

Zinc-Vegetable Bark Extract Combination Tanning System for Leather Production

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ABSTRACT: Interest in clean technologies and standards established by regulations, led tanners to increase its efforts to develop new processes that add the qualities that already have the traditional tanning, such as hydrothermal high stability, good dyeing characteristics and smoothness. The shrinkage temperature has been used to measure the degree of tannage as a means of comparison of the different types of tannages. The shrinkage temperature is one of the properties, and probably the most important, that has been used lately in the study of the combination tannages. When vegetable tannins are combined with metallic salts (not including chromium) a combination tanned leather is produced with good resistance to hydrothermal shrinkage. This is an example of synergy, when the total effect is greater than the sum of the parts. In this study, combination tanning process based on Sunt (garad) bark extract–Zinc sulphate tannage for the production of upper leathers is presented. The bark powder of *Acacia nilotica* (Sunt bark) from Sudan has been utilized in the combination tanning system with zinc sulphate. Both tanning methodologies Sunt bark extract followed by Zinc sulphate (Sunt bark-Zinc) and Zinc sulphate followed by Sunt bark extract (Zinc- Sunt bark) have been attempted. All combinations tanning systems resulted in leathers with shrinkage temperature above 90°C. However, Zinc- Sunt bark leathers tanned using 9% Zinc sulphate; followed by 20% Sunt bark resulted in shrinkage temperature of 95°C. It is seen that combination tanning using Sunt bark (20%) followed by Zinc (9% Zinc sulphate) resulted in leathers with shrinkage temperature of 99°C, which is 15°C more than the control (Sunt bark tanned) leathers. The spent tan liquor analysis shows significant reduction in COD and TDS loads compared to a conventional vegetable tannage. Sunt bark-Zinc combination system resulted in leathers with good organoleptic and strength properties. The combination tanning using Sunt bark and Zinc sulphate presented in this work proved to be a preferred alternative to chrome tanning.

KEYWORDS: Sunt bark, Garad bark, Zinc sulphate, Combination tannage.

I. INTRODUCTION

Chrome leathers offer excellent hydrothermal stability, mechanical properties and good dyeing characteristics. However; the negative side of chromium tannage is that only 60-65% is taken up by the leather [1] and hence, the remaining chromium constitutes a major component of the effluent. The discharged chromium may have adverse effects on the environment, [2] it has been reported that Cr (VI) which may be formed at various stages of leather processing is a carcinogen [3-5]. Chromium is toxic to plant growth at higher concentrations [6,7] and chrome tanning effluents are toxic to aquatic life and may also pollute ground water [8].

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When certain polyphenols and polycarboxyl group compounds are combined with metallic salts (not including chromium) a combination tanned leather is produced with good resistance to hydrothermal shrinkage and a “chemical effect” near to chrome tanned leather [9]. Also able to produce leather having higher hydrothermal stability by using some metallic salts such as Al, Co, Mg, Ni, Fe and Zn together with vegetable tanning agents [10-12]. The work with Al-vegetable tannins has been extensively reported [13].

Zinc is a necessary trace element for living organisms. It affects protein and mineral metabolism, improves digestion and appetite of organisms and contributes to the immune and genetic system [14,15]. Amino acid chelated zinc compounds have been widely studied in pharmacy and nutrition for their biological activities. The effect of zinc-tanning has been previously tested research [16]. Biological researches found that zinc ions and short peptide segments, which compose special structural domain of biomacromolecules, form a stable combination under certain conditions [17, 18]. It has the characteristic of maintaining the stability of the structure of polypeptide. Zinc as a tanning agent has been little explored, it has been used as a preservative in curing and a few patents regarding its use exist [19]. A vegetable-zinc combination tannage has given shrinkage temperatures close to 100°C [20]. A tanning agent based on an aluminum-zinc combination has been shown to increase the shrinkage temperature of leather above 90°C, [21] more recently, zinc in combination with chromium has been used to produce leathers with shrinkage temperatures above 100°C [22]. Leather processed with the chrome-zinc tanning agent shows the zinc imparts tremendous physical strength and enhances the grain smoothness of the leather [23]. Zinc can effectively improve the crosslinking degree of collagen fiber by fully using the amino groups, and the crosslinking of collagen fiber and zinc promotes the combination of chromium and collagen through the more compact state (Fig. 1) [24].

