer wavelengths are characteristic of pyrogallol fractions and to shorter wavelengths are due to catechol type tanstuffs. Hence, fresh babul appears to be a mixture of both catechol and pyrogallol tannins. Chemical tests also confirm the mixed nature of the material. All the fractions obtained by using the solvents mentioned, exhibit this striking feature in showing up two peaks but differ only in their intensities showing thereby that only partial purification has been achieved. The differential absorbance values for the buffer added vs. buffer acid added were negative in the range $260-80 m\mu$ which is characteristic of pyrogallol type tanstuffs.⁶

The methyl derivatives of the fractions show only one peak at $275 m\mu$ in all cases probably due to OMe group thus confirming the observation⁷ that diazomethane methylates all the ionisable phenolic groups.

The acetyl derivatives indicate a maximum at a wavelength slightly lower than that of phenolic fractions ($272-4 m\mu$). However, when buffer is added to the solutions the two peaks appear with more or less the same shifts (12 to $14 m\mu$ and $32-37 m\mu$) suggesting partial acetylation of the phenolic groups of both catechol and pyrogallol type. With

aluminium chloride the second peak alone got replaced by a well-defined inflexion occurring at $38 m\mu$ difference while the first peak was hardly shifted (slightly shifted by $4 m\mu$ in one case). The absence of shift in the 1st case may probably be attributed to the difficulty in the formation of $AlCl_3$ complex if the acetylation is carried to a high degree. The absorption curve of the acetyl derivative of the ether soluble fraction alone (Fraction II) is peculiar in not giving any maximum.

The data obtained by photometric titration technique and non-aqueous titration method for acidities due to carboxyl groups, activated phenolic groups and total acidity due to carboxyl and phenolic groups and the percentages of each of these to the total are given in Table 3.

The acidity due to carboxyl groups compares fairly well by both the methods keeping in mind the dilution factors involved and the small amounts taken for the determinations. They are of the order of 5-10%. It is not possible to attribute the carboxyl acidity to either the COOH groups of the tanning molecule or contamination with weak organic acids (though it has been attempted to purify the extracts by the respective solvents).

It is seen from the table that Fraction I gives 52% activated phenols by non-aqueous titration technique and only 37% by photometric titration technique. The difference in acidities due to the total and activated phenolic groups by the two techniques is somewhat marked because of lack of sharpness of the end points in the nonaqueous method for total acids and the difficulty of using water which acts as an acid at higher pH values for titration in photometric titration. However, the general trend indicates that by ether extraction more of the activated phenolic groups are removed from the system which is apparent by lower percentage of activated phenolic groups in the ether insoluble fraction (57% and 46%) as compared to the original fraction. Since

TABLE 3
Acidities of different fractions of Babul in m.eq. per 100 g. of the Substance

Fraction	Nonaqueous titration				Photometric titration			
	Total acids	COOH acidity	%	Activated phenol	Total acids	COOH acidity	%	Activated phenol
I.	600	60	10	310	575	50	9	213
IA.	320	Nil	Nil	80	263	Nil	Nil	100
II.	370	30	8	210	558	70	13	255
III.	340	10	3	165	588	Nil	Nil	238
IV.	284	Nil	Nil	90	538	30	6	140
V.	770	60	8	270	563	69	12	156

generally more activated phenolic groups are contained in pyrogallol tanstuffs,⁸ probably the pyrogallol type of fraction is being preferentially removed by ether extraction since babul is of mixed type. But whether ether can extract any tannin fraction at all is a moot point. Since the yield obtained with ether is, about 9-10%, it is difficult to attribute the entire amount to nontan components. The techniques, however, will not be distinguishing the total and activated phenolic part of the tannins from the nontannins. It is only possible to conclude with the data available that ether soluble fraction may contain more of the strongly acidic phenolic groups. Further fractionation of the ether soluble fraction may yield more fruitful results.

The data further show that fraction IA differs from fraction I in that it has no carboxylic acids. This is shown to be the case by both the techniques. Hence it is clear that by varying the duration of extraction with ethyl acetate different components could be obtained.

Fraction III contains about 40-46% of activated phenols but very little of carboxylic acids whereas fraction IV contains 6% of carboxylic acids (by photometric technique) and less of activated phenols, ranging from 26-32. This might be expected to be the case as the larger polyphenolic materials which are precipitated by benzene go into Fraction III and the small molecules and acids may go into Fraction IV.

Derivatives: The methyl and acetyl derivatives prepared from the various fractions have also been examined by all the techniques. The methyl derivatives did not give any photometric or nonaqueous titration curve nor a polarographic wave showing thereby that all the ionizable phenolic groups have been methylated by diazomethane and confirming the data obtained with the absorption spectra of the derivatives. However, data obtained on the acetyl derivatives indicated incomplete acetylation. The phenolic groups remaining unattacked could be titrated in nonaque-

ous and photometric method and showed less diffusion currents in the polarographic method. It is possible to compute the percentage acetylation by the different techniques if the values are compared with the original data for unacetylated fractions. The results obtained for the percentage of phenolic group unattacked, by the different techniques are indicated in Table 4.

TABLE 4

Percentage free phenolic groups in the acetyl derivatives by different techniques

Fraction	% Free phenolic groups			
	Polarogram	Absorbance (Buffer added)	Nonaqueous titrn.	Photometric titrn.
I.	63	42	40	53
IA.	56	60	—	48
III.	56	64	—	50
V.	25	19	30	38

It is seen that the values more or less agree excepting those by spectrophotometric buffer addition method. However, the main drawback in these methods is that the m.eqs. of the remaining OH groups are calculated with respect to 100 gm. of the derivative which has increased its molecular weight by substitution whereas % is worked out with 100 gm. of original substance. The correction cannot be applied because the number of phenolic groups present are not known. Further standardization of the techniques with acetyl derivatives of homogeneous fractions, by comparing with acetyl values obtained by chemical method are contemplated and will be published in a future communication.

In conclusion, it can be said that the various physico-chemical techniques play an important role in the characterisation and the study of the nature of the various fractions of tanning materials.

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RESEARCH BEARS FRUIT

Practical Demonstration No. 3

(Second Series)

Manufacture of Glazed Kid Leather by combined one and double bath tanning method with indigenous material*

Process No. 1.

BY

J. C. DEB and G. N. HORE

Raw Materials: Wet salted (Erode) Prime quality goat skins of average size 30" to 32" are taken.

Soaking: The skins are soaked for 2 hours in plain water, taken out, trimmed, broken on the flesh side over the beam by a blunt knife and washed thoroughly in running water for one hour to remove the salt. The skins are then taken out, drained and weighed for liming.

Liming: First liming is for three days, in a drum with the following:

Sodium sulphide liquor (4.1° B $\acute{e}$): 100% on the soaked wt.

Slaked lime: $2\frac{1}{2}\%$ on the soaked wt.

The drum is run for five minutes of each working hour of the day, and left stationary overnight. On the fourth day the skins are taken out, washed and unhaired. They are then washed with $\frac{1}{2}$ per cent sulphuric acid on the soaked weight of the skins in a paddle with sufficient float

of water, just to remove the excess sulphide, the drum being run for 10-15 minutes only.

The stock is then transferred to a paddle containing 10% lime and 300% water on the soaked weight, and left there for two days, the paddle being run for 10-15 minutes of each working hour of the day. On the third day, they are scudded, fleshed and then weighed for deliming and bating.

Deliming: The skins are then delimed in a paddle with the following:

Ammonium chloride: $\frac{1}{2}\%$ on the fleshed wt.

Water: 200% „ 331

They are run in the deliming bath for 30 minutes and then washed in plain water.

Bating: Overnight bating method is followed.

The washed skins are bated in a covered paddle using 1 to 1.5 per cent (on the fleshed weight of the skin) CLRI Bate No. 1, the bath being made up with half the quantity of bate and one paddle of water (300% water at normal temperature). The skins are put in and the paddle run for one hour. After running the paddle for one hour, the goods are left in the bath overnight.

Next morning the remaining half of the bate is added and the temperature of the bath is raised to 100.4° F by passing steam. The skins are paddled in the bath keeping the temperature of the bath at 98° - 100° F until satisfactorily bated. The pH of the bate liquor before unloading should be 7.8 to 8.0, if not it should be adjusted to that pH.

