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Ultrasound scouring of wool and its effects on fibre quality

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Wool scoured using ultrasound irradiation at intermediate stages has been analysed and compared with the wool scoured without ultrasonic energy. The conventional recipe is modified with 25% reduction in chemical concentration, time and temperature. The scouring efficiency is measured in terms of residual grease content. Ultrasound energy effectively removes grease with lower concentration of chemicals, temperature and time. Ultrasound subjected to all bath scouring has lowest residual grease content. However, the highest improvement in whiteness is observed during ultrasound irradiation on rinsing baths. It is also observed that the combination of ultrasound and alkali adversely affects whiteness and yellowness. Scanning electron microscope analysis shows no cuticle damage after ultrasound exposure on wool fibre. In addition, the chemical properties of wool are not changed due to ultrasound treatment. The mean fibre diameter, single fibre strength and moisture content do not show significant change after ultrasound irradiation.

Keywords: Fibre properties, Residual grease content, Scouring, Ultrasound, Whiteness, Wool

1 Introduction

The objective of wool scouring is to remove grease, dirt and suint from the wool without damaging the fibre properties. The scouring method and technique applied have a direct effect on the quality of scoured wool[1]. Emulsification of the grease on the fibre surface in aqueous scouring is the most important process. The detergent, water, temperature and mechanical movement of the scouring liquor act to remove the contaminants from the wool. Aqueous scouring is usually carried out under conditions of high temperature (> 50°C) and high detergent concentrations[2]. The use of high amount of chemicals and detergents in the conventional methods of wool scouring generates some serious consequences both for the environment and the industry while effluent treatment and disposal. Moreover, conventional mechanical scouring can result in poor whiteness and a higher level of residual dirt[3, 4].

Ultrasonic-assisted scouring of wool has the potential to save water and energy, improves product quality, and reduces the time and use of auxiliary chemicals[5,6]. The ultrasound rinsing can remove effectively different substances and pollutants from the textile surfaces even without use of surfactants in the rinsing bath. This could be attributed to the cavitations occurring at certain parameters of the ultrasound field[3, 7]. Ultrasound is defined as the sound with frequency beyond the audibility limit of humans, that is higher than 16 kHz (16000 cycles/s)[8]. When the ultrasonic waves are propagated in liquid medium, sudden drop in acoustic pressure causes the scouring liquor to fracture and the longitudinal vibrations of molecules to generate compression and rarefaction waves, giving rise to cavitation. During compression phases, vapour filled microscopic bubbles of size 10–100 μm expand and collapse violently, thus generate shock waves[9-11]. Acoustic cavitation, the process of bubble formation and collapse, is considered responsible for most of ultrasound's physical and chemical effects observed in solid/liquid or liquid/liquid systems[12]. Ultrasound assists cleaning in two ways namely micro-jetting caused by cavitation bubble implosion and micro-streaming resulting from cavitation bubble oscillation[13]. The combined effect of cavitation and micro-streaming results in intermolecular tearing and fibre surface scrubbing during the ultrasound treatment[11].

Scouring process consists of different sub-processes to clean the wool. It includes alkali treatment, detergent treatment, and rinsing treatment. So far it is not known at which stage of scouring the ultrasound is more effective. In the present work, ultrasound irradiation was selectively subjected to these intermediate sub-processes of wool scouring. Its influence on scouring efficiency was studied in terms of residual grease content. The study was extended to

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examine degree of damage to wool fibre for its chemical properties due to ultrasound scouring. The effect of ultrasound on important properties of fibres, viz. fibre whiteness, yellowness, mean fibre diameter, single fibre strength and moisture content was also studied.

2 Materials and Methods

2.1 Materials

The raw crossbred wool obtained from Himachal Pradesh, India was used for all scouring experiments. The chemicals used were sodium carbonate, Lissapol-N (non-ionic detergent) and ethanol (L.R. Grade) for soxhlet extraction. The scouring bath ratio of 1:50 was kept constant for all the treatments.

