Waste Water Reutilization in the Leather Industry using Membrane Technology

by

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Abstract

This paper documents a technical feasibility study of tannery wastewater which was reclaimed (treated) and reutilized in tannery leather making processes. The studied waste water was processed in the waste water treatment plant owned by Igualada tanners (IDR), in Spain. The objectives included reduction of city fresh water consumption by the tanners of this city.

A pilot plant using membrane technology, determined to be the most appropriate technology in this case, was installed at the IDR wastewater treatment facility. It worked in three consecutive stages: pre-treatment by sand filtration, subsequent ultrafiltration, and then reverse osmosis.

The reclaimed water was of the highest quality, superior to any other water currently available to city tanners.

The right sides of 24 calf hides were fully processed with the reclaimed water, while the left sides of the same hides were processed with softened tap water. Three different leather articles were produced. No significant differences were detectable between the left and right sides, neither organoleptically nor analytically.

Resumen

Esta comunicación presenta un estudio sobre la viabilidad técnica de la reutilización por parte de las empresas curtidoras de una parte de las aguas depuradas por la planta de tratamiento conjunto de aguas residuales de curtidos de Igualada, con la finalidad de reducir el consumo de agua por parte del sector curtidor de esta ciudad.

Considerando la tecnología de membranas como la más adecuada para caso concreto, se ha instalado una planta piloto en la estación depuradora de aguas residuales que realiza los procesos consecutivos de pretratamiento por filtro de arena, ultrafiltración, y ósmosis inversa.

El agua regenerada obtenida tiene una muy alta calidad, superior a la de cualquier fuente de suministro actualmente accesible a los curtidores igualadinos.

Con el agua regenerada obtenida se ha procedido a la fabricación a escala semi-industrial de pieles bovinas en 3 artículos distintos, los cuales se han comparado con los mismos artículos obtenidos con el agua fresca que usan convencionalmente los curtidores de Igualada.

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Manuscript received May 15, 2008, accepted for publication October 13, 2008

Introduction

The processing of skins and hides from their fresh or cured condition to their finished state, ready for being used by the manufacturing industries, implies a high consumption of water as a vehicle for carrying the substances that have to be eliminated from the raw material, and the chemicals that have to be incorporated in the collagen to obtain a marketable leather.

Leather production is a water intensive industry. Water usage is 15 to 40 m^3 of water for the production of 1 ton of wet-salted raw hides and 110-260 liters per sheepskin [1].

In many countries water has become an insufficient commodity and the costs for water supply and discharge increases regularly. In addition, its availability depends on the variability and seasonal variation of the climatic conditions. It is foreseeable that in the future these dynamics will probably become more serious.

The International Union Environment Commission of the IULTCS recommends several options available to minimise the overall consumption of water: reduction, recycling, and re-use [2].

Reduction: Low float processing and batch-type washing instead of rinsing are practical examples of technologies to reduce water consumption.

Recycling: Certain processes are suitable for recycling of floats, although in most cases installations for treatment are necessary. Examples are; soaking, liming, unhairing, pickling and chrome tanning liquors, which can reduce the overall water consumption. However, suitable facilities and strict controls are necessary.

Re-use: Biologically treated effluent offers the opportunity of replacing a certain amount of the process floats, such as the beamhouse process floats, with treated water. However, additional operations might be necessary, such as filtration and disinfection.

BLC Leather Technology Centre has demonstrated the benefits and economical feasibility of membrane applications for chemical recovery in the leather industry [3]. Currently, the membrane systems have the capacity to eliminate practically the totality of the salts and the organic matter, and it is possible to fulfil the highest exigencies of quality of the water. For that reason the regenerated water could be reused in substitution of the fresh water in all the operations of the tannery, even in most delicate as dyeing, fatliquoring and finishing.

One of the great advantages of the regenerated water is that it is not put under the variability of the climatic conditions. It is guaranteed water, always available, with independence of if it rains or not.