The exact stage when the bating can be regarded to have been satisfactorily completed is generally judged by the following indications:—

(1) The skins should be thoroughly delimed and phenolphthalein applied to the edge of a cut piece of bated pelt should not produce a red colouration.

* Process demonstrated during the month of June 1958.

(2) The bated skins should be absolutely fallen and flaccid and should lie in any position in which they are kept. They should not show any tendency to spring back. The absence of this tendency is generally tested by pressing the grain of the skin with the thumb; the thumb impression should remain.

(3) When bated skins are scratched on the grain by the thumb nail, the scud consisting of broken hair roots, other epidermal remnants, dirt, etc., should easily come out showing that the scud has been thoroughly loosened and rendered easily removable.

(4) Air is enclosed by folding the bated skin in the form of a pouch and the pouch is squeezed by the hand; then all the enclosed air should come out producing air bubbles on the surface of the pouch. This shows that the pores of the skin have been thoroughly opened out.

(5) When the flesh side is scratched with the thumb nail the adhering flesh should be removable when some pressure is applied, but the flesh should not be too easily removable as in the case of overbating.

After completion of bating the skins are taken out, scudded well, washed in cold water of 86° to 90°F, weighed and then immediately pickled.

Pickling: The pelts are pickled overnight in a drum using the following composition:

Common Salt: 8-10% on the fleshed weight.

Hydrochloric acid: 2-2½% fleshed weight.

Water: 75-100% on the fleshed weight.

The bath is made with the above chemicals in a drum. The skins are put in the drum which is run for one hour and they are then kept in the bath overnight. Next morning the skins are drummed for ½ an hour, taken out and horsed up for sometime and then taken up for tanning.

Tanning: Tanning is done by the combined single and double bath process as follows:

First Bath:

Chrome alum: 8-10% on the fleshed weight.

Bichromate of soda: 1½-2% „

Water: 150-200% „

The pickled pelts are then put in a tanning drum together with the requisite quantity of solid chrome alum. Then 0.5% of bichromate previously dissolved in a wooden vat with 100% water is added and the skins are set in motion immediately. The remaining 1.5% bichromate dissolved in the remaining 50% water is slowly added to it in six equal instalments within ½ hour. The skins are run in the drum for 2½ hours and kept overnight. Next morning, the skins are run for 30 minutes. The bath is drained and the skins are put in the reducing bath made as follows:

Reduction Bath:

Anhydrous sodium bisulphite: 8-10% on the fleshed wt.

Water: 250-300% „

The bath is made in a drum with ⅛ of the above bisulphite. The skins are quickly put into it and the drum is set in motion. The remaining bisulphite is added in seven equal instalments at intervals of ½ hour. The drum is then run for four hours more and kept overnight. Next morning the pH of the bath is adjusted to 3.5 to 4 before unloading by addition of soda ash. The tanned skins are then washed for 10-15 minutes by diluting the reduction bath and piled up on the platform for 2-3 days to drain off the excess of moisture.

Sammying and shaving: The leathers are then slicked or struck out on the flesh side, sammed, shaved and weighed.

Neutralisation: After, thorough washing in a single change of water the tanned stock is neutralised for half an hour with sodium bicarbonate solution of the following composition.

Sodium bicarbonate: $\frac{1}{2}\%$ on the shaved weight.
 Water: 200% „

The bath is made in a drum at room temperature. The leathers are then put in, drummed continuously for half an hour and tested for proper neutralisation with bromocresol-purple. A purple colour on the leather surface and a yellow colour in the middle layer indicate proper neutralisation. The leathers are then taken out and washed twice with plain water. They are then ready for dyeing and fat liquoring.

Dyeing and Fatliquoring. For blacks the leathers are dyed with the following compositions:—

Chlorozal black: 1% on the shaved weight.
 Nigrosine: $\frac{1}{2}\%$ „
 Methic leather black D: $\frac{1}{4}\%$ „
 Formic acid: $\frac{1}{2}\%$ „
 Water at 130 F: 200% „

Before being put in the dye bath the leathers are first drummed in water at 104°F for 10 minutes to attain that temperature. The conditioned skins are then put in, grain side turned out, into the drum containing 200% water at 130°F. Chlorozal black separately dissolved in water is added into the drum. The drum is run for 15 minutes. Nigrosine, dissolved separately is added and the drum run for 15 minutes more. The methic leather black D is also dissolved separately and added to the drum which is run for further 15 minutes. Lastly formic acid is added and the drum run for 10 minutes more.

The leathers are then fatliquored in the same bath with the following composition for 30-45 minutes at 130°F.

Foots free fish oil (Sardine oil): 1% on the shaved weight
 Castor oil: 1% „
 Sulphated fish oil: $\frac{1}{2}\%$ „
 Bar soap solution (10%): $\frac{1}{4}\%$ „
 or 20% Casein solution: 3-4% „

The pH of the emulsion of the fat liquor is adjusted to 7.8-8.0. Then 2% cutch extract dissolved separately is added to the exhaust fatliquor bath and run for 10 minutes. The leathers are then taken out and horsed up overnight.

Striking out and oiling up. Next day the leathers are struck out on the flesh and grain sides. A mixture of 2 parts foots free fish oil and one part kid finishing oil is applied on the grain side and the leathers hung up in the shed for sammying.

Sammying, setting and drying: After oiling up, the leathers sammed, set out and dried in the shed and then crusted for some days for conditioning.

Finishing: (a) Sawdusting, staking and buffing: The crusted leathers are then saw-dusted overnight in moist saw dust to condition them for staking. The conditioned leathers are staked first slightly by a slocomb staking machine. Then they are piled up overnight on the floor in several packs each pack containing one dozen leathers. Next morning the leathers are staked again by the same machine with considerable pressure. They are then aired off a little and buffed on the flesh side using 120 grit emery paper.

(b) Trimming and ironing: Next morning the buffed leathers are trimmed and ironed on the grain side with hand

electric iron or an ironing machine. The leathers are now ready for seasoning.

(c) Clearing, seasoning and glazing: The leathers are first cleared with a mixture of 10% acetic acid solution and $\frac{1}{4}$ of basic black per gallon of water, on the grain side, dried and then given two successive bottom season coats on the grain with the following bottom season.

Bottom Season:

Nigrosine: 1 oz.
Lissamine green: 7 gms.
CLRI black pigment: 6 ozs.
CLRI binder (Mg.): 4 ozs.
CLRI Glazing finish: 8 ozs.
Ox blood: 20 ozs.
Milk: 16 ozs.
T.R.O.: 1 oz.
Alcohol: 2 ozs.
Ammonia: 1 oz.
Water to make one gallon.

After application of two successive pad coats with the above, the leathers are dried, glazed and then given a top season coat.

Top Season.

Bottom season: $2\frac{1}{2}$ lbs.
CLRI binder (M.g.): 4 ozs.
CLRI glazing finish: 8 ozs.
15% Casein solution: 8 ozs.
Formaldehyde: 6 ozs.
Water to make one gallon.

One coat of the top season is applied to the grain side with pad, dried and then the following fixing coat is sprayed.

10% Casein solution: 1 part.
Formaldehyde (40% strength): 1 part.
Water: 6 parts.

The leathers are then dried and put to final glazing.

Final Glazing: Inclined bed glazing machine is the best. First glazing is done under heavy pressure moving the leather all round. The glazing produces glass marks on the grain which are smoothed out by glazing under light pressure, the second time. The glazing is skillfully done without producing pleats.

Measuring, ironing, smooth plating, grading and packing: The glazed leathers are then measured, ironed with hand electric iron or by a machine and then smooth plated by hydraulic press at 150°F with light pressure. The leathers are then sorted, graded according to substance into 3 or 4 classes. They are then packed with 24 pieces in a bundle.

PREPARATION OF SYNTHETIC TANNING MATERIALS*

BY

N. VISWANATHAN and (late) B. M. DAS

Syntans produced till now have been prepared by one or the other of the following methods:

- (1) Sulphonating a phenolic body and condensing it with a suitable condensing agent like formaldehyde.
- (2) Condensing a phenolic body with an aldehyde such as formaldehyde and subsequently sulphonating the same with sulphuric acid to impart water solubility. Syntans of this type are limited in number due to certain drawbacks.