2.2 Methods

Conventionally, wool was scoured in aqueous condition in five baths using a combination of detergent and alkali like sodium carbonate. The conventional recipe (CR) of wool scouring is presented in Table 1 (ref. 14). In order to study the efficacy of ultrasound in wool scouring the recipe was modified. In the modified recipe (MR), alkali, detergent, time and temperature were reduced by 25% to that of conventional recipe, as shown in Table 1. Five different scouring experiments with modified recipe were conducted, namely ultrasound to alkali bath (US-A), ultrasound to detergent baths (US-B), ultrasound to rinsing baths (US-C), ultrasound to alkali and detergent baths (US-D) and ultrasound to all baths (US-E). The ultrasound treatment was given only to specific baths at a time and its intensity is progressively increased (Table 2).

2.2.1 Ultrasonic Treatment

A lab scale ultrasonic cleaner from M/s Oscar Ultrasonic Ltd., India (OU-mini 120 Model) was used for ultrasonic treatment. The ultrasound was generated at 33 kHz frequency with output power of 120 W. All ultrasound experiments were conducted on the same equipment as per protocol given in Table 2.

2.2.2 Residual Grease Content Measurement after Scouring

The grease content of raw wool and residual grease content after scouring were measured as per the standard method IWTO-19-03. The soxhlet extractor of capacity 250 mL assembled with ground glass joints to a 250 mL distillation flask and reflux condenser was used for accurate measurement of residual grease content.

2.2.3 Whiteness and Yellowness Measurement

The ASTM whiteness index (WI) and yellowness index (YI)-E313 of samples, before and after scouring, were determined by using spectrophotometer colour I-match (version 7) according to IWTO-35-03 standard test method. The improvement in whiteness and reduction in yellowness are expressed as the percentage change, relative to the original whiteness and yellowness respectively.

2.2.4 Surface Characterization and Alkali Solubility Test

The surface morphologies of scoured wool fibres after gold coating were observed under scanning electron microscope (SEM) (Philips model XL30). The alkali solubility test determines the amount of wool substance soluble in alkali under standard conditions. The test was carried out according to the standard test method ASTM D 1283-05.

2.2.5 Moisture Content Measurement

All the samples were preconditioned in a stability chamber for 24 h at 65% RH and 27°C. The moisture content has been determined before and after ultrasound scouring. The oven dry mass was determined according to standard IWTO-34-85(E) method.

Table 1—Conventional and modified wool scouring recipe

Recipe	Bath 1	Bath 2	Bath 3	Bath 4	Bath 5
Conventional (CR)	Na_2CO_3	Lissapol-N	Lissapol-N	Rinse	Rinse
	(1.33 gpL)	(0.5 gpL)	(0.5 gpL)	-	-
	60°C	55°C	50°C	45°C	45°C
	3 min	3 min	3 min	2 min	2 min
Modified (MR)	Na_2CO_3	Lissapol-N	Lissapol-N	Rinse	Rinse
	(1 gpL)	(0.5 gpL)	(0.5 gpL)	-	-
	55°C	50°C	50°C	40°C	40°C
	2 min	2 min	2 min	1.5 min	1.5 min

Table 2—Ultrasound scouring experiment protocol

Sample ID	Ultrasound treatment				
	Bath 1	Bath 2	Bath 3	Bath 4	Bath 5
CR	×	×	×	×	×
MR	×	×	×	×	×
US-A	√	×	×	×	×
US-B	×	√	√	×	×
US-C	×	×	×	√	√
US-D	√	√	√	×	×
US-E	√	√	√	√	√

CR–Conventional recipe, MR–Modified recipe, US-A–Ultrasound treatment for alkali bath), US-B– Ultrasound treatment for detergent baths, US-D–Ultrasound treatment for alkali and detergent baths, US-E–Ultrasound treatment for all baths.

