Development of Natural Garment Leathers: A Metal-Free Approach

by

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Abstract

Growing concerns over the toxicity of chromium and its influence on the health of customers using chromium tanned garment leathers has forced the leather industry to search for alternative tanning agents. Semi-metal tanning agents, aldehyde based systems, etc. have been considered but the toxicity issues are lingering. Hence, in the present study an attempt has been made to develop vegetable tanned garment leathers. The indiscriminate use of vegetable tannins and subsequent stripping prior to retanning and fatliquoring has been evaluated. It has been found that an 8% offer of wattle for tanning provides better physical and organoleptic properties. A polyamide based retanning system was found to offer better perspiration and organoleptic properties. Amongst the various fatliquoring compositions, a 50:50 combination of synthetic and natural fatliquors was found to offer higher softness. The optimized tanning—retanning—fatliquoring combination was found to be comparable to chrome tanned garment leathers and superior to semichrome garment leathers.

Resumen

Crecientes preocupaciones acerca de la toxicidad del cromo así como su influencia sobre la salud de clientes utilizando cueros de vestimenta curtidos al cromo ha obligado a la industria del cuero a buscar agentes curtientes alternativos. Agentes curtientes semi-metálicos, sistemas basados en aldehídos, etc. han sido considerados pero las preocupaciones toxicológicas siguen todavía presentes. Entonces, en el presente estudio se ha tratado de desarrollar cueros de vestimenta al tanino. El uso indiscriminado de taninos de origen vegetal con subsecuente parcial remoción antes de recurtir y engrasar se ha evaluado. Se ha encontrado que una oferta de 8% de extracto de mimosa para el curtido produce las mejores propiedades físicas y organolépticas. Un sistema de recurtido basado en poliamida se encontró ofreciendo mejor solidez al sudor y propiedades organolépticas. Entre las varias composiciones de engrases, se encontró que una composición de 50:50 de engrases sintéticos y engrasantes naturales ofreció gran blandura. La optimizada combinación curtido-recurtido-engrase se encontró comparable a la vestimenta curtida al cromo y superior a la de semicromo.

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INTRODUCTION

Since the advent of chrome tanning, the tanning industries have taken over chrome for vegetable tannins in the process of tanning. The biotoxicity of chromium has been a subject of active discussion. The biological implications of chromium are known to vary with the oxidation state of the metal ion.[1,2] The implication of chromium(III) in glucose metabolism has been considered beneficial,[3,4] while oxyanions like chromates are well-established human carcinogens.[5] The other mineral tanning systems such as aluminum, zirconium, and titanium are known to be preferred in the wet-white tanning.[6] The environmental issues regarding the mineral tanning systems make us to move towards the mineral free tanning such as aldehyde/polymer tanning systems. But even these systems are not environmentally sustainable because of toxicity issues with regard to monomers used in such tanning systems.[7] So the only way left is reverting back to Mother Nature i.e, vegetable based tanning systems. However, this option seems disadvantageous for garment leather application as there would be a decrease in softness, drape, strength, dry cleanability/washability, light fatness, perspiration resistance, etc. Hence, to achieve garment leather from vegetable tanning process three aspects of concern are a) loading/fullness, b) strength and c) softness.

As a general practice vegetable tanning process employs 20–25% of tannins on the pelt weight.[8,9] This process is then followed by stripping of some vegetable tannin already offered in tanning.[9] A survey of literature indicates that there has been no critical analysis on the offer of vegetable tannins for varied applications and this could be related to the loading factor.[9-11] The enhanced loading of leather with vegetable tannins reduces the softness and strength characteristics. Further, specific properties like perspiration resistance needs to be built into the vegetable tanning process by way of combination tanning systems either during tanning or as a post tanning system. It is known from previous studies that combination tanning system based on vegetable tannins and aldehydes results with higher shrinkage temperature, perspiration resistance, etc.[12]

The main objective of the present study is to avoid the use of metal ions to produce garment leathers. Keeping in view the growing concern on aldehydes and polymers, vegetable tannins have been chosen for tanning trials. An evaluation of the offer of tannin, *vis-à-vis* the physical, organoleptic and environmental characteristics form the yet another objective of the present study. The offers of retanning and fatliquoring have also been optimized on the basis of physical and organoleptic properties of the final leathers. The choice of retanning systems and fatliquors are based on commercial products for which adequate literature is available.