One example of a commercial syntan belonging to this class is 'Tanigan Extra B' which is produced by condensing phenol with formaldehyde in acid medium, sulphonating the condensate and recondensing the same with formaldehyde.

Syntans prepared by method (1) are not very satisfactory as replacement tanstuffs because of the fact that they are highly acidic in character due to the presence of too many sulphonic groups in them. The sulphonic groups combine with the free amino groups in the collagen of the pelt and when the amino groups are neutralised the sulphonic groups remain free in the tanned leather. As the sulphonic groups are strong acids, they act upon the leather fibres and deteriorate the quality of the leather. Hence in

*These processes originally covered by Indian Patent Nos. 52798 and 52799 are now released to the Industry, free of cost.

using these syntans it must be taken to ensure that just enough of the syntan is used so that an excess of sulphonic groups may not be present in the leather. Hence these syntans cannot be used in sufficient quantity to produce well tanned leather. They cannot, therefore, be used in place of vegetable tanning materials. They have also poor filling properties and cannot produce a full empty leather. They are, however, found to possess certain distinct advantages such as quick penetration, ability to solubilise the insolubles in vegetable tan liquors and lighten the colour of both vegetable and chrome tanned leathers. Consequently, such products have been widely marketed as pretanning, combination and bleaching agents. They are used in combination with vegetable tanstuffs.

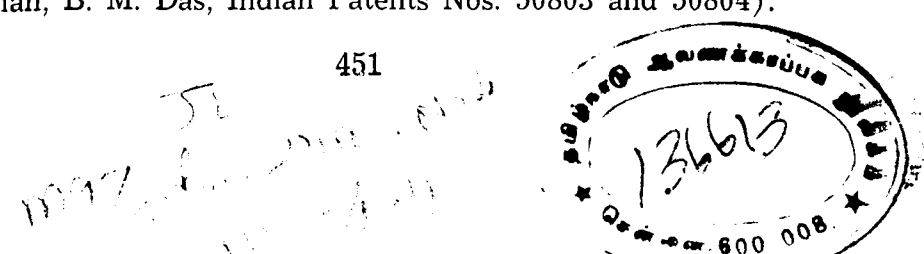
Syntans produced by the method mentioned at (2) above have been more suitable for replacing vegetable tanning materials but have been found to be lacking in penetration, colour and ability to produce smooth and light grained leathers. Such tanstuffs, when dissolved in water have been found to give very astringent liquors. Products of this type have been marketed for the purpose of substituting vegetable tanning materials and are found to give full leathers of usually brown or reddish brown colour.

A survey of available literature does not reveal any method to make the two types of syntans complementary to each other in a single composition with the advantages of both and draw-backs of neither.

The poor filling properties of the condensed sulphonic acids is due to the small molecular size of the product which is again too big in the case of the sulphonated condensates, making them poor tanstuffs.

Investigations regarding the lightening of the colour of the products of the sulphonated condensate type have already resulted in some successful products. (N. Viswanathan, B. M. Das, Indian Patents Nos. 50803 and 50804).

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While the methods described in the above two processes improve very considerably the colour of such products, they need modification as regards penetration and also ability to produce smoother grained leathers.

The two processes described presently, are concerned with the use of (1) condensed sulphonic acids of non-phenolic aromatic compound and (2) condensed sulphonic acids of phenolic compounds respectively for the sulphonation, solubilisation and /or dispersion of the insoluble condensates of phenols with aldehydes.

The processes have in common condensation of phenol with an aldehyde. Then follow sulphonating, solubilising and/or dispersing the water insoluble condensate (novolak resin) with a condensed sulphonic acid of a non-phenolic aromatic compound in the first one and the same with a condensed sulphonic acid of a phenolic compound in the second one.

Raw materials: Phenol, formaldehyde, concentrated sulphuric acid or oleum, sodium sulphite and acetic anhydride for both processes, naphthalene for Process-1 and β -naphthol for Process-2.

The initial step for both the processes is the same and is given below:—

One mole of phenol is condensed with 0.6 to 0.8 moles of formaldehyde in presence of 0.25 to 0.5 cc. of concentrated sulphuric acid dissolved in 12-20 cc. of water at about 90 to 100°C. on a water bath for a period of 60 to 90 minutes. The condensate is separated from the water of condensation in a separating funnel and dried under vacuum for a period of 24 to 36 hours. This is now dissolved in 25 to 40 cc. of acetic anhydride.

In process-1, one mole of naphthalene is sulphonated with 1 to 1.2 moles of concentrated sulphuric acid by heat-

ing them together at 95 to 100°C on a water bath for a period of 2½ to 4 hours. This is now condensed with 0.15 to 0.25 moles of formaldehyde added as 35 to 40% of W/V aqueous solution. The product is kept at the boiling temperature of water for a period of about 10 hours. The solution of the novolak in acetic anhydride is now added to the condensed naphthalene sulphonic acids and the mixture stirred till the product is completely soluble in water. The product is of a thick syrupy consistency and can be easily poured into an open vessel.

In Process-2, equal weights of β -naphthol and sulphuric acid of 1.84 sp. gr. are heated at 115°C for a period of 4 hours under constant stirring in an oil bath. The sulphonated product is dissolved in about 50-70 cc. of water and condensed with 0.25 mols of formaldehyde. The condensate is now capable of precipitating gelatin salt solution. The condensation is completed by keeping the product at a temperature of 95 to 100°C on a water bath for a period of about 8 hours. At the end of this time the product is cooled. This is weighed out and taken in a beaker and heated on a water bath. For every 100 gms. of the condensed β -naphthol sulphonic acid, about 40 gms. of the novolak resin as solution in acetic anhydride is added under efficient stirring conditions. The product is heated on the water bath till it is completely soluble in water. The product is of thick syrupy consistency and is of red brown colour giving a clear dark brown solution in water.

The subsequent step is common for the two processes and is as follows:

Saturated solution of sodium sulphite is prepared and added to the product in order to neutralise it partially. The sulphitation process is carried out in a fume chamber because of the large quantities of sulphur dioxide evolved. Finely powdered, anhydrous sodium sulphite is next added in the solid form stirring

continuously to maintain a uniform consistency of the product. Frothing is vigorous and by controlling the addition of sodium sulphite this can be minimised. After the frothing is stopped, sodium sulphite is added in large quantities till the final product is of a cream white colour. This is now acidified with acetic acid in order that a solution of the product in water will have a pH of about 3.4 in process (1) and 3.2 in process (2).

The syntans so prepared can be used either as an independent tanning agent, or combination tanning agent with vegetable or mineral tanning materials to give very satisfactory light coloured leathers.

The following are among the noteworthy features of these processes:

- (1) The processes give synthetic tanning materials of good molecular size that produce full leathers.
- (2) The synthetic tanning materials produced have good penetrating properties.
- (3) They produce a tanning material of universal application by combining all the desirable properties of various types of syntans into a single compound.
- (4) Use of β -naphthol in process-2 along with ordinary phenol introduces a larger number of phenolic groups which are responsible for the tanning action.
- (5) There is reduction of the ratio of sulphonic acid groups to the phenolic OH groups by use of β -naphthol and also by sulphonating the the novolak with β -naphthol sulphonic acid instead of ordinary sulphuric acid. This reduction of the ratio of sulphonic acid groups to the phenolic OH group per molecule of the syntan to a minimum for complete solubility is highly desirable.

- (6) Utilisation of naphthalene like non-phenolic bodies along with the phenols results in reducing the cost.

Such syntans can be applied for the production of leathers either as independent tanning agents or as pretanning, co-tanning, retanning and/or bleaching agents. The second type of syntans can be applied for the production of particularly heavy leathers as they give good fullness. However, the presence of β -naphthol makes the colour of almost white leather a little brown when exposed to light.

TECHNICAL SEMINAR

Leather Industry in Bihar State*

BY

A. GANESAN

A preliminary survey of the problems facing the leather industry of Bihar State was made with a view to plan our extension services.