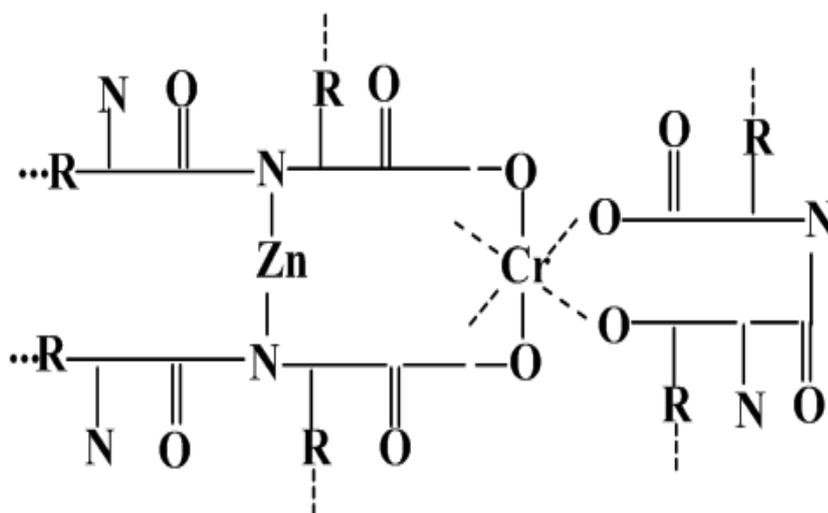


Fig. 1: Model of Zn-Cr combination tanning structure [24].

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The Zinc tanning agent could produce white leathers with properties comparable to the chrome tanning agent, and the tanning agents based on Zinc salts were prepared using different masking agents such as sodium formate, sodium acetate, sodium tartrate and potassium hydrogen phthalate. The selection of the zinc tanning agents has been optimized based on the properties like hydrothermal stability and percentage exhaustion of zinc. The results indicated that zinc sulfate can be used as tanning agent under acidic conditions (50g/L, pH=3, 25°C) and is an effective and superior method. Moreover, Sodium citrate (0.1mol/L) was selected as masking agent compared with other masking agents. The hydrothermal stability was improved effectively with the action of masking agent [16].

Bio-active ingredients in the form of tannins are present in some of the plant materials capable of imparting tanning effect. Vegetable tanning is one of the traditional and eco-friendly processes in leather making involving plant materials [25]. The vegetable tannins are water-soluble polyphenolic compounds having molecular weight in the range of 500 –3000 Daltons [26,27]. Based on their chemical structure, the vegetable tannins are classified as Hydrolysable type (e.g. Myrobalan) and Condensed type (e.g. Wattle). *Acacia nilotica* (Sunt in Arabic) is a member of the subfamily Mimosoideae of Leguminous trees [28]. The tree is characterized by the long white spines, yellow head inflorescence and the gray necklace-like pods [29]. It has multiple uses in Africa and many Arab countries because it is abundant [30]. Sunt is traditionally used for tanning in tropical Africa in a primitive way, and is one of the most important tanning materials in Northern India [31, 32]. Although both pods and bark of *Acacia nilotica* contain high concentrations of tannins, the tannin percent using pods extract is more efficient than that extracted from the bark, and ranges from 26 - 70% [29, 30, 31, 33, 34].

In tropical and subtropical areas of Africa and Asia, the acacias are one of the most important tannin-bearing trees. Several species, such as *Acacia Arabica*, *A. nilotica* and *A. adamsonia*, have supplied pods and bark since immemorial times. The leather produced by acacia pods is soft, plump, light colored and durable, and it can be readily dyed. The acacia pods and bark are known variously in the countries where they grow as babul (hindustan), babar (Sind) garad or sunt (Sudan), neb neb (*West Africa*) and gabarua (Nigeria). According to the conditions of the soil and the climate, the tannin-content of the pods varies from 20 to 30%. The material contains an undue proportion of nontans and a high proportion of sugary matter in the seeds. This results in a rapid fermentation of the liquor [35]. Our aim is to utilize the garad bark extract in combination tannage with zinc sulphate for production of shoe upper leathers, since the garad bark extract contains natural products of relatively high molecular weight which have the ability to complex strongly with collagen.

