

Study on the Eco Processed Scouring Liquor Effluents of Bharat Merino, Sandyno and Coimbatore Kurumba Wool Fibres

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Abstract: *Wool fibers are hydrophilic in nature. There are variable contaminants in greasy wool and the same has to be removed in scouring. This study was focused on scouring of the selected wool fibres using a non - ionic detergent and studying its characteristics on Textile effluents for BOD (Biological Oxygen Demand), COD (Chemical Oxygen Demand), pH, TSS, Colour, Turbidity, Alkalinity, TDS, Oil and Grease. The results thus reveal substantial effective Scouring which are not harmful to the ecology.*

Keywords: wool fibre, scouring, effluent, characteristics

1. Introduction

Wool fibers are hydrophilic, meaning they readily absorb moisture, but are not hollow. Wool can absorb moisture almost one-third of its own weight. Wool absorbs sound like many other fabrics. It is generally a creamy white color, although some breeds of sheep produce natural colors, such as black, brown, silver, and random mixes.

Wool is the textile fiber obtained from sheep and certain other animals, including cashmere from goats, mohair from goats, angora from rabbits and other types of wool from camelids. Wool's scaling and crimp make it easier to spin the fleece by helping the individual fibers attach to each other, so they stay together. Because of the crimp, wool fabrics have greater bulk than other textiles, and they hold air, which causes the fabric to retain heat. Insulation works both ways such as Bedouins and Tuaregs use wool clothes to keep heat out and protect the body.

Wool ignites at a higher temperature than cotton and some synthetic fibers. It has a lower rate of flame spread, a lower rate of heat release, a lower heat of combustion, and does not melt or drip; it forms a char which is insulating and self-extinguishing, and it contributes less to toxic gases and smoke than other flooring products when used in carpets. Wool carpets are specified for high safety environments, such as trains and aircraft. Wool is usually specified for garments for firefighters, soldiers, and others in occupations where they are exposed to the likelihood of fire. Wool is considered by the medical profession to be hypoallergenic.

Wet processing is the segment of the textile production that involves cleaning, bleaching, dyeing and finishing of textile fibres and yarns in aqueous solutions. Most fabrics regardless of their end use go through one or all of these wet processing steps, www.elsevier.com. Water is used to clean, dye, apply chemical finishes to fabrics and to rinse the treated fibres or fabrics[16]. Wet processing of textile materials uses large quantities of water, energy and a considerable amount of time [8],[2],[1],[5],[9],[4]. Almost all textile wet processes involve the use of chemicals and processing auxiliaries like

phosphates, sulphates, alkalies, acids, heavy metals which result in contamination of the water used. According to [14], [6] the resultant waste water creates serious ecological effluent problems. [11], [7],[13] further state that this has expanded hands to develop bio-friendly textile products.

2. Processing of Wool

2.1 Shearing

Sheep shearing is the process by which the woollen fleece of a sheep is cut off. After shearing, the wool is separated into four main categories: fleece (which makes up the vast bulk), broken, bellies, and locks. The quality of fleeces is determined by a technique known as wool classing, whereby a qualified person called a wool classer groups wools of similar gradings together to maximize the return for the farmer or sheep owner. All Merino fleece wool is objectively measured for micron, yield (including the amount of vegetable matter), staple length, staple strength and sometimes color and comfort factor.

2.2 Composition of Raw Wool

Wool grease 2-25% of greasy wool weight Suint 2 - 12 % of greasy wool weight (dried perspiration) Dirt 5-45% of greasy wool weight Residues of insecticides, or insect growth regulators (IGR) used as veterinary medicines to protect sheep from ectoparasites, such as lice, mites, blowfly, etc. Fine wool from merino sheep, used apparel, typically contains 13 % wool grease, coarser wool used for carpets contains an average of about 5 % grease. Natural protein fibre obtained from hairs of sheep. Wool protein is known as Keratin. Differs from silk protein presence of sulphur in the form of cystine amino acid containing sulphur (-CH₂S-SCH₂-) cystine linkage. Impurities 30-70% depending on species of sheep.

