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Reusing Limed Fleshing Wastes As A Fatliquor In Leather Processing

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SULPHATED fatliquor was prepared from fats of sheep limed fleshing wastes to be used on small scale leather processing. The chemical properties of fats were determined before sulphation, thereafter they were neutralized with ammonium hydroxide to produce fatliquor. The chemical characteristics of the prepared fatliquor were then determined. The prepared fatliquor was applied onto light and heavy leather processing. Physical, chemical and organoleptic properties were determined, and scanning electron micrographs of finished leathers were obtained. The results showed that total fat content in sheep limed fleshing wastes constituted about 20% of its initial weight, of which unsaturated fats represented 60% and predominated as oleic acid. This indicates suitability of the prepared fatliquor for sheep leather processing. Additionally, the quality of finished leather processed by this technique is comparable to that obtained using a commercial fish oil-based fatliquor . These results imply that sulphated fatliquor derived from flesh waste can be reliably used in leather manufacturing as an appropriate alternative to traditional fatliquor to reduce tannery pollution and production costs.

Keywords: Fleshing waste, Fat, leather, Tanning, Sulphated, Fatliquor.

Introduction

Leather tanning is the process of transforming skins or hides into leathers by sequential mechanical and chemical steps. In this process, skins or hides pass through three consecutive stages namely; pre-tanning (beamhouse), tanning and post-tanning. The first stage aims to isolate collagen by removing non-collagenous components of skin, hence, collagen fibers stick together and leather becomes stiff. Afterwards, the pelts react with tanning agent, during tanning process, thereby gain its resistance to physical, chemical and biological factors. On the other hand, fats, oils, dyes and re-tanning agents are applied in post-tanning stage to prevent re-sticking of fiber structure during drying, as well to promote its desired softness, handle, feel, water resistance and physical strength [1-3]. However, the tanning process brings about diverse types of liquid, gaseous and solid wastes that lead to the categorization of leather industry as a polluting industry [3].

Fleshing skins or hides is the mechanical beamhouse process during which undesirable fats and meats are removed from the flesh side to facilitate chemicals uptake into pelt. This, in turn, improves diffusion of tanning agents from the flesh side. Therefore, skipping this step during tanning process causes wastage of chemicals and, consequently, good quality leather cannot be produced [1,4,5,6].

Limed fleshing waste is considered one of the non-tanned wastes, which is obtained from hides treated with a high percentage of lime and sodium sulfide. It constitutes about 50-60% of total wastes generated in leather industry [7]. Unfortunately, this waste product is considered unusable and is disposed of through landfill, which represents a major issue in leather industry [8,9].

Recently, modern tannage process focuses on reducing pollution effect by using environmentally-friendly tanning technology. This relies on

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maximizing utilization of tanning waste through circumspect management with regard to order of priority: prevention, reduction, re-use / recycling / recovery, and then disposal [9]. However, the main problem in leather tanning industry is that tannery waste treatments are difficult, and there are no methods of its comprehensive utilization. Moreover, non-tanned wastes are used as raw materials for glue, gelatine, technical fats, protein sheaths and even feed and fertilizers. Nevertheless, for economic reasons, waste remains mostly unutilized and goes to landfills [10]. In this regard, different researchers utilized fleshing in feed, compost, biogas, biodiesel production as well as in bioenergy recovery [11-18].

Thus, the present investigation aims to evaluate the efficiency of utilizing fatliquor emulsion extracted from limed fleshing wastes for fatliquoring leathers. The objectives were to: I. utilize the highest quantity of tannery solid wastes to produce a fatliquor as a useful end product, that can be used in leather tanning industry. II. Reduce pollution levels resulting from the tanning process. III. Compare the properties of prepared fatliquor with commercial fatliquors, which commonly used in leather tanning process. IV. Evaluate the effect of application of prepared fatliquor on leather quality.

Experimental

Chemicals and reagents

All chemicals used for fat extraction and emulsion preparation were obtained from El-Gomhouria Company for Chemicals. Other chemicals for tanning leathers used in subsequent processing were commercial grade products, which are routinely used in leather industry.

Experimental design

The study was divided into four main parts as follows:

Fleshing wastes collection and fats preparation

Sheep fleshing wastes were collected from El-Shafei sons' tannery, Alexandria, Egypt. The wastes were taken after the fleshing step for unhaired sheep skins, which was done mechanically by a fleshing machine. After that, the wastes were transported directly to a laboratory for fats extraction.