II. MATERIALS AND METHODS

Materials

Conventionally processed pickled goat skins were taken for the combination tanning trials. Garad (Sunt) barks were sourced from Sudan. Zinc sulphate heptahydrate and chemicals used for post tanning were of commercial grade. Chemicals used for the analysis of spent liquor were of analytical quality.

Tanning Trials

The tanning experiments were carried out on pickled goat skins. Experimental combination tanning trial zinc sulphate followed by garad (Zinc-garad) were carried out as per the process mentioned in Table 1 and combination tanning based on garad followed by zinc sulphate (Garad -Zinc) was carried out as per the process mentioned in Table 2. Control garad tanning trial was carried out as per process given in Table 3. Both experimental and control leathers were processed into upper crusts following the post-tanning process mentioned in Table 4.

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Determination of Shrinkage Temperature

The shrinkage temperature of both control and experimental leathers were determined using the Theis shrinkage tester [36]. A 2 cm sample, cut out from the leather was clamped between the jaws of the clamp, which in turn was immersed in a solution of glycerol: water mixture (3:1). The solution was stirred using mechanical stirrer attached with the shrinkage tester. The temperature of the solution was gradually increased and the temperature at which the sample shrinks was noted. Triplicates were carried out for each sample and the average values are reported.

Visual Assessment of the Crust Leather

Experimental and control crust leathers were assessed for softness, fullness, grain smoothness, grain tightness (break), general appearance and dye uniformity by hand and visual examination. Three experienced tanners rated the leathers on a scale of 0-10 points for each functional property, where higher points indicate better property.

Table 1 Formulation of Zinc-Garad Combination Tanning Process for Goat Pickled Skins

Process	%	Product	Duration (min)	Remarks
Zinc sulphate tanning	100	water		
	8	salt	10 min	2.8-3
	9	ZnSO ₄ .7H ₂ O	90	
Adjustment of pH	1	Sodium Bicarbonate	3×15+30	pH 4.5-4.7
Vegetable tanning	2	Phenolic Syntan	30	
	10	Garad bark extract	120	
	10	Garad bark extract	120	Check penetration
Fixing	0.5	Formic acid	3×15+30	Check pH to be 3.5, Drain/Wash/drain and pile overnight, next day sammed and shaved to 1.2 mm thickness. The shaved weight noted

Physical Testing

Samples for various physical tests from experimental and control crust leathers were obtained as per IULTCS methods [37]. Specimens were conditioned at 20 ± 2 °C and 65 ± 2 % R.H over a period of 48 hrs. Physical properties such as tensile strength, percentage elongation at break, [38] grain crack strength [39] and tear strength [40] were measured as per standard procedures. Each value reported is an average of four (2 along the backbone, 2 across the back bone) samples.

Analysis of Spent Liquor

The spent liquor from control and experimental tanning processing were collected, filtered and analyzed for chemical oxygen demand (COD), Biochemical oxygen demand (BOD₅), and total Dissolve solids (TDS) as per standard procedures [41].

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Table 2 Formulation of Garad-Zinc Combination Tanning Process for Goat Pickled Skin

Process	%	Product	Duration (min)	Remarks
Adjustment of pH	100	water		
	8	salt	10 min	
	1	Sodium Bicarbonate	3×15+30	pH 4.5-4.7
Tanning	2	Phenolic Syntan	30	
	10	Garad bark extract	120	
	10	Garad bark extract	120	Check penetration
	0.25	Formic acid	3×15+30	pH 2.8-3
Basification	9	ZnSO ₄ .7H ₂ O	90	
	0.75	Sodium bicarbonate	3×15+30	Check pH to be 4, Drain/Wash/drain and pile overnight, next day sammed and shaved to 1.2 mm thickness

Table 3 Formulation of Garad Tanning Process (Control) for Goat Pickled Skin

Process	%	Product	Duration (min)	Remarks
Adjustment of pH	100	water		
	8	salt	10 min	
	1	Sodium Bicarbonate	3×30+30	pH 4.5-4.7
Tanning	2	Phenolic Syntan	10	
	10	Garad bark extract	120	
	10	Garad bark extract	120	Check penetration
Fixing	0.25	Formic acid	3 × 10 + 30	pH 3.5
Washing	300	Water	10	Check the pH to be 3.5. Drain the bath and pile overnight. Next day sammed and shaved to 1.2 mm. The shaved weight noted