EXPERIMENTAL

Materials and Methods

The wet salted sheepskins were chosen as a raw material for the manufacture of vegetable tanned garment leather. All the chemicals used for leather processing were of commercial grade, while the chemicals used for analysis of spent liquors were of analytical grade. Wattle tanning agent used in this study was procured from Tanganyika Wattle Co. Ltd., Tanzania. The skins were processed conventionally from soaking to depickling and the depickled pelts (pH 4.2–4.5) were employed for various tanning experiments. The process adopted for the manufacture of vegetable tanned garment leathers is presented in Table I.

Optimization of Offer of Vegetable Tannins

Vegetable tannin was offered as 6, 8, 10, 12, 15 and 20%, respectively on pelt weight. The skins tanned with the higher offer of vegetable tannins such as 12, 15 and 20% were subjected to stripping operation with minimum amount of stripping agents as shown in the Table I. The leathers were then bleached, retanned and fatliquored. Polymeric syntan 2% and high molecular weight alkanol ester based syntan 3% were employed for retanning. Fatliquoring was carried out using 5% sulfochlorinated paraffin waxes based, 4% ester sulphonates based, 6% anionic emulsion of synthetic fats based and 5% sulfoester derivatives based fatliquors. The leathers were then set, dried, staked, buffed and trimmed. Finally, the leathers were dry drummed for 6 hrs. Then the leathers were characterized for physical strength and assessed for organoleptic properties by three experienced tanners and the results reported as an average.

Optimization of Retanning and Fatliquoring Systems

The depickled pelts were tanned with the optimized amount of vegetable tannins and three different retanning and fatliquoring systems were employed, as presented in Fig. 1. The leathers were assessed for physical and organoleptic properties.

Studies on the effect of Other Tanning Agents

Two synthetic tanning agents based on Tetrakis hydroxymethyl phosphonium sulfate (THPS) (1.5%) and poly aldehydes (2%) have been used in this study in addition to an offer of 8% of wattle (Tanwat GS Mimosa). Comparative assessments were made against chrome and semi-chrome tanned garment leathers from commercial sources. Effects of drumming time and ageing on the penetration of vegetable tannins were also studied. The size of the particles present in vegetable tanning liquor after 10 minutes and depth of penetration of vegetable tannins with time and ageing were chosen as parameters.

Physical Testing and Hand Evaluation of Leathers

Samples for various physical tests from experimental and control crust leathers were obtained as per IUP method.[13]