The tanning units in this State may be divided into three categories (i) Modern mechanised units such as at Mokameh Ghat and Pilot project units as at Bettiah, Bihta and Sikri, (ii) tanning co-operatives having a working capital of about Rs. 20,000/- such as those at Gomla, Bihar Sherif, etc., and (iii) unorganised units carried on by Chamars as for example at Gomla. The problems facing the units at these different levels are not all the same. However, shortage of good quality slaughtered hides and certain essential specialised auxiliary chemicals is generally present. Conventional tanning materials like babul are not only becoming costlier but also poorer in quality.

The leather industry in the State needs methods of making best possible use of the hides from fallen animals, and also indigenous substitutes for the imported disinfectants, synthetic tannins, fat-liquors, etc., Supply of tannin extracts of assured standard quality is of importance. The tanning materials of the State may be systematically studied and extracts prepared out of the promising materials. Blended extracts tailored to specific applications also may be made. This state has an abundant supply of Asan (*Terminalia to-*

mentosa) bark. So the tanning characteristics and feasibility of manufacture of its extracts may be immediately investigated. Also processes for the manufacture of high quality Kattai leathers, vegetable tanned kips, similar to Madras tanned ones and utilisation of buffalo splits may be formulated.

The tanning co-operatives need trained technical personnel to supervise and control the process. Some of the tanners working in these co-operatives could be given further training in our Institute in manufacture of special kinds of leathers. A process of manufacturing sole leather using the indigenous Asan bark would be quite useful, as also the development of a simple rolling machine of squeeze roll type. Finishes which could be easily applied and do not require glazing would be quite useful. An embossing machine having engraved roll could be developed to suit the requirements of these small units. A scientific study of the bag tanned leathers produced by these tanning units could be profitably made with a view to improving the water resistance of these. The utilisation of Amla (*Phyllanthus emblica*) and fermented fleshings for deliming and of tehripods (*Caesalpinia digyna*) for tanning also need study. By adopting better techniques these co-operatives could be enabled to work with better returns.

As regards the unorganised units, training could be imparted to some of these chamars at our Institute so that they could replace their wasteful methods by the modern ones. By bringing them also under co-operatives schemes, technical service could be rendered to them more effectively. Small hand operated rolling and embossing machines may be fabricated so that leathers required for making chappals and slippers may be prepared by the chamars in a better, easier and more economical way.

*Condensed from the Seminar talk given on 30-5-1958.

CLASSIFIED ABSTRACTS

RAW MATERIALS

PIG SKIN AND LEATHERS FROM IT. F. Stather, *Das Leder*, **9**, 35 (1958). The several peculiarities in the histology of pig skin are discussed and the results of systematic investigations of suitable methods of manufacturing upper leather, corrected grain leather and suede from pig skin are given.

VEGETABLE TANNING

SULFITING OF GORAN-BARK TANNINS. M. B. Rahman, A. A. Khan and S. M. Imam (*Pakistan Forest Coll. and Research Inst., Abbottabad*). *Pakistan J. Forestry* **7**, 145-50 (1957); *Chem. Abst.*, **52**, 2439 (1958). In East Pakistan an annual harvest of 3300 long tons of dry goran (*Ceriops roxburghiana*) bark is possible. An average tannin content of 35 % for goran and 20 % for 'bush goran' is indicated from available data. Sulfiting prevented formation of the 7-8 % of insoluble matter that is found in unsulfited extract. Extraction experiments were made by heating bush goran in beakers with 400-500 wt. % of water and from 0 to 4 % on the bark weight of $\text{Na}_2\text{S}_2\text{O}_5$ at 90-92° for 1, 2 or 3 hours respectively. The liquors were evaporated to a viscous mass on a steam bath, air-dried 4-5 days, and then analyzed by SLTC (Society of Leather Trades Chemists) methods. Best results were found by heating for 3-4 hours with 2-3 % bisulfite; with 3 % in 3 hours, total solids increased from 17.1 to 24.0 %, total solubles in the extract from 90.8 to 96.5, and tannin in the extract from 56.8 to 68.1%. Colour of the extract was greatly improved by bisulfite.

PURIFICATION AND CHEMICAL PROPERTIES OF GORAN TANNIN. Abdul Jabbar Mian and Mofiz-ud-Din Ahmad (*Dacca Univ. Pakistan*). *Pakistan J. Sci. Research*, **9**, 89-93 (1957). *Chem. Abst.* **52**, 5013 (1958). Methods of purification of goran-bark tannin were investigated. Neither the hide-powder method nor the electrodialysis method was suitable. The Pb-salt method gave 97.9% purity and 25.1% recovery, the salting-out method 96.2 % purity, and 40.1 % recovery, and the mixed solvent gave 96.3 % purity and 48.4 % recovery. Molecular weight seems

to be related to purity of sample; tannins of 97.7, 96.3 and 95.9% purity gave mol. wts. of 840-797, 323-305 and 294, respectively.

AN APPROACH TO THE STUDY OF VEGETABLE TANNINS BY THE OXIDATION OF PLANT PHENOLICS. D. E. Hathway, *J.S.L.T.C.*, **42**, 108 (1958). This work is concerned with such chemical reactions as might be involved in the formation of large aromatic molecules found in plant tissues, and especially those found in tanning extracts. These are of two classes, the one based on gallic acid or gallates and their oxidation products, and the other based on catechin-like substances and their polymerisation products. Since the reactions in question involve dimerisation of semiquinone radicals, and polymerisation through quinones, knowledge of the chemistry of quinones with special reference to their reduction products and reactivity is essential to an understanding of these reactions. The chemistry outlined represents the background of contemporary knowledge from which the present work stems.

SHELL CORDOVAN AND ANALOGIES OF COMBINATION TANNED SOLE LEATHER. W. Frankford and A. Finch. *J.S.L.T.C.*, **42**, 122 (1958). The paper discusses the various raw materials used in making Cordovan leather and the way in which the properties of "Shell Cordovan" are influenced by the fibrous structure of the horse butts used in its manufacture. The methods used for tannage and finishing are outlined as are the advantages of this type of leather. The growing shortage of equine hides has led to a search for an alternative process of making Shell Cordovan from other types of hides, using enzyme unhairing instead of liming in an attempt to obtain high fibre density. This approach did not succeed but resulted in production of experimental leathers resembling older type sole leathers produced by the sweating process. It is suggested that enzyme unhairing without liming may be of value in producing a firmer, better wearing sole leather.

OTHER TANNAGES

OIL TANNAGE. IV. EFFECT OF CONTROLLING FACTORS OF REACTION IN OIL TANNAGE. Minoru Kubota (*Tokyo Univ. Fisheries*). *Nippon Kikaku Gijyutsu Kyokaishi* **2**, 133-9 (1956); *Chem. Abst.* **52**, 3381 (1958). The oil-tannage reaction was studied from the viewpoint that the tannage is due to combination of aldehydes formed by rancid reaction of oil with hide substance. In tanning for 5 days with oleic acid alone, the amount

of aldehydes combined with hide substance was only 22 millimoles/kg., while in commercial oil tannage it was 27-50 millimoles/kg. The shrinkage temperature (T_s) of the oleic acid-tanned leather was 540. The presence of 50-60% water in the oleic acid tannage gave a maximum rate of fixation of the combined aldehyde and a maximum hydroxyl value of the oil. Dihydroxystearic acid had feeble tanning power. The amount of combined aldehyde in oleic acid tanning increased markedly under alkaline conditions. $KClO_4$ and benzoyl peroxide considerably increased the amounts of combined aldehyde, particularly the former. Tannage with rice oil along gave better results, with a larger amount of combined aldehyde and higher T_s than with oleic acid alone. As the tannage proceeded the I and ester values of the oil decreased while the acid, peroxide and hydroxyl values increased. Addition of Co and Cu salts to the rice-oil tannage increased T_s . Inorganic oxidizing agents also increased T_s and the amount of combined aldehyde, but such strong agents as $KMnO_4$ and $(NH_4)_2S_2O_8$ did not increase T_s . Addition of Cu and Co oleates produced an excellent effect on the rice-oil tannage.