On the other hand, it releases part of the water resources currently consumed by the industry to leave them accessible to other uses as for example the use for human consumption. The reclaimed water is highly engineered, more predictable in quantity and quality than many of our existing surface and ground water sources. The availability of a water of high quality allows also the improvement of the quality of the waste water. For example, when avoiding the necessity of the decalcification, the brine wastes derived from the resin regeneration are saved.

Background

The tanneries cluster in Igualada

Igualada (Spain) is a good example of a leather industrial cluster[4]. The waste water from leather industries goes to a common waste water-treatment plant belonging to the tanners (IDR). The IDR waste water-treatment plant is treating all the effluents coming from tanneries together with a portion of municipal waste water which contributes to diminish salinity. About 100 t/day of salt cured hides are treated by this cluster, mainly bovine hides. In Table 1 the daily volume of waste water coming from different process parts is shown.

TABLE I

Average daily water wastes coming from the Igualada leather cluster.

Processes	Average volume of waste water
Soaking, unhairing and washings	1300 m^3/day
From deliming to chromium tanning	480 m^3/ day
From deliming to vegetable tanning	120 m^3/ day
Dyeing and fatliquoring	800 m^3/ day
Softening of water for steam boilers and other applications	50 m^3/ day

TABLE II

Composition of tap water for industrial use in Igualada

pH	7.5 -7.6	$Conductivity_{20C}$	850-1050 μS/cm
Total $solids_{180C}$	660- 840 mg/L	Cl^-	15-28 mg/L
SO_4^{2-}	240-350 mg/L	Si	~ 3 mg/L
Mg^{++}	50-55 mg/L	Ca^{++}	130-160 mg/L
HCO_3^-	~ 300 mg/L	Hardness	500-640 mg/L $CaCO_3$

TABLE III

Composition of the water from the Anoia River.

pH	7.5 -7.6	$Conductivity_{20C}$	2500-3000 μS/cm
Total $solids_{180C}$	2000 - 2500 mg/L	Cl^-	~ 350 mg/L
SO_4^{2-}	900 - 1000 mg/L	Si	~ 4 mg/L
Mg^{++}	~ 140 mg/L	Ca^{++}	~ 330 mg/L
HCO_3^-	~ 350 mg/L	Hardness	~ 1400 mg/L $CaCO_3$

TABLE IV

Characteristics of the water obtained after the biological treatment.

	Mean Value	Relative Standard Deviation
pH	7.5	± 2.0 %
Conductivity (μS/cm)	5.800	± 16 %
COD (mg/L O_2)	270	± 30 %
Suspended Solids (mg/L)	24	± 36 %
Hardness (mg/L $CaCO_3$)	1020	± 2.5 %
Nitrogen Total Kjeldhal	70	± 22 %

The effluent volume per day is 2500 to 3000 m^3/day depending on the day and the season. The average value is about 2750 m^3/day.

Water supply comes from two different origins: tap water and water from the Anoia River. Tables 2 and 3 show the parameters of both kinds of water sources. Both of them are hard water resources, specially the water coming from the river.

These types of water must be softened by ionic exchange columns before being used in steam boilers and in some parts of the post-tanning processes like dying and fatliquoring steps.

While tap water is free of micro organisms because it is chlorinated before distribution, the river water contains about 6000 CFU/mL in 24 hours.

The characteristics of the water obtained after biological treatment in the IDR waste water-treatment plant are presented in Table 4.

Reclaimed water quality requirements

General water quality requirements in the tanning process:

1. Water must be clarified to avoid filter and membrane obstruction risks and to make disinfection easier.
2. To be stored up during long time, a low content in organic carbon is needed.
3. A low salt concentration is needed to accomplish the very strict effluent salinity legislations.
4. It must be disinfected.

The specific quality requirements for tanning uses will depend on the parts of the process where the water will be used. For example, in beamhouse operations quality requirements are moderate-low, but in post-tanning operations the water quality must be higher: no colour, low content of iron and manganese and other contaminants which should affect the dyeing, low turbidity, etc. Hardness in water must be also very low or it has to be softened.

In addition to the biological waste-water treatment (see Table 4), some other advanced treatments should be made to the treated water to accomplish all these requirements.