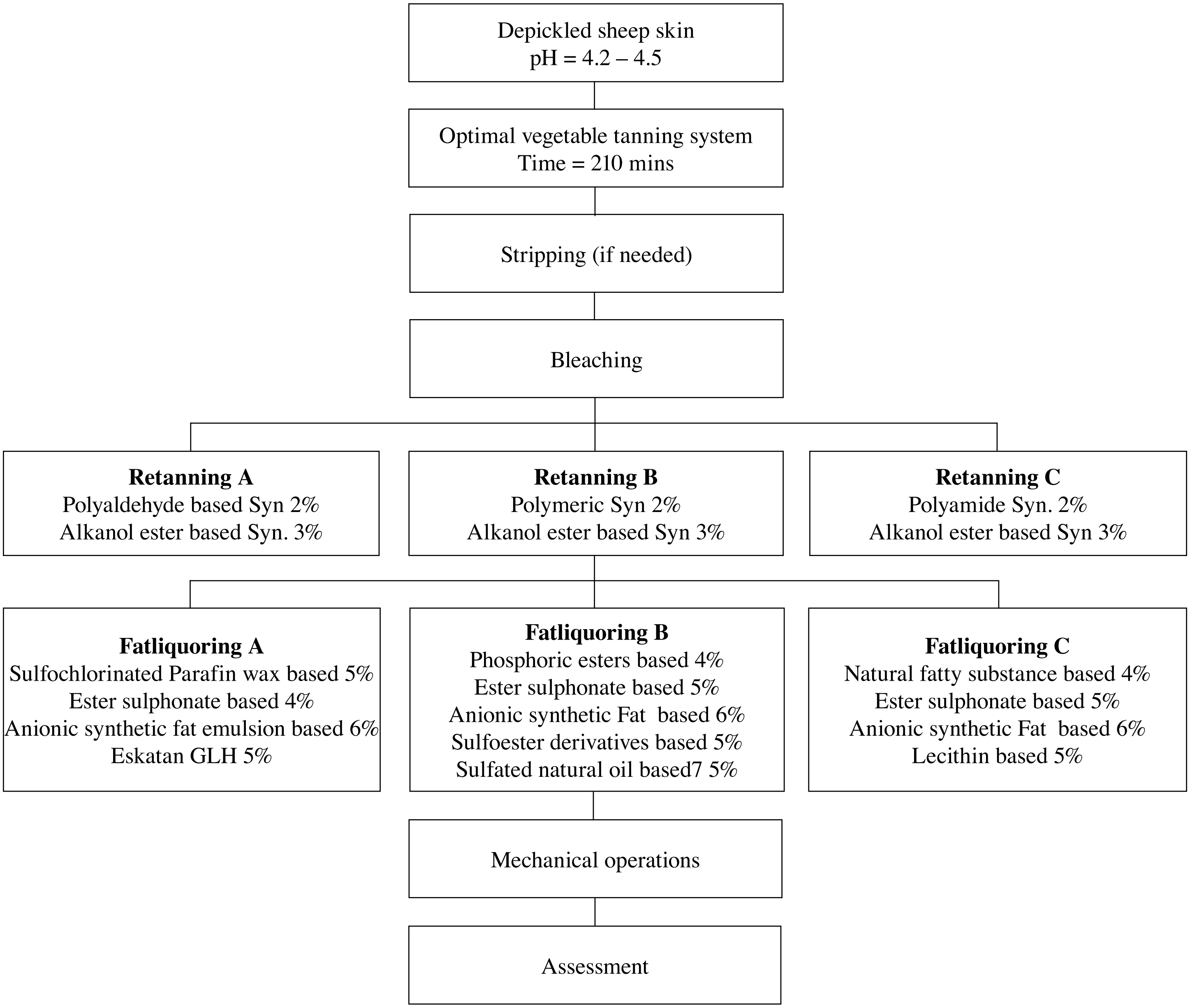

Figure 1. Flow chart for the variations in retanning and fatliquoring systems adopted in the study

Specimens were conditioned at 80±4°F and 65±2% R.H. over a period of 48 hrs.[13] Physical properties such as tensile strength, % elongation at break and tear strength were examined as per the standard procedures.[14,15] The reported values are average of three readings. Crust leathers were assessed for softness, fullness, fluffiness, grain smoothness and general appearance by hand and visual examination. The leathers were rated on a scale of 1–10 points for each functional property by three experienced tanners, where higher points indicate better property.

Objective Assessment of Softness through Compressibility Measurements

Softness of leathers can be numerically measured based on their compressibility.[16] Circular leather pieces (2 cm^2 area) from experimental and control crust leathers were obtained as per IULTCS method[14] and conditioned at 80±4°F and 65±2% relative humidity over a period of 48 hrs.[13] The samples were spread uniformly over the solid base of the compressibility and resilience (C & R) tester. The initial load acting on the grain surface was 100 g. The thickness at this load was measured 60 seconds after the load was applied. Subsequent loads were added (100, 200, 200, 300, 300, 100, 100, 400, 400, 200, 200 g) and the change in thickness was recorded one minute after the addition of each load. Logarithm of change in leather thickness (Y axis) was plotted against logarithm of load (X axis) and $\tan^{-1}$ of the slope is calculated as the negative slope angle. The reported values are average of three readings.

