CHAPTER – III

➢ PROCESS DETAILS OF REPEATED SOAKING AND ALLIED WORKS

CHAPTER III

III.1 Introduction

It has been discussed under section II.7 in chapter II that the swelling of hides and skins reduces gradually in the liming operation with increasing salt content in the lime liquor. This suppresses swelling of collagen fiber and has adverse effects in the successive unit operations. Further the salt concentration in stock of successive lots gradually increases this also creates problem in operations. To overcome the problem, gradually lesser quantity of stock has been used in soaking operation to maintain necessary ratio of stock to water and subsequent operations have been carried out in a single lot for maintaining uniformity. Finally the efficacy of repeated soaking process has been ascertained by comparing physical and chemical properties of finished leather as against the control. Since the physical and chemical properties of finished leather not only depend on the recipe followed for processing but also on the physico-chemical condition of individual raw skin. Keeping this in view the skins is cut into two halves, one half processed by the conventional soaking process and the other half by following the new method.

All skins are cut into two halves along the back bones keeping the grain side up and all left sides are arranged according to their reducing weight giving sample numbers like ε-01, ε-02, ε-03 etc. and corresponding right sides as E-01, E-02, E-03 etc. The necessity of arrangement of samples according to reducing weight has been discussed in section III.3.2. To detect each and every sample from both new and the conventional process finished leather from both the process streams have been suitably marked also. Another important study that has been done here is to find out the gradual changes of different parameters like pH, density, total dissolved solids, salinity, BOD_5^{20} and COD of soak liquor after each and every repetition of soaking.

All the materials and equipment/instruments used with their specifications and source are given below in the section III.2.

III.2 Materials and specification of the Equipment/Instruments

III.2.1 Materials

The materials used in the present study have been listed below with their source in Table III.1.

Table III.1 List of Materials used in the Present Study

Sl. No.	Materials	Source
01	Wet-salted goat skins	Local Tannery (Weblack Tannery)
02	Sodium sulphide flakes (LR)	E. Merck India Ltd.
03	Hydrated Lime (CR)	Local made (Lucky Stores, Kolkata.)
04	Ammonium sulphate (LR)	E. Merck India Ltd.
05	Sodium bi sulphite (LR)	S.D. Fine Chem. Ltd. India.
06	Bate (Vinkol A)	Schill + Seilacher AG. Germany.
07	Wetting agent (Sandozin NIS)	Clariant India Ltd.
08	Common salt (CR)	Local made (Lucky Stores, Kolkata.)
09	Sodium formate (LR)	E. Merck India Ltd.
10	Sulfuric acid (CR)	Biswabharati Chemicals, Kolkata
11	Basic chrome sulphate (CR)	Sun chrome India Ltd.
12	Sodium thiosulfate (LR)	Loba Chemie Pvt. Ltd., India.
13	Sodium bi carbonate (LR)	Loba Chemie Pvt. Ltd., India.
14	Acetic acid (LR)	E. Merck India Ltd.

Table III.1 (Continued)

Sl. No.	Materials	Source
15	Neutralizing syntan (Neutrigan MO)	BASF India Ltd.
16	Fat liquor (Lipoderm Liquor 2 FB)	BASF India Ltd.
17	Dye (Yellow 2RL)	Clarient India Ltd.
18	Syntan (Relugan RE)	BASF India Ltd.
19	Syntan (Basyntan DI)	BASF India Ltd.
20	Vegetable tannin (G.S. Powder)	Unicorn India Ltd.
21	Fat liquor (Lipoderm Liquor SAF)	BASF India Ltd.
22	Fat liquor (Lipoderm Liquor Oil SK)	BASF India Ltd.
23	Fat liquor (Sandolix WWL)	Clariant India Ltd.
24	Preservative (Busan 30L)	Buckman Laboratories (U.S.A.)
25	Formic acid (LR)	E. Merck India Ltd.
26	Fat liquor (Lipamin Liquor FB3)	BASF India Ltd.
27	Protein binder (Eukesol Binder U)	BASF India Ltd.
28	Potassium chromate (LR)	S.D. Fine Chem. Ltd. India.
29	Silver nitrate (LR)	S.D. Fine Chem. Ltd. India.
30	Filter paper, no. 541	E. Mark & Whatman (India) Ltd.
31	Magnesium sulfate (AR)	E. Mark (India) Ltd.
32	Calcium chloride (AR)	E. Mark (India) Ltd.
33	Ferric chloride (AR)	E. Mark (India) Ltd.

Table III.1 (Continued)

Sl. No.	Materials	Source
34	pH Buffer, pH-7	BDH Chemicals
35	Potassium dichromate (LR)	Loba Chemie Pvt. Ltd., India.
36	Potassium Iodide (LR)	BDH Chemicals
37	Mercurous sulphate (LR)	S.D. Fine Chem. Ltd. India.
38	Nitric acid (LR)	Biswabharati Chemicals, Kolkata
39	Calcium carbonate (LR)	S.D. Fine Chem. Ltd. India.
40	Hydrochloric acid (LR)	Biswabharati Chemicals, Kolkata
41	Barium chloride (LR)	S.D. Fine Chem. Ltd. India.
42	Sodium peroxide granular GR	E. Mark (India) Ltd.
43	Starch Indicator (LR)	Loba Chemie Pvt. Ltd., India.
44	Phenolphthalein Indicator	E. Mark (India) Ltd.
45	Bromocresol Green Indicator	E. Mark (India) Ltd.
46	Silica Gel (LR)	Merck Ltd., Mumbai, (India).
47	Glycerol (LR)	Merck Ltd., Mumbai, (India).
48	Alum (Aluminium Sulfate) (LR)	Merck Ltd., Mumbai, (India).

III.2.2 Specification of the Equipments / Instruments

III.2.2.1 Drum for leather processing

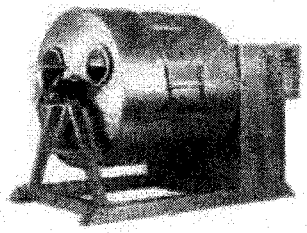


Fig. III.1 Drum

Make: The Bengal Tanning Machineries (Kolkata).

Dia: 2-feet 6-inch.

Width: 1-feet 2-inch.

rpm: 14

Capacity: Up to 6 kg raw skins. The instrument is used for all wet operations like soaking, liming, Deliming, pickling, chrome-tanning etc. for leather processing. That equipment actually acts as reactor for chemical process for leather making from hides and skins.

III.2.2.2 Semi circular fleshing knife

Make: Local made.

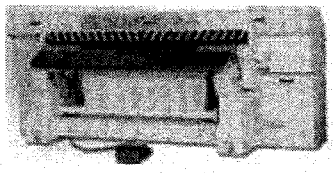
Length: 3-feet with handle. The tool is used to remove fat, flesh, hair and all other tissues still remains on the skin after liming.

III.2.2.3 Shamming machine

Fig. III.2
Shamming machine

Make: Baggio, Italy. The machine is used before shaving and after fat-liquoring of skins for spreading, to render it flat and well spread for pressing, to squeeze out all the excess water.

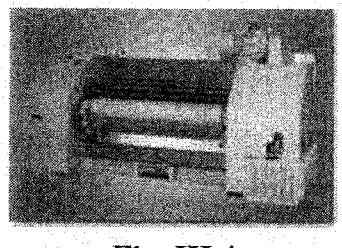
III.2.2.4 Shaving machine



Make: Flamar Spa, Italy. The machine is used to obtain uniform, precise thickness of skins at the wet-blue condition.

Fig. III.3
Shaving machine

III.2.2.5 Setting machine



Make: The Bengal (Machinery) Company, India. The machine is used to make the skin pleasing to eye and touch by spreading and flattening as far as possible.

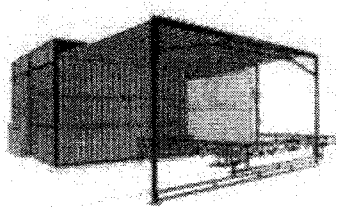
Fig. III.4
Setting machine

III.2.2.6 Staking machine

Make: The Bengal (Machinery) Company, India. The machine is used to separate adhered fibers of skin after conditioning, just prior to crust stage, to make it soft and pliable.

Fig. III.5
Staking machine

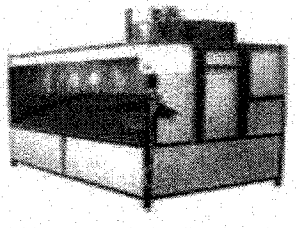
III.2.2.7 Toggling machine



Make: Made in China (HAIAN WEIGUO MACHINERY Co. LTD). The machine is used to spread out the skin and make it dry in that condition.

Fig. III.6
Toggling machine

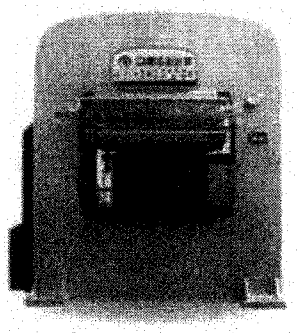
III.2.2.8 Spray booth



Make: Made in India (Auroelectronics). It is used to spry different finish solutions on to the grain side of the leather.

Fig. III.7
Spray booth

III.2.2.9 Hydraulic press



Make: P. Mostardini & Figli Empoli (Italy). It is used for plane platting of leather with pressure and temperature for ironing effect.

Fig. III.8
Hydraulic press

III.2.2.10 Pan balance

Make: Kusum Engineering Works Ltd. India. Capacity: Up to 5 kg. This balance is used to measure different chemicals needed for different wet-unit operations of leather processing.

III.2.2.11 Digital pH Meter

Make: Systronic Instruments Pvt. Ltd., India.

Model: 335, Sr. No. 3137

Temperature range: 0 to 100^0 C.

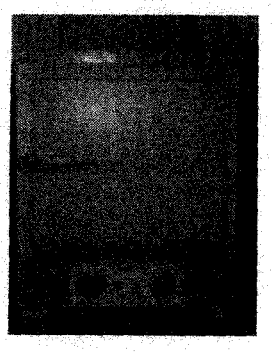
The instrument is used to measure p^H of different solutions for analytical testing purpose.

III.2.2.12 Electronic balance

Make: M/s. Mettler Toledo Co.

Range: up to 130 g. It is used to measure weight of different chemicals for analytical testing purpose.

III.2.2.13 Air Oven

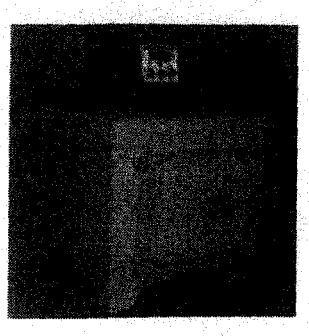


Make: M/s. S.C. Dey & Co. Pvt. Ltd., Kolkata, India.

Temperature range: up to 250^0C. It is used for different physical and chemical testing of leather.

Fig. III.9
Air oven

III.2.2.14 Muffle Furnace



Make: M/s. S.C. Dey & Co. Pvt. Ltd., Kolkata, India.

Temperature range: up to 1200^0C. It is used for different chemical testing of leather.

Fig. III.10
Muffle furnace

III.2.2.15 BOD Incubator

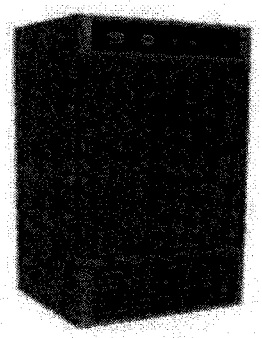


Fig. III.11
BOD incubator

Make: M/s. S.C. Dey & Co. Pvt. Ltd., Kolkata, India.

Temperature range: -5 to 60^0C. It is used for incubation of soak liquors for BOD_5^{20} measurement.

III.2.2.16 Digital Turbidity Meter

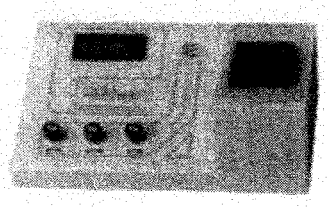


Fig. III.12 Digital turbidity meter

Make: Testing Instruments Mfg. Company, India.

Model: 331, Range: 0 to 1000 NTU,

Accuracy: ± 3% of full scale in 0 – 1000 NTU.

The instrument is used to determine turbidity of Alum treated soak liquor.

III.2.2.17 Water bath

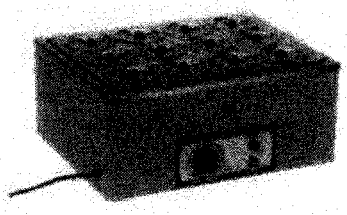
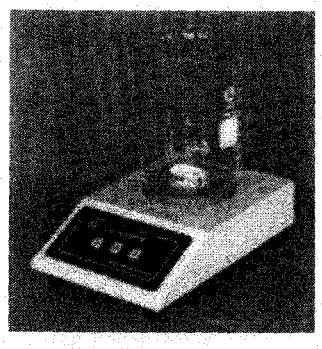


Fig. III.13
Water bath

Make: Lab Equipments & Chemicals, India.

12 – Holes, Electrical immersion heater fitted for heating of water. The instrument is used to determine different analytical testing.

III.2.2.18 Magnetic Stirrer

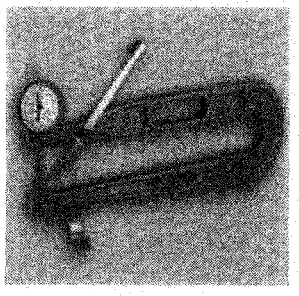


Make: Schott Instruments GmbH.

Model: D-55122 Mainz. It is used to dissolving solute in the solvent for different Analytical testing purpose.

Fig. III.14
Magnetic stirrer

III.2.2.19 Thickness Measuring Gauge



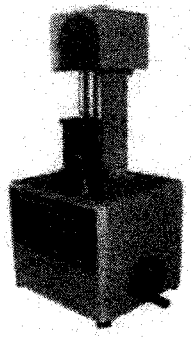
Make: Mitutoyo (Japan),

Range: 0.01 – 10 mm. Model No. : 2046-08.

The instrument is used to measure thickness of finished leather.

Fig. III. 15 Thickness measuring gauge

III.2.2.20 Shrinkage Temperature Tester



Make: Testing Instruments Mfg. Company, India.

The instrument is used to measure shrinkage temperature of finished leather.

Fig. III.16
Shrinkage temperature tester

III.2.2.21 Water Vapour Permeability Tester

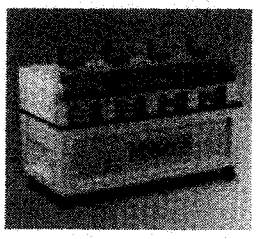


Fig. III.17 Water vapour permeability tester

Make: Testing Instruments Mfg. Company, India.

The instrument is used to measure water vapour permeability of finished leather.

III.2.2.22 Tensile Strength & % Elongation Measurement Tester

Fig. III.18 Tensile strength tester

Make: Precision Scientific Equipment Corp. New-Delhi (India).

Model No. : RT-5,

Sl. No. : 790082.

The instrument is used to measure tensile strength and percentage of elongation of finished leather.

III.2.2.23 Humidity & Thermometer Meter

Make: Testing Instruments Mfg. Company, India.

Temp. Range: -20°C ~1000°C.

Resolution: 0.1^0 up to 200^0C, 1^0 over 200^0C.

Humidity Range: 5% RH ~98% RH.

It is used to measure temperature and percentage humidity for different testing purpose.

III.2.2.24 Kubelka Apparatus for the Measurement of % Water Absorption

Make: Testing Instruments Mfg. Company, India.

With Borosil ® -500 mL beaker (27^0C).

It is used for different testing purpose.

(sketch of kubelka apparatus has been shown in the section III.5.8)

III.2.2.25 Double Hole Stitch Tear Strength Tester

Make: Precision Scientific Equipment Corp. New-Delhi (India).

Model No. : RT-5,

Sl. No. : 790082.

It is used for measuring stich tear strength of finished leather.

(It has been shown in the section III.2.2.22)

III.2.2.26 Grain Crack & Ball Bursting Strength Tester

Make: Lab Equipments & Chemicals, India.

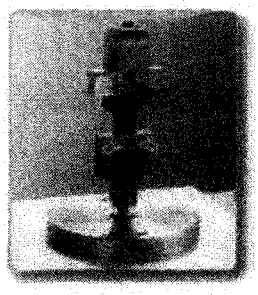


Fig. III.19 Grain Crack and Ball Bursting Strength Tester

The instrument is used to measure grain crack and ball bursting strength of finished leather.

III.2.2.27 Colour Rub fastness Tester

Make: Lab Equipments & Chemicals, India.