THEORIES

OBSERVATIONS ON THE ISOIONIC AND ISOELECTRIC POINT OF ACID—PROCESSED GELATIN FROM INSOLUBLE AND CITRATE EXTRACTED COLLAGEN. D. S. Jackson & A. Neuberger. *Biochem. et. Biophys. Acta* **26**, 638 (1957). The isoelectric and isoionic points of gelatin derived from insoluble collagen and citrate extracted collagen from rabbit skin were determined. The isoelectric point was determined by electrophoresis and the isoionic point by de-ionization on a mixed bed resin column. The results indicate that the isoelectric and isoionic point of the gelatin derived from the insoluble collagen and citrate soluble collagen are the same and behave in the same manner with varying ionic strength.

TESTING

MOLASSES FOR FILLING STIFF LEATHERS. F. B. Blanshei. *Legkaye Prom.* **17**, No. 8, 17-18 (1957); *Chem. Abst.* **52**, 1662 (1958). The sugar content of molasses to be used in filling leather is determined by boiling 1 g. molasses with 20 ml. of a 60% solution of H_2SO_4 and 25 ml. of a standard solution of $Na_2Cr_2O_7$. Excess hexavalent Cr is titrated with $Na_2S_2O_3$. Each

100 parts by weight of $Na_2Cr_2O_7$ consumed is equivalent to 17.2 parts of sugar (computed as glucose).

PREPARATIVE FRACTIONATION AND QUALITATIVE ESTIMATION OF CONSTITUENTS OF SYNTANS PRECIPITABLE WITH METAL SALTS. A. Haglund, *Das Leder* **9**, 29, (1958). In earlier work, the author has attempted to use precipitation with metal salts for estimation of vegetable tannins, the salient features of this procedure being the employment of hydrogen sulphide for freeing the non-tans solutions from the added cations and addition of pyridine to effect complete precipitation. Lead acetate has been found to precipitate not only tannins but also non-tans. So zinc or cadmium acetate is best suitable for this purpose; acetate is chosen since resultant acetic acid could be removed easily. In applying this method to syntans hydrogen sulphide has been substituted by a cation exchange resin both for obtaining the free tannins and for freeing the nontans solution from the added metal ion. The method now was therefore as follows: —

To 100 ml. of an analytical solution containing 5 to 10 grams of dry substance per litre is added 5 ml of N-metal acetate solution (zinc or cadmium) and 2 ml pyridine. The precipitate is filtered off or centrifuged, washed with the dilute solution of metal acetate and pyridine and suspended in water. The cation exchange resin (Amberlite IRA 120) is added in small portions. The free tannins which in this case are mostly novolaks form dispersions. The filtrate of non-tan solution obtained first, is passed through on IRA 120 resin column on H^+ cycle, washed out, brought to pH3 and carefully evaporated, too low a pH and temperature being avoided. The residue gives the non-precipitable components of the syntans.

The tans and the non-tans, that is, the precipitated and unprecipitated components respectively, were used in tanning pieces of pelt. The non-tans had practically no tanning action as shown by comparison of shrinkage temperatures in the two cases.

Novolaks of different degrees of sulphonation and of different degrees of condensation were prepared. The tannin values obtained by the metal acetate precipitation were in each of these cases compared with those obtained by the hide powder method. The discrepancy between the two increased with increasing degree of sulphonation but decreased with increasing degree of condensation, that is, precipitability of the syntans is controlled by these

two characteristics. A highly condensed product, though highly precipitable has low tanning action; the tanning quality of a syntan is not therefore always dependent on the degree of precipitation.

A number of commercial syntans have been analysed by this precipitation method and the analytical results compared with those got by the hide powder method. The ratio of the value got with zinc acetate precipitation to that got by the hide powder method was 1 for the Baykonals; it varies from 0.58 to 0.93 with replacement syntans, from 0.63 to 0.73 with the syntans for white tanning, from 0.44 to 0.48 with the pre-tanning ones and 0.00 to 0.14 with the bleaching ones. In contrast to vegetable tannins, the syntans give higher values with zinc than with cadmium. Substances which do not give precipitate with pyridine and zinc or cadmium acetate cannot therefore be considered as real tannins. On the other hand, all that is precipitated with zinc or cadmium is not a tannin.

Mixtures of vegetable tannins and replacement syntans give almost the same values as correspond to the sum of the values for the individual components. However, when instead of the replacement syntans, auxiliary syntans or ligno tannins are present along with vegetable tannins, invariably higher values are obtained. Substances like lignin extract resembling tannins are not precipitated. However, in admixture with typical tannins, they are co-precipitated. Similar higher values occur even in the case of hide powder. The results obtained with mixtures of mimosa tannin and lignin extract seem to indicate that substances not precipitated by zinc acetate and pyridine also are absorbed by hide powder. The difference in the values obtained by the two methods is designated as due to hemi-tannins. In all these cases, tan values were determined and the residual solutions from these were analysed by both hide powder and zinc acetate/pyridine methods. The difference between the values got by these two methods is again taken as due to hemi-tannins. More of these are found after determination of tan values.

Mixtures of lignin and mimosa extracts prove to be of very high hemi-tannin content. By precipitation with zinc acetate and pyridine it is possible to separate the true tannins from the hemi-tannins and non-tannins. The combining value of the extract is improved by removal of the hemi- and non-tan for these latter interfere in the tanning process. Further, they, by themselves are not leather forming though partly taken up by hide powder.

The precipitating action of metal salts with pyridine can thus be utilised to fractionate the tannin mixtures.

—V. S. P.

MICRODETERMINATION OF ZIRCONIUM IN SULFURIC ACID SOLUTIONS WITH PYROCATECHOL VIOLET. *J. P. Young, J. R. French and J. C. White, Anal. Chem.* **30**, 422 (1958). The blue color of the zirconium pyrocatechol violet complex has been utilized in the development of a very sensitive method for the colorimetric determination of zirconium in sulfuric acid solutions. The molar absorbance index of this complex is 32,600 at a wave length of 650 mμ. Its absorbance conforms to Beer's law up to a concentration of zirconium of 2 γ per ml. Trace amounts of zirconium can be determined in the presence of thorium, uranium, lanthanum, cerium, iron, nickel, and chromium. Fluoride, citrate, oxalate and tartrate ions interfere by decolorizing the complex. The co-efficient of variation for the determination of zirconium by this method is 2%.

MICRODETERMINATION OF CHROMIUM WITH 1, 5-DIPHENYLCARBOHYDRAZIDE by *Thomas L. Allen, Anal. Chem.* **30**, 447 (1958). Operational variables in this microdetermination have been investigated. Included are the effects of pH, redistillation of water, nature and concentration of organic solvent, concentration and quality of 1, 5-diphenylcarbohydrazide, age of stock solutions of this reagent, order of mixing, temperature, ionic strength and shelf life of solid reagent. Also studied were the suitability of sodium peroxide for chromium oxidation and the solubility of 1, 5-diphenylcarbohydrazide in water and dilute sulfuric acid. With fresh solutions of a good grade of 1,5-diphenylcarbohydrazide, the molar absorbance index of the colored product at the absorption maximum of 546 mμ is $(4.17 \pm 0.04) \times 10^4$, based on the concentration of chromium (VI).

STUDIES ON PICKING BAND LEATHER. *Sanjoy Sen, N. Das Gupta & Asis Chakraborty, J. Ind. Leath. Tech. Assoc., New Volume No. 1*, 34 (1958), Symposium special. The article seeks to establish that picking bands made out of the indigenous buffalo leather are in no way inferior to those from imported cow hides. However, the picking band industry is faced with acute shortage of buffalo hides over 65 lbs weight yielding a finished leather with a substance of 60.5 m.m. and possessing uniform thickness.

Tensile strength, though not an absolute criterion does determine the quality and life of the picking band to a large extent. By noting the fibre structure during the different processes of picking band leather manufacture, the authors proceed to show the important role that the fibre structure plays in determining the tensile strength: tensile strength is a function of both the angle of weave and the degree of splitting of fibres and fibrils.