Advanced wastewater treatment

The advanced wastewater treatment processes are also called tertiary treatment processes because are used beyond the traditional secondary treatment to produce high quality water. The main advanced processes are the following [5]:

- Filtration
- Nitrification
- Denitrification
- Coagulation-Sedimentation
- Carbon Adsorption
- Membrane Processes
- Disinfection.

The continuous improvements in the membrane technologies and the ability to obtain regenerated water of high quality, capable to use it for almost all type of applications, are favouring its use in more and more facilities.

Membrane treatment systems are broadly classified by the size particles rejected by the membrane, or by the molecular weight cut off (MWCO), as it can be seen in table 5.

The table 6 shows reverse osmosis contaminants removal efficiency data.

As it can be seen from the table, the reduction in organic carbon and dissolved salts is very high, allowing to fully satisfying these two quality requirements in the water regenerated.

In relation to the first requirement, the absence of suspended solids, the reverse osmosis eliminates practically the totality of these contaminants. However, it is mandatory that the

TABLE V

Categorization of membrane treatment systems

Membrane System	Size of particles rejected	Molecular weight cut off
Microfiltration (MF)	0.1 µm	500,000 MWCO
Ultrafiltration (UF)	0.01 µm	20,000 MWCO
Reverse Osmosis (RO)	0.0001 µm	< 100 MWCO

TABLE VI

Reverse osmosis performance data[6]

Constituent	RO Influent	RO effluent	Average reduction
Total Organic Carbon	9 - 16 mg/L	< 0.5 mg/L	> 94 %
Chemical Oxygen Demand	16 - 53 mg/L	< 2 mg/L	> 91 %
Total Dissolved Solids	498 - 622 mg/L	9 - 19 mg/L	> 97 %
Total Suspended Solids	< 0.5 mg/L	~ 0 mg/L	> 99 %

influent has a concentration very low of suspended matters to avoid fouling problems in the membranes. To accomplish that, a pre-filtration system must be set up. As it can be observed in the example of table 6, the water to be processed by reverse osmosis has less than 0.5 mg/L of TSS.

Respect to the fourth requirement, the treatment with membranes of reverse osmosis eliminates bacteria and other biological polluting agents of the water[6]. However, if a prolonged storage is desired it will be necessary to add and maintain a residual amount of chlorine to avoid later contaminations.

EXPERIMENTAL

Brief description of the reclaimed water plant

A semi-industrial pilot plant has been devised in order to obtain the sufficient amount of water to be able to carry out full tanning tests (from salted hides to the final processes) under real conditions at a sufficiently large scale to be considered representative.

A three-stage treatment has been outlined:

1. Sand filter treatment
2. Treatment with ultrafiltration membranes
3. Treatment with reversed osmosis membranes

The design and setting up took into account the fact that the plant will be automatic and will work continuously, as the results can therefore be more reproducible and any possible practical problems arising can be more easily assessed. The pilot plant was subcontracted with the firm Astramatic [7].

The pilot plant's flow sheet is shown in Figure 1.

The average flow volume for each of the process stages are presented in Table 7. Any surplus flows are flowed back into the waste water treatment plant.

The plant was set in the area where the IDR treatment plant is located. The services required by the pilot plant are nearby and both the place and space are appropriate to be able to work and keep the plant in good conditions. In addition, flows from the washes and rejected flows of the membranes can be adequately returned to the waste water treatment plant.

Pilot plant main systems

a) Sand Filter dual, 900 mm of diameter, with automatic backwash by means of an independent pump. Maximum flow rate is 6.5 m^3/h.

b) Ultrafiltration unit from Norit[8], with 2 elements of hydrophilic membranes of Xiga SXL225 class with an internal diameter of 0.8 mm. The membrane fibers have a high temperature and chemical resistance and excellent mechanical strength. The SXL225 contains 40 m^2 of membrane area. It incorporates filtration, backwashing of the elements and chemical cleaning.

c) Reversed Osmosis system from Hydranautics [9]. Spiral wound membrane of composite polyamide, model LFC3 4040, of neutral ionic charge. It contains 6 elements of membranes (3V2, in 2 steps). Each element is 102 cm in length and 10.1 cm in outer diameter.