The fats were extracted by exposing fleshing wastes to a water vapor steam and, then, fats were condensed by heating to remove excessive water [16].

Fatliquor emulsion preparation

Sulphated fatliquor was prepared from extracted fats [16]. In this regard, the extracted fats were slowly treated with 20% sulfuric acid (w/v) under controlled temperature. Thereafter, water was added to wash and remove the un-reacted acid, and then the sulphated fatliquor was neutralized with ammonium hydroxide to reach a final pH of 6.2-6.8.

Chemical characteristics of fleshing wastes, extracted fats and prepared fatliquor emulsion were determined. Moisture, ash, fat contents and pH were determined in fleshing wastes sample, whereas moisture, identification of fatty acids, acid value, iodine value and saponification value were determined in extracted fats sample. Fatty acids were quantified, according to Salimon, *et al.* [19], using Hewlett Packard 6890 GC-FID system, which depends on gas liquid chromatography with a flame ionization detector**.** On the other hand, moisture, ash contents and density were determined in fatliquor emulsion sample.

Leather processing

Leather tanning was performed at El-Shafei sons' tannery, Alexandria, Egypt. Two experiments were designed for testing the prepared fatliquor emulsion on leather tanning.

The first experiment aimed to evaluate the efficiency of using a fixed concentration of the prepared fatliquor on the properties of finished leathers in comparison to commercial fish oil fatliquor. Ten Barki sheepskins at marketing age, 12- 18 months, were used. Initially, all skins shared the same beamhouse and chrome tanning stages. At post-tanning stage, each skin was divided longitudinally into two sides. Right and left sides groups were fatliquored separately with 3% of prepared fatliquor and commercial fish oil, respectively.

In the second experiment, prepared and commercial fish oil fatliquors were used in fatliquoring cow wetblues with three fatliquor concentrations, 5%, 10% and 15%. Three cow wetblues were divided into 6 sides. Each two sides for a cow wetblue were fatliquored with the same fatliquor concentration. The right side was fatliquored with a prepared fatliquor, whereas the other side was fatliquored using commercial fish oil. The sheep and cow wetblues were post-tanned according to the explained recipe in Table 1.

TABLE 1. Executed recipe for post-tanning wet-blues.

Step	Description		Time (min.)	Remarks
	%	Added		
Washing	100 2	Water Soap	10	Drain float
Naturalization	100 X	Water Sodium bicarbonate	60	To adjust pH= 5
Dyeing	3	Black dye	90	Check dye penetration before next step
Fatliquoring	150 X	Water Fatliquor	90	Adding fatliquor quantity and type according to treatment
Fixation	1 1	Formic acid Formic acid	30 60	Check dye & fatliquor in float pH = 4
Washing	100	Water	10	Out & overnight as horse up then samming.
Finishing	--	--	--	Drying in shaded place.

Leather testing

All finished leathers were assessed for softness, grain smoothness, loose, fullness, and general appearance by standard tangible evaluation technique. Three experienced tanners rated the leathers for each functional property on a scale from 0 to10 (0= the lowest appreciated in organoleptic property whereas 10= the highest appreciated in organoleptic property).

Qualitative and operational properties of obtained leather were assessed according to indices of chemical analysis as well by physico-mechanical investigation of the finished leather. Thickness, tensile strength, elongation, split tear strength, water absorption, colorfastness of crocking of leather, pH, ash, fats and moisture contents were obtained according to standard procedures [20].

Scanning Electron microscopic analysis

A leather sample (1 cm^2) of each treatment was cut from official sampling position (ASTM) and subjected to sputter coating with gold ions which acted as a conducting medium during scanning, and was observed using a JEOL JSM-5300 electron microscope.

Results and Discussion

Limed fleshing wastes properties

Chemical properties of limed fleshing wastes are shown in Table 2. The results demonstrated that limed fleshing wastes were characterized by high content of moisture (63.09%) and moderate amounts of both ash (13.08%) and fats (20.12%). This was initially speculated since the first steps of beamhouse stage utilize high amounts of water in soaking, washing and unhairing steps. Furthermore, in curing and unhairing steps, different salts such as sodium chloride, sodium sulphide and lime were added and caused the increase in ash content [4,5]. The percentage of fat content is considered acceptable to fat content of sheep skins, ranging from 5% to 30% [1]. The basicity of limed fleshing wastes recorded high alkalinity (11.30) due to treating the skins with different strong alkalis such as sodium sulphide and lime during unhairing and liming steps to remove wool and fats [5].