The raw tensile strength of slaughtered buffalo hide is very much greater than that of wet-salted buffalo and imported ox hides. Preliminary findings appear to show that greater tensile strength in the raw stage leads to superior picking band. The criterion in the liming operation is not the duration but the degree of splitting of fibres and fibrils. A one-day lime sulphide painting followed by a day in old lime liquor and 3 to 4 days in new lime liquor should achieve the desired splitting. The final tensile strength of limed and pickled leather is found to be higher than that of leather which is only pickled. 2.5% Cr_2O_3 based on limed pelt-weight has been found to be optimum. In dry tanning 20% basic liquor enables rapid penetration and quick tannage. Uniform distribution as well as proper fibre splitting in the pre-tanning processes is ensured by this lower basicity. It has been the experience of the authors that tensile strength of chrome tanned picking band is higher than that of vegetable tanned picking band and of sulphur and oil-tanned picking band also. Uniform distribution of fat in stuffing is achieved by having the optimum moisture content of about 40% and by ensuring that leather dries out at the same rate at which oil penetrates into the inner layers. In selecting grease for stuffing, high iodine value, high free fatty acid content and high dehydrating power property are to be avoided.

—V. S. P.

BYE-PRODUCTS

TREATMENT OF TANNERY WASTE BY ACTIVATED SLUDGE. B. L. Rasenthal. *Lea. Manuf.* 75, No. 2, 26 (1958). Laboratory scale treatment of a combined tannery waste by activated sludge indicated that better than 95% BOD removals could be obtained with loadings upto 100 lbs. per 1000 c. ft. abration capacity per day and with 170 lb. loading, 90 per cent could be removed. Necessary pretreatment included equalization and carbonation with flue gas. The study indicated that where disposal of a primary treated tannery waste is a problem, pilot plant

experiments on the specific waste could prove that activated sludge secondary treatment is feasible because of the high loadings that could be attained.

PATENTS

DELIMING AGENTS FOR ANIMAL HIDES. *Deutsche Gold-und Silber-Scheideanstalt vorm. Roessler (Ulrich Hoffmann and Erich Bohme, Inventors)* Ger. 962,025, Apr. 4, 1955 (Cl. 28a, 2) *Chem. Abst.* 52, 4222 (1958) Pentaerythritol (I) is better than molasses or waste products of the beet-sugar industry when used in weak organic acid deliming liquor to obtain a fine, tight, smooth grain surface on skins. After deliming 100 kg. of dehaired hide with AcOH 0.5, technical NH_4OH 0.2, and I 0.1 kg., the residual CaO in the hide was 16.5% of that originally present; without I, the residual CaO was 28.5%. Instead of I, 0.4 kg. of the mother liquor from the preparation of I by alkaline condensation of AcH with HCHO can be used with 0.8 kg. of a $\text{HCOOH-NH}_4\text{COOH}$ mixture or an AcOH-AcONH₄ mixture.

LEATHER WITH AN ARTIFICIAL GRAIN. *Wickrather Lederfabrik (vorm. Z. Spier) A. G. (Dietrich Grosse, inventor)*. Ger. 926,875, Apr. 25, 1955 (Cl. 28a, 11). *Chem. Abst.* 52, 4222 (1958). Artificial grain can be produced on all types of leather by embossing the dehaired or preferably pickled skin and then tanning without motion. For example, bated lambskins were pickled for 90 minutes in a solution of water 100, NaCl 20, alum 5, and a tech. 30% mixture of low-mol. C acids 2%. After draining overnight and wringing, they were embossed in a hydraulic press at 35°, then tanned overnight by hanging in a chrome liquor containing 30 g. of commercial liquor (25% Cr_2O_3) per 1. and 0.5 g. ethoxylated fatty alcs. ($\text{C}_{12}\text{-C}_{18}$), using ultrasonics during the first 3 hours.

PRIMING-COAT MATERIAL FOR UPPER-LEATHER FINISHING: *Badische Anilin- & Soda-Fabrik Akt.-Ges. (Helmut Lidle and Gerhard Otta, inventors)*. Ger. 928,361, May 31, 1955 (Cl. 28a, 9) *Chem. Abst.* 52, 4222 (1958). The advantages of oil- or wax-containing binders, without their disadvantages, are obtained by use of dispersions or water-miscible solutions of cation-active fats. Thus dyed, dry, chrome side leather, before application of a casein finish, is brushed with a 20% solution of octodecyldimethylbenzylammonium acetate. Chrome sheep garment leather, before a collodion finish, is treated with the glycolate of

the condensation product of diethylenetriamine and coconut oil fatty acid 30, olive oil 30, and water 40 parts. Chrome retan side leather is treated with the chloride of the condensation product of oleic acid and diethanoethylenediamine 25, peanut oil 25, and water 50 parts. With these binders, good grain, good pigment coverage, and high fastness to wet rubbing are obtained.

PROTEINACEOUS ADHESIVES. *David H. Read (to American-Marietta Co.). U.S. 2,813,037, Nov. 12, 1957. Chem. Abst. 52, 4224 (1958).* Aqueous alkaline soya-bean and blood-protein plywood glues are stabilized against the loss of thixotropy due to the presence of approximately 5% of pentachlorophenol (fungicide) by the addition of 0.1-0.5% of a cobaltous salt. The principal action of these salts is on the blood protein. Nickelous and ferrous salts have only a minor effect.

DISINFECTING HIDES. *E. S. Lavrova. U.S.S.R. 102,505, Apr. 30, 1956, Chem. Abst., 52, 5014 (1958).* Hides of animals infected with hoof-and-mouth-disease virus are washed or sprayed with a saturated solution of Na_2SiF_6 and then evenly dusted on both sides with a mixture containing NaCl 40-50 and Na_2SiF_6 28% of the weight of the hides. The latter are stacked and thus kept for 7-10 days at 15-20°.

GENERAL

DRUMS VERSUS PADDLES. *A. W. Goetz, Leather Manufacturer, 75, No. 1, 10 (1958).* In this article, an attempt is made to categorise those factors of tannery processes that are most affected by the equipment employed. The four factors of concentration, time, agitation and heat are the bases of any process whether it be done in drum or in paddle.

With drums one can employ almost unlimited ratio of stock to liquor whereas with paddles the ratio is restricted to a definite low limit. In drums and paddles quite accurate ratios or volumes could be established and maintained by use of volume to inches curve. Another use of the capacity curve is the determination, by liquid displacement, of the weight of the stock.

When individual processes are transferred or changed from a paddle to a drum, considerable alteration in the processes may be required because of the effect of usually diminished ratio, e.g., in pickling. The ratio with drums sometimes is so low that reaction proceeds too rapidly to ensure uniform take-up by the skin or

hide. However, the lower ratios in drums and the resultant higher concentrations contribute to the fact that for certain processes there can be a quicker equipment process turnover with drums than with paddles.

That lower ratios or shorter floats contribute to temperature rise and to more agitation is well-known. Because of the impact and frictional heat generated during milling, external heating is not necessary with drums. The same process and mechanical features that contribute to the rise in temperature during milling also bring about a proportionate increase in the agitation of the stock in the liquor.

The processes of fatliquoring, colouring and in most instances, one bath chrome tanning, carried out in a paddle would be more timely and at the same time expensive in so far as the materials required in temperature controls are concerned. Drum operations usually require less water and so there is less effluent. For this reason, drums are replacing paddles in fatliquoring of vegetable grain leather and tanning. As regards pickling, the same percentage of acid is used in drum as well as in paddle. However, less salt is used with drum. The agitation and shorter liquor contribute to a faster pickling action, in the drum. In bating the drum does not allow ready escape of ammonia. So addition of dilute acid is necessary. An outstanding contribution made by drum bating is the so-called combination drum bating pickling and tanning. By not handling the stock between these operations, considerable labour and time is saved.

A few direct comparisons between drums and paddles are made with regard to loading and unloading, materials used, power used, repairs, etc., feeding, washing, draining, observation and taking samples.

—V. S. P.

THE CARE OF LEATHER, *Anon. Leather World, 2, No. 1, 10 (1958).* In spite of the ubiquity of leather, the care and treatment of leather articles is too frequently neglected. Cracks and a stiff unyielding surface are the commonest blemishes in old uncared for leather.

Leather being an animal substance, needs appropriate treatment; undue heat is harmful, so do not put wet shoes or bags near a fire. Let them dry out naturally by placing them near an open window or where there is draught.