2.2.6 Fibre Mean Diameter Test

Fibre diameter measurement was carried out with OFDA 100 as per the standard IWTO 47-2011. The fibre samples were cut into 2 mm snippets and spread on a 70 mm square glass slide. The whole slide was scanned with a minimum of 6000 fibres measured in each measurement. For each sample, three measurements were taken. The mean diameter and standard deviation of the sample were then calculated.

2.2.7 Single Fibre Strength Test

The single fibre strength of raw and all scoured wools was measured on Shimadzu tensile strength tester according to ASTM D 3822 standard test method. The instrument was based on constant rate of elongation (CRE) principle. The distance between jaws was 10 mm and the traverse rate was 6 mm/min.

3 Results and Discussion

3.1 Effect of Ultrasound Irradiation on Residual Grease Content

For ease of processing on the worsted system the residual grease content of wool fibre needs to be below 2%. The residual grease content (RGC) obtained by different treatments is shown in Table 3. It is observed that RGC of US-E is superior to that of MR and is comparable with CR. It infers that desired RGC can be obtained by using ultrasound energy with 25% reduction in chemical concentration, time and temperature. Goud *et al.*[14] reported that the ultrasound assisted scouring can be carried out at much lower temperature (50°C), utilizing lesser amount of chemicals (1gpL of both alkali and detergent) with substantial saving in time and comparable value of RGC. Hurren *et al.*[2] studied the micro scale high fluid pressures caused by the ultrasonic agitation emulsify and remove the grease more effectively than the conventional method at the lower scouring temperature.

Table 3—Residual grease content, whiteness and yellowness index values of scoured wool

Sample ID	Residual grease content, %	WI[a]	% Improvement in whiteness	YI[a]	% Reduction in yellowness
Raw wool (before scouring)	11.68	-18.2	-	46.9	-
CR	0.93	11.5	162.9	22.4	52.3
MR	1.33	11.2	161.3	24.6	47.5
US-A	1.18	11.1	160.9	27.2	41.9
US-B	1.13	20.8	214.1	21.1	55.1
US-C	1.2	24.3	233.6	20.0	57.4
US-D	1.11	14.3	178.3	22.1	52.9
US-E	0.96	11.2	161.3	26.2	44.2

WI– Whiteness index, YI–Yellowness index.
[a]Measured using American Standards Test Method E313.

The RGC for intermediate ultrasound treated scouring baths (US-A, US-B, US-C, US-D) is found lower as compared to that of modified recipe. It indicates cavitation to the smallest extent also assists in grease removal during scouring. The micro-agitation occurring in the vicinity of the cavitation bubble effectively wets out the fibre surface and helps to displace particulate contaminants and grease[13]. Fibre surface is wiped out by micro-brushing action of ultrasonic waves and effectively cleans the fibre surface. It is also observed that ultrasound treatment for all five baths of scouring was among the lowest residual grease content. It leads to derive the fact that the grease removal efficiency will increase, as the number of times this micro-agitation increases. When the high pressure bubble implodes near a hard surface, it changes its size into a jet about one-tenth the bubble size. Because of the inherently small size of the jet and the relatively large energy, ultrasonic cleaning has the ability to reach into small crevices and remove entrapped particles effectively.

3.2 Whiteness Improvement and Yellowness Reduction

Hurren *et al.*[2] reported that fibre colour is not significantly affected by the scouring method. They did not find any significant difference in the colour of the wool scoured with ultrasonic agitation in comparison with fibre scoured with conventional method. The whiteness improvement in CR and US-E is found to be comparable and agrees with earlier reported study[2]. However, a different phenomenon of whiteness improvement for US-C (ultrasound subjected to rinsing baths only) has also been observed. The result deducted mainly due to two reasons. Firstly, detergent forms micelles over an outer layer on fibre surface and avoids dust particle to redeposit. During rinsing with ultrasound, the shock waves greatly speed up the breaking of the hanging contaminants, evenly distribute cavitation implosions in a liquid medium and enhance displacement with the detergent film. Secondly, the ultrasound is more effective in only aqueous medium and not along with chemicals, especially alkali. When ultrasound is used along with chemicals, its dirt removal efficiency is being reduced. This is because the cleaning bath consists of detergents and a chemical has lot of air bubbles which adversely affects the ultrasound cavitation. Therefore, whiteness improvement and yellowness reduction in US-A, US-B and US-D is relatively lower than in US-C.