Suint Dried perspiration Soluble in water. Removed by washing Wool Fat or Wool wax Complex mixture of esters, diesters and hydroxyesters fatty alcohol like lanoline and fatty acids. Wool fat is yellowish in colour. Soluble in

organic solvents like trichloroethylene or perchloroethylene which can be easily hydrolyzed in presence of mild alkali like ammonia at moderate temperature. Wool wax can be isolated and used in preparation of good quality soap and cosmetics.

Dirt is held by adhesive action of suint and wool fat which is removed during scouring and washing operation. Burrs Vegetable fragments consisting of dried grass, straw, sticks etc. are collected on the body of sheep during grazing and scratching the body against bush or tree to relieve itching.

2.3 Preparatory Processes for Wool

2.3.1 Wool Scouring

Wool straight off a sheep, known as "greasy wool" or "wool in the grease", contains a high level of valuable lanolin, as well as dirt, dead skin, sweat residue, pesticides and vegetable matter. Before the wool can be used for commercial purposes, it must be scoured, a process of cleaning the greasy wool. Scouring may be as simple as a bath in warm water or as complicated as an industrial process using detergent and alkali and specialized equipment. In North West England special Potash pits were constructed to produce Potash used in the manufacture of a soft soap for scouring locally produced white wool.

In commercial wool, vegetable matter is often removed by chemical carbonization. In less-processed wools, vegetable matter may be removed by hand and some of the lanolin left intact through the use of gentler detergents. This semi grease wool can be worked into yarn and knitted into particularly water-resistant mittens or sweaters, such as those of the Aran Island fishermen. Lanolin removed from wool is widely used in cosmetic products such as hand creams.

Scouring is a process by which all natural and additive impurities such as oil, wax, fat, hand dust etc., are removed to produce hydrophilic and clean textile material. It is one of the vital processes of wet processing. Scouring of wool differs from cotton scouring. Wax content of wool is higher than that of cotton. Cotton can withstand strong alkaline conditions at high temperature. Wool is sensitive to alkali. Sodium hydroxide is never used for wool scouring. Wool wax can be hydrolyzed by mild alkali like sodium carbonate or ammonia.

Objects of scouring

- To make the fabric more hydrophilic
- To remove impurities such as oils, waxes, gum, husks as nearly as possible
- To improve absorbency of fabric or textile materials without physical and chemical damage
- To produce a clean material by adding alkali
- To make the fabric ready for next process Wool Scouring is done in many ways.

Emulsion Scouring

Raw wool scoured in tanks filled with detergent or oleate soap (2-4% conc. Owf) pH 8-10, Temp. 55-60°C, time 30-45 min. Wash with water.

Solvent Scouring

Widely used ready solubility of wool wax in solvents like Trichloroethylene, perchloro ethylene or carbon tetrachloride. No danger of wool degradation. Wax can be easily recovered in pure form by solvent evaporation during solvent recovery. Solvent recovery is essential for economic benefits.

Freezing Scouring

Raw wool is subjected to low temperature of -30deg.C Wool wax becomes hard crushed mechanically at low temperature removed by mechanical shaking of wool on a sieve. All operations to be carried out at low temperature.

Chemical Scouring

Peroxide also gives mild scouring action. It doesn't give any scouring action. Simultaneous scouring and bleaching is possible in continuous process. It doesn't affect the coloured material so it can't be used over coloured material can be used for coloured materials. With H₂O₂ there is no need of danger of a problem of corrosion and equipment corrosion and no unpleasant odours. Only rinsing after bleaching is sufficient.

3. Methodology

3.1 Selection of Wool Fibre

Bharat Merino wool fibre, Sandyno wool fibre, and Coimbatore Kurumba wool fibre were procured and selected for the study.