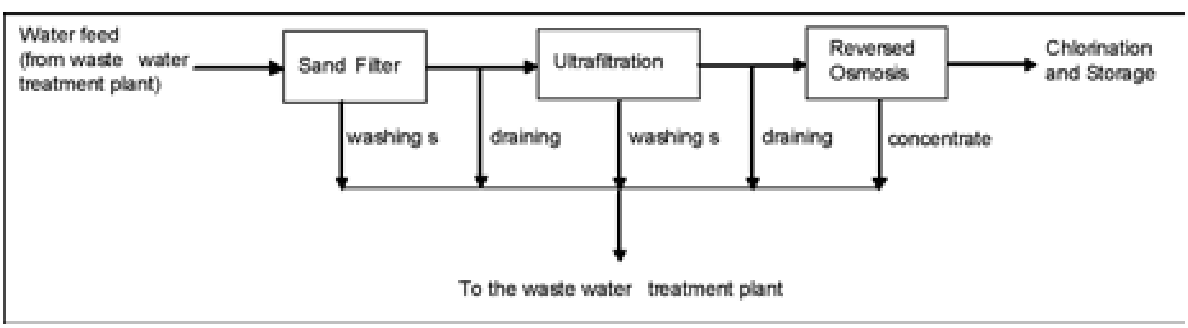


Figure 1. Flow sheet for the reclaimed water pilot plant

TABLE VII
Flow of each process stage

Water feed flow	4 - 5 m^3/h
Ultra filtration influent flow	3 – 3.5 m^3/h
Reversed Osmosis influent flow	~ 2 m^3/h
Reversed Osmosis permeate flow	1 - 1.1 m^3/h

RESULTS AND DISCUSSION

Although the sand filter was set up with manual wash initially, it was soon observed that it became dirty too quickly and that it was difficult to maintain it in optimal functioning conditions. The flow decreased in such a way that the ultrafiltration plant came to a stop due to lack of water.

Hence, an automatic washing system was set up instead in order to solve this problem. The filter is washed automatically every three hours with filtered water adjusted with sodium hypochlorite to disinfect the sand stratum.

The ultrafiltration plant was adjusted to work at a flow volume of 3 m^3/h (which corresponds to a flow of 50 $L/h•m^2$) with 15-minute process cycles every 20 minutes.

Aluminium polychloride was dosed in the membrane input feed stream in order to improve its effectiveness. Adjustments to the washing program involved a 30-second long back flush every twenty minutes, at a flow volume of 20 m^3/h. In addition, an automatic wash with sodium hydroxide and sodium hypochlorite was set every 6 hours, and one with hydrochloric acid every 12 hours.

The osmosis plant was fed by a flow volume of 1.6 m^3/h approximately. Recovery was adjusted at 63%, with a flow rate of permeate of 1 m^3/h and a rejection of 0.6 m^3/h.

Sodium bisulfite and a phosphonate antiscalant chemical were introduced in the RO feed to enhance the operation of the system. Antiscalants are a family of chemicals designed to inhibit the formation and precipitation of crystallized mineral salts that form scale, like calcium carbonate and the sulphates of barium, calcium and strontium. Phosphonates avoid also the scaling of calcium fluoride and inhibit the sediments of aluminium, iron and silica. Sodium bisulfite removes any residue of chlorine added in the water pre-treatment that could damage the RO membranes.

During the project it has been necessary to clean up and to change the cartridge filters of the membranes. A progressive fouling was observed, so the solids retained on the membrane surface were washed away periodically by means of a permeate backwash not involving any chemicals. However, this light fouling did not affect the right operation of the plant during the length of the project. Adsorbed substances were removed at the end of the project by a chemically enhanced backwash restoring the membrane to its original, clean state.

The permeate was collected in containers for 4 weeks, and picked up to AIICA facilities to carry out the planned tanning studies.

Room temperature for the operation period of the plant was quite constant, between 17 and 20 ºC.

Analytical control

The proper working of the pilot plant was subjected to analytical control. Samples were taken from the outfeeders of each of the systems employed, i.e. sand filter, ultrafiltration, osmosis (permeate and rejection).