TABLE I

Vegetable tanning process for the making of sheep garment leathers

Process/chemicals	Percentage	Duration	Remarks
Soaking			Conventional soaking was followed.
Paint liming		12 hrs	
Water	20		
Lime	5		
Sodium sulphide	1.5		Skins were kept overnight and unhaired on the next day conventionally.
Unhairing enzyme	0.5		
Reliming		4 days	Skins were handled thrice a day and on the 5th day the skins were subjected to fleshing operation. The percentage chemicals were based on the fleshed weight.
Lime	10		
Water	300		
Deliming			
Ammonium chloride	1		pH: 8.0–8.5
Water	150	1 hr	Washing was done.
Bating			
Bating enzyme	1	1 hr	Washed after completion of the process.
Degreasing			
Degreasing agent	2	1½ hr	Washing was done.
Pickling			
Water	100		
Salt	10	30 min	Acid was given in 3 feeds at 10 min interval.
Sulphuric acid	1	1 hr	pH: 2.5–3.0
Depickling			Given in feeds upto the pH: 4.0–4.5
Sodium bicarbonate	0.5	45 min	
Vegetable tanning			
Pretanning agent	2		End pH: 3.0–3.5.
Wattle Powder	8 to 20%	210 min	Pile overnight.
Stripping*			
Borax	0.25		Chemicals offered on sammed wt basis.
Sodium sulphite	0.25	45 min	Washing was done.
Bleaching			
Bleaching syntan	2		
Oxalic acid	1	45 min	Washing was done.
Retanning	5	1½ hrs	Variation in Retanning system.
Fatliquoring	20	3 hrs	Variation in fatliquoring system.
Fixing			
Formic acid	2	30 min	Given in feeds.

*Only for 12 - 20% offer of tannins (The chemical bases of the commercial products mentioned are listed in Annexure I)

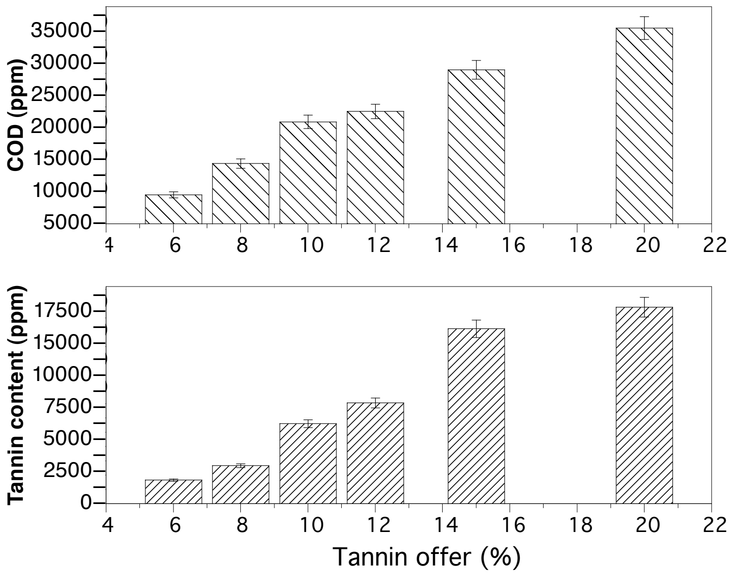

Figure 2. Variation in the tannins and COD in the spent vegetable tan liquors

Analysis of Spent Liquors

The spent vegetable tanning liquors as well as spent tanning liquors after stripping were analysed for chemical oxygen demand (COD) and tannin content. The COD was determined by the dichromate method and tannins using folin-ciocalteu agent method.[17,18] The reported values are average of three readings. The size of the particles present in vegetable tan liquor was measured in a Zetasizer 3000HSA using light scattering and the technique of photon correlation spectroscopy (PCS).

RESULTS AND DISCUSSIONS

The use of vegetable tannins like tara for upholstery, strap/case leather is popular. The inability of vegetable tanning process to enable the production of garment leathers is related to the high quantities of vegetable tannins employed. Several of the properties desired of garment leathers can be achieved by proper choice of raw material, tanning, retanning and fatliquoring. The processes prior to tanning have been designed to obtain a desired level of fibre opening and clean up of the pelt.