3.2 Processing of Wool Fibre

3.2.1 Scouring of Wool

Scouring, the first step in wool processing removes oily and greasy impurities from the fibre and also improves the absorbency and dyeability of the fibres [3],[12],[10]. According to the [21], the impurities present in wool are classified as natural, acquired and applied impurities. Natural impurities are the glandular secretions adhering to the fleece. The two major components are suint and wool grease. Suint is the dried perspiration of sheep and is water soluble. Wool grease is water insoluble matter and special type of scouring is required for its removal which contributes to significant pollution load. The acquired impurities include soil, dust, dirt, straw, vegetable and fecal matter. Applied impurities occur due to treatments given to sheep against insect and pests. So, prior to spinning the procured wool was subjected to careful scouring and drying. Traditionally, scouring employs cleaning in warm detergent solution and soft water for four to six times.

The scouring of wool in neutral or very lightly alkaline baths reduces the fibre damage and enhances the rate of processing. A satisfactory scouring should be given using appropriate detergent and minimum alkali. In preparatory proceedings, the starch degrading enzymes are sensitive to ionic wetting agents and hence only non-ionic wetting agents have to be used. Both neutral and alkaline aqueous scouring systems based on non-ionic surfactants are currently used.

Wool can also be scoured with organic solvents and chemicals. In chemical scouring, the scales are affected very badly. Hence, solvent scouring is usually followed by a detergent wash as recommended by [20]. The water insoluble wool grease is removed by treating the desuinted wool with a mixture of detergent and sodium carbonate. Hence, the selected Bharat Merino, Sandyno and Coimbatore Kurumba wool fibres were first soaked over night in soft water. This soaking process removes dirt like sand, cow dung and urine extractions. The soaking resulted in dark grey or black coloured effluent water. The rinsed wool is further scoured and rinsed.

A specially formulated non-ionic detergent was used for the study. It is a biologically degradable wetting agent with emulsifying and dispersing action for the scouring and removal of mineral oil contamination from textiles. Therefore, thousand grams of selected wool fibre was treated in a solution of eleven ml of non - ionic liquid detergent and three grams of sodium carbonate keeping M:L ratio as 1:60 of soft warm water at $52 \pm 2^\circ\text{C}$ temperature. The selected wool fibres were immersed in the first bowl for five minutes and agitated gently at intervals. Later, the wool fibres were squeezed without rubbing and transferred to the second bowl, for three minutes and finally transferred into the third bowl. The wool fibres were squeezed, rinsed thoroughly with plain soft water after three minutes, so that all soapy materials were removed in the fourth bowl following three rinses. The wool fibres were dried at room temperature in open air.

3.3 Characteristics of Wool Scouring Liquor Effluent

The environment problems associated with the scouring and dyeing of textiles as effluents discharged by textile processing are typically related to pollution caused by the discharge of untreated effluents. Textile effluents are generally grey (in pre-treatment processes) or coloured (colouration processes) and have a high BOD (Biological - Oxygen Demand), COD (Chemical Oxygen Demand), high solids and have high temperature in some cases. The constituents of the liquors with respect to pH, TSS, Colour, Turbidity, Alkalinity, COD, BOD, Oil and Grease and TDS can be analyzed.

Considering this, the scouring effluent was collected by mixing the scouring waters from three bowls and the required quantity of scoured effluent was taken as a sample from each variety of Bharat Merino, Sandyno and Coimbatore Kurumba wool fibres and transferring them in an air tight container. The scouring effluents were analyzed chemically as suggested by [21],[15] for their pH, Temperature, Total Suspended Solids, Colour, Turbidity, Total Alkalinity,

Chemical Oxygen Demand, Biological Oxygen Demand, Oil and Grease and Total Dissolved Solids.