Extracted fats properties

Data presented in Table 2 illustrate the chemical properties of extracted fats. The results showed high extraction yield (62.3%), high fatty matter content (79.8%) and low moisture content (3.16%), which reflects the economical feasible ratios of this technique to produce good amounts of fatliquors.

The properties of different oils and fats sources which can be used in leather fatliquoring were introduced by different researches [1,5,21]. The iodine, saponification and acid values for extracted fats were found to be near similar to the corresponding values of palm oil, neatsfoot oil and beef tallow fats, and therefore that provide further indication to the possibility of its utilization in preparation of leather fatliquor.

Iodine value is often used to determine the amount of unsaturation in fatty acids. Dutta [21] classified the fats and oils according to their iodine values; the non-dry oils has an iodine value lower than 95, whereas the corresponding values for dry oils and semi-dry oils are higher than 140 and ranged between 95 and 140, respectively. Hence, the obtained fats are classified as non-dry fats, which are insusceptible to autoxidation and polymerization.

TABLE 2. Chemical properties of limed fleshing wastes and extracted fats.

Item	Value	Unit
Limed fleshing waste		
Moisture	63.09	%
Ash	13.08	%
Fat	21.76	%
pH	11.30	--
Extracted fats		
Extraction yield*	62.3	%
Fatty matter	79.8	%
Moisture	3.16	%
Acid value	1.16	mg KOH/g
Iodine value	55.49	gm I_2 /100 gm fat
Saponification value	199.97	mg KOH/g

** calculated based on dry weight of limed fleshing waste.*

The acid value is defined as the number of milligrams of potassium hydroxide required to neutralize 1 g of fat. The content of free fatty acids expresses how many parts of free fatty acids are contained in 100 parts of fat [22]. Though not always the case, high acid value for oil indicates that the bondages between fatty acids and glycerol are not very strong and so the oil will hydrolyze very easily setting the fatty acids free from glycerol molecule [21]. Therefore, the obtained low acid value of extracted fats (1.16 mg KOH/g) indicates the difficulty to hydrolyze these bonds.

The saponification value is the number of milligrams of potassium hydroxide required to saponify (hydrolyze) 1 g of fat and is related to the molecular mass of the fat [22]. In the current investigation, the obtained saponification value of extracted fats (199.97 mg KOH/g) was considered moderate-high value. This may explain the increased number of fatty acids, penetration power and softness effect due to the decrease in molecular sizes and weights of the fatty acids [5,16].

Nevertheless, the values of moisture, fatty substances, saponification and iodine values were similar to those previously obtained when fleshing wastes fats were used [14,17,18].

Fatty acid analysis of extracted fats (Table 3) showed that about 60% of fatty acids are unsaturated, with oleic acid as the predominant fatty acid (45.87%), whereas about 35% are saturated with palmitic constitutes the higher percentage (21.43%). Unsaturated fatty acids are considered the most important for preparing a fatliquor. It tends to be liquid and may react better with sulfuric acid and, therefore, it can be used in leather fatliquoring [5,16]. On the other hand, the main problems of saturated fatty acids are their solid shape at normal temperature [5], difficulty to react due to linkage saturation [16], and the possibility of developing spue spots in finished leathers as a result of migration from internal structure of the leather to the grain surface and visible as a white fluorescence [21].

From chemical properties of extracted fats, the results elucidate the ability of converting their fats into sulphated fatliquor emulsion. This is in agreement with previous reports [16,17,18,20].

Fatliquor emulsion preparation

The preparation of fatliquor emulsion was done by sulphation reaction between extracted fats and sulphuric acid. The (-SO_3H) radical is thus linked up with a carbon atom through an oxygen atom (R-O-SO_3H). This sulphuric acid ester is then neutralized with alkalies as such as sodium or ammonium hydroxide to produce sulphated oils [4,5].

Fish oil fatliquor is considered the most commercially used sulphated fatliquor in leather processing. Therefore, samples from prepared sulphated fatliquor and commercial fish oil fatliquor were taken to compare their chemical characterizations. The results are displayed in Table 4. The pH and density values were similar between two fatliquors, while moisture and ash percentages were different. The ash value, unlike moisture, was higher in prepared fatliquor than commercial fish oil fatliquor. This may be due to the low efficiency of washing step, which was done after sulphation reaction.