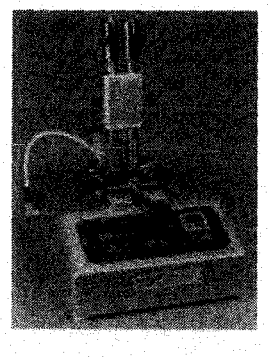


Fig. III.20 Colour rub fastness tester

Grey Scale: Used as designed by The Society of Dyers and Colourists of England.

The instrument is used for wet and dry colour rub fastness of finished leather.

(Sketch of grey-scales have been shown in the section III.5.9)

III.3 PROCESS DETAILS OF LEATHER PREPARATIONS

There are various types of finished leather with different physical and chemical properties for different applications, such as shoe-upper, bag, shoe lining, garment etc. All finished leathers can be categorized almost in three groups according to their feel i.e. hard, medium-soft or soft. These variations can be controlled in processing mainly by controlling shaving thickness, extent of neutralization and choosing of quality and quantity of syntans and fats. Thus for the judgment of efficacy of the proposed soaking, medium-soft type leathers, like shoe lining leather, bag leather etc. has been considered so that the followed process equally viable to all. Therefore in the present study shoe lining type of medium-soft leather has been made following the proposed method of soaking. It may be mentioned here that the physical and chemical properties of lining leather is more or less similar to many other varieties of finished leather. In conventional method of soaking, raw skins are drummed for one hour with 400% water based on weight of raw skin and then left in the drum over night. It has been discussed in Chapter I under section I.2 that the main objective of soaking is to rehydrate the skins by dipping in water to enable absorption

of sufficient water. In the new method of soaking the skins instead of being left in the drum overnight are dipped in dilute sodium sulphide liquor after a certain minimum period of drumming to avoid deterioration of the skins by bacterial action. The minimum period of drumming has been determined following the procedure described in section 3.III.1.

III.3.1 Optimization of drumming period for soaking

Three types of wet-salted raw goat skins sample of different curing period has been collected from local raw hide market like; Sample C-1, Sample C-2 and Sample C-3 having cured for 15 days, 1 month and 2 months respectively. Five small pieces of almost equal weight (about 15g) of each category of wet-salted skins has been taken for the experiment. Then each sample is taken separately in a Kubelka apparatus with 400% water on sample weight. The volume of water is measured and placed on a magnetic stirrer for agitation of liquor with stirring paddle speed 20 rpm approximately. The volume of water absorbed (in mL) by wet-salted skin is measured from Kubelka apparatus for each 10 minutes interval up to 90 minutes with stirring and finally after one day without stirring. The percentage (V/W) of water absorption (in mL) on the basis of wet-salted skin weight (in g) is calculated. Each experiment has been done with replication of five. The average results obtained from the experiments are given bellow in the Table III.2. The amount of water absorption by wet-salted skins depends on several factors, like; curing period, thickness of the raw skin, amount of adipose tissue attached on the flesh layer etc. But, it is found from the results of the experiment that the wet-salted skins absorb water exponentially with a high rate of absorption initially. It has been observed that 20 to 40 percent water uptake takes place within first 10 minutes of the soaking. 48 to 58 percent is absorbed within 60 minutes and after that up to 24 hours soaking, the water absorption is hardly

increased by only 5 to 10 percent against the wet salted weight. In case of drum soaking, the rate of absorption and also total amount of water absorption within 60-minutes may be more due to more mechanical action instead of stirring.

Table III.2 Percentage of Water Absorption by Wet-Salted Raw Skins with Time

Category	% of water absorption by wet-salted raw skins after (V/Wx100)									
	10′	**20′**	**30′**	**40′**	**50′**	**60′**	**70′**	**80′**	**90′**	**1-day**
C-1	35	41	42.5	47.5	49	50.5	52.5	54	54.5	58
C-2	27.5	34	40.5	51	53	56	57.5	58.5	59	60.5
C-3	21	24	30	41.5	48.5	55.5	58	59	61	66.5

The average value of percentage water absorption at different time interval up to 90 minutes is calculated for five samples of each category of wet-salted skins. The result is presented in a graphical form in **Fig III 21.** It is evident from the results that one hour drumming is sufficient for satisfactory soaking of the skins.

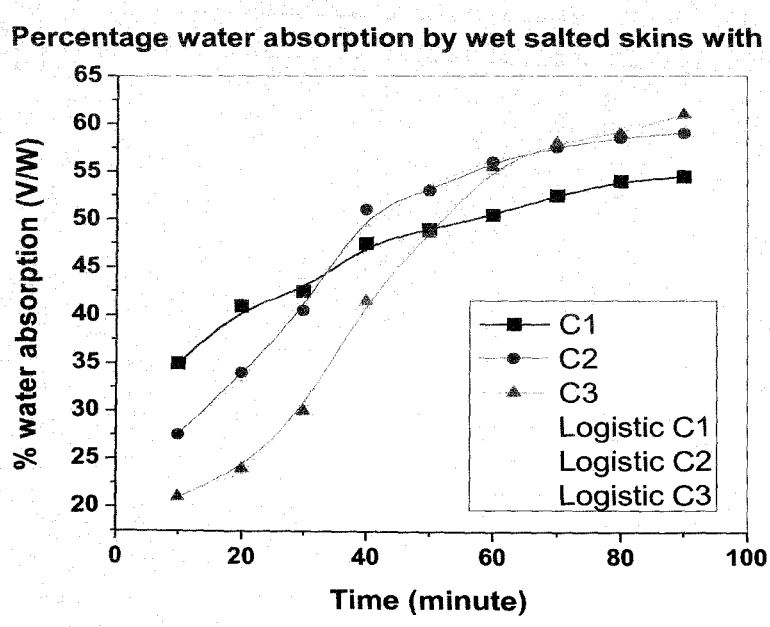


Fig. III.21 Percentage water absorption (100xV/W) by wet-salted skins with time

Table III.3 Fitment parameters of various samples fitted with logistic model equation*

Sample	A_1	A_2	x_0	p	R^2	Chi^2/DoF
C1	26.55722	72.47211	52.87871	0.88329	0.99001	0.70105
C2	26.91182	60.13649	31.79667	3.14729	0.99402	1.30372
C3	21.06335	62.57557	41.0448	3.86628	0.99851	0.59313

* Model: Logistic; Equation: $y = A_2 + (A_1-A_2)/\{1 + (x/x_0)^p\}$

III.3.2 Preparation of sample numbers

Now, fifteen pieces of wet-salted raw goatskins are taken for processing following the new soaking method. Raw skins of different curing period as described in Chapter III under section3.1 have been selected for processing. All the skins are cut into two halves keeping the grain side ups, along the backbone by a sharp cutting knife. Special care has been taken for handling the skins to minimize loss of adhered salts during trimming and cutting. The mechanical beating of skins is very much dependent on the percentage of float used for drum soaking. Mechanical beating increases with the decreasing amount of float present in the drum. High mechanical beating always deteriorates the strength of leathers. Generally, 200 to 400 percent float on the basis of raw or pelt weight of stock during drumming is considered very much convenient to minimize the loss of physical strength of leather. For reusing the soak liquor in the new method, 400 percent float (water) on the basis of wet-salted goat skins has been maintained. During soaking, it is generally found that a considerable amount of water is absorbed by the skins and with the simultaneous removal of adhered dirt, dung, salt, loose hair and soluble proteins into the soak liquor. Due to this reason, the weight of soak liquor after soaking is decreased. Thus, soaking of the next sample (wet-salted raw skin) reusing the spent soak water, gradually reducing sample weight will be

convenient to maintain 400% float on raw weight basis. Thus, we need a sequence of gradually reducing weight of the sample for present studies. The left sides of all wet-salted raw goat skins are arranged according to their reducing weights and treated as experimental sides i.e. where the new method of recycling and reuse of spent soak liquor has been adopted. On the other hand, all right sides are to be treated in the conventional way. Thus, samples have been numbered as ε-01, ε-02, ε-03 .... ε-15 against left sides for proposed method of study i.e. experimental samples and E-01, E-02, E-03 .... E-15 is being marked against right sides for conventional method of study i.e. conventional samples. Raw weights of sides are being expressed in Table III.4.

Table III.4 Raw Weights against Experimental and Conventional Sample Nos.

Experimental sample nos.	Weight of the left sides (g)	Conventional sample nos.	Weight of the right sides (g)
ε -01	528	E-01	488
ε -02	512	E-02	493
ε -03	438	E-03	435
ε -04	420	E-04	431
ε -05	415	E-05	395
ε -06	408	E-06	364
ε -07	363	E-07	360
ε -08	314	E-08	324
ε -09	284	E-09	296
ε -10	276	E-10	269
ε -11	268	E-11	258
ε -12	248	E-12	304
ε -13	210	E-13	196
ε -14	202	E-14	198
ε -15	189	E-15	223

These sample numbers has been maintained through out.

III.3.3 Procedure of marking against sample numbers

Now each side of experimental and conventional is being marked against their sample numbers by punching holes. The samples are being differentiated by the following factors:

I. Number of holes (e.g. 1 or 2),

II. Position of the holes on the side (e.g. neck, fore shank or hind shank),

III. Orientation of the holes (as shown in the sketch follows) and

IV. The relative distance between two holes.

For better understanding, the following **figures III.22 to III.37** are being given:

ILLUSTRATION OF IDENTIFICATION MARKS OF THE SAMPLES

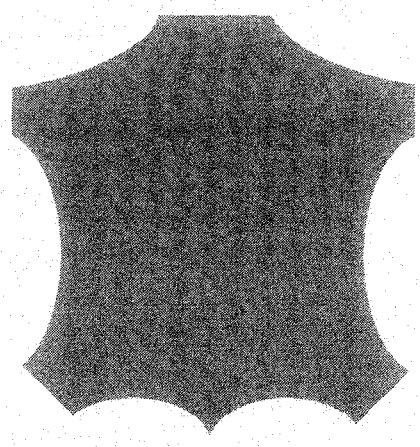


Fig. III.22

Pattern of full piece goat skin

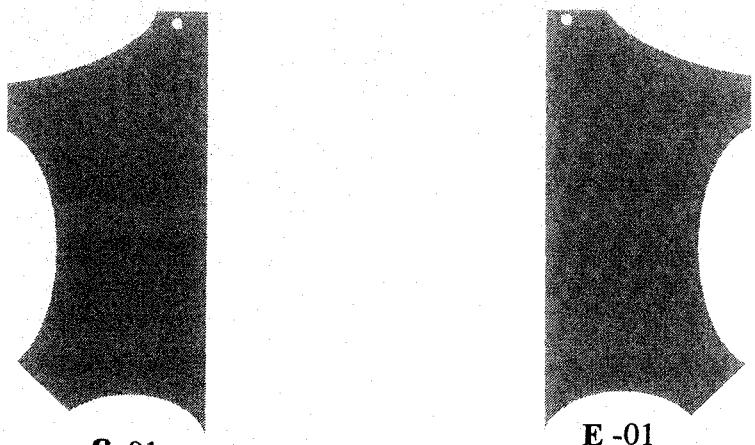
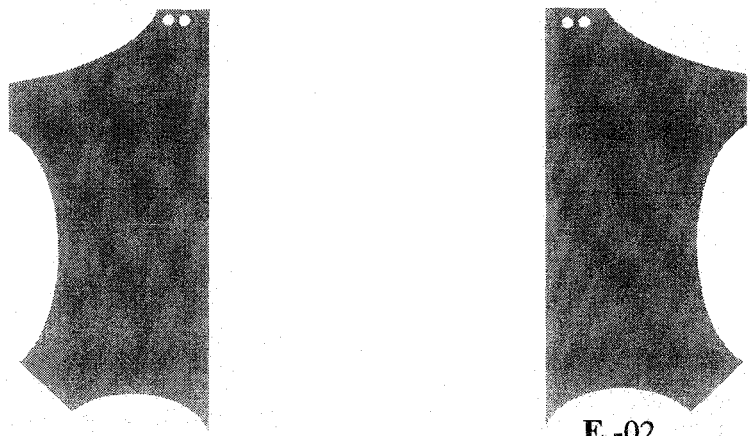


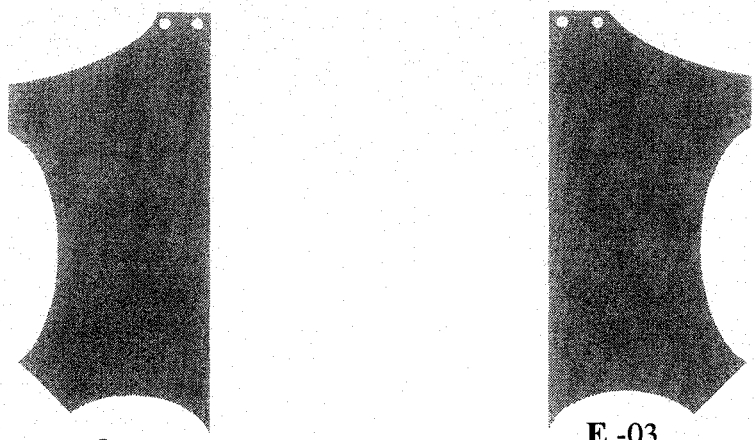
Fig. III.23

Experimental (Ɛ–01) and Conventional (E-01) sample sides



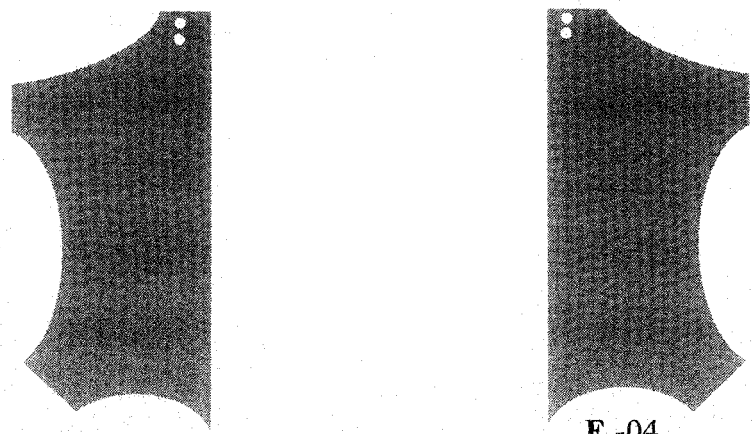
Ɛ–02 E -02

Fig. III.24
Experimental (Ɛ–02) and Conventional (E-02) sample sides



Ɛ–03 E -03

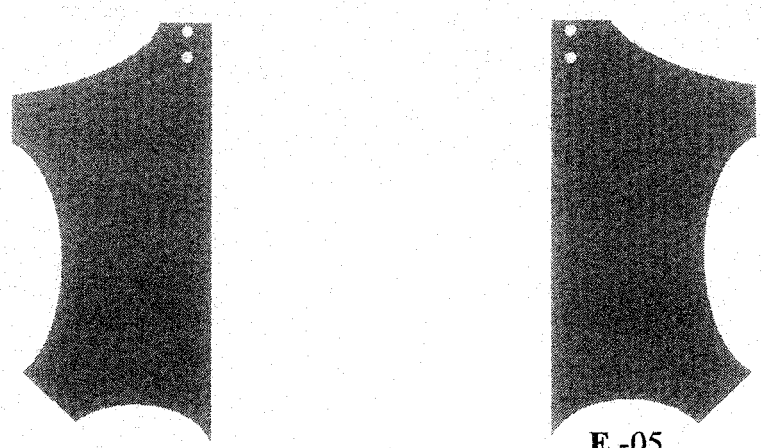
Fig. III.25
Experimental (Ɛ–03) and Conventional (E-03) sample sides



Ɛ–04 E -04

Fig. III.26

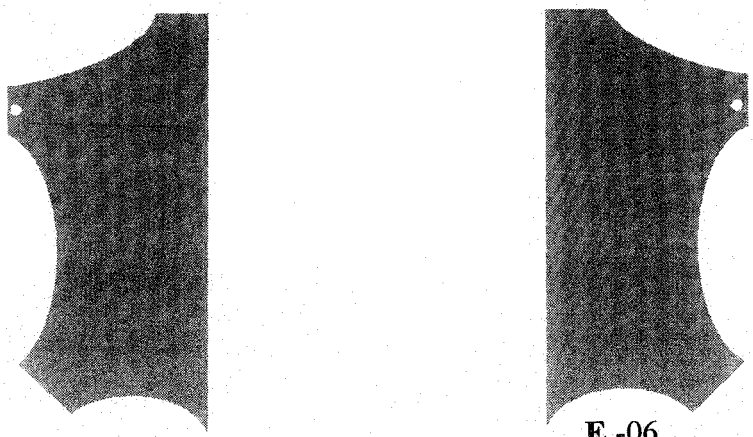
Experimental (Ɛ–04) and Conventional (E-04) sample sides



