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### Ultrasonics - Sonochemistry



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## Effect of sonochemical scouring on the surface morphologies, mechanical properties, and dyeing abilities of wool fibres



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#### ABSTRACT

Ultrasonics has the potential to reduce the cost and environmental impact of textile processing. This work investigates the effects of ultrasonic irradiation during wool scouring on fibre surface morphologies, fibre mechanical properties, and fibre dyeing abilities. A range of ultrasonic frequencies were used in the scouring bath to examine the forms of fibre cuticle damage. It is observed that wool fibres underwent ultrasonic irradiation at a low frequency have severe modifications of the fibre surface structure. Despite some visible disruptions to the fibre scale structure however, ultrasonic irradiation has shown a negligible impact on the fibre mechanical properties, especially bending abrasion resistance which depends largely on the fibre surface conditions, and is responsible for the handle and pilling propensity of the resultant fabrics. Dyeing abilities were investigated on wool samples using commercially available acid dye and reactive dye. It is found that ultrasonically scoured wool has a quicker dye uptake in the early stage of low temperature dyeing for both acid dye and reactive dye, than the conventionally scoured wool.

#### 1. Introduction

Developments in ultrasonic cleaning technology have enabled new and environmentally friendly uses of ultrasonics. As a water and energy intensive process, textile industry has never been as desperate as it is now in searching for new technologies to reduce the cost and environmental impact of textile processing. Studies have been widely conducted in the use of ultrasonics to provide mass transfer [\[1\]](#page--1-0) within the processing liquor or provide the energy to enhance chemical interactions (sonochemistry) [\[2,3\].](#page--1-1)

Scouring is the first stage of wool processing, which is conducted to remove contaminants from the surface of the wool that may impede its manufacture into a textile or affect the aesthetics of the finished textile. Raw wool contaminants include wool grease, suint (secreted potassium salts) and dirt [\[4\]](#page--1-2). Wool scouring is normally conducted by mechanically agitating the fibre in water under warm neutral to alkaline conditions (pH 7–9 and temperatures of 50–65 °C) in the presence of a nonionic detergent [\[5\].](#page--1-3) The agitation in warm water can cause significant fibre entanglement.

The adoption of ultrasonic irradiation on wool scouring process has received various attention in the past decade. Studies on both Merino wool and alpaca have shown that the use of ultrasonic technology in the scouring process has the potential to provide significant improvement in the removal of wool contaminants and the reduction of fibre entanglement [6–[9\].](#page--1-4) Laboratory studies have shown that ultrasonic irradiation can provide up to 20–25% reduction of water, detergent and energy use in conventional non-ionic detergent wool scouring in achieving similar scouring efficiency [10–[13\].](#page--1-5) The study is not restrained in laboratory. Recently an industrial trail has been successfully conducted and it is shown that the adaption of ultrasonics can be a viable industrial application for wool scouring [\[14\].](#page--1-6) These changes brought about by the development of ultrasonic technology are rather revolutionary in the wool industry.

Ultrasound is effective in dirt removal because the sudden drop in acoustic pressure causes the fluid to fracture, generating vapour filled cavitation bubbles of about 10–100 μm in diameter [\[15\]](#page--1-7). The lowered acoustic pressure of the sound wave leads to bubble growth and the raised acoustic pressure causes the bubbles to rapidly collapse. The bubble undergoes a violent implosion, generating fluid jets, localized heating and shock waves [\[2,16\].](#page--1-1) The micro-agitation occurring in the vicinity of the cavitation bubble effectively wets out the surface and helps to displace particulate contaminants and grease.

Results from previous studies on ultrasonic induced fibre surface disruptions have not been consistent. In as early as 1963, Bradbury

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et al. reported a release of cuticle materials under ultrasonic irradiation [\[17\]](#page--1-8). Recently, Kadam et al. [\[12\]](#page--1-9) has concluded that there is no cuticle damage after ultrasound exposure on wool fibres during scouring, while studies done by Li and Hurren et al. [\[7,11\]](#page--1-10) found that fibre scale structure is subjected to some minor changes, in the form of micro cracks and scale peeling, under ultrasonic irradiation. These work however, were conducted under different working conditions and only ultrasound with a fixed frequency of 35–45 kHz was used. Therefore in this work, it is decided to conduct a comprehensive investigations on forms of fibre surface damage which were resulted from ultrasonic irradiation at a variety of frequencies.

For the successful adoption of ultrasonics in aqueous wool scouring, it is important that the process has little or no adverse impact on fibre properties. Although previous studies have showed that a prolonged ultrasonic treatment can provide wool with slightly better mechanical and thermal properties [\[18,19\],](#page--1-11) given the time required for wool scouring, ultrasonics was found to have no impact on fibre properties [\[12\]](#page--1-9). These studies were either conducted on wool in the fabric form or only tensile strength was investigated. During scouring process however, wool fibres are freely placed in the scouring bath with no restrictions, hence the energy received from the ultrasonics is expected to be different to that of fabric form.