3.3.1 Potential Hydrogenii (pH)

pH is a measure of hydrogen ion concentration in water. pH is the most important parameter, as it indicates instantaneously the acidic or alkaline conditions of an effluent water [22]. The pH value which is determined according to ISO 3071 is restricted to the range of 4.0 to 7.5 because it corresponds to the natural conditions of undamaged human skin. According to [20], waters with pH below 7 are acidic and pH above 7 is alkaline. pH of the waste water indicates the acidic or alkaline nature of the effluent. It helps in two ways as it is useful in determining the type of the treatment to be applied to the effluent and it determines the efficiency of the applied treatment method. It is commonly known that during the process of colouration, there are possibilities of the pH drifting to the higher side owing to several reasons like poor water quality, presence of residual alkali, improper washing, faulty pH meter, weigh errors and low strength of the acid. The pH should be between 5.5 to 9.0. The measurement is made using a pH meter.

pH can be measured electrometrically or calorimetrically. Electrometric methods are by far the most accurate and suffer with little or no interference. To test the scouring and dyeing effluents for pH, the electrodes were washed thoroughly with distilled water before measurement. The system was allowed to stabilize before making the final reading. The samples containing oil or grease were removed by filtration and then measured for pH. If any oily film sticks to the electrodes, it can be removed with a soft tissue soaked in suitable solvent or detergent followed by thorough washing with distilled water. Care should be taken while filling the solution in the reference electrode so that no air bubbles are entrapped in the solution or in the liquid junction. After measurement of pH, the electrodes should be thoroughly washed with distilled water.

3.3.2 Temperature

Temperature measurements are usually made with mercury filled centigrade thermometer. The reading should be made by dipping the thermometer in the sample (water or waste water). The temperature should be expressed to the nearest centigrade.

3.3.3 Total Suspended Solids (TSS)

The undissolved matter present in the effluent water is usually referred to as suspended solid. Organic matter and living organisms can account for suspended solids. It is one of the valuable parameters in judging the pollution potential of an effluent. It is determined by filtering or centrifuging the sample, drying the residue and determining its weight by difference. The procedure for testing TSS is by taking 100 ml of the effluents and the same was filtered through a preweighed whatman No.40 filter paper. The filter paper was then dried and weighed again. The difference in weight is the weight of TSS. The calculation for TSS is as given below:

$$\text{TSS} = [\text{Weight of residue (mg)} / \text{Volume of effluent (ml)}] \times 1000 \text{ (ppm)}$$

3.3.4 Colour

Colour is a common constituent of many natural waters. Dye house wastes are the most important effluent as they are intensely coloured and impart turbidity. Observation of the colour is the primary test to determine the line of treatment. The standard method for colour measurement involves the use of "standard colour solution" prepared by using potassium chloroplatinate and cobaltous chloride and comparing the sample with the standards. Turbidity in excess of 5 mg/l seriously affects the true colour determination. Hence it should be removed prior to determination by centrifugation. Care should be taken not to filter the sample to remove turbidity or suspended matter, since filtration has a decolourising effect. To prepare the standard colour solution, dissolve 1.246g of potassium chloro-platinate, K_2PtCl_6 and 1.0 g cobaltous chloride hexa hydrate, $CoCl_2 \cdot 6H_2O$ in distilled water. Add 100 ml concentrated HCL and make up to 1000 ml in a volumetric flask with distilled water. This standard solution should be equal to 500 Hazen units. Place 50 ml of the effluent sample (centrifuged) in a 50 ml nessler tube.

Compare the colour of the sample with that of the working colour standards by looking vertically downwards against a pure white surface placed at such an angle that light is reflected upwards through the columns of liquid. If the colour exceeds 70 units, dilute the centrifuged sample with distilled water such that the colour is within the range.

3.3.5 Turbidity

Turbidity is caused by the final particles present in suspension. It is to be mentioned that waters having the same turbidity may possess different types of solids in different quantities and their effects may vary. Normally ground waters from deep wells and brine holes are clear [20]. However, they may also become turbid or attain colour on standing.

3.3.6 Alkalinity

Alkalinity in water is not a specific substance but rather a combined effect of several substances and conditions. [20] Alkalinity is caused by the presence of bicarbonates, carbonates, hydroxides and to a certain extent by borates, silicates, phosphates and organic substances. Various limits for alkalinity in water have been prescribed by different authors, the maximum being 150 mg/l as $CaCO_3$ (as per the Bureau of Indian Standards). However, the upper limit of alkalinity depends entirely on the intended use. Four drops of mixed indicator (or methyl orange) is added to the effluent sample in which phenolphthalein alkalinity has been determined and titrate against sulfuric acid to pH 4.5. The colour change is from emerald green to light pink, with methyl orange, the colour change is from yellow to orange red.