Ɛ–05 E -05

Fig. III.27

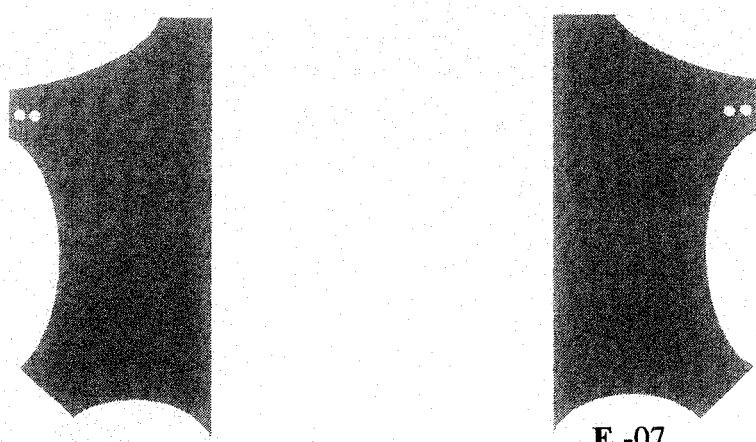
Experimental (Ɛ–05) and Conventional (E-05) sample sides



Ɛ–06 E -06

Fig. III.28

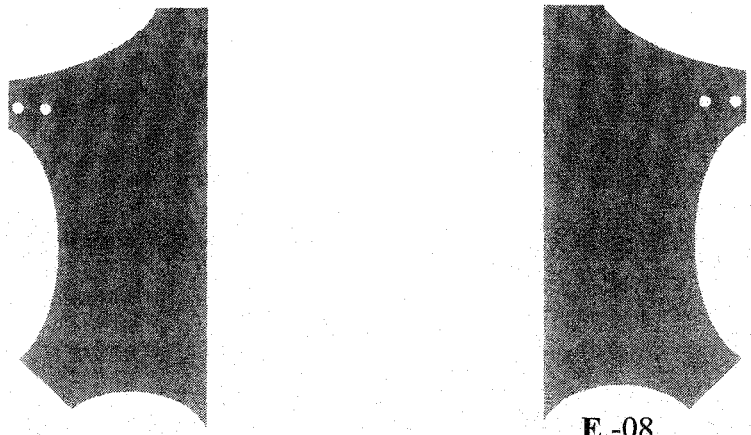
Experimental (Ɛ–06) and Conventional (E-06) sample sides



Ɛ–07 E -07

Fig. III.29

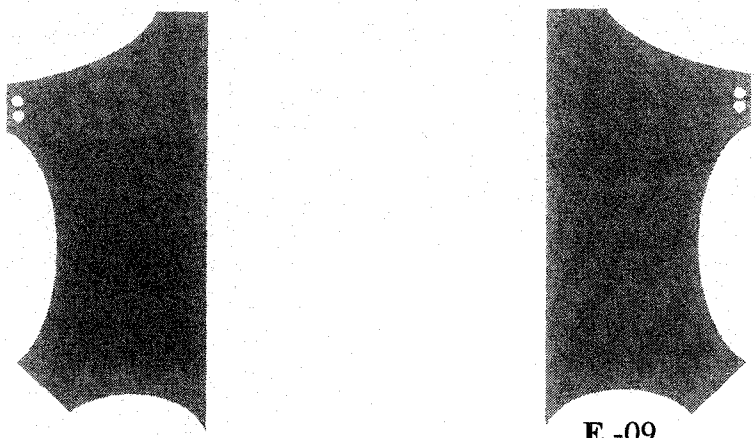
Experimental (Ɛ–07) and Conventional (E-07) sample sides



Ɛ–08 E -08

Fig. III.30

Experimental (Ɛ–08) and Conventional (E-08) sample sides



Ɛ–09 E -09

Fig. III.31

Experimental (Ɛ–09) and Conventional (E-09) sample sides

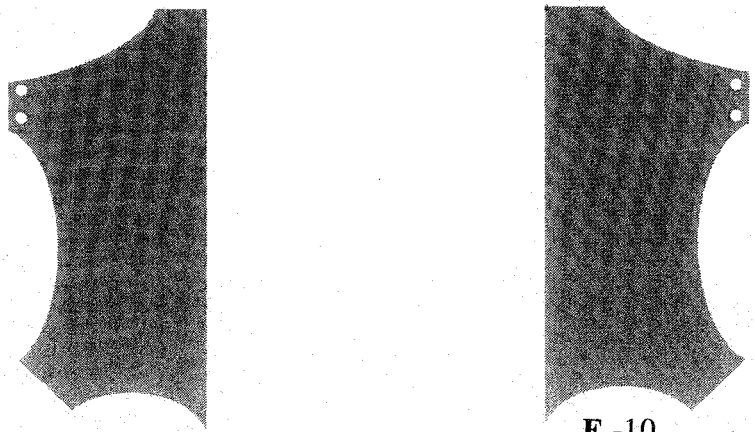


Fig. III.32
Experimental (Ɛ–10) and Conventional (E-10) sample sides

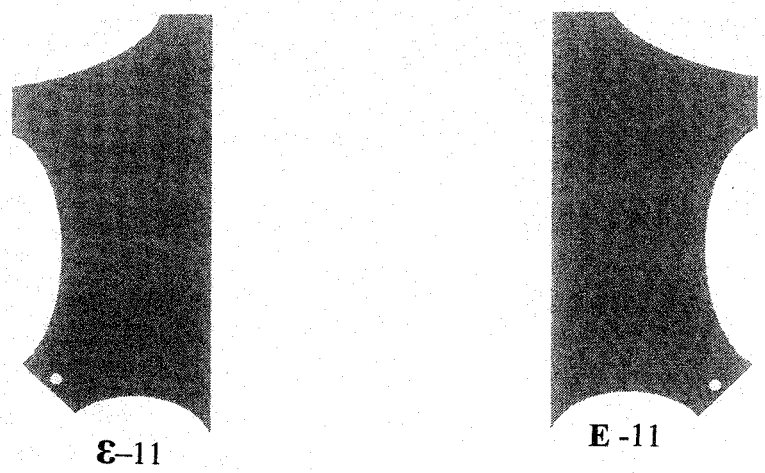


Fig. III.33
Experimental (Ɛ–11) and Conventional (E-11) sample sides

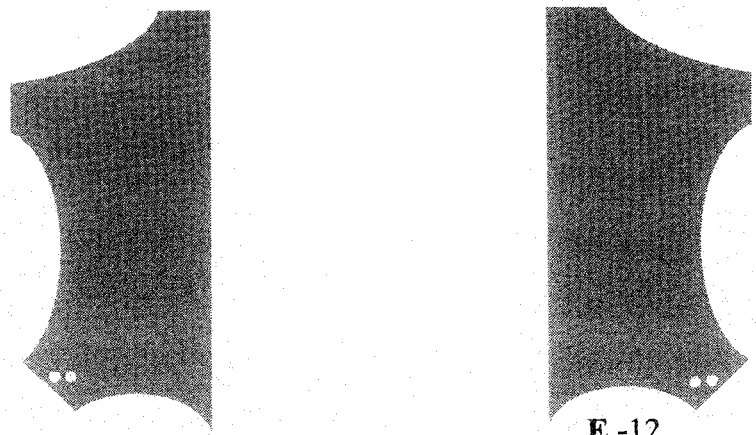


Fig. III.34

Experimental (Ɛ–12) and Conventional (E-12) sample sides

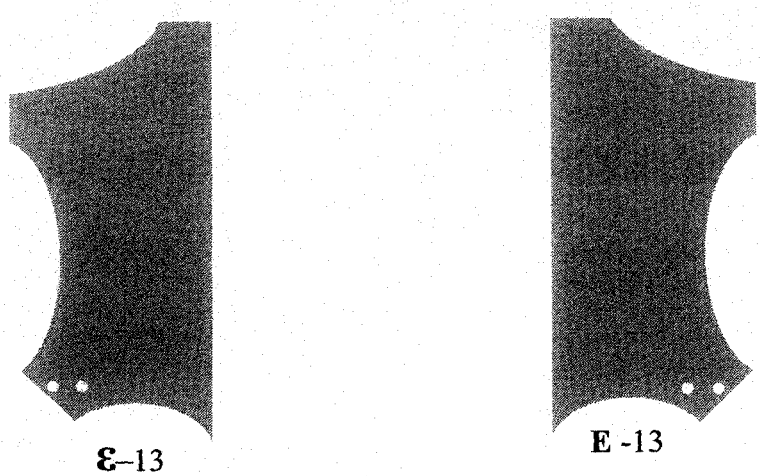


Fig. III.35

Experimental (Ɛ–13) and Conventional (E-13) sample sides

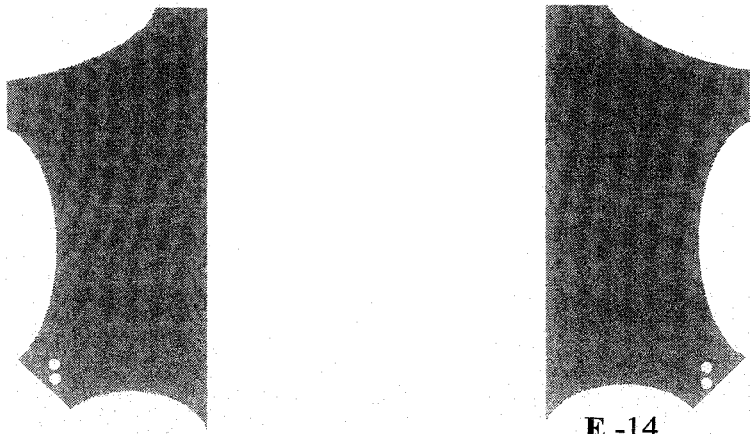


Fig. III.36

Experimental (Ɛ–14) and Conventional (E-14) sample sides

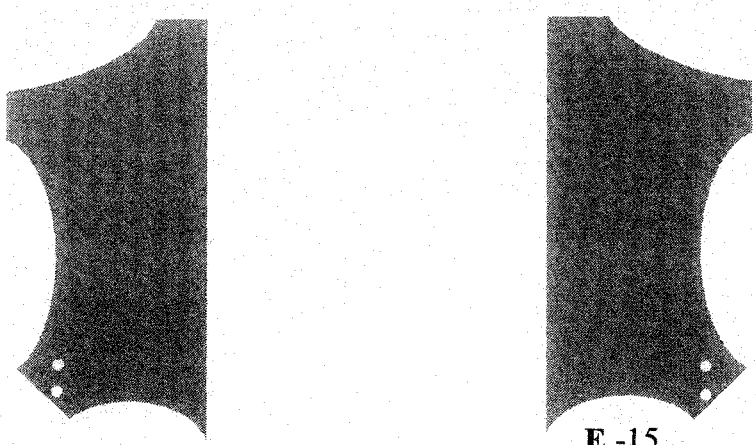


Fig. III.37

Experimental (Ɛ–15) and Conventional (E-15) sample sides

III.3.4 Recipe for experimental (laboratory) processing of leather

Sample numbers denoted by 'ε' are soaked one by one starting from ε-01 in a drum containing approximately 400% water on raw weight basis. Drum is run for 1 hour with 14 rpm for each soaking. Sample of soak liquor collected for analysis to determine its characteristic viz; Density, BOD_5^{20}, COD and such other. The detail procedure for testing has been described in Chapter III under section III. 4. While adding fresh water in different stages care has been taken to maintain the salinity level of the soak liquor by adding some salt wherever necessary. Since the objectives of the studies are to find out how many times a certain amount of water can be reused for soaking till the water is saturated with salt this process step will not in any way affect the generality of the objective. It is presumed that the volume of the solution will not change with the addition of salt, as the volume of salt is much less as compared to the volume of the water. Following this procedure it has been found that eleven repetition of soaking is possible up to a sample marked ε-11. The different data for soaking of experimental sample has been presented in Table No.-III.5. Since each side was soaked separately, it was necessary to put them after soaking in a stock solution of 1% Na_2S and 400% water (% based on w/s weight). Then all the eleven samples (ε-01 to ε-11) are drummed for 5-minutes in the same solution and kept overnight. Next day liming operation is done with 6% - hydrated lime and 3% - sodium sulfide with the basis of w/s weight. The addition of chemicals and the duration of drumming are maintained as followed in conventional process in Chapter III under section 3.7. After that the whole stock is finished for beige coloured medium soft leather following the same recipe as mentioned in conventional process in Table III.7. In following the repeated soaking operation it has been observed that there was no qualitative difference in the processed leathers as compared to leathers produced by the

conventional route. The physical and chemical properties of finished leathers of both the samples denoted by 'E' and 'ε' have been determined and presented in Table B.14. It is observed from this table that some of the properties of the leather produced following the repeated soaking route are in fact better. Physical and chemical properties of finished leathers have been determined following standard procedure as described in Chapter III under section III.5.

Table III.5 Soaking table for the Experimental samples

Sample No.	Raw weight of skins (g)	Weight of water before soaking (g)	Weight of water after soaking (g) [excluding 20 ml for testing]	Reduction of water after soaking (g)	Density of soak water (g/c.c.)	pH of soak water after soaking	Soaked weight of skins (g)	Adjustment to maintain 400% soak water		Observations
								Water (g)	Salt (g)	
ε - 01	528	2112	1832	280	1.03	7.9	631	210	6	Removal of salt with some loose hair and the feel of soaked skin is achieved.
ε - 02	512	2048	1791	257	1.07	7.9	622	-	-	Same as previous sample.
ε - 03	438	1791	1510	281	1.09	8.0	534	156	14	Same as previous sample but the soak liquor becomes more opaque and dirty.
ε - 04	420	1680	1370	310	1.12	7.9	517	259	31	Same as previous sample and the dirtiness of the liquor gradually increases.
ε - 05	415	1660	1307	353	1.13	8.1	515	288	37	Removal of salt and the feel are satisfactory.

Table III.5 (Continued)

Sample No.	Raw weight of skins (g)	Weight of water before soaking (g)	Weight of water after soaking (g) [excluding 20 ml for testing]	Reduction of water after soaking (g)	Density of soak water (g/c.c.)	pH of soak water after soaking	Soaked weight of skins (g)	Adjustment to maintain 400% soak water		Observations
								Water (g)	Salt (g)	
ε-06	408	1632	1287	345	1.15	7.9	508	143	22	Swelling of skin is suppressed to some extent but salt removal is satisfactory.
ε-07	363	1452	1081	371	1.16	7.9	454	151	24	Till now everything is o.k. except suppressed swelling and more dirty liquor
ε-08	314	1256	876	380	1.18	7.8	402	220	40	More or less same as previous results but liquor is nearly saturated by salt.
ε-09	284	1136	862	274	1.19	7.8	371	203	39	The crystal size of salt reduces remaining with the skin surface but till now the feel of soaking is satisfactory.

Table III.5 (Continued)

Sample No.	Raw weight of skins (g)	Weight of water before soaking (g)	Weight of water after soaking (g) [excluding 20 ml for testing]	Reduction of water after soaking (g)	Density of soak water (g/c.c.)	pH of soak water after soaking	Soaked weight of skins (g)	Adjustment to maintain 400% soak water		Observations
ε - 10	276	1104	855	249	1.21	7.9	364	179	38	Salt adheres with the skin surface and inside the drum, liquor becomes extremely dirty but soaking is till nearly satisfactory.
ε - 11	268	1072	853	219	1.22	7.8	370	-	-	It seems, the adhered salts with the skin become more, least feel of soaking and the liquor becomes exhausted to reuse any more.

III.3.5 Important information regarding recycling of soak liquor

Important information regarding recycling of soak liquor obtain from the data appeared in the Table III.5 are given bellow point wise:

1. Total data are created from the experiment of repeated soaking of wet-salted raw goat skins with very short hair of one to two months old preserved stock.

2. The weight of soak water always maintained 400% on the basis of wet-salted raw weight of goatskins to be soaked, except sample No. ε -03, where slightly excess amount of water (39 g) has been used by default.

3. Soaked weight of each skin has been measured in a digital balance taking in a plastic tub allowing loose water left in the drum as much as possible. The weight is expressed in the table to the nearest integer.

3. Weight of water after soaking has been measured in a digital balance taking in another plastic tub after collecting 20ml for testing TDS, salinity, COD and such others and the weight is expressed in the nearest integer.

4. Density of soak liquor is measured by weighing the 20ml soak liquor keeping in a sample bottle in a digital balance.

5. pH of the liquor after soaking has been measured in a digital pH meter.