Finished leather properties

The effect of adding fatliquors with fixed concentration

Fatliquoring step in leather tanning mainly depends on the reaction between fatliquors and

TABLE 3. Fatty acid composition of extracted fats.

Structure		Fatty acid	%
Saturated	C10:0	Capric	0.20
	C12:0	Lauric	0.16
	C14:0	Myristic	3.32
	C15:0	Pentadecanoic	1.36
	C16:0	Palmitic	21.43
	C17:0	Margaric	1.35
	C18:0	Stearic	7.54
	C20:0	Arachidic	0.05
Total saturated acids			*35.41*
Unsaturated	C14:1	Miristoleic	2.03
	C15:1	Pentadecenoic	0.80
	C16:1	Palmitolic	6.29
	C17:1	Margoleic	3.02
	C18:1	Oleic acid	45.87
	C18:2	Linolic	2.27
	C18:3	Linolenic	0.46
	C20:1	Gadoleic	0.44
Total unsaturated acids			*61.18*
Total unknown acids			*3.41*
Overall percentage			*100*

TABLE 4. Chemical characterization of prepared fatliquoring and commercial fish oil emulsions.

Property	Prepared fatliquor	Fish oil
pH of 10% solution	8.80	8.10
Moisture (%)	32.60	57.83
Ash (%)	3.87	0.49
Density (gm/ml)	0.88	0.84

collagen fiber bundles. Sulphated fatliquors are mainly anionic because it is ionized in water into negative charged (Oil-O-SO_3^-) and positive charged (NH_4^+) ions. The positive basic group of protein reacts with negative sulphated fatliquors molecules and thus fixed up with leathers [21].

In this experiment, the low percentage (3%) of fatliquor was added to compare the behavior of prepared fatliquor with commercial fish oil fatliquor on sheep leather properties. The data for finished leather properties are shown in Table 5.

The fatliquoring step was done after naturalization step at pH 5 and, thus, fatliquors were penetrated easily into middle layer of leathers. Different researchers [5,16,21] illustrated that in fatliquoring of chrome tanned leathers, the negatively charged (-OSO_3^-) ions penetrate into the chrome complex and therefore also act as masking agents. The number and the strength of the positively charged groups of protein which are dependent upon the acidity of the leather actually control the penetration of the oil into leather. In very acidic leathers, the oil remains to the surface only, whereas the oil molecules go right up to the middle layer if the acidity of the leather is very low.

Furthermore, the results of organoleptic properties clarified that the spue spots were not observed in all leathers as was speculated due to the low concentration of added fatliquors and penetrate it into middle layers.

Leathers fatliquored with fish oil fatliquor were fuller, grain looseness and softer than those fatliquored with prepared fatliquor, whereas grain smoothness property was similar between the two groups. In this regard, Heidemann [4] explained the positive relation between grain looseness and softness by a good penetration of fats. Another relation was illustrated by Dutta [21] who reported that penetration of fatliquors into middle layer of leathers increases grain looseness property. For these reasons, leathers fatliquored with prepared fatliquor was superior in the general appearance property due to decrease its grain looseness.

The physical properties showed that prepared fatliquor had less effect on both fullness and softness as reflected in decreasing thickness and elongation values, respectively. Moreover, the negative relation between leather thickness and strengths led to increasing tensile and tearing strengths in prepared fatliquored leathers [1,23].

With respect to the effect of fatliquoring on water absorption, lubricating effect of sulphated fatliqours is mainly due to hanging insoluble protein fibers, which makes the other neutral oils molecules adhered by cohesion [21]. Thus, coated fibers might be less susceptible to water absorption. In addition, the obtained data showed that prepared fatliquor was superior in decreasing water absorption than fish oil fatliquor after 2 and 24 hr, which gave an indication of their better distribution on collagen fibers.

TABLE 5. Physical and chemical properties of finished sheep leathers.