The whiteness and yellowness indices of wool, before and after scouring are presented in Table 3. The high whiteness improvement percentage after scouring, ranging from 160 % to 233%, is attributed to negative whiteness values because of grease present in raw wool and effective scouring. Further, the trend observed in whiteness improvement and yellowness reduction is the same. The increasing order of improved performance is US-A < MR < US-E < CR < US-D < US-B < US-C. The lowest improvement in US-A is due to the reduction in scouring recipe and combination of alkali-ultrasound, which adversely affected the cleaning process. In case of US-E (ultrasound exposure to all baths), whiteness is deteriorated and is found to be comparable to CR. This may be because of partial re-adhesion in addition to alkali-ultrasound combination. Dynamic waves of ultrasound throughout scouring might not allow the dirt particles to become stable and forced to redeposit on the fibre surface. Sonic shock waves on loaded micelles might result in partial re-adhesion.

When ultrasound is exposed to rinsing baths only, the yellowness reduces to its maximum extent (57%), as shown in Table 3. It is followed by US-B where ultrasound is used along with detergent. It means ultrasound assists in cleaning action of detergent.

3.3 Surface Characterization

Goud *et al*.[14] reported that ultrasound has not created any surface topographical changes and caused no damage to the wool fibres. Surface scales are found to be intact and no surface cracking is observed after ultrasound treatment. However, it is reported that ultrasound treatments for wool scouring have shown fibre surface modifications and wool cuticle disruption[15]. Fibre surface scale cracking is occurred during the ultrasonic treatment[2]. Wool cuticle disruption can be related to the ultrasonic frequency employed. The scale peeling and severe surface damage are evident at low frequency ultrasound irradiation (28 kHz). However, it is also reported that not all fibres within the bath suffer fibres scale damage and it is observed for only a small portion of the total fibres[13].

The SEM images of MR, US-C and US-E are compared in Fig. 1. No cuticle damage is observed to all the fibre samples at 33 kHz ultrasound frequency for a maximum 9 min irradiation time. On the other hand, US-C surface is found to be smooth and clean as compared to other two samples that resulted at higher reflectance. Thus, it confirms the improvement in whiteness of wool.

3.4 Effect of Ultrasound on Wool Fibre Properties

Alkali solubility is an indication of the degree of damage to wool due to chemical treatments. Undamaged scoured wool has typical alkali solubility in the range of 9-15 %. The alkali solubility determined for all the samples is found in the range of 9-11 % (Fig. 2). This indicates that ultrasound exposure for scouring has no harmful effect on chemical properties of wool fibre.

The ultrasound energy is locally impacting only on the cuticle and not on the cortical cells of the fibre. The increased moisture uptake can be directly attributed to the surface cracking as the moisture is able to diffuse into the fibre through the cracks as well as between the scales[2]. It is reported that the

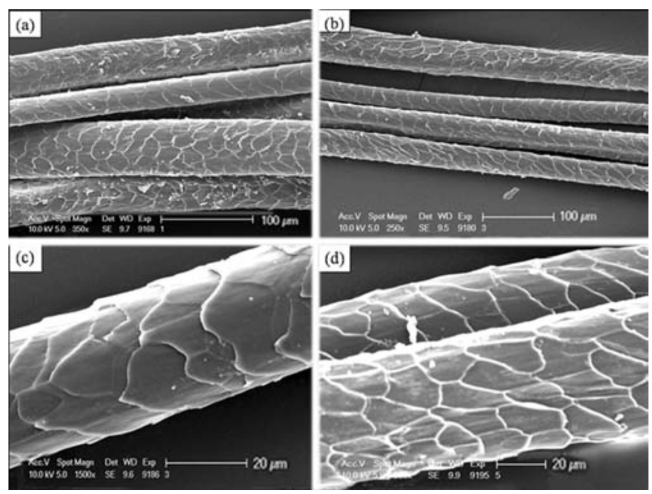

Fig. 1—SEM images of wool fibre scoured with (a) modified recipe (MR); (b) and (c) ultrasound irradiation to washing baths (US-C); (d) ultrasound irradiation to all baths (US-E)