One of the most important parameters was the suspended solids. It is fundamental that the input flow to the reversed osmosis membrane was free of suspended solids[5]. The sand filter showed an efficiency of 50% in diminishing this parameter. The analytical results showed an efficiency of more than 90% in the ultrafiltration stage. It was probably higher but it's beyond the sensitivity of the analytical method. The turbidity measurements before and after ultrafiltration also showed an efficiency higher than 90%.

As expected, ultrafiltration did not cause any effect on the salinity. On the other hand, the total organic matter was reduced by 22%. This effect was not only produced in the suspended organic matter (which is almost completely removed) but also in the one that is dissolved. This is believed to be owing to the retention of those organic molecules with high molecular mass by the membranes, whereas molecules of smaller size freely penetrated through the filter.

TABLE VIII

Average performance reduction data of different water constituents.

Constituent	Week within the period of thorough control of the plant 1st	2on	3rd	4th
Conductivity reduction	96.7 %	95.8 %	95.8 %	94.4 %
COD reduction	99.6 %	99.5 %	99.4 %	99.3 %
Ammonia reduction	88.8 %	92.1 %	92.9 %	89.6 %
Hardness reduction	99.6 %	99.6 %	99.6 %	99.5 %

The quality of the permeate produced by osmosis was thoroughly controlled for 4 consecutive weeks. Permeate is presented completely odorless, transparent and colorless. The relevant analytical measures by turbidimeter and colorimeter confirmed this observation. The conductivity of permeate was held below 400 μS/cm in all the measurements carried out. The average measurement obtained was 240 μS/cm.

It was observed that permeate conductivity reflected variations in conductivity of the water supplied by the waste water treatment plant since the performance of removal is quite regular.

In the table 8, the removal performance data of some constituents within the exhaustive pilot plant control period are presented. The effectiveness of the chemical oxygen demand reduction has been calculated rating the permanganate oxidizability of the permeate by the COD of the influent.

The efficiency of reduction of salinity of the plant decreased slightly during the course of the last operating days. This was attributed to a possible minor fouling of the membranes, and can be solved by carrying out the appropriate washes.

Table 9 presents an average analysis of the obtained permeate. As it can be seen, the quality of the water is excellent, with values better than any other source currently available in Igualada (tables 2 and 3).

In relation to the microbiological stability, it has been realized that the reclaimed water may be stored without any problem for a period of 2-3 days (table 10). For longer times, a treatment of disinfection would have to take place, preferably with chlorine to provide to water with a long-lasting protection.

The chlorine dosage required to disinfect water to any desired level is greatly influenced by the constituents present in the water. The main interfering substances are:

- Organic constituents, which consume the disinfectant
- Particulate matter, which protects microorganisms from the action of the disinfectant.
- Ammonia, which reacts with chlorine to form chloramines, a much less effective disinfectant species than free chlorine.

Given that the reclaimed water is practically free from all these three parameters, the amount of chlorine added to give a residual content will be very low.

TABLE IX
Average chemical composition of the obtained permeate.

pH	6.11
Conductivity at 20 ºC	236.0 µS/cm
Suspended Solids	< 1.5 mg/L
Chemical Oxygen Demand	< 10 mg O_2/L
Permanganate oxidability	0.7 mg O_2/L
Total Kjeldahl Nitrogen	< 7 mg N/L
Ammonium Nitrogen	4.1 mg N/L
Phosphates	< 0.10 mg P/L
Phosphorus, total	< 0.10 mg P/L
Chlorides	60.3 mg/L Cl^-
Sulphates	5.3 mg/L SO_4^{2-}
Sulphides	< 0.05 mg/L S^{2-}
Nitrates	< 0.1 mg/L NO_3^-
Carbonates	-
Bicarbonates	25.1 mg/L CO_3H^-
Total Hardness	4.0 ppm $CaCO_3$
Turbidity	< 1.0 UNF
Adsorbable organic halogens (AOX)	< 0.1 mg/L
Iron, total	< 0.01 mg/L Fe
Manganese, total	< 0.005 mg/L Mn
Aluminium	< 0.01 mg/L Al
Silicon	0.3 mg/L Si
Calcium	1.23 mg/L Ca
Magnesium	0.38 mg/l Mg
Sodium	46.2 mg/L Na
Potassium	1.11 mg/L K
Barium	< 0.005 mg/L Ba
Strontium	0.02 mg/L Sr
Colour (ISO 7887, a 20 ºC) :	
λ = 436 (m^{-1})	0.0
λ = 525 (m^{-1})	0.0
λ = 620 (m^{-1})	0.0