3.3.7 Chemical Oxygen Demand (COD)

COD is called as dichromate value. An indication of the organic content of water can be obtained by measuring the oxygen required for its stabilization. To measure the content of organic matter of an effluent, normally COD test is carried out. In COD test a strong chemical oxidizing agent is used in acid medium and the oxygen equivalent of the organic matter

is determined. The test is normally carried out using $K_2Cr_2O_7$ at temperature in the presence of some catalyst like silver sulphate. The COD can be determined in 3-4 hours.

To determine COD, 20 ml effluent sample was taken and 10ml of acid $K_2Cr_2O_7$ (0.25 N) along with a pinch of mercuric sulphate and one glass bead. Then 30 ml of COD acid through condenser was added in an ice bath. The prepared solution was kept in a COD digestion apparatus for 2 hours at 150°C. Then, it was taken out, rinsed through condenser with little distilled water. It was then cooled in an ice bath by adding 3 drops of ferroin indicator and titrating it against 0.1N ferrous ammonium sulphate. The end point shows the appearance of wine red colour. The above seven steps are repeated for blank solution preparation. The COD was thus calculated as:

$$COD = [(B - A) \times \text{Nor. of FAS} \times 8 \times 1000 \times \text{dilution factor}] / \text{Volume of the sample taken.}$$

3.3.8 Biological Oxygen Demand (BOD)

BOD is defined as the amount of oxygen required to carry out the biological decomposition of dissolved solids in effluent under aerobic conditions at standard temperature [22],[18]. The BOD test measures the oxygen consumed by bacteria during the process of oxidizing organic matter under aerobic conditions. In dilution requirements, often the BOD concentration in waste / effluent water will be more than the DO. Hence dilution was required, bacterial growth requires nutrients such as N_2 , P and trace metals. Addition of buffer maintains pH. To determine BOD, in a 500 ml BOD bottle, 5ml of effluent sample was taken and 1ml each of four buffers was added with 1ml seed and diluted with distilled water to find out the DO of this mixture immediately (Blank value -A). Two other effluent samples as above were prepared and kept for incubation one at 20°C for 5 days and another at 27°C for 3 days. The DO of this mixture is found out on completion of the period (Sample value-B). BOD is calculated as:

$BOD \text{ (mg / lit.)} = (A - B) \times \text{Dilution factor}$. According to the Textile Committee (1983), the strength of the effluent is given in ppm which ranges from very strong – above 550; strongly effluent -450; average effluent – 350; weak effluent – 250; standard filter effluent – 20 and very good filter effluent – 5 to 10 ppm.

3.3.9 Total Dissolved Solids (TDS)

The determination of dissolved solids helps in the estimation of dissolved mineral matter content of the effluent [23]. The dissolved inorganic matter, mostly sodium salts increase the salinity of water and consequently it becomes unfit for irrigation. The term dissolved solids is applied to the soluble substances present in the water. Determination of dissolved solids is useful in deciding the mineral matter content.

The procedure for TDS is that by taking 100 ml of the effluent, filtered through a whatman No.40 filter paper in a preweighed evaporating dish. The filtrate was evaporated and the residue was weighed. The difference in weight is the weight of TDS and calculated as follows:

TDS = (Weight of residue (mg) / (Volume of effluent (ml) x 1000 (ppm)).

3.3.10 Oil and Grease

Oils, fats, soaps and greasy substances gain access into water through the discharge of industrial effluents. These contaminants coat the materials with which they get contact. They have to be removed from the water, otherwise they may soil the fabric. Oil and grease is normally determined by solvent extraction with dense low – boiling solvent such as Methylene chloride or Petroleum ether.