Once the leather is dry, it needs nourishment and protection. Both these are supplied with saddle soap (not the transparent glycerine soap used only on saddles). Moisten the soap with a damp sponge and with the same sponge work the soap well into the leather. (There is no lather with this soap). When the soap has been worked in and left to dry for a few minutes, polish vigorously with a soft brush or cloth. Snow stains too are removed by saddle soap. Saddle soap cannot be used with those coloured leathers for which a special polish is recommended to keep the colour at its original shade. Benzine or other spirit cleaners are not recommended.

Once the leather has been treated with saddle soap, a good shoe polish can be applied. Patent leather surface is not absorbant, so use less polish. Polishing is to be done regularly not only when the articles become dirty but also when they have been in use for some time. Leather upholstery can be treated with the hide foods available in the market.

Leather articles unused or stored for sometime such as holiday suitcases, should be treated before they are put away, lest they become stiff and cracked when not used. Dubbin will keep the articles soft and supple and quite clean, even in dust grimy surroundings. It is simply rubbed fairly thickly all over the clean articles which are given a wipe with an old cloth when brought out again to remove any surplus. Oils like neat's foot oil, keep leather supple but stain clothes. Lastly if you have goods of certain shape, help them to keep that shape. Insert shoe trees into shoes after use. Do not overload bags.

NOTES & NEWS

Sulphur fed sheep produce stable wool

Wool that will not shrink is being produced in the United States with sheep at the University of Illinois experiment station as the result of an accidental discovery in a series of experiments aimed at finding the effect of mixing different chemicals with animal fodder. After adding a bit of sulphur to the sheeps diet, the scientists measured the stretch of wool fibres and found wool of the sulphur-fed sheep remained stretched, while that of others returned to its original length. The effect became the same as that of stabilising methods used to prevent changes in woollen garments in washing and cleaning. Fibres that do not change length are in great demand in the textile industry and the new discovery is expected to cause a degree of revolution in sheep production.

— *The Tanner*, 12, 272 (1958).

Disposable shoe shine pads

Men who like to give their shows a quick shine before going to work and who hate to fuss with cans of polish, daubers, brushes and buffers, will welcome new, disposable shoe shine pads. The pads are made of circular cotton pieces saturated with a polishing agent. Disposable after use, the quick shine pads provide a water-repellent finish as well as protection against mold and mildew. The pads are manufactured by Pet Chemicals, Inc., of Miami, Fla.

— *Science Digest*, 43, No. 2, 96 (1958).

Dry cleanable, water repellant and oil resistant suede leather

Dry cleanability and water repellant and oil resistant properties can now be imparted to suede leather. The new Minnesota Mining and Manufacturing treatment — Scotchgard brand stain repeller for leather — has a fluoro chemical base as does its textile treatment but in addition to its water repellant and oil resistant properties, makes suede soil resistant. At the same time, says the company, it permits it to breath', aids in dye fixing, provides added softness and dry-cleanability, properties which steam pressing will not remove.

— *Chem. & Eng. News*, 35, No. 10, 91 (1957).

Bromelain — a new proteolytic enzyme

A new protein digesting enzyme, bromelain is being produced from the lower stem of the pine apple plant by Dole Hawaiian Pine Apple Co. The 'mother' stumps are washed and crushed and the extracted juice filtered. Bromelain is precipitated by addition of acetone, centrifuged off and over dried. The product, a very fine powder is a mixture of several enzymes. Most of these compounds are protein digesting enzymes. Other ingredients include acid phosphatase, amylase and peroxidase. The action of the enzymes in bromelain increases as temperature goes up to about 70°C, at which temperature the enzymes are destroyed.

Bromelain has a rather limited proteolytic action compared with papain, ficin and pepsin. It attacks the protein molecule at fewer places and generally hydrolyses it without breaking it down into small fragments. In addition to tenderizing meat, other uses envisaged are *leather processing*, textile production, etc.

—*Chem. & Eng. News*, 35, No. 38, 83 (1957).

New Leather Colors for fall and winter 1958

Men's Colours: The Copper Beech Browns — Ranger, Beechwood, Vintage, Juniper.

The Mahogany Browns: Meerschaum, Bruin, Mahogany, American Burgundy.

The Forest Browns: Olivewood, Dark Oak, Hemlock.

The After Dark Colors: Midnight Blue, Black.

The Brushed Leathers: Greige, Wheat, Peat Moss, Vicuna, London Grey.

Women's Colours

The Winter Neutral: Oyster.

The Deerskin tans: Vicuna, Nut Brown.

The Copper Tan: Benedictine.

The Two best Medium Brown: Briarwood, Perfect Brown.

The Blackened Browns: Golden Walnut, Java, Town Brown.

The New Winter Green: Tartan Green.

Reds: Comanche, Scarlet, Basque Red, Cherry Red, Damask Rose, Beet Root.

The Winter Grays: Gunmetal, Gray Seal, Chinchilla, Graphite, London Gray.

Children's Colours: Campus, Benedictine, Papoose, Glen Brown, London Gray, Scarlet, Cherry Pie, Raspberry, Blue Ribbon, Royal Blue, Sapphire.

—*Leather & Shoes*, 135, No. 3, 39 (1958).

Market Survey of Hides and Skins in Sweden, Denmark, Norway and Finland

The total demand for imported hides and skins into these countries is of the order of 36 thousand tons a year. Imports of hides and skins from India are, as a rule, very small in all the four countries. Most of the Indian hides and skins are, however, being bought from the United Kingdom through various broker firms.

According to the people in the Trade, the domestic production of hides and skins is of a higher quality than anywhere else in the world except Switzerland and New Zealand. Unprepared Indian goat and sheep skins are considered to be among the very best which are available. These types are, however, of relatively little importance except for Sweden. The standard of living is rising in these countries; so the quality is more important than the price. Importers, of course, attach importance to the time of delivery, price and quality agreed upon. There should be no confusion regarding nomenclature and classification.

The quality of the unprepared skins must be improved at all stages of production — before and after slaughter. The difficulties in the way of marketing unprepared hides and skins from India could be avoided by delivering tanned hides and ready-made leather to larger extent than at present. An attempt to simplify the quality, size and classification, would be welcomed by the Scandinavian customers. It is felt that exports of Indian hides to the Scandinavian market could be considerably increased if a special agent were appointed to deal exclusively with this problem. An Indian Agency situated in one of the four countries, preferably in Sweden and represented by a person possessing the confidence of the market, would probably have better qualifications to attend to progressive sales especially if backed by guarantees regarding quality.

Sweden: Indian skins from goat and sheep are used to a rather large extent. As a result of fast growing motorism, con-

sumption of leather jackets has increased considerably. Sometime back light ladies' shoes of goat skins also were popular.

Denmark: The Danish leather trade is aware of India as a supplier of raw materials and would probably regard any Indian offer seriously on condition of a fair relation between price and quality.

Finland: Sheep skins for gloves and leather clothing and also some other special types of leather are imported from India and are considered satisfactory. It is understood that Indian as well as Australian and South African hides are mostly sold to Finland through London brokers.

— *Supplement to J. Ind. & Trade*, March 1958. p. 13.

Impressive Progress of Chemical Industry

The chemical industry in India has made rapid strides during the last few years. Production of various chemicals has increased to a remarkable extent and the industry has been able to take up many new lines of manufacture.

The country has before it a large production and development programme for the chemical industries in the Second Five Year Plan. The following are the targets for some of the chemicals.