Optimization of Vegetable Tannin Offer

Conventional leather processing was dependent on the concentration gradient of vegetable tannins for penetration of tannins into the skins. The availability of sulfited tannins and pretanning agents, which provide the desired levels of penetration and uniformity of tanning, has opened up avenues for a relook into the offer of vegetable tannins. In the conventional tanning, the tanning is followed up by stripping, which removes the excess or low quality tannins. Other aspects related to stripping are the breakdown of collagen-tannin bond, providing uniform color, etc.[9] Further, vegetable tannins are hard biodegradables; meaning higher inputs into the pollution control devices.[19] The higher offers of tannins result in higher tannin input for effluent treatment plant (ETP). Stripping adds further load to the ETP. In this study, the offer of vegetable tannins was varied from 6-20% and the influence of the same (after the common post-tanning) on the properties of leather and wastewater has been analyzed.

TABLE II

Physical characteristics and organoleptic properties* of leathers tanned with varying percentage of vegetable tannins

Parameter	Offer of Tannins (%)					
	6	8	10	12	15	20
Shrinkage Temperature (°C)	78	80	81	83	84	86
Tensile strength (kg/cm^2)	187	199	178	175	168	151
Tear strength (kg/cm)	25	39	35	32	27	23
% elongation at break	40	46	62	60	47	41
Softness*	9	9	8	9	8	9
Fluffiness*	9	9	8	8	8	9
Fullness*	8	9	9	9	8	8
Grain Smoothness*	9	9	8	9	9	9
General Appearance*	9	9	9	9	8	9

Rating on a Scale of 1–10 with 10 being the best

Table II shows the effect of vegetable tannin offer on various physical properties of leather. The shrinkage temperature as expected increases with the offer of vegetable tannins However, the variation in the shrinkage temperature when the offer of vegetable tannins is increased from 6–20% is only 8±1°C. The tensile strength and the tear strength were optimal at 8% offer of wattle, which then decreased with tannin offer. A 24% decrease in tensile strength and 41% in tear strength was observed when tannin offer was increased from 8 to 20%. Another significant observation in this study is the decrease in tensile and tear strength properties as the tannin offer is increased from 8–10%. For achieving desired properties for garment applications, loading of skin has to be avoided. Therefore, 8% tannin offer was taken as optimal. Organoleptic properties of the leathers have been computed on a scale of 1–10 with 1 being the lowest. The results are presented in Table II. The tanners evaluated the leathers with 8% offer of wattle better in all properties compared to other leathers.

Fig. 2 provides the amount of chemical oxygen demand (COD) and tannins present in wastewater in relation with the tannin offer. An increase of 59% COD load and 81% tannins in wastewater has been observed as the tanning offer was varied from 8% to 20%. These results when viewed in conjunction with the observed results on physical and organoleptic properties indicate that higher offer should be avoided in the making of garment leathers. Also, the load due to stripping needs to be calculated to analyze the cumulative effects on the environment.

Effect of Stripping on Effluent Quality

Effect of stripping on leathers tanned with 12–20% wattle needs consideration. The stripping process breaks down the collagen-tannin bonds and hence it is possible that the strength properties decrease. In the absence of breakdown of tannin-collagen bond, the decrease in strength has to be related to the loading by tannins. The stripping process removes the excess and low quality tannins.[9] This results in a significant amount of tannins in wastewater and hence an increased COD load. The results are presented in Table III. It can be seen that tannins discharged by stripping a leather tanned with 20% wattle is 34% higher than that discharged from a tanning process employing 8% wattle. The COD from these processes are above 20,000 ppm, indicating an additional cost for treatment of these wastewaters. Based on the physical, organoleptic and emission loads, the offer of 8% vegetable tannins was considered optimal and employed for further studies.