For this, 250 ml or appropriate volume of the sample effluent was placed in a separating funnel. To this 5 ml. of sulphuric acid per litre was added to the effluent sample. The sample bottle was rinsed with 15 ml petroleum ether and the same was added to the separating funnel. 25 ml. of ether was added to the funnel and shaken vigorously for two minutes. The aqueous phase was drawn into a clear container. The ether layer was filtered through a filter paper (whatman No. 42) containing sodium sulphate in its cone and moistened with petroleum ether, into a tared distilling flask. Continue the extraction of the aqueous layer is continued twice and the

ether extract is added to the distilling flask, after passing through sodium sulphate in the filter. The ether in the distilling flask is distilled until about 10 ml. remains in the flask. Then it is dried on a water bath and cool in a desiccator and weighed.

Oil and Grease mg/l = {(mg. residue distilling flask) / mg. sample taken for determination} x 1000

The wool scoured effluents of 100% Bharat Merino (BM), 100% Sandyno (S) and 100% Coimbatore Kurumba (K) wool fibres were analysed for the above mentioned parameters for the study.

4. Results and Discussion

4.1 Characteristics of Wool Scouring Effluent

The characteristics of wool scouring effluents (waste scour liquor + rinse water) are shown in Table I.

Table 1: Characteristics of Wool Scouring Effluent

| S. No | Parameters | Wool Scouring Effluent | | | BIS IS:2490 (PART- I) |
|---|--|------------------------|-------|-------|--|
| | | BM | S | K | PCB NORMS TESTING (BIS) PUBLIC SEWERS |
| 1 | pH at 30°C | 8.53 | 7.75 | 8.33 | 5.5 – 9.0 |
| 2 | TSS mg/l | 570.5 | 546.5 | 330.5 | 600 |
| 3 | Colour Hazen | 325 | 400 | 210 | - |
| 4 | Turbidity NTU | 540 | 695 | 290 | - |
| 5 | Total Alkalinity as CaCO ₃ mg/l | 80 | 100 | 115 | - |
| 6 | COD mg/l | 768 | 1122 | 384 | - |
| 7 | BOD 27°C mg/l 3 days | 102 | 106 | 12 | |
| 8 | BOD 20°C mg/l 5 days | 110 | 120 | 15 | 350 |
| 9 | Oil & grease mg/l | 44 | 74.5 | 19 | 20 |
| 10 | TDS mg/l (Evaporation Method) | 436 | 488 | 464 | 2100 |
| IS:2490 (Part –I) General Limits (Second Revision with 1985 Amendments) (Superseding IS:3306-1974, IS:3307-1977 and IS:7968-1976) Note:1 All efforts should be made to remove colour and unpleasant odour as far as practicable. Note:2 Further relaxation may be decided by the concerned agencies. Note:4 For paper, dye stuff, pesticide and certain chemical and petro chemical industries, these values may be relaxed, whenever these values are relaxed, it shall be ensured that the effluent passes the test for toxicity as given in IS:6582-1971. | | | | | |

From the Table I, it could be inferred that scoured effluent of the Sandyno (S) variety of wool has the highest values for colour, turbidity, COD, BOD at 27°C and 20°C, oil and greases, TDS as 400hazen, 695NTU, 1122mg/l, 106mg/l, 120mg/l, 74.5mg/l and 488mg/l respectively. The highest value for TSS and alkalinity as 570.5mg/l and 115 mg/l is observed in Bharat Merino (BM) and Coimbatore Kurumba (K) wool varieties. Hence, it could be concluded that the Sandyno scoured effluent has the maximum pollution when compared to Bharat Merino and Coimbatore Kurumba wool varieties. However on the whole, the scoured effluent of all the three varieties proved to be lesser than BIS sewage norms.

5. Conclusion

Eco-friendly processing and increasing concern for ecological preservation has led to the quest for resources that are safe, bio-degradable and re-cyclable. National and International awareness about milieu and ecology has increased the use of eco-friendly processing methods and techniques all over the orb. The National Water Quality Management Strategy (NWQMS) which aims to achieve the sustainable use of the nation's water resources by protecting and enhancing their quality, while maintaining economic and social development.

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