6. Water and salt have been calculated for adjustment to make up 400% for the next soaking as follows:-

Weight of Water = [weight of 400% water required for soaking of sample no. ε-(n-1) – weight of water after soaking of sample no. ε-n] / Density of water after soaking of sample no. ε-n (considering the nearest whole number), where n stands for sample number .

Weight of Salt = [weight of water before soaking of sample no. ε-(n-1) – weight of water after soaking of sample no. ε-n] – weight of water taken for adjustment.

7. Total raw weight from sample no. ε–01 to ε–11 (W_R) = 4226 g.

8. Total weight of fresh water used for soaking (W_W) = 2112 g (400% of raw weight of sample no. ε–01) + 1809 gm (for adjustment to make up 400% water for soaking) = 3921 g.

9. Weight of salt used for adjustment (W_{ST}) = 251 g.

10. The weight of water remained after complete soaking (W_U) = 853 g.

11. Total soaked weight from sample no. ε–01 to ε–11 (W_S) = 5288 g.

12. Total weight of water taken (20 ml for each soaking) for testing (W_T) = 251 g (i.e. 20 x summation of densities).

13. Thus the weight loss during process = $(W_R + W_W + W_{ST}) - (W_U + W_S + W_T)$ = 8398 – 6392 = 2006 g which is nearly 24% of the total weight $(W_R + W_W + W_{ST})$.

14. Total water used in the process for study = $W_W + W_T$ = 3921 + 251 = 4172 g which is nearly **800% of the raw weight of the sample no. ε–01.**

15. **The raw weight of sample no. ε –11 is nearly half of the same of sample no. ε–01.**

III.3.6 Process optimization for soaking

Based on the information obtained from the repeated soaking operation already done, the process has been repeated in the way described below for process optimization. Eleven pieces wet-salted raw goat skins of different curing period as described in Chapter III under section 3.1 have been taken and processed. The highest raw weight of a single piece in the lot has been found to be1200 g and the lowest 650 g. The skin of raw weight 650 g has been trimmed slightly from the edge to make it 600 g i.e. half of the 1200 g. The raw weights of the other nine samples are maintained so that the differences between two adjacent samples are 1200/20 =60 g. In this manner, the raw weight of any sample number can be determined by the formula;

$W_{\varepsilon\text{-}n} = \{20 - (n - 1)\} \times W_{\varepsilon\text{-}01} / 20$ [where $W_{\varepsilon\text{-}01}$ denotes raw weight of sample no. ε –01 and $1 \leq n \geq 11$] and the raw weight (W_R) of total samples from ε –01 to ε –11 can be calculated simply by **multiplying the number 8.25 with $W_{\varepsilon\text{-}01}$.**

All these data can be calculated out simply by application of the properties of Arithmetic Progression. Let, the first term (T_1) i.e. weight of sample no. ε –01 of the series, $W_{\varepsilon\text{-}01} = W$, so consequently the eleven term (T_{11}) i.e. weight of sample no. ε -11 of the series will be $W_{\varepsilon\text{-}11} = W/2$, as it was in experiment. Thus according to the formula we can write:-

$T_{11} = T_1 + (11 - 1)\, D$. [here D is common difference]

⇨ $W_{\varepsilon\text{-}11} = W_{\varepsilon\text{-}01} + (11 - 1)\, D$. [since, $T_{11} = W_{\varepsilon\text{-}11}$ and $T_1 = W_{\varepsilon\text{-}01}$]

⇨ $W/2 = W + 10D$. [since, $W_{\varepsilon\text{-}11} = W/2$ and $W_{\varepsilon\text{-}01} = W$]

⇨ $D = - W/20$

⇨ **$D = - W_{\varepsilon\text{-}01} /20$.** [since $W = W_{\varepsilon\text{-}01}$]

In this case, $D = -1200/20 = -60$ g [since $W_{\varepsilon\text{-}01} = 1200$ g]

Now, the term T_n (where $1 \leq n \geq 11$) i.e. the weight of sample no. ε-n ($W_{\varepsilon\text{-}n}$) can be calculated out from the formula:-

$T_n = T_1 + (n - 1)\, D$. [Where $1 \leq n \geq 11$]

⇨ $W_{\varepsilon\text{-}n} = W_{\varepsilon\text{-}01} + (n - 1)\, (-W_{\varepsilon\text{-}01} /20)$ [putting $T_n = W_{\varepsilon\text{-}n}$, $T_1 = W_{\varepsilon\text{-}01}$ and $D = - W_{\varepsilon\text{-}01}/20$]

⇨ $W_{\varepsilon\text{-}n} = W_{\varepsilon\text{-}01} - (n - 1)\, W_{\varepsilon\text{-}01}/20$

⇨ **$W_{\varepsilon\text{-}n} = \{20 - (n - 1)\}\, W_{\varepsilon\text{-}01}/20$.**

From this formula, weight of any lot for repeated soaking can be found.

Now, the summation of the terms from T_1 to T_{11} i.e. the summation of the weight of the sample nos. ε –01 + ε –02 + ε –03 + ……+ ε –11 can be derived from the formula:-

$S_{11} = 11/2 \{2 T_1 + (11 - 1) D\}$ [where S_{11} stands for summation of the series up to term no. 11]

⇨ $W_R = 11/2 \{2 W_{\varepsilon\text{-}01} + 10 (-W_{\varepsilon\text{-}01} /20)\}$ [putting $S_{11} = W_R$, $T_1 = W_{\varepsilon\text{-}01}$ and $D = - W_{\varepsilon\text{-}01} /20$]

⇨ $W_R = 11/2 (2 W_{\varepsilon\text{-}01} - W_{\varepsilon\text{-}01} /2)$

⇨ $W_R = 11/2 \times 3 W_{\varepsilon\text{-}01} /2$

⇨ $W_R = 33/4 \times W_{\varepsilon\text{-}01}$

⇨ $\mathbf{W_R = 8.25\ W_{\varepsilon\text{-}01}}$

Contrary, the weight ($\mathbf{W_{\varepsilon\text{-}01}}$) of first sample (ε–01) for soaking can be determined by dividing the lot weight ($\mathbf{W_R}$) by **8.25** and the weight of successive samples (ε–02, ε–03 … ε –11) can be determined by using the formula: -

$$\mathbf{W_{\varepsilon\text{-}n} = \{20 - (n - 1)\} W_{\varepsilon\text{-}01}/20.}$$

Now, 800% water on the basis of raw weight (1200 g) of sample no ε –01 i.e. 9600 g, has been taken into drum and drumming for 1-hr. maintaining 14 rpm with the sample for soaking. Then the soaked sample is transferred to another drum containing 400% - water and 1% - sodium sulfide based on the raw weight of the lot. Then the next sample is soaked with same manner in the same water and transferred after 1-hr. drumming into the sodium sulfide liquor. In this way up to sample number eleven has been soaked in the same water successfully without any operational problem. The soak liquor at final stage has been found 3850 g, which is nearly 40% of the initial water taken. The salinity content of the final soak liquor has been found around 270 g/L. The lot has been processed as usual as followed in the previous case up to chrome tanning and found very good results in look, feel and also shrinkage

temperature. Some lime liquor was collected to determine the salinity and found the result around 30g/L.

In practice, processing is always been done in lots of about 1000 kg. For industrial purpose, the procedure of this type of soaking operation should be as simple as possible. Otherwise it will be problematic to follow up with the infrastructure of the industries. To follow up the procedure, one separate soaking drum with one-tenth dimension of the main processed drum is required. The rpm of the drum should be around 14 and there should have suitable arrangement for collecting final soak liquor for subsequent treatment for salt recovery. Simultaneously another processed drum should be ready with 400% water and 1% sodium sulphide based on total raw weight of lot for continuation of repeated soaking of sub lots. A generalized procedure may be understood from the following Table III.6.

Table III.6 Generalized soaking (Proposed) process for 1000 kg's lot

Nos. of sub lot	Actual weight of sub lot (kg)	Approx. practical weight of sub lot (kg)
1	$W_{\varepsilon-01}$ =121.21	121
2	$W_{\varepsilon-02}$ =115.15	115
3	$W_{\varepsilon-03}$ =109.09	109
4	$W_{\varepsilon-04}$ =103.03	103
5	$W_{\varepsilon-05}$ =96.97	97
6	$W_{\varepsilon-06}$ =90.91	91
7	$W_{\varepsilon-07}$ =84.85	85
8	$W_{\varepsilon-08}$ =78.79	79
9	$W_{\varepsilon-09}$ =72.73	73
10	$W_{\varepsilon-10}$ =66.67	67
11	$W_{\varepsilon-11}$=60.61	60

[$W_{\varepsilon-01}$ =1000/8.25=121kg (approx.), D= $W_{\varepsilon-01}$ /20=6 kg (approx.), $W_{\varepsilon-n}$ = {20-(n-1)} $W_{\varepsilon-01}$ /20, water= $W_{\varepsilon-01}$ x8=970 kg (approx.), Duration of drumming=1hr, RPM=14]

970 kg water is to be taken into soaking drum with the first sub lot of 121 kg. Then drum is to be run for 1 hr. with 14 rpm. After that, the sub lot is to be transferred to the drum containing 1% sodium sulphide with 400% water as stated earlier. During transfer of soaked material to the liming drum loose water should be retained in the soaking drum as far as possible. Then the next sub lot is to be soaked in the similar way. In this way up to sub lot no. 11 are to be soaked and transferred to the drum containing dilute solution of sodium sulphide. Next day, the liming operation is to be started using 6% lime and another 3% sodium sulphide and adding no more water

further to maintain the float at 400% water. Percentages are based on w/s weight. After liming, fleshing is to be done and taking pelt weight; the rest operation is to be followed as per requirement.

III.3.7 Recipe for conventional process of leather

Now, from sample no. E-01 to E-15 have been taken as a lot and processed for chrome-tanned, beige-coloured, medium-soft leather in conventional way. The recipe has been followed for conventional process as given in Table III.7.

Table III.7 Recipe Followed for Conventional Processing of Leather

(Raw weight = 5034 g)

Unit operations	Reagent used	Amount of reagents (%)	Duration (minutes)	Temp. (^{0}C)	pH of liquor in the Process and Leather cross section and Comments and Observations
Soaking (% based on raw weight)	Water	400%	60	30 ± 2	pH of bath around 8, almost 30% hairs are removed; satisfactory soaking achieved, stock is left over night in the drum. Next day, drum is run for 5 minutes and taken 20 cc soak liquor for experiments and rest part is drained out. Soaked weight is taken and found= **6020 g.**

Table III.7 (Continued)

Unit operations	Reagent used	Amount of reagents (%)	Duration (minutes)	Temp. (^{0}C)	pH of liquor in the Process and Leather cross section and Comments and Observations
Liming (% based on soaked weight)	Water +Sodium sulfide	400 1	10	31 ± 2	Next day, again drum is run for 10 min./h for another 6h.Then the pelts are found with ideal condition of liming i.e. swelled, without keratinous matter on grain layer and with loose-adipose layer on flesh side. The cross sectional pH is examined by phenolphthalein indicator solution and found deep pink-colour as because the value is around 11.Then fleshing is being done in a machine and taken the pelts weight= **4500 g.** Then pelts are washed for 10 minutes and drained out the bath.
	+ Hydrated lime*	2	30		
	+Sodium sulfide	1.5	20		
	+ Hydrated lime	2	30 then drum is stopped for 30 minutes.		

Table III.7 (Continued)

Unit operations	Reagent used	Amount of reagents (%)	Duration (minutes)	Temp. (^{0}C)	pH of liquor in the Process and Leather cross section and Comments and Observations
Liming (% based on soaked weight) continued.	+Sodium sulfide	1.5	20	31 ± 2	
	+ Hydrated lime	2	30 then drum is stopped for 30 minutes, after that it is run 10 minutes/hr for next 5hr on the same day. Then stock is left over night in the drum in immersed condition.		

Table III.7 (Continued)

Unit operations	Reagent used	Amount of reagents (%)	Duration (minutes)	Temp. (^{0}C)	pH of liquor in the Process and Leather cross section and Comments and Observations
Deliming & Bating (% based on pelt weight)	Water + ammonium sulfate + sodium bi-sulfite	200 2 0.5	60	30 ± 2	Bath pH is found 8.3. The cross sectional pH is examined by phenolphthalein indicator solution and found colourless as because the value is around 8.
	Bate (Vinkol A)	0.2	30		The pelts are found slightly flaccid, smooth and almost white, except the few edges due to short hair roots appeared slightly black. The thumb impression is retained on the grain surface for a few seconds. All these are indicated that the deliming operation completed. Then the bath from drum is drained out and following reagents are added for dry bating.

Table III.7 (Continued)

Unit operations	**Reagent used**	**Amount of reagents (%)**	**Duration (minutes)**	**Temp. (^{0}C)**	**pH of liquor in the Process and Leather cross section and Comments and Observations**
Deliming & Bating (% based on pelt weight) continued.	+ ammonium sulfate + wetting agents (Tergolix IA) +common salt	0.5 0.8 2	20	30 ± 2	The short hair-roots are removed by hand scudding. Then pelts are washed for 10 minutes in running water.

Table III.7 (Continued)

Unit operations	Reagent used	Amount of reagents (%)	Duration (minutes)	Temp. (^{0}C)	pH of liquor in the Process and Leather cross section and Comments and Observations
Pickling (% based on pelt weight)	Water + common salt + sodium formate + sulfuric acid (98% pure, 1: 10 dilution)	80 7.5 0.5 2	10 3x20 + 60	30 ± 2	Drum is run without pelts. Specific gravity of the bath liquid is measured and found 5.6^0 Be'. Pelts are then added into the drum and it is run for 5minutes.
	+ sulfuric acid (98% pure, 1: 10 dilution)	2	3x20 + 60		The pH of the bath liquid is found to be around 3 and pelt is left in the bath for overnight. Next day drum is run for 20 minutes. Then the cross sectional pH is examined by bromo-cresol green indicator solution and found deep yellow colour which indicated the value around 2.8. Then half of the pickle bath is drained out and started next operation.

Table III.7 (Continued)

Unit operations	Reagent used	Amount of reagents (%)	Duration (minutes)	Temp. (^{0}C)	pH of liquor in the Process and Leather cross section and Comments and Observations
Single bath chrome-tanning (% based on pelt weight)	+ basic chrome sulfate (basicity-33.33%)	4	120	30 ± 2	Cross section of the pelt is being checked and found total green colour that indicated the chromium has been penetrated through and through. Then 100% water (based on pelt weight) is added into the drum and run for 20 minutes and then started next operation in the same bath.
	+ basic chrome sulfate (basicity-33.33%) + sodium formate	4 0.5	120		

Table III.7 (Continued)

Unit operations	**Reagent used**	**Amount of reagents (%)**	**Duration (minutes)**	**Temp. (^{0}C)**	**pH of liquor in the Process and Leather cross section and Comments and Observations**
Basification (% based on pelt weight)	+ hypo (1:10 diln.)	0.5	3x10	30 ± 2	pH of the bath is checked by pH paper and found around 4. The cross sectional pH of the pelt is also checked by bromo-cresol-green indicator solution and found green colour that indicated the value around 4. Then a small piece is cut out from thickest portion (near butt) of the pelt for boil-test. 10% shrinkage is found, which is allowable in this stage i.e. indicated completion of tanning. Then wet-blues are washed in running water and piled on floor for 7-days.
	+ sodium bi carbonate (1:10 diln.)	2	3x20 + 120		
Thereafter usual mechanical operations such as shamming shaving and trimming have been carried out. The shaved weight of the wet blue stock has been found to be **3800 g**. The stock is then ready for the next operations viz; rechroming, neutralization, dyeing, retanning and fatliquoring. Following the recipe given below.					

Table III.7 (Continued)

Unit operations	**Reagent used**	**Amount of reagents (%)**	**Duration (minutes)**	**Temp. (^{0}C)**	**pH of liquor in the Process and Leather cross section and Comments and Observations**
Rechroming (% based on shaved weight)	Water + acetic acid	200 0.5	15	28 ± 2	The bath pH is checked by pH paper and found around 3.7. The wet-blues are allowed to remain in the bath for night. Next day, the bath is drained out and wet-blues are washed in running water and then started next operation.
	+ basic chrome sulfate (basicity-33.33%)	4	45		
	+ sodium formate	0.5	20		

Table III.7 (Continued)

Unit operations	Reagent used	Amount of reagents (%)	Duration (minutes)	Temp. (^{0}C)	pH of liquor in the Process and Leather cross section and Comments and Observations
Neutralization (% based on shaved weight)	Water + sodium formate + sodium bi carbonate + neutralizing syntan (neutrigan MO)	200 0.5 1.2 1	45	28 ± 2	The bath pH is checked by pH paper and found around 5. The cross sectional pH of the wet-blue is also checked by bromo-cresol-green indicator solution and found greenish-blue colour, which indicated the value around 4.8. Then the bath is drained out and wet-blues are washed in running water and then started next operation.
Pre-fat Liquoring (% based on shaved weight)	Water +Fat-liquor (lipoderm liquor 2FB)	200 1	20	28 ± 2	Bath is checked for exhaustion of fat. The bath is found very clear and transparent which indicated excellent exhaustion. Dyeing is continued in the same bath.