Studies on the Effect of Other Tanning Agents

Effect of adding other tanning agents such as THPS and polyaldehyde syntans on the size of particles of tannins have been studied. It had been observed that in a wattle alone system, 55.4% of particles had a volume average diameter of 1199 nm while the same decreased considerably when combined with THPS and polyaldehyde. In the case of Vegetable tannin-THPS, 65% of the particles had a volume average diameter 32 nm and in the case of polyaldehyde, 72% of the particles had only 38 nm size. This reduction in size of the particles is possibly due to the fact that the tannins-THPS and tannins-polyaldehyde reaction prevented the agglomeration of tannins.

TABLE III

Influence of Stripping on the COD and tannin load

Wattle Offer (%)	12	15	20
COD (ppm)	2340	3120	4680
Tannins (ppm)	3755	4703	5089

At an offer of 8% of vegetable tannins the penetration of tannins in to the middle layers is not complete even after 4 hrs of drumming. Hence, the influence of drumming time and ageing on penetration of tannins has been studied. It has been observed that a complete penetration of tannins has been observed for a drumming time of 8 hours. A complete penetration of tannins is also observed by drumming the leathers for 2 hrs and dipping them overnight (12 hours) in vegetable tannin liquor. A complete penetration is further possible when skins drummed in wattle liquor for 4 hrs is aged in wet condition for 48 hrs. This penetration is possibly promoted by the concentration gradient of tannins that existed between an un-penetrated layer and the penetrated one. In all further experiments the ageing of vegetable tanned leathers for 48 hrs after tanning was adopted. A complete penetration by the process is further justified from the results of the tear strength carried out after 2 months. Negligible variation in tear strength (less than 2%) has been observed indicating a stable tanning. The organoleptic properties and physical properties of the leathers from combination tanning systems (THPS-Vegetable Tannin and Polyaldehyde-Vegetable Tannin) is comparable with that of 8% tannin alone system. Hence, for further studies vegetable tanning alone has been employed.

Optimization of Retanning

The choice of retanning agents for vegetable tanned garment leather applications has been designed to achieve necessary degree of perspiration resistance, rub fastness and uptake of fatliquors without additional loading on the fibers. The retanning agents employed during the optimization of vegetable tannin offer have been designed with an aldehyde syntan (Retan system A); a polymeric composition (Retan system B), which provides lubrication and lesser loading on

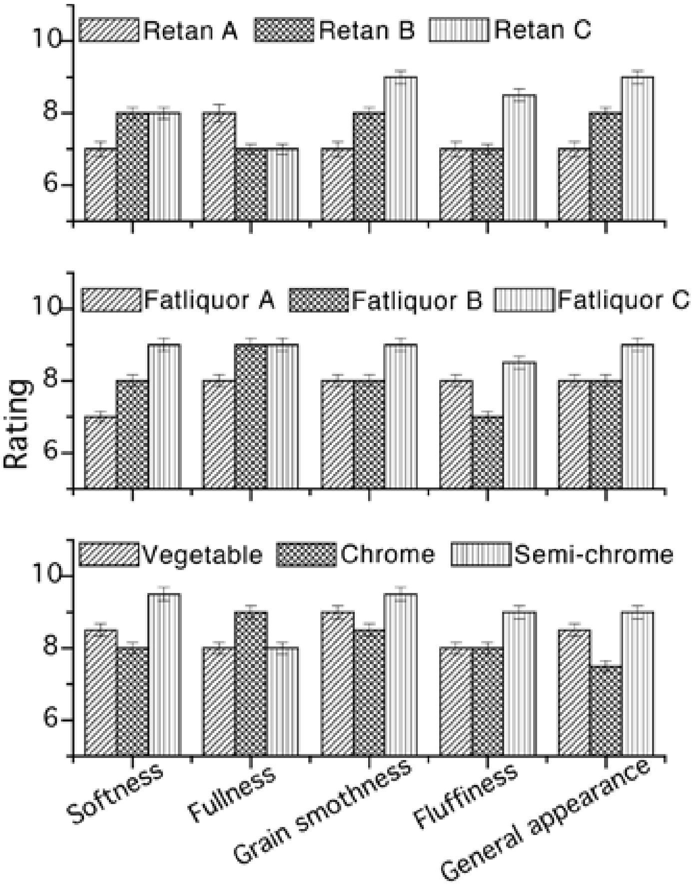

Figure 3. Influence of retanning and fatliquoring agents on the organoleptic properties of resultant garment leathers and comparison of organoleptic properties between vegetable, semi-chrome and full chrome garment leathers