TABLE X
Summary of the results of the microbiological analysis of reclaimed water

microbiological contamination (average values)	Storage time of reclaimed water (at 20ºC) 1 day	2 days	1 week
CFU/mL at 24hr	< 15	570	10500

Leather production with reclaimed water

A study of the production of three different end products was carried out, starting from 24 bovine raw hides. Water use from the regeneration treatment was compared to that supplied by the industrial network (i.e. tap water). The flow diagram in the figure below outlines the operations involved in each of the processes for each of the leather goods produced.

The end products obtained are the following:

1. Chrome tanned leather upper.
2. Fully vegetable tanned kip leather aimed at fancy leather goods.
3. Mixed tannage (chrome-vegetable) aimed at leather uppers or fancy leather goods.

Each of the raw hides was cut into two sides by the backbone; each of the sides were marked accordingly. The left sides were employed to carry out the different processes and operations using tap water, whereas the right sides were employed to effect the same operations but using regenerated water obtained in the tertiary treatment pilot plant.

The different operations were carried out in identical drums and machinery and applying the same formulas, chemical products and under the same working conditions. Therefore, the only variation between each pair of sides produced from the same leather was the type of water employed in the processes.

Assessment of leather quality

A sample from each of the 24 sides processed with tap water and from each of the 24 sides processed with regenerated water was taken in order to carry out different chemical and physical analysis. Sampling was carried out in accordance with standards IUP2-IUC 2 (EN ISO 2418).

The chemical tests carried out were pH determination of the aqueous extract from the leather, chrome content and percentage of extractable fatty matter.

The results showed differences which were not very relevant, except for the pH values. Hence, despite not being highly appreciable, these values were significant in the sense that in all the cases the pH values of the leathers processed with regenerated water were around 0.2 units lower than those treated with tap water. This can be attributed to the influence of the two final washes of each of the formulas, with 400% water. In contrast with the other operations, the pH is not controlled in this one and no product is used which might either mask or diminish the distinctive effect of washing with a type of water that is practically free of bicarbonates at pH ~6.1, or wash with a type of water with 300 mg/L bicarbonates at pH 7.5.

The organoleptic characteristics with regard to feel, softness, grain firmness, smoothness, dye quality and possible defects were also assessed comparatively. In addition, the following physical tests and fastnesses were evaluated in accordance with standards EN ISO corresponding to IUP and IUF:

- Lightfastness
- Wet and dry rubbing fastness
- Tear resistance
- Grain crack resistance
- Tensile strength and elongation at break

To serve as an example, figures 2 and 3 show the distension of the grain and the tearing load measurements of the six lots of hides.

It seems obvious from these figures that there aren't important differences between hides processed with both kinds of water. But furthermore, the results from all the trials were statistically evaluated by means of the paired Student's t test [10].

No significant differences were found between the leathers treated with regenerated water and those treated with tap water with regard to the three leather goods and the properties measured. Even with regard to colour and touch, no differences are appreciable.