Table III.7 (Continued)

Unit operations	Reagent used	Amount of reagents (%)	Duration (minutes)	Temp. (^{0}C)	pH of liquor in the Process and Leather cross section and Comments and Observations
Dyeing (% based on shaved weight)	+ beige coloured dye (yellow 2RL)	0.4	20	28 ± 2	Bath is checked for exhaustion of dye. The bath is found very clear and transparent which indicated excellent exhaustion. Retanning is continued in the same bath.
Retanning (% based on shaved weight)	+ Syntan (Relugan RE)	2	20	28 ± 2	Bath is checked for exhaustion of retanning materials. The bath is found almost clear and transparent which indicated good exhaustion. Fat – liquoring is continued in the same bath.
	+ Syntan (Basyntan DI) + Vegetable tannin (G.S. Powder)	10 5	60		

Table III.7 (Continued)

Unit operations	Reagent used	Amount of reagents (%)	Duration (minutes)	Temp. (^{0}C)	pH of liquor in the Process and Leather cross section and Comments and Observations
Fat-liquoring (% based on shaved weight)	+ lipoderm liquor 2FB + lipoderm liquor SAF + Sandolix WWL + lipoderm liquor oil SK + preservative (all are different anionic fat-liquors)	2 2 2 0.3 0.1	60	28 ± 2	Bath is checked for exhaustion of fat liquor materials. The bath is found almost transparent and slightly slippery which indicated satisfactory exhaustion of fat liquor. Top fatliquoring is continued in the same bath.

Table III.7 (Continued)

Unit operations	**Reagent used**	**Amount of reagents (%)**	**Duration (minutes)**	**Temp. (^{0}C)**	**pH of liquor in the Process and Leather cross section and Comments and Observations**
Fixation and Top fat liquoring (% based on shaved weight)	+ formic acid	1	30	28 ± 2	Bath is checked for fixation of fat liquor materials. The bath is found almost clear, transparent and non-slippery which indicated satisfactory fixation of fat liquor. The bath pH is found around 4. Then the leathers are washed in running water and piled for night. Next day, the following mechanical operations have been done.
	+ Cationic fat (lipamin liquor FB3)	1	30		
Mechanical operations: **Shamming** – it is done in a through-feed shamming machine to reduce the water content in leathers. **Setting** – it is done in a reversible setting machine to stretch out the surface of leathers and to give some ironing affect. **Drying** – the leathers are simply hang to dry in the open air under shade and allowed to stay in this condition for 2-days. Then leathers are checked and found completely dried. Following mechanical operations are then executed.					

Table III.7 (Continued)

Mechanical operations continued: **Conditioning** – water is sprayed on the flesh side of the leathers to increase the moisture content for make the next operation easier. Leathers are allowed to stay in this condition for 8-hours. **Staking** – it is done in a slow-cum-staking machine to make the leather fluffy, soft and flexible. **Toggling** – it is done in a toggle bed to stretch out the leathers and allowed to stay in this condition for 15-hours. **Trimming** – in this operation the unusable portions of leathers are cut out for dressing. Then the leathers are finished as follows:
Finishing: Leathers are plane plated at 60^0 C and 100-kg/cm^2 pressure. Then the leathers are sprayed twice with the solution prepared as follows: protein binder – 10 parts + preservative – 1part + water -100 parts. Then leathers are hanged to dry. After complete drying they are again plane plated at 60^0 C and 150 kg/cm^2 pressure.

* All the operations and recipe from the addition of hydrated lime have been followed same for experimental samples also. Only the chemicals for liming process have been calculated on w/s raw weight i.e. **4226 g**. The pelt weight and shaved weight of experimental samples were **3758 g** and **3200 g** respectively.

The finished leathers from both conventional and experimental samples are then tested for determining the physical and chemical properties as per Indian standard method. All these are stated in the section III.5.

III.4 Soak liquor analysis

It has been already stated that the wet-salted skins of sample number ε–01 to ε–11 are being soaked one by one in a drum. Percentage of water has been maintained always 400% based on the raw weight of samples. Soak liquor obtained after soaking of sample number ε–01 has been used for the soaking of sample number ε–02 and likewise we have repeated the soak liquor up to the soaking of sample number ε–11. 20mL soak liquor is collected after soaking of each samples and denoted by the ε–01, ε–02, ε–03 ... accordingly. All collected soak liquors are filtered twice through Whatman filter paper (541). Then the liquors are being used for the testing of TDS, Salinity, BOD_5^{20} and COD. The procedures of the above tests are being described briefly here.

III.4.1 Determination of total dissolved solids in soak liquor

1mL soak liquor is taken in a measuring cylinder by means a pipette. Then the volume is made 10 mL by the addition of distilled water. Dilute sample is being transferred into a clean porcelain basin of known weight. The weight of the empty porcelain basin is being measured previously in a Mettler balance. Then the basin with 10 ml dilute sample is placed on a hot water bath and allowed to evaporate to dry mass. The porcelain basin is then placed in an air oven at 100 ± 2^0 C for 1hr. After that the porcelain basin is allowed to cool in desiccators for 30-minutes and weight is measured in a Mettler balance. The heating and measurement of weight after cooling are being done repeatedly till the weight of the porcelain basin with solid residue become almost constant. Difference in weight is used to compute TDS. In the same way TDS of distilled water has been measured as control and subtracted this value from the value of dilute analyte.

III.4.2 Determination of salinity (as NaCl) in soak liquor

0.5 mL soak liquor is taken in a measuring cylinder by means a pipette. Then the volume is made 50 mL by the addition of distilled water. Then 5mL dilute sample is taken in a conical flask and added 2-drops of 5% potassium chromate solution as indicator. The solution is converted into orange colour and then titrated with (N/10) silver nitrate solution until the colour changed to permanent brick-red. The volume required of (N/10) silver nitrate solution is used to compute salinity (as NaCl) in soak liquor. In the same way salinity (as NaCl) of distilled water has been measured as control and subtracted this value from the value of dilute analyte.

III.4.3 Determination of BOD_5^{20} value in soak liquor

In this method, an air tight bottle of specified volume, completely filling with dilute soak liquor maintaining the salt concentration 1.015g/L (out of four different concentrations it has given the best result), is allowed to incubate at 20^0 C for 5-days. Dissolved oxygen is measured just before incubation and after 5-days incubation and BOD value is calculated from the difference. This method is an empirical test (Lenore S. et al 1998) to determine the relative oxygen requirements of biodegradable matters present in water.

Preparation of reagents:

1. Phosphate buffer solution: KH_2PO_4 – 0.85g, K_2 HPO_4 – 2.175g, Na_2HPO_4, $7H_2O$ – 3.34g and NH_4Cl – 0.17g are dissolved in minimum quantity of distilled water and diluted to100 mL. pH is measured 7.2.

2. Magnesium sulfate solution: 2.25g of $MgSO_4$, $7H_2O$ is dissolved in distilled water and made 100mL.

3. Calcium chloride solution: 2.75g of $CaCl_2$ is dissolved in distilled water and diluted to 100mL.

4. Ferric chloride solution: 25mg of $FeCl_3$, $6H_2O$ is dissolved in distilled water and diluted to 100mL.

5. Acid and alkali solution: 1(N) solution, for neutralization of sample.

a) Acid solution: 2.8mL conc. Sulfuric acid is slowly stirred with distilled water and made up to 100mL.

b) Alkali solution: 4g sodium hydroxide pellets are dissolved in distilled water and made up to 100mL.

6. Seeding: putrid cow dung is treated with distilled water which is being pre-saturated with oxygen by passing air for 2 hours. 10% solution of this liquor is used as seed water.

7. Preparation of dilution water: 1mL each of phosphate buffer, magnesium sulfate, calcium chloride, ferric chloride and seed water solution is added into 1L distilled water.

Procedure: Sample soak liquor is taken 2ml in a measuring cylinder and diluted to 100ml by the addition of distilled water. Then a specific volume of dilute sample is taken in an incubation bottle of known volume and the vacant portion of the bottle is filled to the brim with the dilution water. The specific volume of the dilution water is calculated so as to maintain the salt (as NaCl) concentration in the incubation bottle at 1.015g/L. Solution from the incubation bottle of volume 50mL is taken to measure its dissolved oxygen content (D_1) through titration with 0.025 (N) Hypo solution. Again the incubation bottle is filled with the dilute soak liquor and dilution water in the same way as stated earlier. The bottle is sealed and kept in the incubation chamber at 20^0 C for 5-days. After

incubation, again 50mL solution is taken to measure dissolved oxygen content (D_2) in the same way.

^{20} BOD $_5$ = (D_1 - D_2) x 4 x 100 / mother soak liquor present in 100mL dilute sample.

III.4.4 Determination of COD value in soak liquor

This is a rapid method to determine the carbonaceous organic matter content in sample material. It simply consists of refluxing the sample water with potassium dichromate, the strong oxidizing agent, in acidic condition. Remaining oxidizing agent is titrated with sodium thiosulfate solution. The difference measures the value of chemical oxygen demand (COD).

Preparation of reagents:

1. 0.25(N) potassium dichromate solution: 3.0625g of potassium dichromate is dissolved in 18(N) sulfuric acid and made up to the mark in 250mL volumetric flask with the same acid solution.

2. Sodium thiosulfate solution: 3.95g of sodium thiosulfate is dissolved in distilled water and made up to the mark in 1L volumetric flask with distilled water.

3. Potassium iodide solution: 25g of potassium iodide (AR grade) is dissolved in 250mL of distilled water to make a 10% solution.

Procedure: 1mL soak liquor is taken in a measuring cylinder and made up to 20mL with distilled water. 1mL of dilute sample is taken in a round bottomed 250mL flask with a ground glass joint. 20mL of 0.25(N) potassium dichromate in 18(N) sulfuric acid is added to the sample along with 0.1 g of silver sulfate and 0.1 g of mercuric sulfate.

The mixed solution is refluxed for 4-hours. Then the whole mass is cooled and the excess potassium dichromate is titrated with 0.025(N) sodium thiosulfate solution using 10% KI

solution and starch as indicator at the end of the reaction. The difference in the hypo required for the corresponding amount of potassium dichromate computes the COD.

III.5 Physical and Chemical Testing of Finished Leather

All physical and chemical testing are being done as per Indian Standard methods. This Indian Standard was adopted by the Indian Standard Institution on 7 January (for methods of physical testing) and 16 July (for methods of chemical testing) 1970, after the draft finalized by the Leather Sectional Committee had been approved by the Chemical Division Council.

This standard is the amalgamated revision of all physical and chemical methods of test specified in IS: 582-1954 and IS: 1016-1960. These methods of test were originally based on B.S. 1309: 1946 'sampling and analysis of vegetable and chrome tanned leathers', and 'official methods of sampling and tests (1954)', adopted by the Society of Leather Trades Chemists (SLTC), UK.

In the formulation of this standard, assistance has been drawn from the following publications:

B.S. 1309: 1956 Methods of sampling and analysis of vegetable tanned and chrome tanned leathers. British Standard Institution.

ALCA Methods of analysis, 1957. American Leather Chemists Association, USA.

Book of ASTM standards---part 15, 1966. American Society for Testing and Materials, USA.

Official methods of analysis, 1965. Society of Leather Trades Chemists, UK.

III.5.1 Conditioning of leather and sampling position

During 48 hours immediately preceding its use in a test, each test piece is to be kept for physical testing at a standard atmospheric temperature of 27 ± 2^0 C and relative humidity 65 ± 2% (IS: 196-1966). On the other hand, all results of test should be expressed on zero percent volatile matter. In this study all physical and chemical tests result are compared between leathers obtained from conventional process and from experimental process. That is why; the temperature and % R.H. have been maintained strictly same for the same type of physical and chemical tests. Sampling position for physical and chemical testing of leather has been selected as per IS norm as showing in the following **figure III.38.**

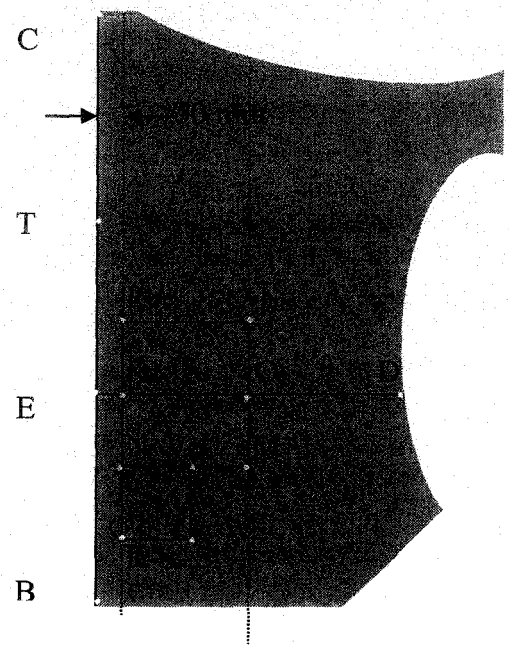


Fig. III.38
Sampling position for physical and chemical testing of leather

Sampling positions for physical and chemical testing of leather as per IS: 3840 – 1966 are being described. Let BC be the backbone line where B is the tail-root position. BC is divided into three equal parts by the points T and E. So, BE=ET=TC=1/3 BC. A line PR is drawn parallel to BC 50 mm apart. Another line ED is drawn perpendicular to BC at the point E, which meets the edge of the leather at the point D. The point O has divided the straight line ED into two equal parts. K is the intersecting point of two lines PR and ED. The line LM is perpendicular on ED through the point O. Now the four points P, K, N and R are selected on the line PR so as to satisfy PK = KN = NR = ½ KO. Similarly OL = OM = ½ KO. PL and NM are joined. The point J is selected as mid. Point of the line MN. The line JS is drawn perpendicular to line MN at point J. S is a point on JS so that JS = JN. The line RS is drawn.

The square PLMN is collected as sample on the right side of the leather for physical testing. Similarly the square NJSR is collected as sample on the right side of the leather for chemical testing.

In the same manner the sampling positions both for physical and chemical testing has been located on the left side of the leather.

III.5.2 Method for measurement of thickness and length of finished leather

Thickness of leather is measured with the help of a analog micrometer type of gauge whose presser foot is flat and exerts pressure of 500 ± 2 g/cm^2 on the leather (grain side up) placed on a glass topped wooden table.

Leather is placed on a flat glass-topped wooden table and flattened it out without pulling and stretching. Length is measured with the steel scale to the nearest 0.5mm at the location required.