the fibers; polyamide syntan based (Retan system C); along with an ester based syntan. A total retanning of 5% has been offered, while maintaining 3% offer of ester based product as constant. This syntan is reported to provide enhanced tear strength. In all the three systems the strength properties are higher than that required for garment leathers (120 kg/cm^2) as seen from Table IV. However, the Retan system C based on polyamide syntan offered higher tensile and tear strengths than aldehyde based Retan system A and polymeric based Retan system B.

The organoleptic properties of the garment leathers are presented in the form of a graph in Fig. 3. It could be observed from the figure that all values obtained for Retan system C are comparable to that of leathers from that processed using other Retan systems A and B. However, the fullness as desired is lower for a polyamide based retanning. Therefore a polyamide-based system has been taken as optimal. Further, the softness of the leathers was measured by compressibility method. The negative slope angles of 14.6, 8.9 and 15.1 have been observed for retanning system A, B and C respectively. A higher negative slope angle indicates greater softness. Accordingly, Retan system C is better, hence was chosen as an optimal retanning combination.

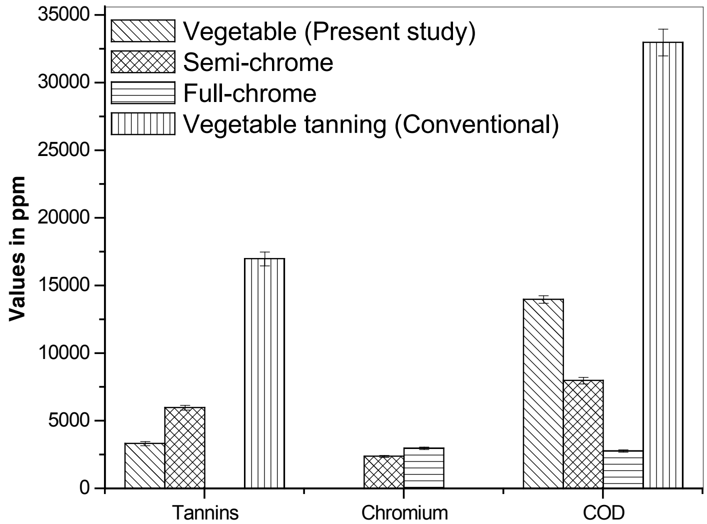

Figure 4. Characteristics of spent tanning liquors from vegetable, semi-chrome and full-chrome garment liquors

Optimization of Fatliquoring System

Garment leathers require good lubrication, grain smoothness and softness without loading of the fibres. While a 100% use of synthetic fatliquors would result in less loading of the fibres, it would also result in a dry feel. Hence, various fatliquoring systems adopted in this study have aimed in employing varying combinations of synthetic and natural fatliquors, such that the ratio of natural to synthetic fatliquor would be 0:100, 25:75 and 50:50. In order to understand the influence of varying combinations of fatliquors, 50% of the total fatliquors were maintained constant. Vegetable tanned leathers (with 8% vegetable tannin offer), retanned with a Retanning system C (Tergotan EF), and fatliquored with three fatliquoring systems. The organoleptic properties are presented in Fig. 3. The physical characteristics of leathers are presented in Table IV.

It can be seen that 50:50 combination of synthetic and natural fatliquors provide good softness, coupled with desired levels of grain smoothness and general features without any major alterations in the tensile and tear strengths. Predominantly, the tensile and tear strengths have remained more or less constant except in the case where synthetic fatliquors have been employed to the extent of 100%. The softness of the leathers has been measured by compressibility method. The negative slope angle values for the three fatliquoring systems are 10.2, 10.9 and 18.4 for fatliquoring system A, fatliquoring system B and fatliquoring system C, respectively. Thus a comparative assessment of the three fatliquoring systems based on organoleptic, physical and softness measurements indicates that the fatliquoring system C having the natural to synthetic fatliquor ratio of 50:50 has given better properties when compared to other fatliquoring systems.