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Table 4 Formulation of Post-tanning Process for Making Crust Leathers

Process	%	Product	Duration (min)	Remarks
Washing	200	Water	10	
Neutralization	0.75	Sodium bicarbonate	3 × 15	pH 5-5.5
Retanning	100	Water		
	8	Synthetic tannin (Syntan)	90	
Fatliquoring	9	Synthetic fatliquor	40	
Dyeing	3	Acid dye brown	30	
Fixing	1	Formic acid	3 × 10 + 30	pH 3.5

Chemical Analysis of Leathers

The chemical analysis was carried out for control and experimental leathers according to the standard procedures [42] for total ash content, % moisture, % oils and fats, % water soluble, % hide substance, % insoluble ash and degree of tannage. Triplicates were carried out for each sample and the average values are reported.

III. RESULTS AND DISCUSSION

Combination tanning trials using garad bark extract and zinc sulphate were carried out with 9% offer of zinc sulphate and 20% offer of garad bark extract. The shrinkage temperature data of leathers tanned with Garad-Zinc and Zinc-Garad combination along with garad control is given in Table 5. From the table it is seen that both the combination resulted in leathers with good shrinkage temperature. The shrinkage temperature of leathers obtained from Garad-Zinc combination tanning is slightly higher than Zinc-Garad. However, Garad-Zinc combination tanning resulted in leathers with shrinkage temperature close to 100°C, which are better than control leathers from Garad of Ts 84°C.

Table 5 Shrinkage Temperature of Crust Leathers for Experimental and Control

Experiment	Shrinkage temperature, Ts (°C)
Zinc - Garad	95±1
Garad - Zinc	99±2
Garad (Control)	84±0.5

Ecological Impact - Spent Liquor Analysis

The spent tan liquor contains highly organic matter in both control and experimental process liquor and it contributes to extremely high COD, dissolved and suspended solids. Hence, it is vital to assess the environmental impact from control and experimental tanning process. The COD, BOD₅, and TDS of the spent liquor for experimental and control trials have been determined and are given in Table 6. From the table, it is observed that the COD, BOD₅ and TDS of the spent liquor processed using both the experimental tanning system are lower than the spent liquor from Garad tanning (control). The BOD₅ and TDS of the spent liquor processed from Garad and Zinc combination tanning trials have significantly reduced compared to the spent liquor of control Garad tanning trial.

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Table 6 Characteristic of Spent Liquor for Experimental and Control

Experiment	COD (mg/l)	BOD ₅ (mg/l)	TDS(mg/l)
Garad (control)	114800±2660	27800±1200	99850±1250
Zinc- Garad	101500±1250	18500±1200	78500±1200
Garad-Zinc	95500±1650	15450±850	65650±1350

Tactile Properties of Crust Leathers for Experimental and Control

The organoleptic properties (visual assessment) of crust leathers for experimental and control are given in Fig. 2. (Higher numbers indicate superior properties). From the figure, it is observed that crust leathers processed by experimental combination tanning system exhibited good softness, fullness, smoothness, general appearance and dye uniformity compared to control leathers from Garad tannage. The organoleptic properties of the Garad-Zinc crust leathers are slightly better compared to Zinc-Garad crust leathers.