III.5.3 Method for measurement of shrinkage temperature of finished leather

The apparatus used for shrinkage temperature measurement is shown in the following **figure III.39**:-

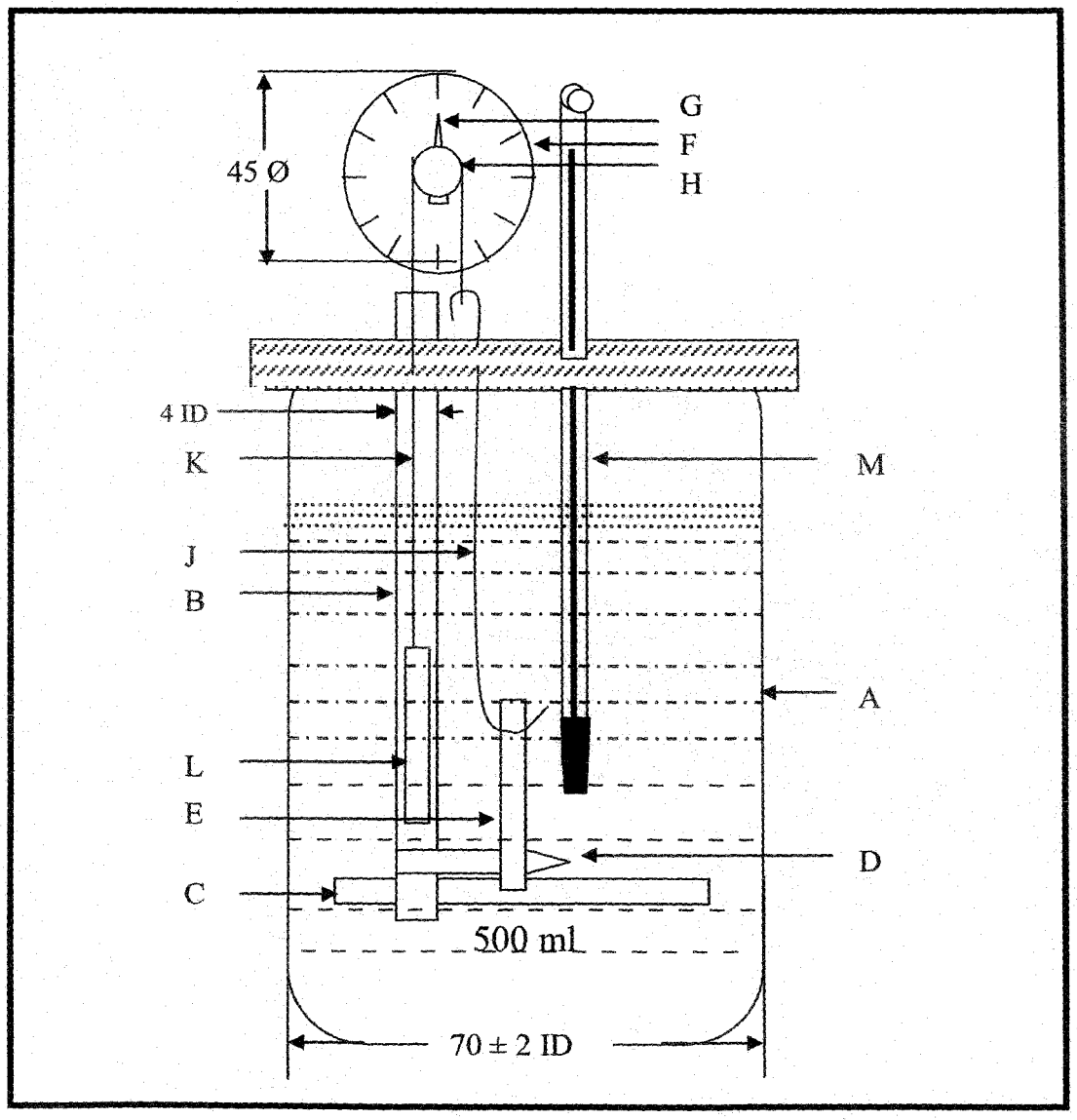


Fig. III.39
Sketch of shrinkage temperature tester

A= glass beaker, B= brass tube, C= rod, D= rod for sample holding, E= test piece, F= circular scale, G= pointer, H= pulley, J= hook, K= thread, L= weight, M = thermometer. (All dimensions are in millimeters.)

Procedure: Two sample pieces, one along the back-bone and another across the back-bone, in rectangular shape of dimension 50 mm x 3 mm are cut out from the sampling position specified for physical testing. Two small holes are punched at middle of width and the ends of 50 mm length from each side to hold the test piece from hooks and sample holding rod. Samples are then wetted for conditioning before testing as per IS: 5914 – 1970 (LP: 10). Then sample is attached to the hooks D and J. Around 400 mL glycerin solution (75%) is poured into the beaker A and placed on electric heater fitted with magnetic stirrer. The control of heating is maintained so that the rise of temperature would be as nearly as possible at 2^0 C per minute. The temperature and the corresponding reading of the pointer G are noted at half-minute interval until the test piece shrinks considerably. The temperature at which the test piece has shrunk to such an extent as to move the pointer half a division from the position corresponding to the maximum length of the test piece is considered shrinkage temperature. The average value of two shrinkage temperatures, corresponding to along and across the backbone is denoted the shrinkage temperature of the sample.

III.5.4 Method for measurement of water vapour permeability of finished leather

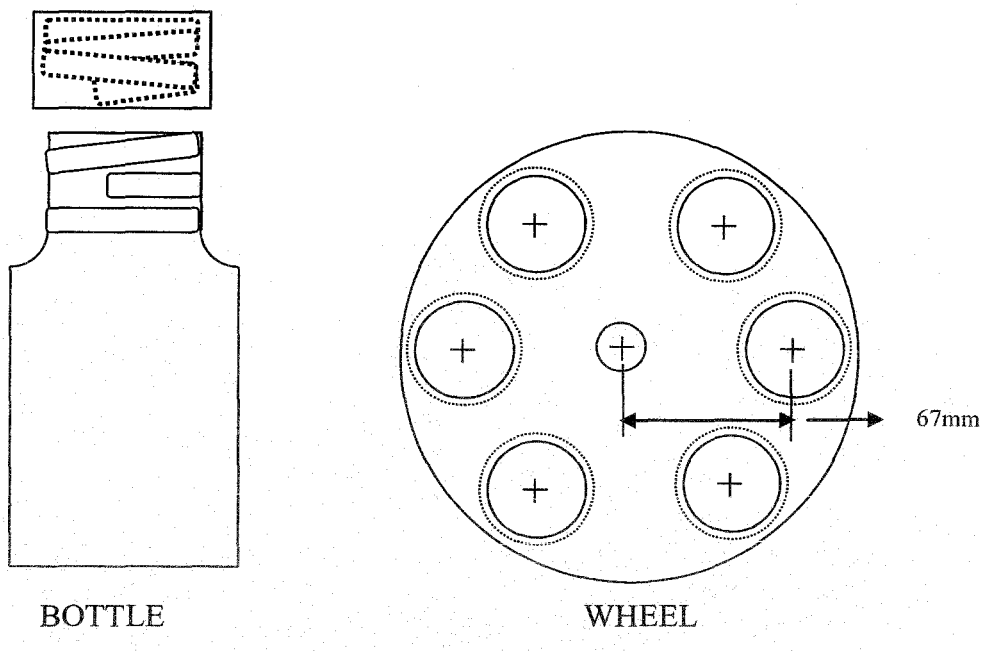


Fig. III.40
Sketch of a part of water vapour permeability tester

Bottles of the approximate shape shown in the **figure III.40** with screw tops cut away to leave a circular opening. The neck of each bottle is ground to give a flat end surface which is perpendicular to the interior wall of the neck, and the circular opening in the cap has the same diameter as the interior wall (each approximately 30 mm).

A bottle holder in the shape of a wheel that is rotated at 75 ± 5 rpm by an electric motor. The bottles are mounted on the wheel with their axes parallel to the axle and at a distance 67 mm from it.

Silica gel which has been freshly dried for at least 16-hrs in a ventilated oven at 125 ± 5° C and cooled for at least 6-hrs in a closed bottle. The particle size of the gel is sufficiently large to prevent it passing a 2 mm IS sieve.

A fan has been used to agitate the air outside the bottle. All dimensions and specifications are maintained as per IS: 5914 – 1970 (LP: 21).

A circular piece of around 38 mm diameter is cut out from the sample position of leather. The test piece is clamped; grain inwards, across the mouth of the bottle, which contains conditioned silica gel as desiccant nearly half of the volume of it. Then the bottle with silica gel and sample is weighed in an electronic digital balance. The bottle is fitted in the bottle holder wheel and circulated by keeping the desiccant in motion in a rapid current of air in a conditioned room. The bottle is weighed after 1-hr and 3-hrs (i.e. after another 2-hrs from first weight) to determine the mass of vapour transmitted through the leather and absorbed by the desiccant. The weight differences are used to compute the water vapour permeability in g/ min/ cm^2.

III.5.5 Method for measurement of tensile strength and % elongation at break of finished leather

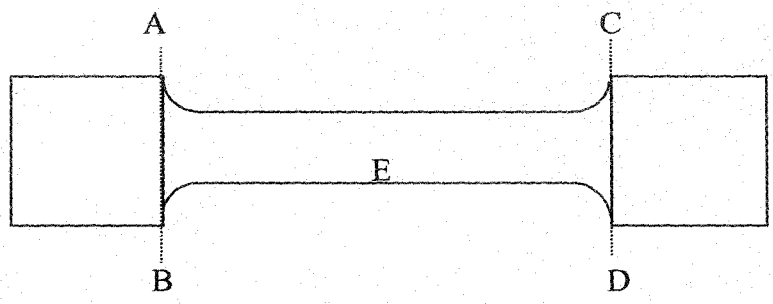


Fig. III.41
Sketch of sample piece for tensile strength and percentage elongation at break testing

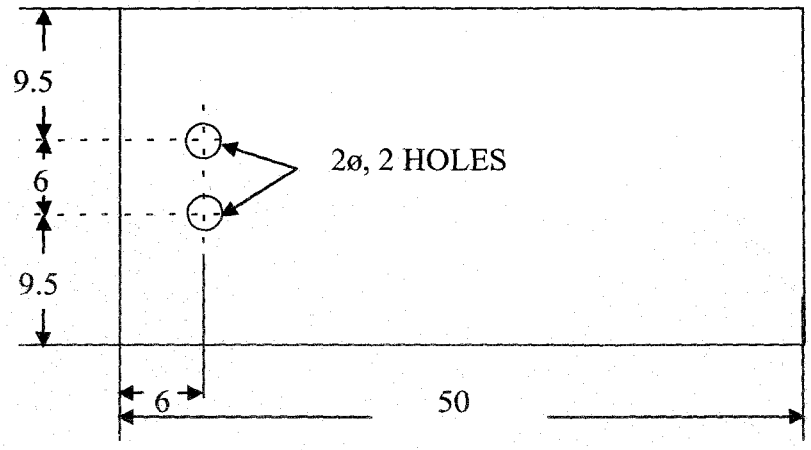
Tensile strength: - The force per unit area of the cross-section of non-stretched test piece required at the time of rupture. It is calculated by dividing the breaking force in kilograms-force by the cross-section of the non-stretched test piece in square centimeters.

Percentage Elongation at break: - The extension between benchmarks produced by a tension force applied to a test piece, at the time of its rupture. It is calculated by taking

the difference between the original length and the length at the time of rupture under the tension, expressed as a percentage of the original length.

Procedure: - Test pieces are cut out from the sample position by means of press-knife as per **figure III.41** shown above. These methods supersede those given in 23 of IS: 582-1954 and are partly based on IUP/6. The width of each test piece is measured to the nearest 0.1mm at three positions on the both grain and flesh sides. In each group of three, one measurement is taken at the mid point E of the waist of the test piece and the other two positions are selected approximately midway between E and the lines AB, CD. The arithmetic mean of the six measurements is taken as the width of the test piece. The thickness of the test piece is measured at three positions as mentioned in width measurement and the arithmetic mean of the three is taken as thickness of the test piece. Then the cross-sectional area of each test piece is measured by multiplying its width by its thickness. Now the jaws of the tensile strength machine are set according to the test piece. The test piece is clamped in the jaws so that the edges of the jaws lie along the lines AB, CD and at same plane. The distance between the jaws is measured to the nearest 0.5mm and taken as the initial length of the test piece for the purpose of the test. Then the machine is run until the test piece is ruptured. The instant distance between two jaws are noted and treated as the length of the test piece at break. On the other hand the load required for this rupture is taken as the breaking load. From breaking load and area of cross-section of the test piece, the tensile strength is measured. The % elongation at break is computed from initial length and the length of the test piece at break.

III.5.6 Method for measurement of double-hole stitch tear strength of finished leather



(All dimensions in millimeters)

Fig. III.42

Sketch of sample piece for double-hole stitch tear strength testing

The two test pieces, one along the back bone and another perpendicular to the back bone, are cut out as per dimensions shown in the **figure III.42** from sample position of finished leather for physical testing. Two holes of 2mm diameter are punched as shown in the figure. The method is followed here as IS: 5914 – 1970 which is adopted from E 13 of ALCA methods of analysis. Thickness of the test pieces is measured as per IS – Specification. A steel wire (1.000 ± 0.025 mm in diameter and 100 mm in length) is bent into U-shape loop and passed through the two holes so that both ends projected from the flesh side of the test piece. The two ends of the wire are clamped in the wrapped grips of the testing machine. The free end of the test piece is clamped in the other grip of the machine. The machine is started to apply force to the test piece at such a rate that the accurate grip travels at a uniform speed of 25 ± 5 cm / min. The load required at the moment of initial tear of the test piece is noted. Then the double-hole stitch tear strength is computed in kg / cm thickness of the sample.

III.5.7 Method for measurement of grain crack strength and ball bursting strength of finished leather

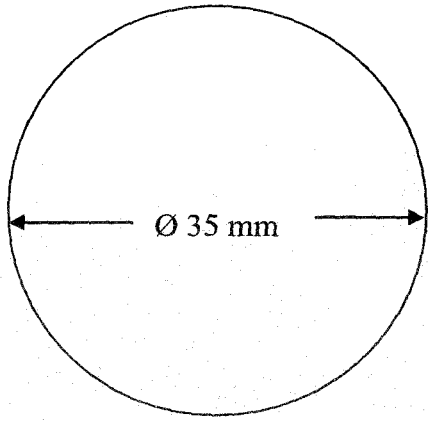
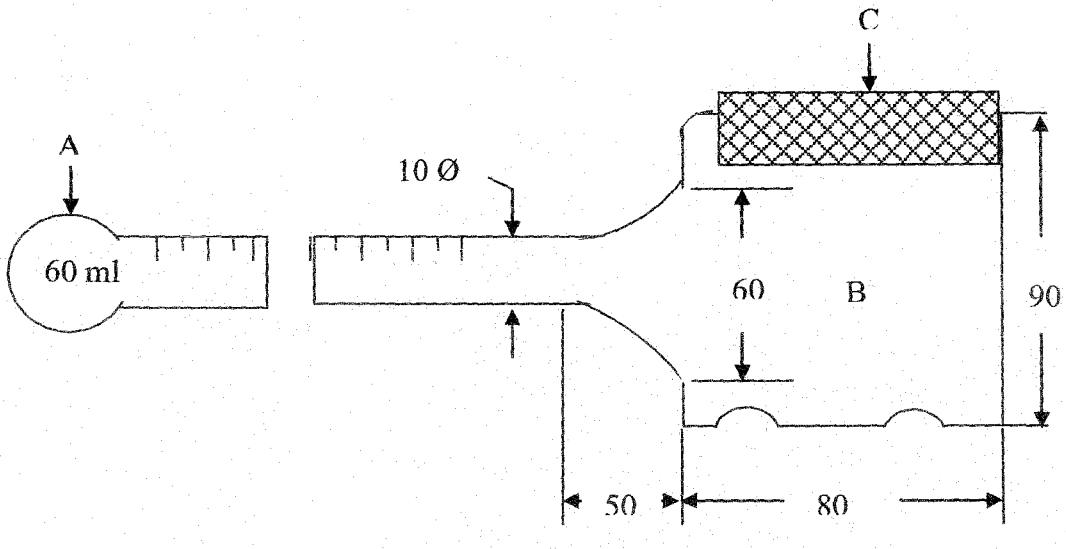


Fig. III.43
Sketch of sample piece for grain crack and ball bursting strength testing

The test is carried with an instrument named 'Lastometer'. In this instrument, the pressure is applied on the test piece with the help of a rising road with a spherical metallic ball of diameter 6.25 mm at the top. A circular test piece of 35mm diameter as shown in the **figure III.43** is cut out from the sample position for physical testing. Thickness of the leather sample is measured as usual. The test piece is clamped horizontally to the clamping device of the instrument with a circular metallic ring for grip and a screw. The central portion of the test piece is formed a circle of diameter 25 mm remains free from both sides. The test piece is clamped with the grain side up. The load is applied directly to the center from the bottom with a round ball on the top of a rod which is raised by rotating the handle of the instrument. The load required just to crack the grain and vertical lift of the rod, called distension, are noted from the respective dials provided in the instrument. The grain crack strength is then calculated in kg / cm thickness of test piece. Then the raising of rod is continued to exert more pressure to the sample till

bursting out the ball. At the bursting point the load and corresponding distension are noted for the determination of bursting strength of the test piece in kg / cm thickness.