Property		Prepared fatliquor	Fish oil fatliquor
Organoleptic properties	Fullness	8	9
	Grain looseness	2	3
	Grain smoothness	7	7
	Softness	6	8
	General appearance	8	7
Physical properties	Thickness (mm)	1.23	1.36
	Tensile strength (kg/cm^2)	167.06	152.26
	Tearing strength (kg/cm)	25.95	21.10
	Elongation (%)	58.00	69.57
	Water absorption (%) after 2 hrs	194.48	219.71
	Water absorption (%) after 24 hrs	207.03	234.65
	Colorfastness of Crocking of Leather*	Good	Good
Chemical properties	Moisture (%)	14.90	14.33
	Ash (%)	5.26	5.21
	Fat (%)	6.13	6.18
	pH	3.64	3.65

** According to ASTM D-5053, the specimens classify as follows:*
- *Good: if no appreciable staining of either the wet or dry cloth.*
- *Fair: if appreciable staining of wet cloth but no appreciable staining of dry cloth.*
- *Poor: if appreciable staining of dry cloth.*

On the other hand, the chemical properties of the two fatliquored leathers groups were similar due to the similarity in all tanning steps and fatliquor concentration.

The SEM analysis of sheep leather samples showing surface and cross sections, are depicted in Fig. 1. The micrographs showed that prepared fatliquor leathers possess more flat surface with less wrinkles. In addition, the cross section showed that fiber bundles of prepared fatliquor leathers group were more open and less in fibers separation when compared with the other group. This may explain the trends of organoleptic and physical properties data.

Increasing the distances among fiber bundles led to show it as separate sheets [24]. It is identified as a shape of looseness in leathers which decrease its toughness or increase softness. In micrographs, the distances among fiber bundles in fish oil fatliquored leathers were higher than the other group, which indicates the changes in fullness, softness, thickness, water absorption and strengths within the two groups.

The effect of adding fatliqours with different concentrations

Three concentrations of prepared or fish oil fatliquors, 5%, 10% and 15%, were used. Leather properties of finished cow leathers are shown in Table 6.

At the same concentration of fatliquor, as previously described in sheep leathers experiment, organoleptic properties showed the same differences between leathers fatliquored with prepared fatliquor and corresponding leathers fatliquored with fish oil fatliquor.

By increasing the fatliquor concentration, the fullness, grain looseness, smoothness and softness

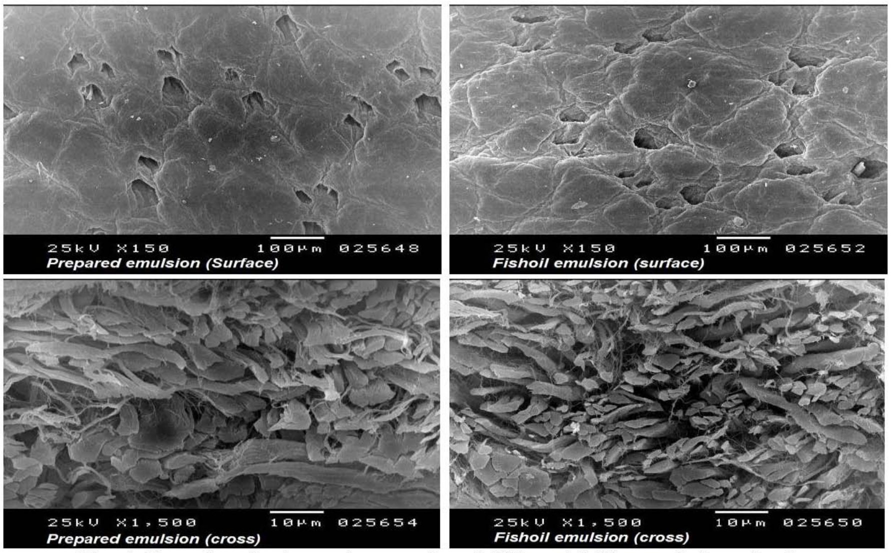

Fig. 1. Scanning electron micrographs of different fatliquored sheep leathers.

TABLE 6. Physical and chemical properties of finished cow leathers.

Property		Prepared fatliquor			Fish oil fatliquor		
		5%	10%	15%	5%	10%	15%
Organoleptic properties	Fullness	6	8	8	7	8	9
	Grain looseness	2	3	4	2	4	5
	Grain smoothness	7	7	8	7	7	8
	Softness	6	7	9	6	8	9
	General appearance	9	9	7	8	8	6
Physical properties	Thickness (mm)	1.20	1.30	1.33	1.25	1.30	1.35
	Tensile strength (kg/cm^2)	134.28	128.66	119.99	130.65	122.26	113.26
	Tearing strength (kg/cm)	41.54	34.20	27.97	34.70	29.44	24.66
	Elongation (%)	34.10	38.80	43.87	37.31	46.82	47.42
	Water absorption (%) after 2 hrs	72.36	70.33	66.82	106.52	76.87	74.20
	Water absorption (%) after 24 hrs	76.91	75.45	71.58	110.28	87.73	81.86
	Colorfastness *	Good	Good	Fair	Good	Good	Fair
Chemical properties	Moisture (%)	15.52	15.69	15.35	15.66	15.28	15.12
	Ash (%)	5.61	5.80	6.62	4.68	5.39	5.74
	Fat (%)	4.41	8.05	10.79	4.44	9.52	12.79
	pH	3.79	3.84	3.84	3.79	3.83	3.96