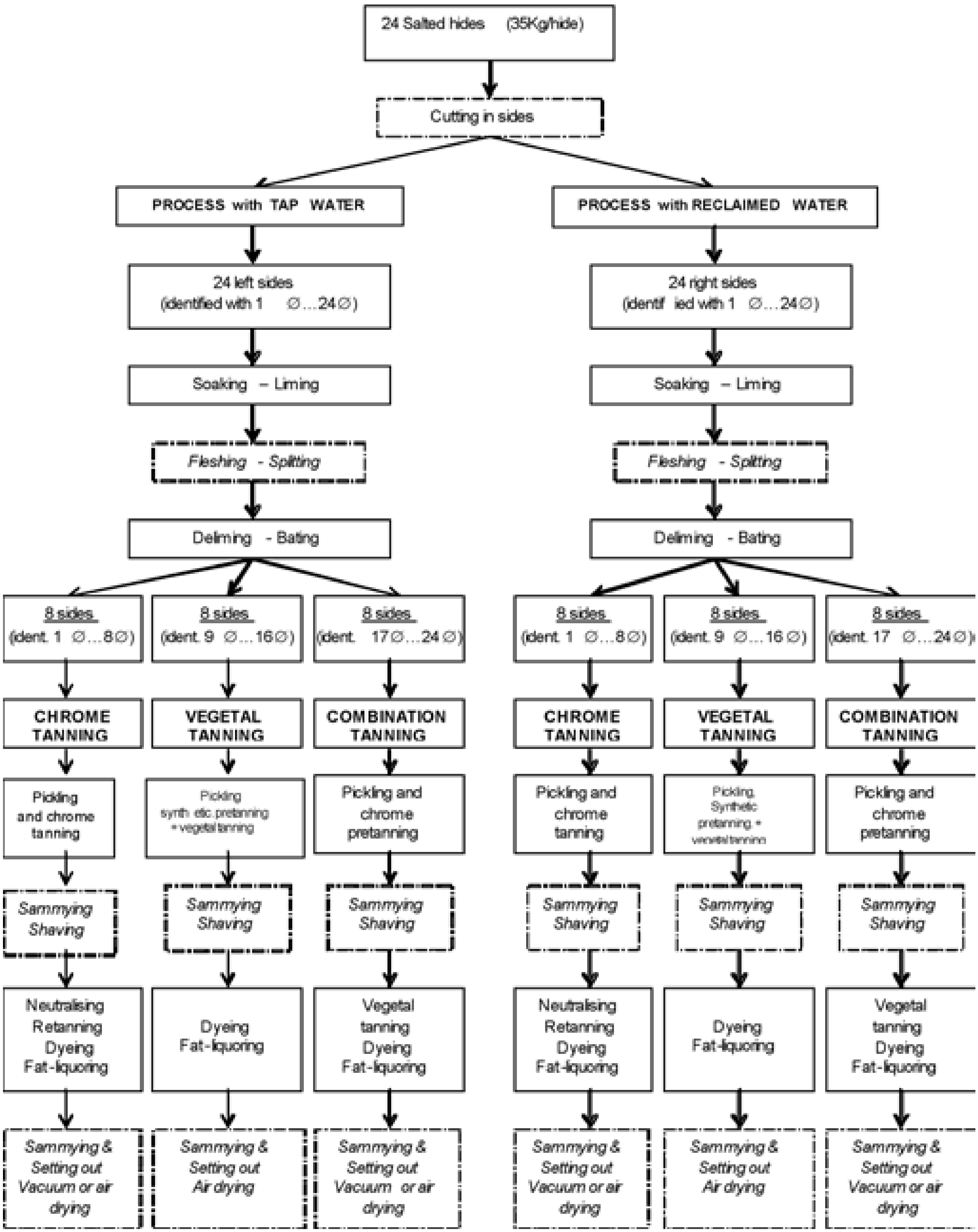
It must be borne in mind that the tap water from the AIICA Technological Centre where the processes were carried out is softened by resins of ionic interchange. Therefore, tap water hardness could not have any effect on the quality of the leathers.

Conclusions

A pilot plant at semi-industrial level has been working continuously and in an automatic manner for two months in order to obtain regenerated water by means of a treatment using of sand pre-filtration, ultrafiltration and reversed osmosis membranes. The monitoring of both the working of the plant and the quality of the waters produced in the plant has proved to be a highly successful technical experience. With some slight modifications to the design of the sand filter and in the chemical treatment previous to the ultrafiltration, the plant has been working properly. A high standard of reliability was obtained. After the initial fine tuning period, the plant worked fully automatically for more than 4 weeks without any problem or interruption and only a gentle diminution of the removal performance data, diminution that may be easily solved by cleaning the RO membrane.