III.5.8 Method for measurement of absorption of water: Kubelka method- of finished leather



(All dimensions in millimeters)

Fig. III.44
Sketch of Kubelka apparatus

Kubelka apparatus is used to measure water absorption in milliliters of water per 100 g and also per 100 mL of leather. Kubelka apparatus is made of glass as shown in the **figure III.44** with dimensions as per IS – specification. To the rubber stopper C a steel wire of diameter one millimeter is fastened, to keep the test piece at the end of B distant from C. The test piece is cut out from the sampling position with the help of a steel press knife, the inner wall of which is a right circular of diameter 70 mm. Then the length,

breadth, thickness and weight of the test piece are measured as per IS-norms. The method followed here is superseded the method given in 21.1 of IS: 562 – 1954 and is adopted from IUP / 7. Before starting the experiment, the apparatus is thoroughly cleaned by chromic acid solution. The apparatus is then finally cleaned by washing three times distilled water. The bulb and tube up to the zero mark are then filled up with distilled water and the test piece of known weight is put into the cup B with flesh side up. The entire quantity of water is then transferred to the cup by tilting the apparatus. The open top surface of the cup is covered with a rubber stopper C, to prevent evaporation losses. The leather test piece is allowed to remain immersed in water in the cup for 30 minutes. After the period, the entire water is carefully transferred into the bulb by tilting the apparatus and the reading is taken. The water is again transferred from the bulb to the cup and allowed to remain the test piece in immersed condition for another 30 minutes. After the period, again the entire water is carefully transferred into the bulb by tilting the apparatus and the reading is taken to measure the volume of water consumed by the test piece. These two readings are used to calculate the percentage of water absorption in volume / volume and volume / weight of the sample test piece.

III.5.9 Method for measurement of dry–rub and wet–rub colour fastness of finished leather

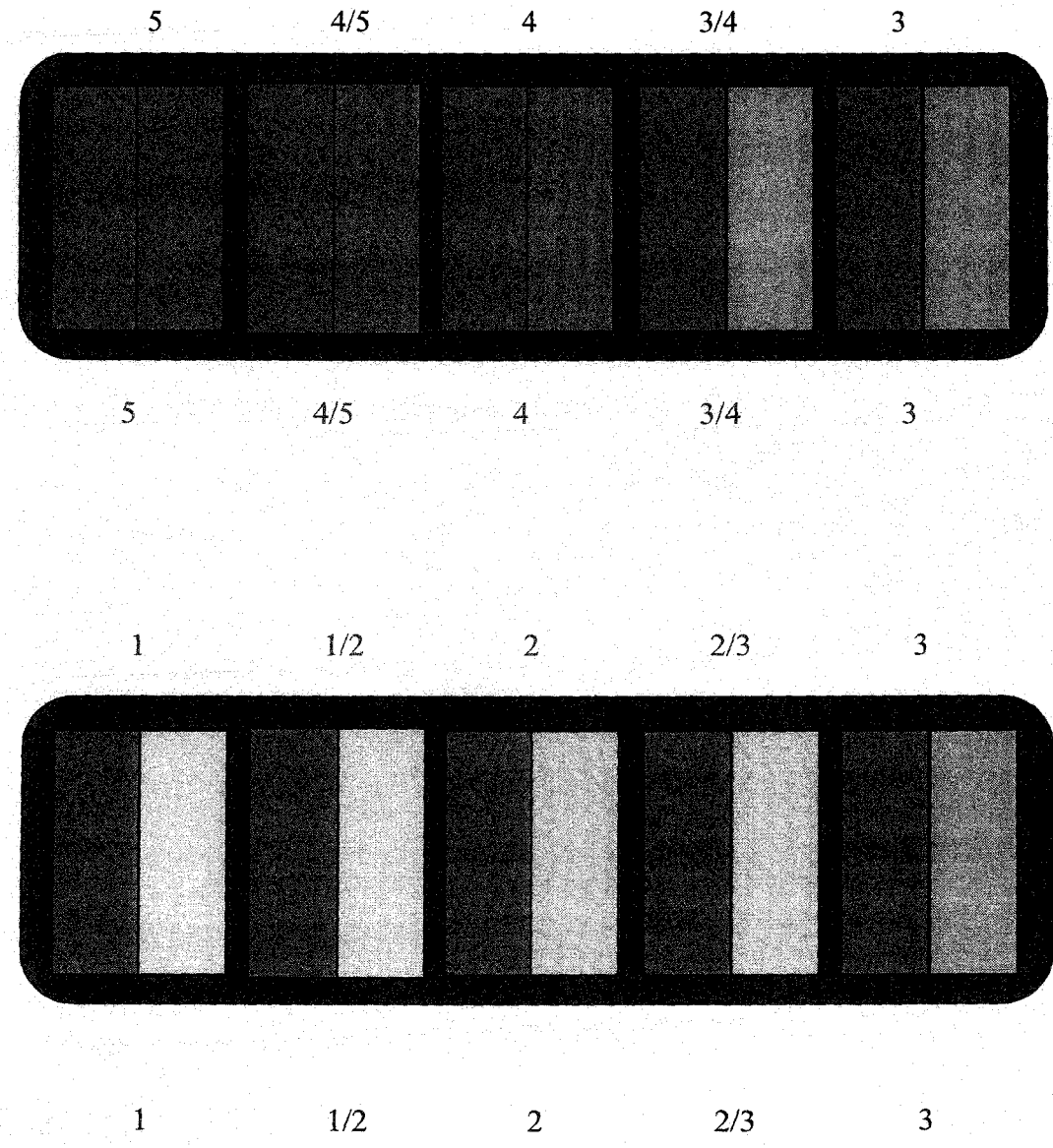
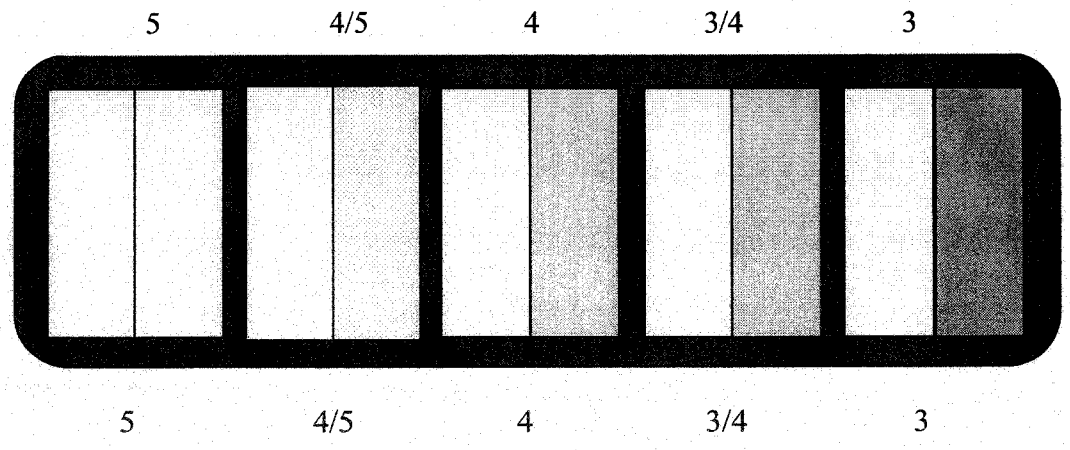


Fig. III.45
Sketch of Grey scale for assessing changes in colour of leather



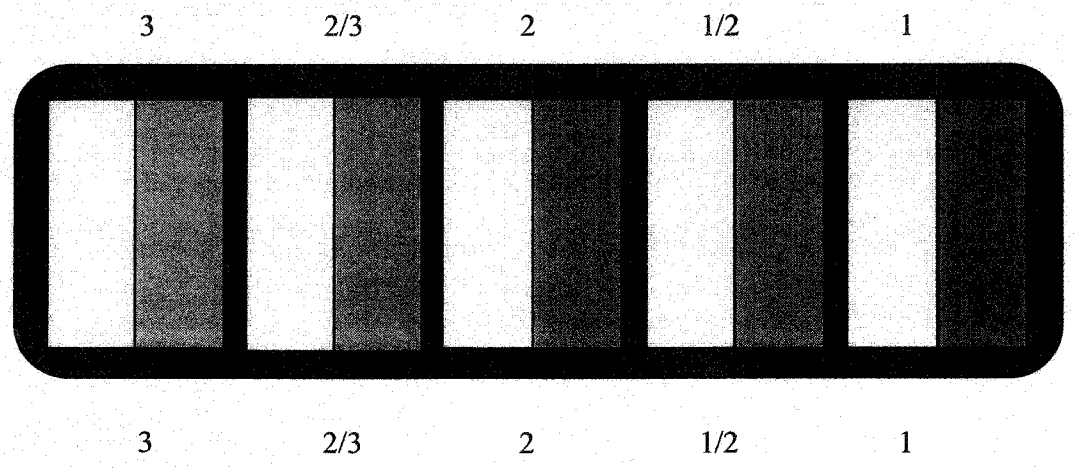


Fig. III.40

Sketch of Grey scale for assessing degree of staining by dyed leather

The wet and dry rub fastness of leather is measured with the wet and dry rub fastness tester, designed by SATRA of England. In this instrument a perfect white, medium soft, circular textile pad of diameter 25.4 mm and thickness 6.35 mm is allowed to rotate on

the coloured leather surface, placed on a hard flat base, at the rate of 150 rpm. The test piece of 50 mm square is cut out from the sampling position for physical testing of leather. Then the test piece is placed on the flat base and the dry pad is allowed to rotate on the leather surface with a dead load of 2.5 kg for 2048 revolutions. In case of wet rub fastness test, a shammed pad in place of dry one is used with a dead load of 0.73 kg for 1024 revolutions. In both the cases the intensities of staining adhered to the pad are compared with an unstained pad with the help of a grey scale as shown in **figure III.45** and **III.46** recommended for assessing degree of staining by dyed leather. The contrasts between the rubbed and un rubbed portions of the leather for both dry and wet tests are also determined with the help of another grey scale recommended for assessing changes in colour of leather.

The grey scale rating: The scale consists of nine pairs of grey colour chips each representing a visual difference and contrast.

The fastness rating goes step – wise from:-

5 = no visual change (best rating) to

1 = a large visual change (worst rating)

The grey scale has the nine possible values:-

5, 4/5, 4, 3/4, 3, 2/3, 2, 1/2, 1.

III.5.10 Method for determination of volatile matter present in finished leather

Sample is collected from leather for chemical testing in accordance with IS: 5868 – 1969 as shown in the figure III.38 above. The test portion is ground by scissor so that it passes through the sieves of 1-square mm. Approximately 3 g ground leather is weighed to an accuracy of 0.001 g and allowed to dry in an oven at 102 ± 2^0 C for five hours. Sample weight is taken after cooling in the desiccators for half an hour. The weight of the sample is checked after a further one hour of drying and half an hour of cooling. In the same way of drying and cooling, the weight of the sample is taken third time and find the reduction in weight less than 0.1 % of the original weight of the sample (i.e. 3 g). The difference in weight is used to compute the volatile matter present in leather in percentage.

III.5.11 Method for determination of total ash content in finished leather

The sample is taken from the portion as specified for chemical testing and ground as stated above. Approximately 5 g ground leather is weighed to an accuracy of 0.001 g and allowed to carbonize it over a low flame in a Nickel crucible which has previously been heated to 800 ± 25^0 C, cooled and weighed. After carbonizing the whole sample, the nickel crucible with the carbonized sample is allowed to ignite in a muffle furnace at 800 ± 25^0 C for four hours. After cooling the ash obtained in the crucible is checked and find on trace of black particle which indicated that the ash is totally free of carbon. Then the crucible is allowed to dry in an air oven at 102 ± 2^0 C for one hour. Sample weight is taken after cooling in the desiccators keeping for half an hour. The weight of the sample is checked after a further one-hour of drying and half an hour of cooling. In the same way of drying and cooling, the weight of the sample is taken third time and find the reduction in weight less than 0.1 % of the original weight of the sample (i.e. 5 g). The difference in weight is used to compute the percentage ash content of finished leather sample.

III.5.12 Method for determination of total chlorides (as NaCl) in finished leather

5 g ground leather sample is taken to an accuracy of 0.001 g and made it ash as stated in the section III.5.11. Then the ash is moistened with 5 drops of concentrated nitric acid in water, and made up to 250 mL with distilled water in a volumetric flask. 50 mL from the mother solution is taken in a glass beaker and filtered through a filter paper (whatman 541) and collected into a conical flask. Then the pH of the solution is checked and found slightly acidic in nature. A small amount of solid calcium carbonate is added into solution to bring the pH at neutral condition. Then ten drops of 5% solution of potassium chromate is added into solution as indicator and titrated by 0.01(N) solution of silver nitrate till the colour of solution converted into permanent brick red. The titration is repeated thrice and the average reading is used to determine the total chlorides content (as NaCl) in finished leather.

III.5.13 Method for determination of total sulphates (as Na_2SO_4) in finished leather

50 mL extract, as prepared in total chlorides determination, is taken in a glass beaker and filtered through a filter paper (whattman 541) into a conical flask. 5 mL Concentrated hydrochloric acid is added into solution and then filtered into a conical flask and transferred the filtrate into another glass beaker of volume 300 mL. Then the solution is diluted up to 175 mL with distilled water and heated to boil. 5 mL of 12% hot solution of barium chloride is added into the solution containing in the beaker and kept the reaction mixture on a water bath for half an hour and allowed to stand over night. Next day the solution is filtered through ash less filter paper (whatman 541) with repeatedly washing the beaker with distilled water. Then the filter paper is allowed to dry and ignited in a muffle furnace at 400^0 C for 4 hours taking in a previously weighed nickel crucible. To have a constant weight the same procedure is followed as mentioned in the section

III.5.11. A blank test is also carried out using the same quantities of reagents as used for the experiment with the materials. From the result obtained in blank test and the weight of the precipitate, the total sulphate (as Na_2SO4) in finished leather is determined.

III.5.14 Method for determination of chromium (as Cr) in finished leather

5 g ground leather sample is taken to accuracy 0.001g and made it ash as stated in the section III.5.11. The sodium peroxide as oxidizing agent is taken nearly 5-times of the weight of ash into nickel crucible and mixed well. Then the mixture is fused gradually and heated to moderate red heat for 5-minutes. After cooling, the crucible with melt is placed in a large glass beaker and poured almost boiling distilled water to dissolve the melt. The beaker is covered with a clock glass to prevent loss by spurting. Then the solution with crucible is transferred gently to a 500 mL boiling flask. The crucible is removed carefully and washed thoroughly adding the washing to the boiling flask. Then the solution is cooled and filtered through a filter paper (whatman 541) and made up to 500 mL with distilled water in a measuring flask. 100 mL of filtrate is taken in a conical flask with a glass stopper and acidified with 15 mL of concentrated hydrochloric acid (sp. gr. 1.16). Then 10 mL of 10 percent (w/v) potassium iodide solution is added into the solution containing in the conical flask. The flask is closed by the glass stopper and allowed to stand for 2-minutes. Then the iodine liberated in the reaction mixture is titrated immediately by N/10 Hypo solution, using starch as indicator towards the end of the reaction. The volume required of hypo solution in the titration is used to compute the percentage chromium present in the finished leather.

III.6 Desalination of Final Soak Liquor

The final soak liquor what obtained after eleven times repetition of soaking of wet-salted raw skins in the same water is collected for desalination. The soak liquor is appeared very dirty, full of mud, coagulating proteins, loose hairs, saturated saline water with some bad odor. Since the effective method of desalination of saturated salt solution is evaporation, solar evaporation method has been followed here for desalination. Before expose the soak liquor on sun ray, Alum (aluminium sulphate) solution is used to separate out the suspended and coagulating matters present in the liquor.

III.6.1 Alum treatment of final soak liquor

To separate out the course materials present in the liquor, the soak liquor is filtered through a porcelain funnel fitted with perforated bed of pore diameter 200 – 300μm. After that, six glass beakers (Borosil) of 100 mL capacity are taken and labeled as experiment nos. 1 to 6 and 50 mL filtered final liquor is transferred into each beaker. Alum (Aluminium Sulfate; $Al_2(SO_4)_3$, $18H_2O$, slightly yellowish lumps) solution of strength 50,000 ppm is prepared previously in a 250 mL high neck flat bottomed flask. Then, 2-mL, 4-mL, 6-mL, 8-mL, 9-mL and 10-mL solution of alum are mixed with the soak liquor containing in the glass beakers from experiment nos. 1 to 6 respectively. The temperature of the soak liquor is maintained at 25 ± 2^0C i.e. the atmospheric temperature. The mixture is stirred well on a magnetic stirrer instruments for 5-minutes with the stirring paddle speed 150-250 rpm. Then the mixture is allowed to stay for 3-hours to settle down the suspended matters present in the soak liquor. After that, the mixture is filtered through a Whatman 541- filter paper which can retain the coarse-size particle only. The filtered solution is collected into a measuring cylinder of 100-mL capacity to measure the volume of cleared-slightly yellowish- filtered saline water directly and also

time is noted for filtration. Then the turbidity of filtered solution is measured in a digital turbidity meter against distilled water of turbidity value 0-NTU. All the results obtained from the experiments will be very clear from the following Table III.8.