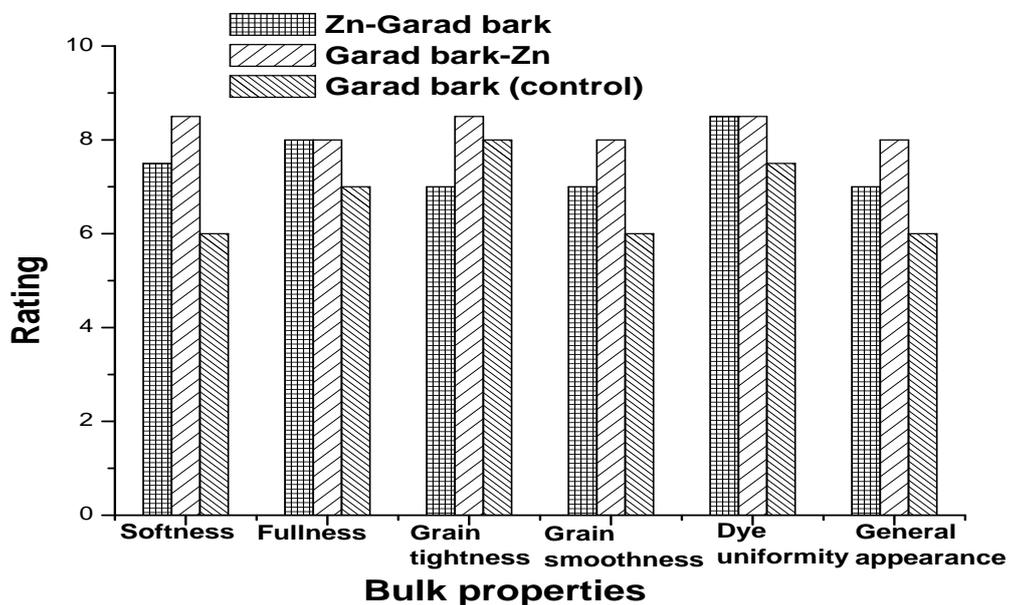


Fig. 2 Graphical representation of organoleptic properties of the Experimental and control leather

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Strength Characteristics of Experimental and Control Crust Leathers

The physical strength measurements of experimental and control leathers are given in Table 7. The physical strength measurements viz., tensile strength, tear strength has been found to be better for experimental leathers. The experimental Garad – Zinc tanning system resulted in leathers with good tensile and tear strength characteristics. The values for load at grain crack for both experimental and control leathers were comparable. All the physical strength parameters for both control and experimental leathers are found to exhibit the requirement of standards. It is seen that the softness of experimental leathers are better than that of the garad control leathers.

Chemical Analysis of the Crust Leather

The chemical analysis values of experimental crust leathers (Zinc - Garad and Garad - Zinc) and control (Garad) are given in Table 8. The chemical analysis data for the experimental leathers is comparable to that of control leathers. However, the water soluble matter for the control leathers is more compared to the experimental leathers.

Table 7 Physical Strength Characteristics of Experimental and Control Crust Leathers

Parameter	Zinc-Garad	Garad-Zinc	Garad (control)
Tensile strength (Kg/cm ²)	225±2	248±2	212±2
Elongation at break (%)	52±1.40	57±1.50	42±1.6
Tear strength (Kg/cm)	51±1.50	62±1.50	48±0.70
Load at grain crack (kg)	25±0.52	28±0.52	24±0.52
Distention at grain crack (mm)	11±1.28	12±1.35	10±0.7

Table 8 Chemical Analysis of Experimental and Control Crust Leathers

Parameter	Garad bark (control)	Zinc–Garad bark	Garad bark-Zinc
Moisture %	14.2	12.55	13.10
Total ash content %	2.8	2.35	2.65
Fats and oils %	3.3	2.95	3.20
Water soluble matter %	4.8	3.55	3.30
Hide substance %	51	52.50	51.40
Insoluble ash %	1.3	1.25	1.35
Degree of tannage %	49.8	51.81	53.79

IV. CONCLUSION

Tanning is one of the most important processes in leather production, which plays an important role in the change of the rawhide performance. Tanning agents are chelated with the leather collagen in peptide bond so that the stability of the hide can be more firmly. The Zinc tanning agent could produce white leathers with properties comparable to the

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chrome tanning agent. In the present study, an attempt has been made to produce upper leather using combination tanning process based on Garad bark extract and zinc sulphate. It is seen that combination tanning system with 20% Garad extract and 9% Zinc sulphate results in leathers with a maximum shrinkage temperature of 99°C, which is 15°C greater than the control leathers. The physical and chemical analysis indicates that the experimental leathers are comparable to control leathers in terms of all the properties. The Garad -Zinc tanned leathers are softer than control. The bulk properties for the experimental leathers are better than control leathers. One of the main benefits of this work is the lower environmental impact. The spent tan liquor analysis shows significant reductions in COD and TDS loads compared to control. It is possible to manufacture lighter shade leathers from garad - Zinc combination with a shrinkage temperature of 99°C. These results make the Garad-Zinc tanned leather a promising alternative for the production of high quality upper leather'

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