* *According to ASTM D-5053, the specimens classify as follows;*

- *Good: if no appreciable staining of either the wet or dry cloth.*
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properties increased, whereas the spue spots were not visually observed.

Both prepared and fish oil fatliquors exhibited similar effect on all physical properties of corresponding finished leathers. With respect to the concentration effect of adding fatliquor, an increase of concentration caused an increase in elongation and thickness values whereas other properties decreased. Increased lubrication and decreased friction between fibers and fiber bundles at high concentrations of fatliquors may be the reason for these changes in organoleptic and physical properties. Furthermore, the degree of colorfastness was decreased by adding fatliquor with high concentration 15% but the property is still acceptable in manufacture using [1]. The reason may be due to the effect of increase fatliquor on the surface layer by increasing the addition of fatliquor.

From chemical properties of finished leathers,

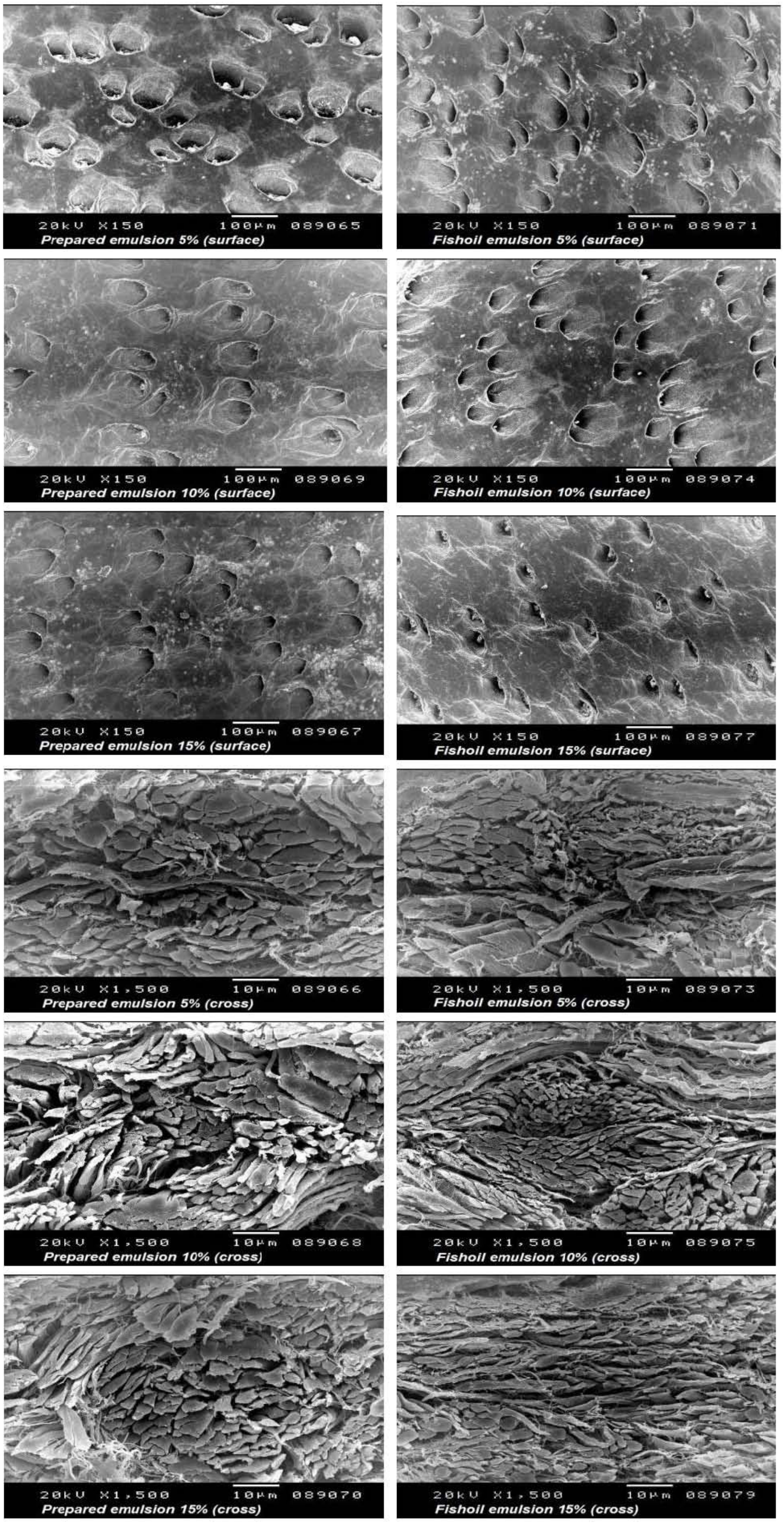

Fig. 2. Scanning electron micrographs of different fatliquored cow leathers.

moisture contents and pH values were comparable among all finished leathers. On the other hand, ash and fat contents increased within each fatliquor group along with increasing fatliquors concentration, which was expected.

The SEM analysis of cow leathers samples, showing surface and cross sections, are depicted in Fig 2. Surface micrographs showed increased wrinkles in leather fatliquored with fish oil fatliquor. In addition, these wrinkles also increased by increasing fatliquor concentration. Although the spue spots were not visually observed on the surface, it was shown on the surface as white fluoresce spots in the micrographs especially with the leathers that were fatliquored with 15% prepared fatliquored.

On the other hand, the cross sections micrographs showed that finished leathers exhibited slightly dispersed fiber bundles structure, although it would exhibit principally better opening of fiber bundles and, thus, leather fullness as a shape of looseness among bundles [24].