Table III.8 Alum treatment in final soak liquors

Experiment Nos.	Volume of final soak liquor taken (mL)	Volume of Alum solution (50,000 ppm) added (mL)	Time required for filtration (minutes)	Volume of filtered solution obtained (mL)	Turbidity reading of filtered solution (NTU)	Weight of the filtrate just after 1-hr of filtration (g)
01	50	2	120	40	74	6.1537
02	50	4	60	41	06	6.1205
03	50	6	35	41	03	6.1120
04	50	8	25	44	01	5.8674
05	50	9	20	45	01-00	5.5521
06	50	10	15	47	00	5.2312

The turbidity of filtered solution of experiment no.-05 is found frequently changes from 01 to 00 in the digital display screen. It has been experimented to see the turbidity of filtered solution by increasing the alum solution dosing up to 20-mL into same 50-mL final soak liquor, but the turbidity is gradually increased and it becomes 07-NTU at 20-mL dosing. Slightly yellowish colour of the filtered solution and the high turbidity (slightly higher than 1000-NTU in fully dispersed condition) of 50,000 ppm alum solution may be the cause for the result. Some weight and volume of the liquor are being lost due to incapability of complete transfer of alum treated liquor into filter funnel due to its high consistency and the consumption of certain volume of liquor into the mud like

filtrate respectively. The curd like muddy filtrates what are arrested on the filter paper takes around 6 days for complete drying and in this condition the average weight of the filtrate is found nearly 77 g/L of soak liquor. From this data it is clear that the considerable amount of alum-treated liquor has been arrested by the filter paper and coagulating materials, and the water of this arrested liquor has been evaporated out during these 6-days. Otherwise the weight of the filtrate does not reduce to 77 g/L from nearly 120 g/L what we have had after 1-hour of filtration. It is also very important to mention here that at this stage a white film of salt crystals is found on the top of the filtrate.

III.6.2 Solar evaporation of filtered solution

Five pieces circular porcelain basin of specific dimensions have been taken for solar evaporation of filtered liquor. The basins are marked as PB-01 to PB-05. Then the weight of the each empty-cleaned-basin has been taken in a digital balance. Filtered liquors obtained from experiment no.-05 and 06 have been mixed in a 100 mL beaker as their turbidity was nearly 00-NTU. Then 10 mL mixed liquor has been taken for each basin and poured in to them gently. The surface area of each 10 mL mixed liquors has been converted as per respective inner surface area of porcelain basin. Then the basins are placed on a concrete roof in an open sunny area from 6:00 am to 5:00 pm at the middle of the month of November. The day was fully sunny with clear sky. All water of the liquor has been found evaporated out within this time leaving clear white crystals of common salt in the basin. Then the weight of the basins has been taken again in digital balance to measure the weight of the salt present in 10 mL liquor considering TDS of the liquor only

contributed by common salts. The experimental data can be clearly understood from the following Table III.9.

Table III.9 Solar evaporation of filtered soak liquors

Expt. nos.	Inner dia. of the basin (mm)	Weight of the empty basin (g)	Vol. of filtered liquor taken (mL)	Surface area of filtered liquor (sq. mm)	Depth of the filtered liquor (mm)	Weight of the basin with salt (g)	Weight of the salt found (g)
01	82	65.2299	10	5285	1.89	68.1484	2.9185
02	80	86.6585	10	5030	1.98	89.5907	2.9322
03	81	55.6021	10	5157	1.94	58.5375	2.9354
04	81	58.9986	10	5157	1.94	61.9276	2.9290
05	80	56.1173	10	5030	1.98	59.0184	2.9011

The average weight of the common salt obtained in five experiments has been found 2.9232 g from 10 mL filtered soak liquor. Therefore the common salt can be recovered from filtered soak liquor nearly 292 g/L. One thing should be noticed that the volume obtained from final soak liquor after filtration via alum treatment is slightly less than its volume taken for treatment. Like in experiment no.-6 of Table-III.8, 60 mL volume (50 mL final soak liquor + 10 mL alum solution) is reduced to 47 mL after filtration followed by alum treatment. Thus the actual salt content in the final soak liquor should be slightly more than the value 292 g/L.

It has been found from more than five experiments that the quality of common salt crystal can be modified in whiteness by treating bleaching powder and size of crystal by controlling the rate of evaporation. But in this regards, further study is to be needed. The

common salt obtained from solar evaporation may be reused for preservation of raw hides and skins and also in pickling operation theoretically. But for the practical implementation we need some supplementary back up in this regard.

III.6.3 Proposed improved method of desalination

Solar desalination method has been followed to recover the dissolved salts from saturated salt solution, there is also scope to recover the solvent i.e. water from the same solution as well. The recovery of water will be an added advantage of the proposed method, as water is a precious natural resource and thus the proposed method is a welcome step in preserving this natural resource. Keeping this view an attempt has been made complete separation of salt and water from saturated brine solution using only solar energy. The proposed method is depicted in **Fig.III.47.**

The unit consists of:

1. Photo Voltaic Array P_V: Meant for supplying power to the two motors M_1 and M_2 as shown in **Fig. III.47** for supplying power to the pump motor M_1 meant for circulation of hot water through the heat exchanger coil and suction pump motor M_2 as shown in **Fig. III.47** meant for creating partial vacuum inside the Saline water tank in order to increase the evaporation rate.
2. Solar water heater Panel S_w: Meant for heating water to be supplied to the heat exchanger coil for enhancing the evaporation rate.
3. Hot water tank T_H: For storing hot water, from the solar water heater S_W to be supplied to the heat exchanger coil E_C for elevation of temperature of the saline water.

4. Glass covered Saline water tank T_S: For holding the saline water to be exposed to the solar radiation for evaporation.
5. Condensed water tank T_C: For collecting the condensed water from the saline water tank after evaporation.
6. Pressure gauge P: For measuring pressure inside the saline water tank.
7. Thermometer T: For measuring the temperature inside the saline tank.

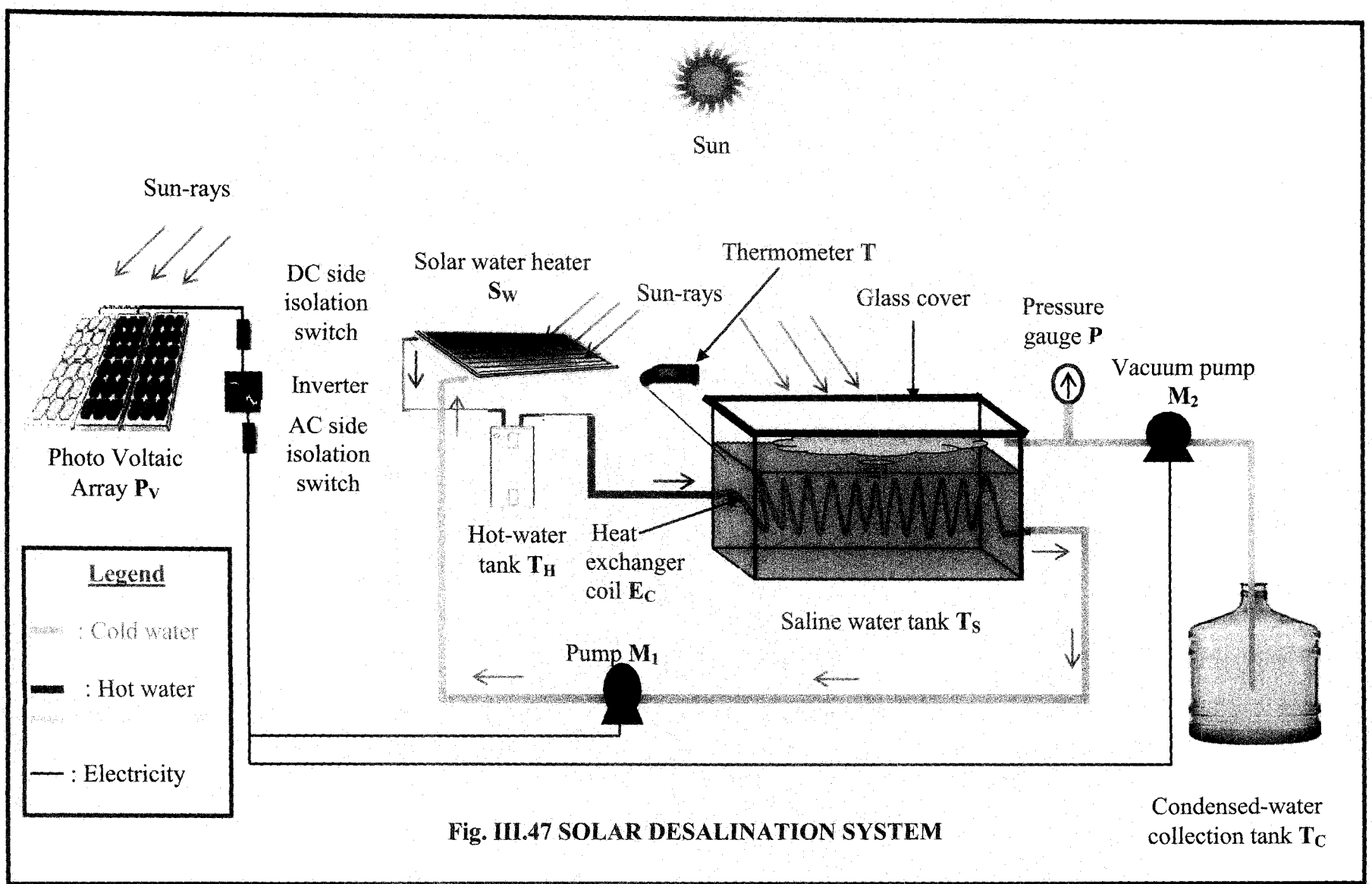


Fig. III.47 SOLAR DESALINATION SYSTEM

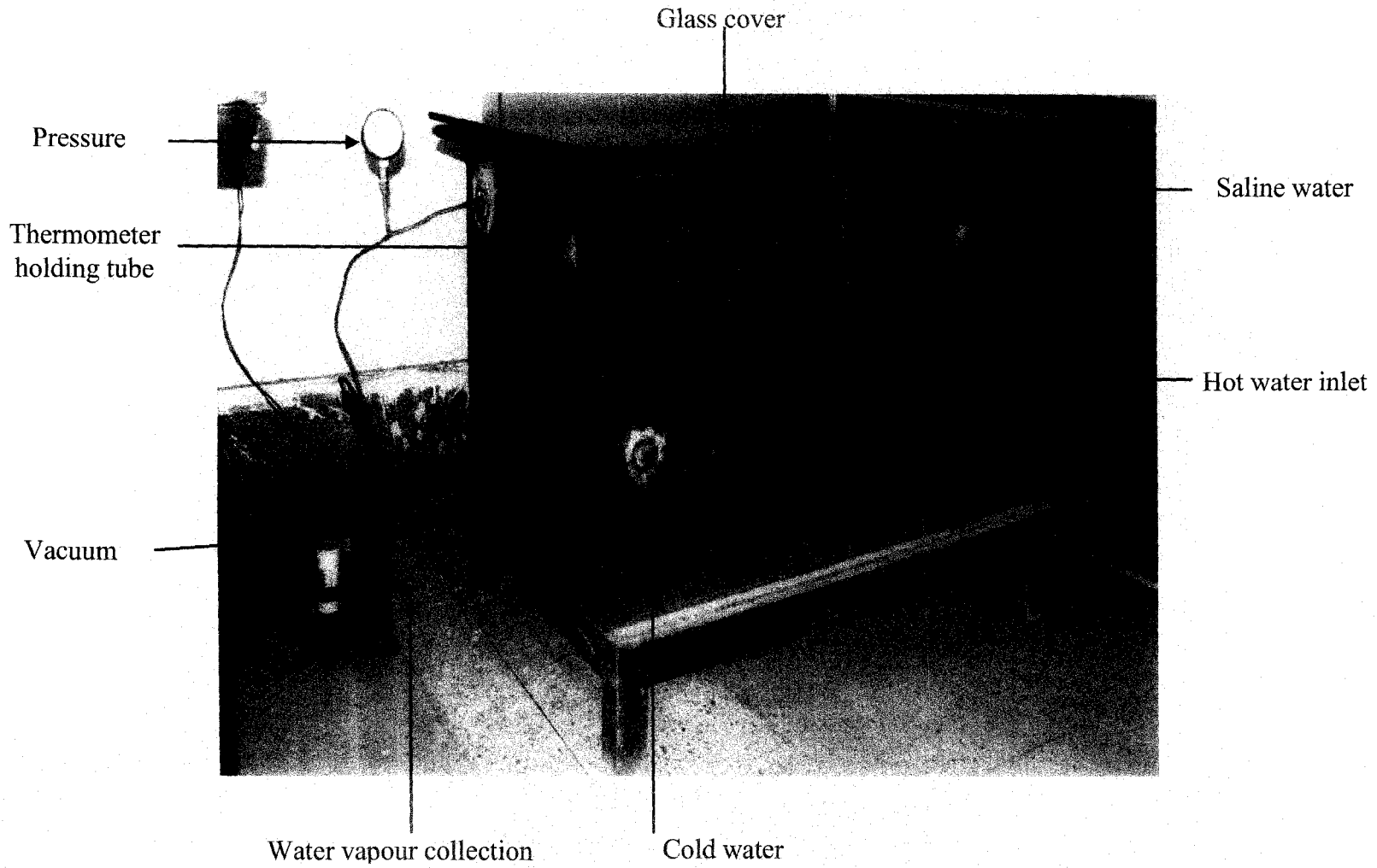


Fig. III.48 Front view of saline water tank

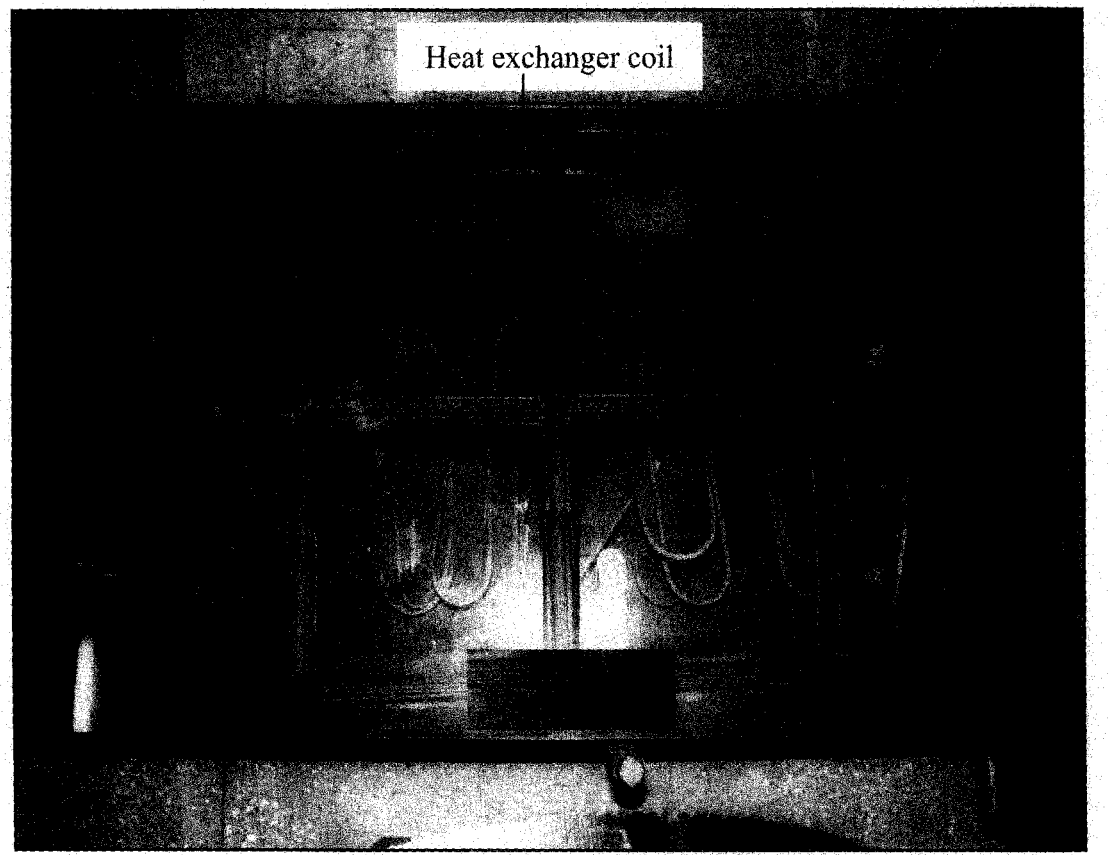


Fig. III.49 Top view of saline water tank