Item	Tons		
	1951	1957 (Estimated)	Targets for 1961
Ammonium sulphate ..	52,604	370,000	1,600,000
Sulphuric acid ..	106,932	195,000	470,000
Soda ash ..	47,532	90,000	230,000
Caustic soda ..	14,724	42,000	135,400
Liquid chlorine ..	5,268	15,500	17,000
Bleaching powder ..	3,588	5,200	15,000
Bichromates ..	3,271	3,500	6,000
Sodium bicarbonate ..	1,630	4,400	8,000
Potassium chlorate ..	1,593	2,300	3,800
Alum and aluminium sulphate ..	21,810	37,150	50,000
Ammonium chloride ..	—	4,800	5,000
Acetic acid ..	—	2,600	—
Benzene hexa chloride ..	—	2,500	3,000
D-D.T. ..	—	(50%) 1,400	3,000
Hydrogen peroxide ..	—	550	1,500
Sodium hydrosulphite ..	—	Nil	4,000

— *J. Ind. & Trade*, 8, 481 (1956)

Annual Convocation of College of Leather Technology, Calcutta

The Annual Convocation of the College of Leather Technology, Calcutta, convened by Shri B. C. Mallik, Director of Industries, West Bengal, was held on 14th May, 1958, at the College premises. Shri P. M. Das Gupta, Deputy Secretary of the Commerce & Industry Department, West Bengal presided over the ceremony. Shri B. Majumdar, Minister for Commerce and Industry, West Bengal, delivered the Convocation Address. On this occasion, certificates of the Calcutta University as well as Departmental certificates from the Directorate of Industries, in tanning and in boot, shoe and leather goods making, were awarded to successful students. Medals were presented to those who topped the list in the different courses.

Shri M. Banerjee, Superintendent of the College and Deputy Director of Industries (Ex-Officio), welcomed the gathering. Shri Majumdar, in his Convocation Address, stressed the importance of technical education in independent India. He said that no education was worth-while unless it could be applied in practical fields. He concluded by exhorting the students to cultivate the essential qualities of self-reliance and integrity.

Next day, the annual re-union and social gathering was held with Shri Sanjoy Sen, Director, Sen Raleigh Industries Ltd., and National Tannery Co., Ltd., as President. Dr. U. P. Basu, Director, Bengal Immunity Research Institute, was the Chief Guest. The Re-union was sponsored by a Committee headed by Shri P. K. Sarkar.

RESEARCH BEARS FRUIT

Practical Demonstration No. 4

(Second Series)

Process: Manufacture of Rapid Tanned Sole Leather using Indigenous Vegetable tanning materials.

Period: 25th July to 13th August, 1958.

Place: Central Leather Research Institute.

The next in the second series of Practical Demonstrations will be on the manufacture of rapid tanned sole leather using indigenous vegetable tanning materials.

It may be recalled that two processes for the manufacture of rapid tanned sole leather were demonstrated in March, 1957. A number of tanners participated in these demonstrations and they have been following up these processes in their tanneries. Since that time additional researches have been made on the manufacture of sole leather using indigenous tanning materials, e.g., *Pithecolobium Dulce*. This bark called Manila Tamarind in English, Kodukkapuli in Tamil, and Seema Chinta in Telugu, is reported to be available in some States. It is now proposed to demonstrate this process in the month of July, 1958. If you are interested in this line of work, you are most welcome to send your representative(s) to get trained in the manufacture of sole leather. You may kindly intimate the name(s) of the representative(s) who is/are likely to attend this demonstration. After following the process, we would request you to try this process in your tannery and give us your valuable comments.

SYMPOSIUM

on

Tanning as a Small-scale and Cottage-scale Industry

The major part of the Indian Leather Industry is at the decentralised small-scale and cottage-level. In fact, next to handloom it is the most important cottage industry; and hence its share in the overall production of National wealth is quite considerable. It has been estimated that about 70% of the raw hides and 35% of the skins produced in India are processed by the Small-scale and Cottage sector of the tanning industry. The overall demand for leather at the end of 1960-61 has been estimated to be equivalent to 23 million hides and 26 million skins and the Planning Commission envisages a substantial part of this increased demand to be met by products of the small-scale units. It is therefore evident that the development of this sector is of vital interest. The Government of India has been alive to this fact and the Small-scale Industries Board, and the All India Khadi and Village Industries Commission, Community Development Wing, the Central Leather Research Institute and other organisations are active in promoting tanning as a village and small scale industry.

The village tanning industry, follows at present, empirical and traditional methods and the artisans engaged in the industry are mostly illiterate. The development programme aims primarily at raising the present level of technical efficiency of the small-scale tanner by providing common service facilities for improved methods of curing, tanning, finishing, etc., at well equipped centres. With this end in view, the establishment of flaying centres, village model tanneries, manufacturing units and training cum-production centres have been included in the development programmes of the Government.

The Central Leather Research Institute has been established for serving the Indian Leather Industry in all its various phases, by carrying to the industry the fruits of its research at all levels.

Symposium at the Institute is an annual feature adopted for renewing and strengthening its contact with the Industry, to pool the ideas and share the available knowledge. As you may be aware of, the Institute has conducted three symposia so far on (1) East India Tanning Industry and Tanning Agents, (2) Raw hides and skins—Curing and Preservation, and (3) Leather Auxiliaries, and it is now proposed to hold the fourth symposium on

'Tanning as a Small-scale and Cottage-scale Industry' in February, 1959, in order that the Institute may be better able to serve this sector of the Industry by getting to know its problems.

The scope of the Symposium is defined in Appendix-A and papers are invited on these subjects. Since suitable translation arrangements will be made, the papers may be presented in English or any other language. An advance copy of the paper together with an abstract not exceeding 150 words may kindly be forwarded before the 1st December, 1958, to enable us to circulate the papers in advance to the delegates attending the Symposium.

You are requested to kindly intimate this office at an early date the names of the representatives of your organisation who will participate in the Symposium. The enclosed form, Appendix-B may kindly be filled in and returned.

Arrangements will be made for the accommodation of the delegates, and visits to some of the tanneries and places of interest in and around the city will also be organized.

It is hoped that you and members of your organisation will be able to attend the Symposium and present papers which would afford an opportunity for establishing personal contacts leading to the exchange of ideas for the development of the Industry.

An early reply which will facilitate drawing up of a detailed programme will be greatly appreciated.

APPENDIX-A

TANNING AS A SMALL-SCALE AND COTTAGE-SCALE INDUSTRY

Scope

- I. Raw Hides and Skins.
 - (a) Collection
 - (b) Preservation
 - (c) Transport
- II. Processing and Treatment materials.
 - (a) Traditional manufacturing processes
 - (b) Modern methods and techniques
 - (c) Tanning agents and treatment materials

- III. Marketing.
 - (a) Methods of collection
 - (b) Grading and quality control
 - (c) Sales
- IV. By-products.
 - (a) Collection and disposal.

APPENDIX-B

CENTRAL LEATHER RESEARCH INSTITUTE MADRAS-20
Particulars to be supplied by those wishing to participate in the Symposium

1. NAME
2. FULL ADDRESS
3. Title of paper
4. Do you intend to take part in the general discussions on the subjects
5. If so, please indicate title and send a short abstract
6. Is accommodation in Madras to be reserved for you
7. Are you interested in visiting tanneries in and around Madras
8. Any other request or suggestion not covered above

C. L. R. I. NEWS

PROF. HUMAYUN KABIR VISITS THE CENTRAL LEATHER RESEARCH INSTITUTE

Prof. Humayun Kabir, Union Minister, for Scientific Research and Cultural Affairs and Vice President, Council of Scientific and Industrial Research, visited the Central Leather Research Institute on the 28th May, 1958, and spent over two hours going round the various sections of the Institute including the tannery and the laboratory.

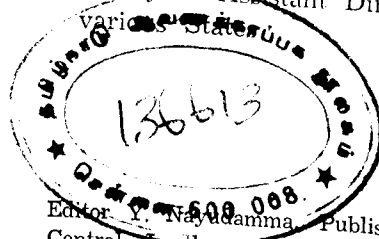
The Union Minister evinced keen interest in the progress of research in the Institute. He was taken round the various departments by Dr. Y. Nayudamma, Director of the Institute.

A party of Assistant Directors of Industries of the various States, now undergoing training at the Small Industries Service Institute, visited the Central Leather Research Institute on 14-6-1958. They were taken round the tannery and the various departments of the Institute. Dr. Y. Nayudamma, Director, Central Leather Research Institute explained to them in the course of a talk the extension service programme of the Institute.

Dr. Y. Nayudamma, Director, has been nominated as Member of the Technological Sub-Committee for 1958-59 of the Indian Central Arecanut Committee.

VISITORS

Name	Date of Visit
Prof. Humayun Kabir, Vice-President, C.S.I.R., New Delhi	28-5-1958
A Party of Assistant Directors of Industries of the various States	14-6-1958



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