From finished leathers properties data, it can be concluded that using prepared fatliquor resulted similar behavior and effects on collagen fiber to those of commercial fish oil fatliquor. Also, all leathers were similar in quality and suitable for leather manufacturing according to quality requirements for the main types of leather [1].

Conclusion

Collectively, the results of the current study pointed out the efficiency of extracting fats from fleshing wastes and converting it into fatliquors in achieving distinctive environmental and economic benefits. Additionally, the results showed that the extracted fats were suitable to prepare sulphated fatliquor and provide satisfactory yields of both total and unsaturated fatty acids. Further, the prepared sulphated fatliquor was stable during fatliquoring of leathers at different concentrations till 15%, and enhanced the organoleptic and physical properties for finished leathers such as synthetic commercial fish oil.

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إعادة إستخدام مخلفات التلحييم كمادة تشحيم في خطوات تجهيز الجلود

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خلال هذه التجربة تم إعداد مادة لتشحيم الجلود في صورة مكبرتة من مخلفات تلحيم جلود الضأن لتستخدم في تجهيز الجلود. تم تقدير الخواص الكيمياية علي الدهون قبل خطوة الكبرته. وبعد خطوة الكبرتة تم معادلة مادة التشحيم باستخدام هيدروكسيد الامونيوم. تم تقدير الخصائص الكيميائية لمادة التشحيم التي أُعدت ثم أستخدم الشحم لتشحيم الجلود بنوعيها الخفيفة والثقيلة ضمن خطوات دباغة الجلود. الخصائص الطبيعية والكيميائية والمظهرية للجلود النهائية تم تقديرها كما ألتقطت صور بالميكرسكوب الاليكتروني للجلود المشطبة. أوضحت النتائج أن محتوي الدهون بمخلفات تلحيم جلود الضأن حوالي ٢٠٪ من الوزن والتي تحتوي بدورها علي نسبة 60% دهون غير مشبعة تركيبها الاساسي حمض الاوليك. بين ذلك التركيب إمكانية استخدامها لتشحيم الجلود. بالاضافة الي ذلك فإن جودة الجلود المنتجة من إستخدام الشحم المُعد كانت مقاربة لتلك التي تم تشحيمها بزيوت الاسماك التجارية . هذه النتائج بينت امكانية تحويل مخلفات التلحييم الي شحم مكبرت يمكن إستخدامة ضمن خطوات دباغة الجلود كمادة بديلة عن الشحوم التجارية التقليدية مما يقلل من ملوثات المدابغ ويقلل تكاليف الانتاج.