This shows that the technology applied is adequate as a tertiary process for the water purified by the biological waste water treatment plant of IDR (owned by the Igualada tanners) to obtain high-quality water.

LEATHER PROCESSES



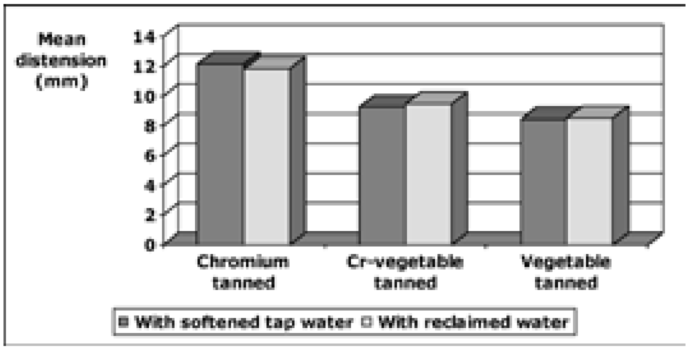


Figure 2. Comparisons between the grain resistance of the left sides made with softened tap water (in dark grey colour) and the grain resistance of the right sides (in light grey colour) made with reclaimed water, for the 3 kinds of leather produced. Measurements were carried out by the IUP 9 – ISO 3379 method.

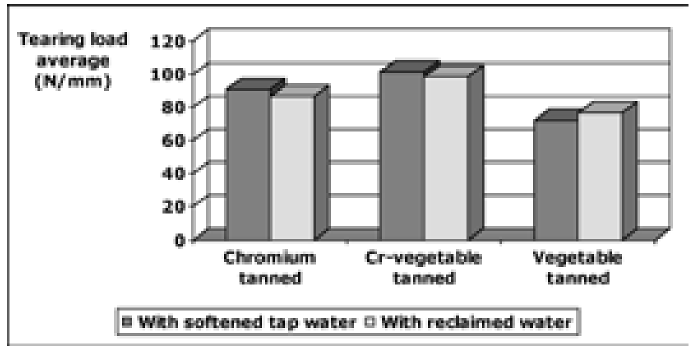


Figure 3. Comparisons between the tearing load resistance of the left and right sides, made with softened tap water and with reclaimed water, respectively. Measurements were carried out by the IUP 8 – EN ISO 3377-2 method.

In order to demonstrate the suitability of the water obtained for its use in tanning, three different leather goods made from bovine hides were manufactured using regenerated waste waters in contrast with the simultaneous production using softened tap water. These tests demonstrate that there are no significant differences between the leathers at sensorial, physical, chemical and fastness levels, and the fact that water regenerated has been employed does not involve a decrease in quality. Hence, the quality and usefulness of the water obtained has been verified and validated.

In addition, the regenerated water has hardness lower than 5 mg/L $CaCO_3$ and can hence be employed as feeding water for boilers. This implies a double saving since water and salt consumption for the regeneration of softener resins is avoided while the conductivity of discharges is reduced.

As for the generated waste waters, these have practically the same properties as those generated in the processes using tap water, with slightly lower salinity. Therefore, the waste waters do not cause a problem prior of their subsequent treatment at IDR.

One of the greatest advantages of using regenerate water is that it is not subject to the variability of climatic conditions. Hence, it is **guaranteed water**, always available, regardless of the rain that falls. On the other hand, it partly frees the tap water resources currently consumed by the industry and makes them accessible to other uses such as human consumption. In front of the enduring drought, and once the technical feasibility of the process has been successfully demonstrated, the investment in infrastructures for the reuse of purified waters should become a priority for all government services.

ACKNOWLEDGEMENTS

This study was supported by the "Consell General de Cambres de Comerç de Catalunya" and the "Agència Catalana de l'Aigua". The authors wish to thank X. Elias from the CGCC and J.M. Puigdengoles from the ACA, M. Vila from IDR, J. Ballester, J. Aribau and M. Gonzalez from AIICA, M. Reyes and M. Cobos from the Leather Technology School, C. Gonzalo from Astramatic, and Curtits Aqualata SA, and Miret y Cía, SA for their assist and collaboration.

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