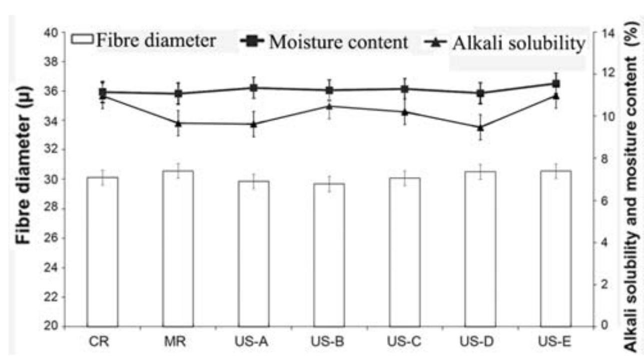

Fig. 2—Effect of ultrasound on wool fibre properties

Table 4—Single fibre strength of wool with and without ultrasound

Sample code	Maximum breaking load, N		Extension at max load %	
	Load	SD	Extension	SD
CR	0.27	9.13	53.38	11.11
US-E	0.3	7.93	60.16	15.99

SD– Standard deviation.

waxy lipid layer on fibre surface gets disrupted upon ultrasonic treatment, providing the fibres with increased water absorption. The decrease in the hydrophobic ingredient makes the hydrophilic component to absorb more water mainly due to high irradiation time of ultrasound[16].

There is no significant change in moisture content because of ultrasound treatment which is also supported by SEM images of undamaged scale surface. As shown in Fig. 2, all readings are nearly equal to 11% and no significant difference is found at 5% significance level between samples scoured with conventional and different ultrasound recipes.

Hurren *et al.*[17] reported that the ultrasound treatment has no impact on mechanical properties of wool fibres. When wool is treated with ultrasound for 90 min there is negligible effect on the tenacity and elongation of wool fibres. This is attributed to high degree of variability in fibre dimensions and non-uniform irradiation energy within the ultrasonic bath.

Prolonged ultrasonic treatment for more than 2 h can cause breakage of bonds and linear segments that link the polypeptide chains of wool fibre, resulting in less coherent interactions within the protein structure and significant reduction in fibre tenacity and extensibility[18]. The present study found that there is no significant difference in single fibre breaking load and extension at 5 % significance level among all scoured samples (Table 4). It has also been found that there is no difference in fibre diameter of all the samples at 5% significance level. As shown in Fig. 2, the fibre diameter range is in between 29.5µ and 30.5µ. It means that the ultrasound treatment used for scouring does not affect the fibre diameter.

4 Conclusion

4.1 Ultrasonic cavitation has enhanced the grease removal efficiency. The scouring with ultrasonic energy has the ability to reach into small crevices and remove entrapped particles very effectively.

4.2 The desired residual grease content can be achieved through ultrasound scouring with 25% reduction in chemical concentration, time and temperature.

4.3 Ultrasound irradiation to only rinsing baths yields wool fibre with maximum whiteness and least yellowness. Ultrasound waves are more effective in aqueous medium only. Combination of alkali and ultrasound adversely affects the cleaning process.

4.4 The ultrasound frequency of 33 kHz does not damage surface scales of wool fibre. Use of ultrasound for scouring does not significantly change/damage mechanical, physical and chemical properties of wool.

4.5 The study has direct implication to wool industry. Application of ultrasound energy to the rinsing baths only instead of all five baths is practically feasible and cost effective. Such strategic use of ultrasound energy for wool scouring could result in clean and white fibre with better removal of grease and lower damage to the wool surface.

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