Wool is a keratin fibre with a complex cellular morphology. The fibres have closely packed cortical cells surrounded by single or multiple layers of cuticle cells [\[20,21\].](#page--1-12) Inter-cellular adhesion is provided by the cell membrane complex [\[20\].](#page--1-12) Each of the morphological components contains various structural elements which affect its tensile, torsional, bending and shear properties. The most common method for evaluating fibre properties is single fibre tensile strength and elongation, which can provide effective way of evaluating any damage to the internal structural elements of the fibre including the cortical cells and inter-cellular adhesion.

While much of the work has been focused on tensile strength and elongation of ultrasonically scoured wool, mechanical properties such as bending and abrasion of the wool fibre seem to have been ignored. The scale structure of wool plays a large part in the interaction of a fibre when it is rubbed against another fibre or hard surface. Friction caused by movement of fibre on fibre or fibre on other solid items can lead to surface damage, fibre deformation and even fibre breakage. The extent of damage depends in part on the type of frictional interaction which can include ploughing, rolling, abrasion etc [\[22\]](#page--1-13). Fibre interaction during wear can result in abrasion fatigue damage and fuzz and pill formation [\[23](#page--1-14)–25]. Measurement of single fibre bending abrasion failure is an effective method for quantifying differences in fibre properties and has been used to search for any effects of the observed fibre surface modification after treatment [\[26\].](#page--1-15) Fibre bending abrasion measurement is therefore considered in this study.

Applications of ultrasonic technology in wool dyeing has been studied widely in the past decade. Much of the work has achieved in the area of low temperature dyeing with acid dyes [\[27](#page--1-16)–32] or at low-liquor ratio [\[33\],](#page--1-17) to reduce the energy and chemical presented in the dyeing process when comparing with the traditional dyeing method. While it is expected that wool cuticle damage resulted from high intensity ultrasonic fields can provide more pathways for dyes to diffuse into the fibres, hence lead to a quick and efficient fibre dye uptake rate, few literature has reported on how wool dyeing ability can be affected by ultrasonic scouring. It is therefore decided in this work to examine the fibre dyeing ability of the ultrasonically scoured wool.

This study extends previous work by using a range of ultrasonic frequencies (28 kHz, 45 kHz, and 80 kHz) at a laboratory scale to investigate the effects of cavitation intensity on wool surface morphologies and the subsequent changes in fibre mechanical properties and dyeing performance. A flexural fatigue combined with surface abrasion test was used to examine if exposure to ultrasonic irradiation during scouring affects abrasion resistance of wool fibres. Commercially available acid dye and reactive dye were used in this study to measure any changes to the dyeing ability of ultrasonically scoured wool.

#### 2. Experimental

#### 2.1. Materials and sample preparation

Australian merino wool was used in this work. The wool fibre specifications after blending were 19.5 µm in average diameter, 0.3% vegetable matter content, and a scouring yield of 64.5%. The fleece was opened before scouring by a single pass through a 500 mm wide single drum Fearnaught opener (Houget Duesberg Bosson, France).

#### 2.2. Scouring equipment

An ECOSCT 10 litre, 28 kHz ultrasonic bath (Ultrasonics Eco Cleaning Solutions, Australia) and a KQ-300VDE 10 litre, 45/80 kHz frequency sweeping digital control ultrasonic cleaner (Kunshan Ultrasonic Instrument Co., Ltd, China) were used for ultrasonic scouring. Both cleaners have a maximum power of 300 W and can be adjusted from 40% to 100% power. Both have adjustable temperature control between 20–80 °C and are capable of frequency sweeping around the working frequency. Frequency sweeping was turned off for all experiments.

A mechanical agitation was utilized based on an industry rake scour motion of 23 rev/min, and was used as a control.

#### 2.3. Scouring bath parameters

A scouring bath liquor ratio of 1:100 was used for each of the different irradiation methods. Wool samples were conditioned and weighed before addition to the first scouring bath. Scouring was undertaken using a simulated conventional 5-bath method. Hydrpol TN450 (Nonyl Phenol Ethoxylate detergent, Huntsman Chemicals) was used as the detergent, and soda ash ( $Na<sub>2</sub>CO<sub>3</sub>$ ) as the builder [\[10,34\]](#page--1-5). Details for each bath of the process are given in [Table 1](#page-1-0).

Fibres were squeezed with a Rapid PA0 pad mangle (Rapid Labortex Co. Ltd., Taiwan) after each bath to remove excess liquor. After padding the water retained in the fibre was around 43% w/w. The samples were then dried in a Binder FED (Binder, Germany) fan forced oven at 105 °C for one hour before conditioning, bagging and labelling.

#### 2.4. Fibre surface analysis

Fibre surface morphology was examined using a Zeiss Supra 55 VP field emission gun scanning electron microscope (SEM). Measurements were conducted with an Extra High Tension (EHT) of 10 kV, working distance (WD) of  $3.8$  mm and aperture size of  $30 \mu m$ .

#### 2.5. Determination of fibre diameter and tensile strength

Single wool fibres randomly selected from each of the washed samples were prepared to assess fibre diameter profile and fibre tensile properties. The fibres were prepared as per the above method and

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