TABLE IV

Influence of retanning and fatliquoring agents on the tear and tensile strength of the final vegetable tanned leathers

Retanning System	Tear Strength (Kg/cm)	Tensile Strength (Kg/cm^2)	Elongation at break (%)
Retan A	36	190	36
Retan B	32	152	41
Retan C	44	214	35
Fatliquor A	32	190	68
Fatliquor B	32	205	65
Fatliquor C	38	208	62

A comparison of leathers made from optimized tanning-retanning-fatliquoring system with semi-chrome and full chrome garment leathers have been carried out. Organoleptic properties, strength characteristics and environmental characteristics have been evaluated. The organoleptic properties of the leathers are given in Fig. 3. From the figure it can be seen that the organoleptic properties are comparable with chrome garment leathers, in terms of softness and general appearance and superior to semi-chrome garment leathers. Interestingly, the fullness of the chrome tanned leathers and vegetable tanned leathers are similar, indicating that the optimized process did not load the leathers. The process developed in this study thus indicates a possibility to produce garment leathers without employing metal ions.

The characteristics of the spent full chrome, semi-chrome and vegetable tanning wastewaters are presented in Fig. 4. It is observed that the tannin load of the wastewater is 44% higher for semi-chrome than the optimized vegetable tanning process. The 8000 ppm COD observed for semi-chrome process is after the chrome tanning of vegetable tanned leathers. Hence, this value needs to be coupled to the 33000 ppm of COD observable for vegetable tanning process prior to the offer of chromium. Thus the present study provides an efficient and environmentally benign route for making garment leathers employing vegetable tanning process.

CONCLUSION

A method for replacing chromium and other metal ions along with polymers in tanning, especially for garment leathers has been demonstrated in the present work. While, vegetable tanning methods offer the most viable environmentally benign methodology, the indiscriminate use of higher quantities of tannins is of concern. The present study focused on the optimization of vegetable tanning system with low offer (8% as wattle) of tannin as against the conventional vegetable tanning (20–25%). The drawbacks of stripping and the emission loads due to such systems have been quantified and highlighted. Conditions for complete penetration of tannins, achieving good softness coupled with necessary strength and perspiration resistance have been optimized. The optimized tanning—retanning—fatliquoring process results in leathers comparable in properties to chrome tanned garment leathers.

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NOTE

List of Commercial Retanning agents

1. **Tergotan EF** (Clariant India Ltd.)—polyamide based syntan.
2. **Sellatan CF** (TFL India Ltd.)—polyaldehyde based syntan.
3. **Tergotan GSI** (Clariant India Ltd.)—Polymeric syntan
4. **Tannit LSW** (Dr. Th.Bohme chemicals)—high molecular alkanol esters and modified ethyleneoxide adducts.

List of Commercial Fatliquors

1. **Cutapol WK** (Dr. Th.Bohme chemicals)—ester sulphonates, alkyl ammonium esters, ethyleneoxide adducts and higher molecular aliphatic hydrocarbons.
2. **Eskatan GLH** (Dr. Th.Bohme chemicals)—sulfoester derivatives and high molecular aliphatic hydrocarbons.
3. **Dermalix SFL** (Clariant India Ltd.)—anionic emulsion based on synthetic fats.
4. **Dermalix CFL** (Clariant India Ltd.)—sulfochlorinated paraffin waxes.
5. **Coripol MB** (TFL India Ltd.)—combination of selected natural fatty substances.
6. **Coripol EF** (TFL India Ltd.)—phosphoric esters.
7. **Remsol ALM7** (Hodgson chemicals Ltd.)—sulfated natural oil and phospholipids with synthetic softening agents.
8. **Cremol L57** (Hodgson chemicals Ltd.)—blends of solvent and lecithin.